Practical course M

Experiment M2.4
Magnetization of a superconductor

Room 126

Universität zu Köln
II. Physikalisches Institut

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1 Preparation

This experiment should acquaint you with magnetic properties of a superconductor, in particular, with the Meissner-Ochsenfeld effect. Superconductivity occurs in many elemental metals, alloys, and intermetallic compounds. When cooling a sample below a temperature T_c specific for a particular material, its electrical resistivity disappears and any magnetic field is expelled from the material (Meissner-Ochsenfeld effect). Both these effects identify the superconducting transition.

Below T_c, superconductivity is suppressed by a sufficiently large magnetic field ($H \geq H_c$). The critical field H_c depends on the temperature as well as on the material. The function $H_\mathrm{c}(T)$ defines the phase boundary between the superconducting state and the normal state. In this experiment you should observe the Meissner-Ochsenfeld effect for a type-I superconductor at different external magnetic fields via heating and cooling the sample across this phase boundary. In addition, you should evaluate the magnetization of the sample as a function of the applied magnetic field.

For that, you should learn about the meaning of magnetic parameters H, B, M, μ, and χ. Be familiar with the magnetization curves of type-I and type-II superconductors. You need to understand the thermodynamics of the superconducting phase transition in order to interpret your results. There are following questions to answer:

(1) What is the difference between a superconductor and an ideal conductor (shielding *vs.* Meissner-Ochsenfeld effect)?

(2) What happens when a ring-shaped superconductor is cooled below T_c in a magnetic field?

(3) What happens in the same process as in 2. with an inhomogeneous sample? Assume that the inner part of the sample has a lower critical temperature than the outer part.

(4) How does the sample geometry (demagnetization factor) influence the magnetization curve?

(5) What are the London penetration depth and the coherence length of a superconductor?

(6) How does H_c depend on temperature?

(7) Why is the charge transport by Cooper pairs not hindered by scattering?

(8) Which relation exists between the area below the magnetization curve and the free enthalpy difference between the normal and the superconducting phases?

(9) How does the experimental setup work?

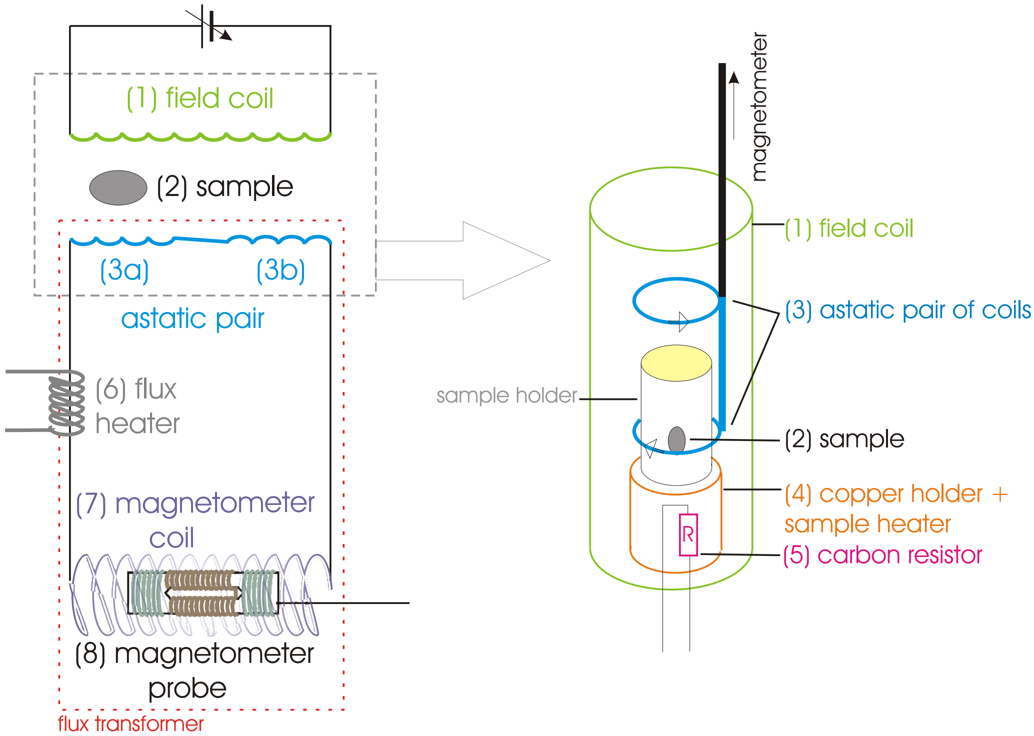

Figure 1: The experimental assembly and the coil system at the sample position.

2 Experimental Setup

The core part of the experimental setup is the system of coils for producing and measuring magnetic fields. The whole setup and the coil system are depicted in Figure 1. The sample (2) and an astatic pair of coils (3), which measures the magnetic moment of the sample, are situated inside the primary coil (1) which produces the external field. For measuring the magnetization of the sample, a superconducting flux transformer, which transfers the magnetic flux to another place, and a flux-gate magnetometer are used (see Fig. 2).

Two coils (3a) and (3b) form a so-called astatic pair. The coil (3a) measures both the primary field and the induction due to the magnetized sample, while the identical coil (3b) measures only the primary field which has the same value as at the sample position. The windings of the two coils (3a) and (3b) are of the opposite sense are connected in a series in order to compensate the signal produced by the primary field. Because in the actual setup the compensation is not exact, some signal proportional to the primary field still survives which will result in a linear background in your field dependent measurements. The flux heater (6) is thermally contacted to a part of the flux transformer circuit and can be used to heat a part of the wire to its normal state thus removing any super current flowing in the circuit. The probe of the magnetometer (8) is situated in the magnetometer coil which is part of the flux transformer.

The operation of a flux transformer is based on the fact that a magnetic flux through an area surrounded by a superconductor remains constant. A change $\Delta\Phi$ in a part of the area generates

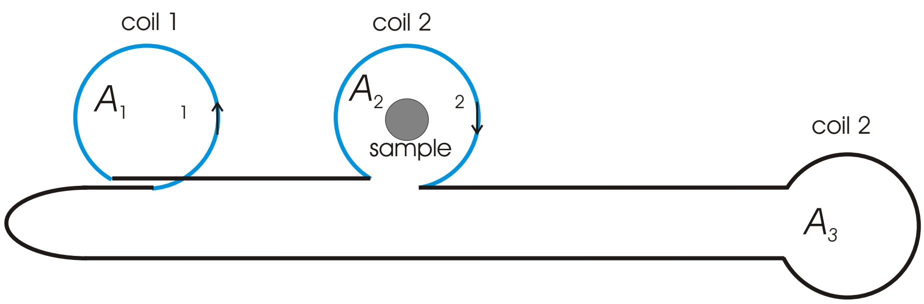

Figure 2: The principle of the flux transformer of the experimental setup. Coils 1 and 2 represent the astatic pair and coil 3 is the magnetometer coil. The entire flux given by $\phi_{ges} = A_1 \cdot \phi_1 + A_2 \cdot \phi_2 + A_3 \cdot \phi_3$ is constant, with $\phi_1 = \mu_0 H$, $\phi_2 = \mu_0(H + M)$, $A_3 = \phi_{mess}$ and $A_1 = A_2$.

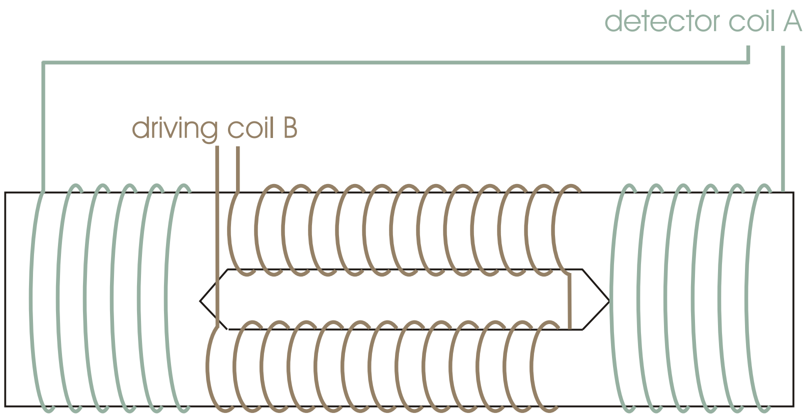

Figure 3: The probe of the flux gate magnetometer.

a super current which, in turn, produces a flux change $-\Delta\Phi$ in the total area. The flux through the whole area remains constant. Considering the flux through two areas A_1 and A_2, it follows that

$$0 = \Delta\Phi_1 + \Delta\Phi_2 = A_1 N_1 \Delta B_1 + A_2 N_2 \Delta B_2, \tag{1}$$

therefore

$$\Delta B_2 = -\frac{A_1 N_1}{A_2 N_2} \Delta B_1, \tag{2}$$

where A is the area of the superconducting circuit and N is the number of windings of a coil.

During the measurements the entire coil system is placed in a bath of liquid ^{4}He (T = 4.2 K). The sample is in good thermal contact with a copper holder, on which the sample heater (4) is wound. Using a current source, the sample can be heated by a few Kelvin. The temperature measurements are done with the help of a carbon resistor (5). The temperature can be determined from the measured resistance using the calibration curve (see Fig. 6).

Fluxgate magnetometers are a very sensitive technique for measuring small changes in magnetic fields and were invented in the 1930s by Victor Vacquier at Gulf Research Laboratories.

The principle of operation of the flux gate magnetometer can be described as follows. The magnetometer consists of a ferrite core wrapped by two coils, the driving coil and the detector coil, see Figure 3. Since the permeability μ of a ferromagnet is field-dependent, an alternating electrical current passed through one coil drives the core through the hysteresis curve $M(H)$. This constantly changing field induces an electrical current in the second coil, and this output current is measured by a detector. In a magnetically neutral background, the input and output currents will match. Exposing the system to an external constant field H_{G1}, results in a shift of the hysteresis curve, *i.e.* the core will be more easily saturated in alignment with that field and less easily saturated opposite to it. The alternating magnetic field and the induced output current at the detector coil will be out of phase with the input current. Besides the strength of the background field there are additional factors that affect the size of the resultant signal (number of windings, sense of windings, permeability of the core, geometry...).

In our setup, the driving coil does not contribute to the signal, since it is built in such a way that its entire magnetic flux stays within the closed circle of the ferrite core. The detector coil measures only the flux $\Phi = \mu(H(t))\mu_0 H_{G1} A$ produced by the constant field H_{G1}. The induced currents in the output coil are, thus, proportional to the constant field. The currents are integrated, yielding an output analog voltage, proportional to the magnetic field. Using a phase synchronous detection *i.e.* a lock-in amplifier, converts these harmonic signals to a DC voltage proportional to the external magnetic field.

The magnetometer control device is depicted in Figure 5a). (The standard adjustments of the magnetometer are: range= 1A, dc offset=off, function=dc.) The output is analog and is transferred to a *Keithley 2000* to read out the signal digitally, 5b).
The temperature of the sample *i.e.* the resistance of the carbon resistor is obtained in a two-point measurement and is read out by another *Keithley 2000* (use the 10 kΩ range), see Fig.5c). The device *HP E3631A*, Fig.5 D, is a three-terminal current source that is able to set and read out up to 3 different currents simultaneously. This device controls the current supply for the primary coil, the flux heater and the temperature of the sample.
The wires within the sample environment (Fig.5 E) are connected via plug and wire to the distribution box (Fig.5f)) from where the explicit connections are made to the corresponding control and measurement devices.

The devices are controlled via the *LabView* program *XY-M24.vi* which is shown in Figure 4).

A The program will plot the measured voltage signal of the magnetometer against the resistance of the carbon resistor (temperature) or against the current through the primary coil (external magnetic field).

B The program needs to know the devices that have to be addressed and the corresponding GPIB.

C The current through the primary coil can be set to a certain value by pushing the upper slide control to a certain value. The maximum value is restricted to 1 A to prevent damage of the coil. The field can also be sweeped with a certain sweep rate which can be chosen via the lower slide control. Pushing the buttons UP (DOWN) will set the

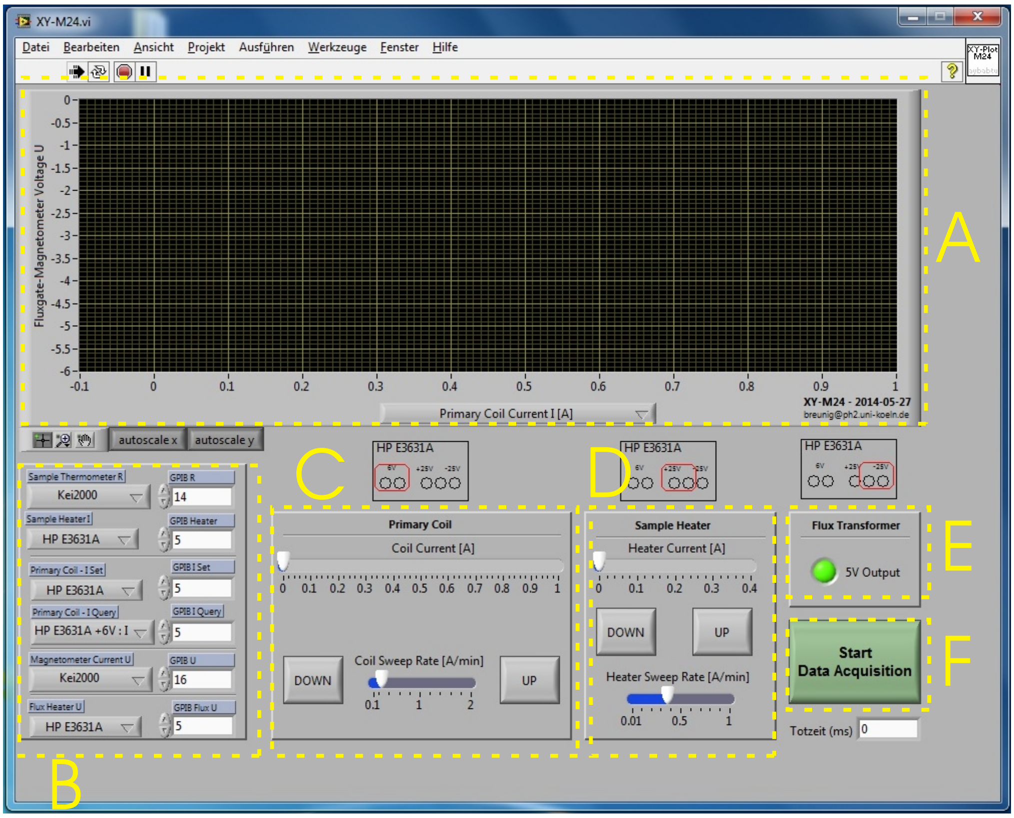

Figure 4: XY-M24.vi

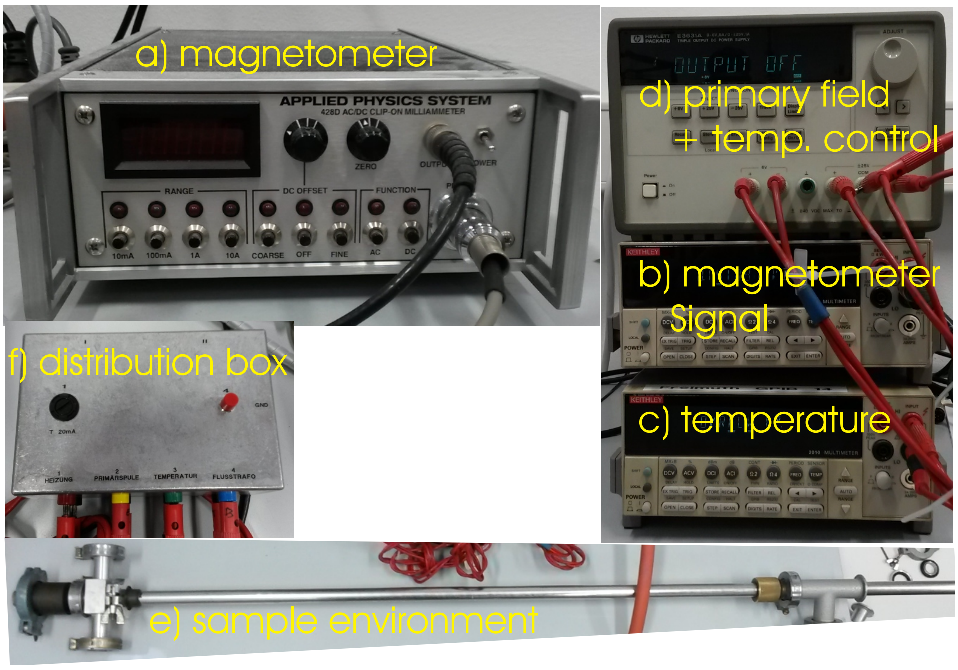

Figure 5: Pictures of the assembly: control devices, magnetometer, sample environment.

current to the minimum (maximum) value and sweep the field with the chosen rate to the maximum (minimum) value.

D The temperature control works the same way like the field control. Note, that the maximum value of 0.4 A is not the current that is running through the heater because the heater is in a parallel circuit with a resistor of about 10 Ω.

E The heater of the flux transformer can be activated here, but has also to be switched on by pushing the red key button at the distribution device which closes the circuit and heats up a small part of the flux transformer.

F Pushing this button to start a measurement results in opening a window which asks you to save the measurement in a file. After naming the data file the measurements will start. Pushing ENTER or ESC will start the measurement without saving the data. Note, that in this case you cannot save this data set anymore.

3 Experiment procedure

At the beginning of the experiment, make yourselves familiar with the equipment by "playing" with the different devices, which are shown in Fig. 5. Make yourself familiar with the measurement environment. Do not mess around with the helium dewar!

Tasks

(1) Record the magnetization as a function of field $M(H)$ at the lowest temperature, in order to specify the field value H_{max}, up to which the Meissner-Ochsenfeld effect will be measured.

(2) Magnetization curves
Record magnetization curves for 10 temperatures above 4.2 K (including one curve for $T > T_{\mathrm{c}}$). Heat up the sample and the flux transformer (field off!) before each measurement. After the temperature is stabilized, wait for about 2-3 minutes, as the readings of the ohmmeter stabilize to make sure that the sample actually reaches thermal equilibrium.

(3) Meissner-Ochsenfeld effect
In order to perform zero-field cooled measurements, before each measurement, the sample and the flux transformer (field off!) should be heated up for removing remaining magnetic flux and then cooled down without field. Set a constant primary field below H_{max} at the lowest temperature. Record the magnetization as a function of temperature, first increasing the current in the sample heater until the sample temperature is above T_c and then reducing the heater current to 0. Try out several sweep rates and choose one for all measurements. Why are the curves for increasing and decreasing temperature different? Repeat the measurement for 10 field strength in the interval between $H = 0$ and H_{max}.

Discuss the experimental errors.

The relation between the primary coil current I and the field H at the sample position is

$$H = kI, \text{where } k = 739 \text{ G/A}.$$

The relation between the resistance R of the thermometer (in Ohms) and the temperature T (in K) in the temperature region between 4.2 and 7.5 K is

$$T = 1.017 + 6.07 * \exp(-R/1700) + 40.6 * \exp(-R/170).$$

4 Analysis

(1) Plot the flux change at the phase transition as a function of the corresponding field strength (warming up and cooling down $M(T)$ curves). Describe your observations and determine how large the Meissner-Ochsenfeld effect in % (fraction of $M_{max,up}/M_{max,dw}$) is. Is the size of the Meissner-Ochsenfeld effect field-dependent?

(2) Subtract the linear background signal (fitted in the high field region) from the original curves. Where does this signal come from? Plot the H_c values, obtained from the magnetization curves $M(H)$, as a function of the temperature $[H_c \simeq H_{c0}(1 - (T/T_c)^2)]$ and make a linear fit by plotting M *vs.* T_c^2. Determine H_{c0} and T_c and compare them with the literature data. Determine the demagnetization factor D using $H_i = H_a - DM$ and the fact that for a superconductor $M = -H$ and relate the resulting equation for D to the field values H_{max}, at which the magnetization is maximal, and H_c, where M becomes zero . As a purely geometrical factor, D should be constant. Explain its possible temperature dependence.

(3) Calculate the integral $A = \int_0^{H_c} MdH \sim H_c^2$ for each of the measured magnetization curves. Which physical quantity does the integral correspond to? Plot the resulting A values as a function of H_c on a double-logarithmic scale and determine the slope. What is the theoretical expectation for its value?

5 Literature

- C. Kittel, *Einführung in die Festkörperphysik*
- W. Buckel, *Supraleitung* (FK-BUC)
- E. Lynton, *Supraleitung* (FK-LYN)
- E. Kneller, *Ferromagnetismus* (FK-KNE)
- M. Tinkham, *Introduction to superconductivity*
- Textbooks on experimental physics
- Open University Course: Superconductivity
  http://www.open.edu/openlearn/science-maths-technology/engineering-and-technology/engineering/superconductivity/content-section-0

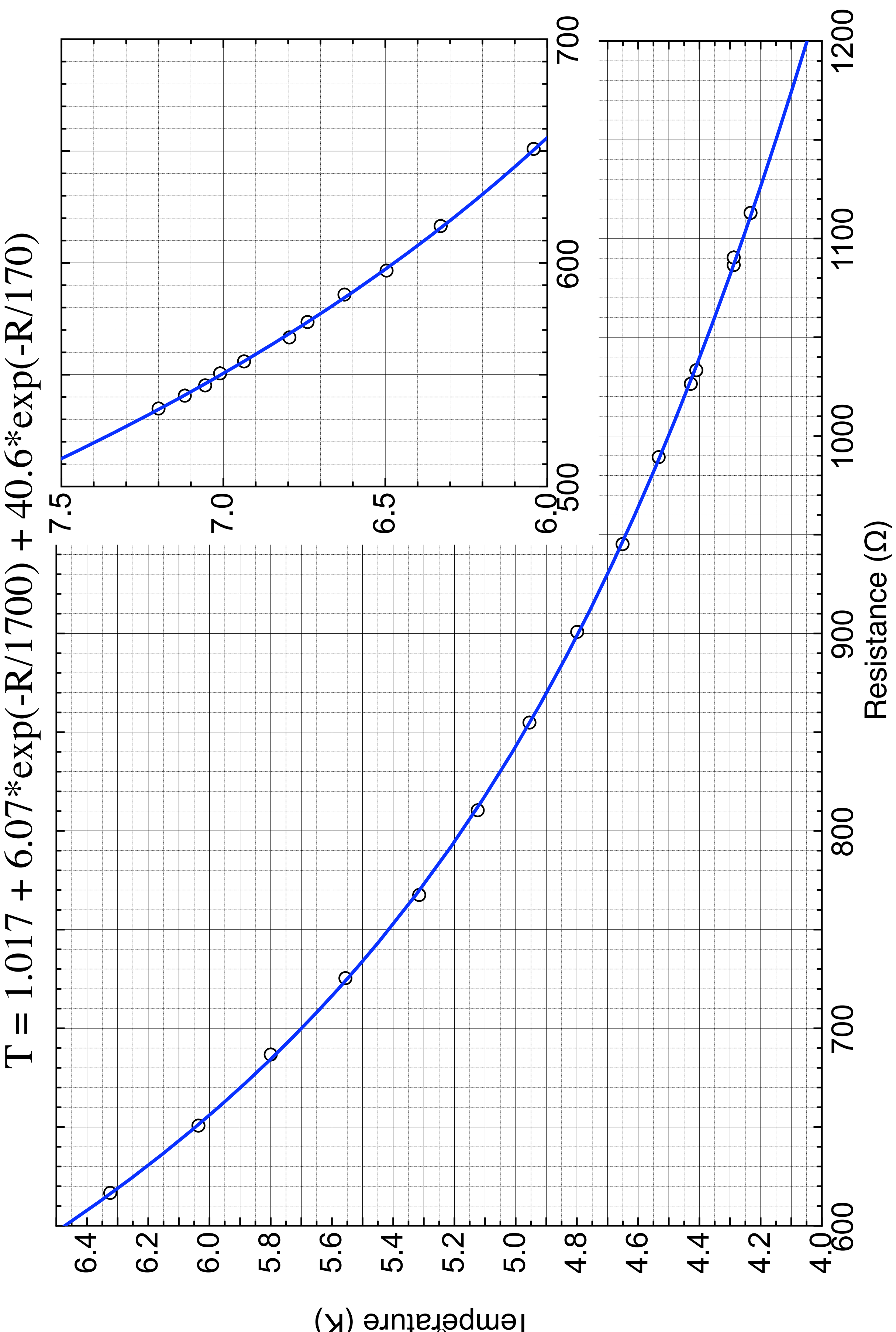

Figure 6: The calibration of the carbon thermometer.

Questionnaire for the M-practical course

Magnetization of a Superconductor

Please fill in the form after you finished the experiment and discuss the criticism with your tutor wherever applicable.

The valuation is based on the german school mark system (1:best – 6:worst)

Semester: Winter 20___/___ Summer 20___

		1	2	3	4	5	6	Abstention
Query 1: How much did you learn from the experiment in terms of concepts in physics as well as technical skills?	**in physics**	□	□	□	□	□	□	□
	tech. skill	□	□	□	□	□	□	□
Query 2: How do you rate the quality of the experimental equipment?		□	□	□	□	□	□	□
Query 3: Is the experiment up-to-date with recent topics in physics?		□	□	□	□	□	□	□
Query 4: What is the advance in your experimental skills?		□	□	□	□	□	□	□
Query 5: Did you have fun carrying out the experiment?		□	□	□	□	□	□	□
Query 6: Is the tutorial of the experiment explicit?		□	□	□	□	□	□	□
Query 7: How much time did you invest in the preparation?		h						
Query 8: How do you assess the experiment in total?		□	□	□	□	□	□	□

study course: **Master** □ **Teacher** □

Comments and suggestions

Overall mark for your tutor (1: excellent, 6: not that good)
Name of your tutor: ___________________________________

1	2	3	4	5	6	Abstention
□	□	□	□	□	□	□

Workplace:	**Operating instruction according § 20 GEFSTOFFV**	As at 5/2011 *Date:* 31.05.2011

Helium, *liquid, cryogenic*

Kp.: -269°C Fp.: -272°C Solubility (H_2O/20°C): 0.86 ml/100 ml rel. Vapour density$_{(Air)}$ = 1 : 0.14

Physical danger:
Cracks/scratches in the Dewar vessel may lead to spontaneous implosion!
If the lid of a Helium tank remains open, air will sublimate into the tank and the overpressure relief valve. Block of pressure release due to this, can cause a life-threatening pressure increase within the tank.

Dangers to health:
In high concentration, Helium leads to suffocation without warning!
H 281 Contains cryogenic gas; may cause cryogenic burns or injury.
Depending on the duration of the contact, deep tissue destruction, frostbite, and severe eye damage may occur.

Attention

Safety instructions Prevention:
P282 wear protective gloves / face protection shield / eye protection with cold insulation.

Technical measures:
P403 Store the Helium tank in a well-ventilated place.
- Conect the Helium tank to the recovery.

Filling of Helium only by instructed persons in a well-ventilated place.
- Liquid Helium should only be filled in dry and well-isolated Dewar vessel.
- Dewar vessel and recovery lines must be labeled unambiguously.
- Shut-off valves should not be apruptly opened or closed.
- Removal of ice on valves and vessels by use of warm air only.
- If a dangerously high pressure builds due to heat, suitable safety devices must be installed. In case of icing, ensure that the overpressure is released.

Organizational measures:
Transport only specially suited Helium tanks or Cryogenic containers in elevators ***without accompanying persons***.

Behaviour in case of danger: In case of massive gas release leave the room. Alert other people. Ventilate the room. Rescue injured persons in compliance with self-security.

First Aid

Skin or eye contact:
P 315 Seek immediate medical advice/attention.
P 336 Warm frozen parts with lukewarm water. Do not rub the concerned area. Cover and keep the burned area sterile. Consult a physician.
Inhalation: High concentration may cause suffocation. Symptoms are loss of mobility and consciousness. Expose the victim to fresh air in compliance with self-security. Keep warm and calm. Consult a physician. If breathing stopped, begin artificial respiration.

Emergency number:

Emergency medical services: 01-112

Nearest hospital: **01-4792213** Evangelisches Krankenhaus, Weyertal 76, 50931 Köln

Maintainance main building: 2200

*If necessary: * Specify location*	*(Signiture)*