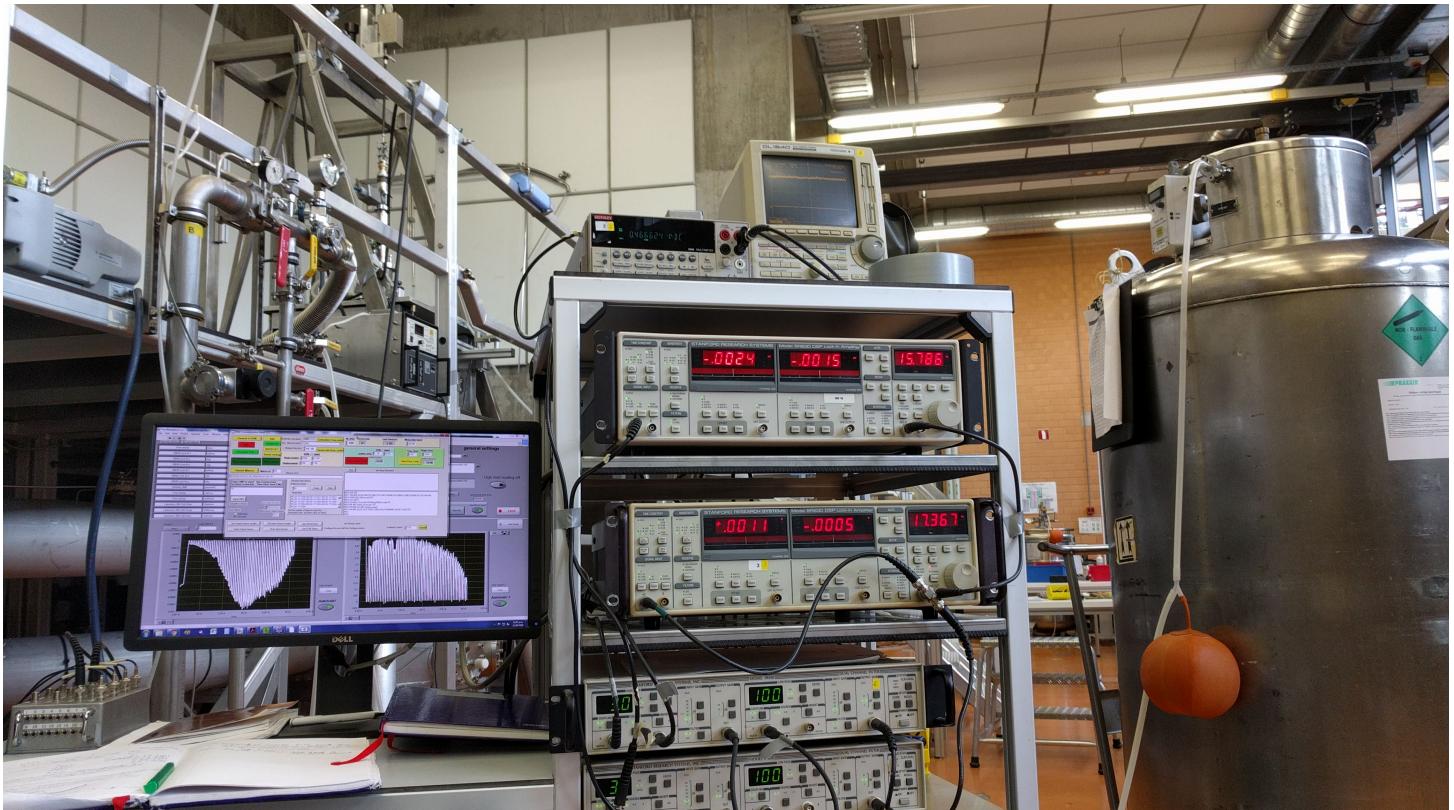


Vibrating Sample Magnetometry

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Abstract

Vibrating sample magnetometry is a technique used to measure the magnetization of a sample. It utilizes the principle of electromagnetic induction; a magnetized sample will induce a voltage across a set of 4 pickup coils. This report contains both a in-depth analysis of the inner workings of the vibrating sample magnetometer (VSM) at HFML, and doubles as a manual for this VSM. Detailed schematics and illustrations with dimensional measurements are therefore included in the appendices. The VSM was used to measure the saturation magnetization of copper sulfate pentahydrate. Furthermore the phase transition of $\text{KEr}(\text{MoO}_4)_2$ was analyzed. Finally, in collaboration with dr. Patricia Lázpita from the University of Basque Country and Anabel Pérez Checa from BCMaterials the temperature and field dependence of the phase transition of $\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$ and $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$ was measured.

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1 Introduction

When analyzing phase transitions or when determining the saturation magnetization of some material, it is useful to measure magnetization as a function of temperature and/or external magnetic field. This can be done using a vibrating sample magnetometer (VSM). The advantage of VSM over similar techniques such as torque magnetometry is that a VSM is also able to measure samples in powder form. It also has a higher sampling rate compared to extraction magnetometry. A disadvantage of the VSM compared to a torque magnetometer is that the VSM is only capable of measuring magnetization (anti-)parallel to the external magnetic field.

This report explains the workings of the VSM and shows how one can do measurements with it. However, this report is more than just a manual, because the latter sections will describe actual measurements preformed with the VSM. Measurements have been done with $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, $\text{K}\text{Er}(\text{MoO}_4)_2$, $\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$ and $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$. Furthermore, the appendices contain a short checklist that might be useful when doing a actual measurement. Also included in the appendices are a manual for using the motor controller software and several detailed schematics and figures.

Throughout the duration of this project improvements have been made to the HFML VSM, in the form of replacing a Keithley 236 Source-Measurement Unit with a dedicated voltage supply, namely a box consisting of among other things 2 AA batteries.

2 Theoretical principle of a VSM

Materials in a magnetic field will react to this field, how they react exactly is an interesting field of study because not all materials will react in a trivial way. Some materials will exhibit phase transitions at certain magnetic field strengths and/or temperatures. By analyzing the magnetization of samples as a function of temperature and/or magnetic field strength, we can not only uncover interesting phenomena but also learn more about the sample material.

A vibrating sample magnetometer is capable of doing these magnetization measurements, using a set of four pickup coils. Two coils are positioned above the sample and two below. The sample is placed in a sample holder attached to a long stick. This sample stick is then screwed onto a connector piece, which in turn screws onto the top of the VSM. The length of the sample stick is adjusted such that the sample is positioned exactly between the pickup coils. The whole VSM is then inserted into a cryostat for temperature control, which in turn is inserted into a bitter magnet. This bitter magnet induces a magnetization on the sample. The connector piece on top of the VSM is attached to a motor, which sends a vibrating motion through the connector piece and causes the sample to vibrate in between the coils. The following sections explain the physical effect of the vibrating magnetized sample on a pickup coil, why 4 pickup coils are required, and why the magnetization can thus be measured accurately. Schematics and pictures of the VSM can be found in Appendix C.

2.1 Magnetization and Faraday's law of induction

This section introduces the principle of induction. This principle is essential to measuring magnetization using a VSM. Faraday's law describes the voltage that is induced in a closed loop due to a change in the magnetic field through this loop. It is given by:

$$V = -\frac{d\phi_B}{dt} \quad (1)$$

Where V is the induced voltage¹, and ϕ_B is the magnetic flux through the loop which is given by:

$$\phi_B = \iint_S \vec{B}(\vec{r}, t) \cdot d\vec{A} \quad (2)$$

Where S represents the surface enclosed by the loop and B the magnetic field through this loop. By taking the magnetic field to be perpendicular to the surface enclosed by the closed loop, and homogeneous throughout the loop ($\vec{B}(\vec{r}, t) = B_{\perp}(t)$), Equation 2 simplifies to $\phi_B = BS$.

Electrons in any material will react to an external magnetic field applied to this material, it will do this paramagnetically and/or diamagnetically.

Paramagnetic contributions are caused by the **alignment of magnetic moments** in the material. This causes an internal magnetic field in the material which is parallel to the external field (positive magnetization).

Diamagnetism on the other hand is caused by the **Lorentz force on the electrons** causing them to circulate creating an internal magnetic field antiparallel to the external field (negative magnetization). All materials are diamagnetic to some extent, however because the electron orbitals are constrained by the Pauli principle these contributions are generally much smaller than paramagnetic contributions [6].

Once a material is magnetized it will have its own magnetic field, and will thus cause a magnetic flux through a closed loop. When the material moves with respect to the closed loop the flux will change, inducing a voltage in the closed loop. Because the induced voltage is directly related to the magnetization, it is possible to determine a sample's magnetization by measuring the induced voltage.

2.2 The response of a pickup coil to magnetized sample

Because the inductive effect on just one closed loop is small, and because the alignment of one closed loop with respect to the magnetic field is prone to errors, a coil is used instead. Since a coil is basically just a series of closed loops, we can multiply Equation 1 with N , the number of windings:

$$V(t) = -N \frac{d\phi_B}{dt} \quad (3)$$

¹also known as the 'electromotive force'

This can be written as:

$$V(z, t) = -N \frac{d(BS)}{dz} \frac{dz}{dt} \quad (4)$$

Where S represents the coils surface area, and the z direction is parallel to the vibrating motion [4].

In an ideal situation the motion of the sample is perfectly sinusoidal, i.e. $z(t) = C \cos(2\pi ft)$, and thus $dz/dt = -2\pi fC \sin(2\pi ft)$. If we further assume that there are no defects or other imperfections in the coil we can also use the following for its magnetic field:

$$B(z) = \frac{\mu_0 I}{2} (z - L) \left[\ln \left(r_2 + \sqrt{r_2^2 + (z - L)^2} \right) - \ln \left(r_1 + \sqrt{r_1^2 + (z - L)^2} \right) \right] + \frac{\mu_0 I}{2} z \left[\ln \left(r_1 + \sqrt{r_1^2 + z^2} \right) - \ln \left(r_2 + \sqrt{r_2^2 + z^2} \right) \right] \quad (5)$$

Where r_1 and r_2 are the inner and outer radius of the coil respectively, and L is the length of the coil [4].

Taking the derivative of Equation 5 and filling in dz/dt , we get:

$$V(z, t) = -NASI \mu_0 \pi f \sin(2\pi ft) \left[\ln \left(r_2 + \sqrt{r_2^2 + (z - L)^2} \right) - \ln \left(r_1 + \sqrt{r_1^2 + (z - L)^2} \right) + (z - L)^2 \left(\frac{1}{r_2 \sqrt{r_2^2 + (z - L)^2} + r_2^2 + (z - L)^2} - \frac{1}{r_1 \sqrt{r_1^2 + (z - L)^2} + r_1^2 + (z - L)^2} \right) + \ln \left(r_1 + \sqrt{r_1^2 + z^2} \right) - \ln \left(r_2 + \sqrt{r_2^2 + z^2} \right) + z^2 \left(\frac{1}{r_1 \sqrt{r_1^2 + z^2} + r_1^2 + z^2} - \frac{1}{r_2 \sqrt{r_2^2 + z^2} + r_2^2 + z^2} \right) \right] \quad (6)$$

From this it is clear that the voltage signal from the pickup coil is also sinusoidal (90° phase shifted). In a experimental setup we will always measure the root mean square (RMS) value of this AC signal, therefore we take the RMS value of Equation 6:

$$V(z) = -NASI \mu_0 \frac{f\pi}{\sqrt{2}} \left[\ln \left(r_2 + \sqrt{r_2^2 + (z - L)^2} \right) - \ln \left(r_1 + \sqrt{r_1^2 + (z - L)^2} \right) + (z - L)^2 \left(\frac{1}{r_2 \sqrt{r_2^2 + (z - L)^2} + r_2^2 + (z - L)^2} - \frac{1}{r_1 \sqrt{r_1^2 + (z - L)^2} + r_1^2 + (z - L)^2} \right) + \ln \left(r_1 + \sqrt{r_1^2 + z^2} \right) - \ln \left(r_2 + \sqrt{r_2^2 + z^2} \right) + z^2 \left(\frac{1}{r_1 \sqrt{r_1^2 + z^2} + r_1^2 + z^2} - \frac{1}{r_2 \sqrt{r_2^2 + z^2} + r_2^2 + z^2} \right) \right] \quad (7)$$

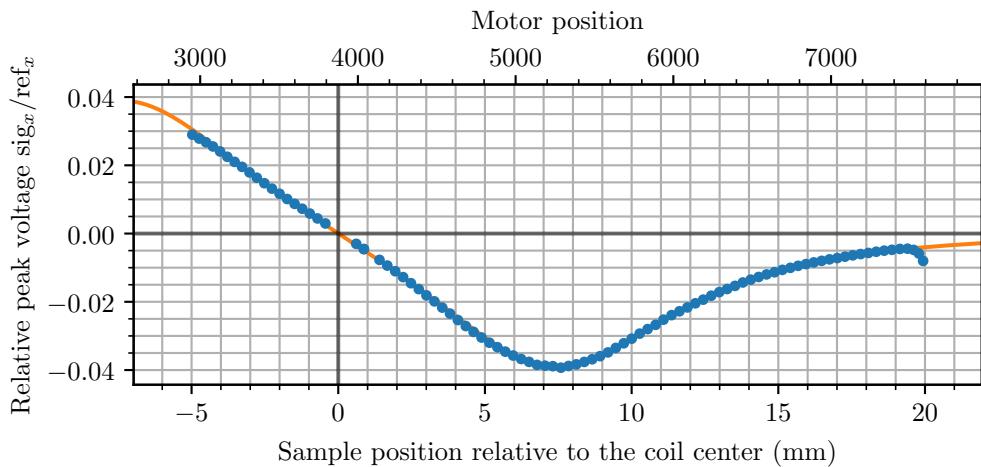


Figure 1: Response of upper large pickup coil, designated ‘1-2’ (18.1 Hz, 1.08 mm) to a permanent magnet (30.9 mg). Coordinates on the lower axis have been shifted such that 0 corresponds to the coil center. Equation 7 has been fitted through the data.

We can check if this equation holds by making a plot of the response of 1 pickup coil as a function of the position at which the sample is vibrating (z), and fitting Equation 7 through this data, as shown in Figure 1.

2.3 Flattening the curve

The attentive reader will notice 2 things in Figure 1. First, the quantity on the y-axis: This is actually not $V(z)$ as in Equation 7 but $V(z)$ divided by a reference voltage that is directly proportional to the vibrating motion of the VSM's motor. The reason for this will be discussed in more detail in Section 3.3. Second, the plot in Figure 1 is not flat anywhere, which is a problem because this means that the sample will induce a different response throughout its vibrating motion. Because of this the signal is not going to be a perfect sine, since the signal will be higher closer to the coil and lower further away. For an accurate measurement we require a (almost) perfect sine wave as VSM signal, thus we need a flat area in the plot that is at least as big as twice the amplitude of the sample's motion.

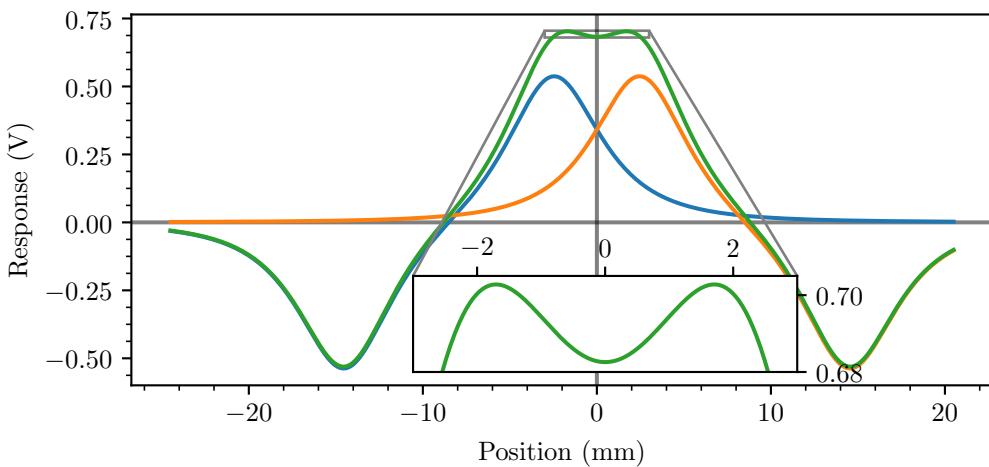


Figure 2: The superposition (green) of the response of 2 coils (blue and orange, 5 mm separation).

This can be accomplished by measuring the combined responses of several pickup coils instead of just one. When a second coil is added on the other side of the sample and connected such that it responds oppositely (its response curve (Figure 1) is mirrored in the z -axis, see Appendix C for a schematic) the peaks of both coil start to merge, as demonstrated in Figure 2. By carefully choosing the separation between these 2 coils, we get a nice and flat (0.1% deviation [4]) area, as shown in Figure 3. For demonstration purposes the separation in Figure 2 has been set to 5 mm, instead of the actual value of 2.5 mm.

2.4 Compensating for motion of the pickup coils

It is impossible to keep the VSM completely still inside the bitter magnet. There will always be unwanted vibrations caused by the various pumps, motors or water currents throughout the lab. These unwanted vibrations cause the pickup coils themselves to move inside the external magnetic field. While this field is quite homogeneous (1×10^{-3} cm DSV in cell 3 [2]) any such movement results in a small magnetic flux that will result in noise on the signal. A solution is to add an oppositely wound twin coil inside both pickup coils. This can compensate for any movements inside the external magnetic field, since these unwanted vibration will cause both this compensation coil and its parent coil to move in exactly the same way. For this compensation coil to cancel out these external vibrations the flux has to be exactly opposite to the flux that these vibrations cause in the parent coil. To accomplish this the compensating coil has to have a smaller radius, and thus needs to have a larger number of windings, such that the surface area of one winding multiplied by the number of windings is equal. When the flux through both coils cancel each other out completely, there will be no overall effect of external vibrations on the signal when measuring across the combination of all coils.

The reader might wonder why this does not cancel out the flux of the sample as well. This does not happen because the sample's magnetic field has a large curvature in the coils, and thus does not

go all the way through the coil. Because the field lines only go through some windings of the coil, the flux through parent and compensation coil is not completely opposite and thus the interference is not completely destructive. Whereas for the external magnetic field the field lines always go through all of the coils windings (even if it is vibrating), by design this causes exactly opposite flux and thus complete destructive interference.

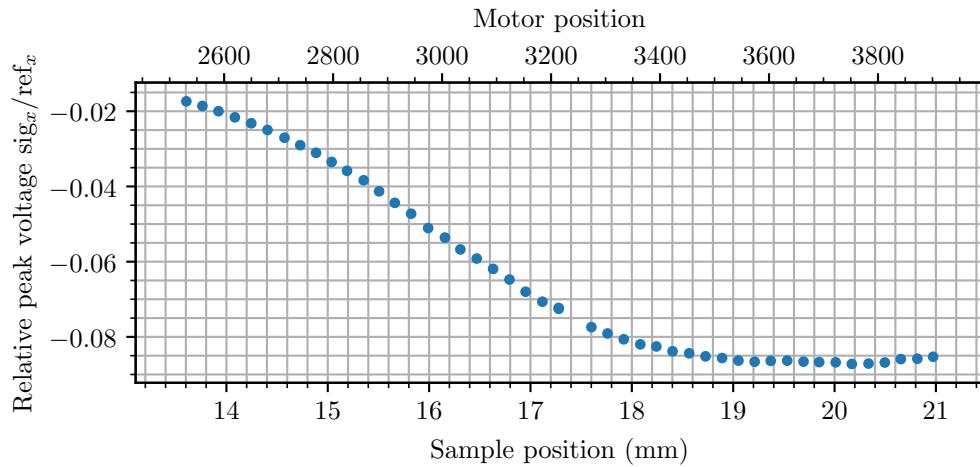


Figure 3: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 1.08 mm) to nickel (42.5 mg), at 2 T (cell 3).

Note that Figure 3 covers a shorter z-axis range than Figure 1, due to restrictions in the movement of the motor driving the VSM (more on this in the Section 3.1 and Section 3.3). If we were to continue the measurement beyond 21 mm, it will of course approximately follow the curve in Figure 2 and eventually go through zero.

3 Setup

The whole VSM setup consists of 3 major components: the motor, the pickup coils and a potentiometer. This section will describe each component, as well as any additional equipment required to correctly analyze and record data from each component. A detailed schematic of the VSM setup can be found in Appendix C on page 30

3.1 The drive motor

The sample's motion is driven by an electric motor, which is in turn controlled by a motor controller (LAC-25, Figure 22). This motor controller can receive commands from a computer using a VSM control program written by Hung van Luong. In this control program the user sets the desired amplitude and starting position, after which the computer will send the necessary commands to the motor. More detailed instructions on how to use this program can be found in Appendix A.

However if the motor were to be connected directly to the sample stick, the pumping up/down motion of the stick would cause undesired pressure changes inside the VSM. Therefore, the motor does not drive the sample's motion directly, instead it drives a connector piece on top of the VSM (Figure 21a). This connector piece seals the inside of the VSM and therefore prevents any pressure changes. The connector piece then passes on the vibrating motion of the motor to the sample stick (Figure 21b).

3.2 The pickup coils

When a sample is set to vibrate such that the sample is within the flat area (in Figure 3), we get a sinusoidal VSM signal from the combination of all 4 pickup coils, the frequency of which should be the same as the frequency of the sample's vibration. Previous research [3] has determined that a frequency of 18.1 Hz causes minimal noise in the VSM's signal, so this will be the frequency we will send to the motor. The VSM signal first goes through a Stanford Research 650 Dual-Channel Filter, set to filter out everything below 4 Hz and above 30 Hz. Furthermore the filter also amplifies the signal, the amplification required depends on the amplitude of the original signal, and thus on the magnetization. The signal is then sent through a Stanford Research 830 Lock-In Amplifier, which measures both the real and imaginary components of the signal and send these back to the computer. The signal can also be monitored visually on a oscilloscope, using the 'monitor out' of the lock-in. This is useful, because it enables us to check if the signal is truly sinusoidal and allows us to choose the correct amplification.

3.3 The potentiometer

Because of friction, the actual motion of the motor is not necessarily equal to what was set by the user on the computer. This would not be a big problem if the friction were to be the same at each motor position. However there is a position dependent component to this friction, caused by the (springs of the) connector piece whose friction depends strongly on how much its springs are compressed (Section 3.1 and Figure 21a). As a result the amplitude when vibrating in a position that leaves the connector piece's springs more or less in their rest position will be significantly larger than when the connector piece's springs are pushed all the way down or up. Furthermore, for high external magnetic fields the sample is going to be attracted to the center of the bitter magnet, thus reducing the amplitude even further. This reduction in amplitude is directly visible in the VMS's signal (right part of Figure 5), which might lead one to conclude that the magnetization has decreased, when in fact it has not.

To record the actual motion of the motor (and thus the sample) we connect a potentiometer (Honeywell SLF01N1500B6A) to the motor. The potentiometer will return a certain fraction of a voltage applied to it depending on the position of the motor (and thus the sample). When the motor starts vibrating this fraction will change accordingly, thus creating a AC (vibration) signal on top of a DC (position) signal. The DC component is then sent to a Agilent 34410 multimeter, which sends it back to the computer. The AC component is sent through a Stanford Research 650 Dual-Channel Filter and Stanford Research 830 Lock-In Amplifier first, after which it can be recorded on the computer and displayed on the oscilloscope.

3.4 Potentiometer power supply

The potentiometer requires some sort of voltage/current source to work. Previously a Keithley 236 Source-Measurement Unit was used for this purpose. However, this device was considered excessive, and thus it was replaced. As a replacement, we have made a box (Figure 4, and Appendix C on page 30) that uses 2 AA batteries to create a 3 V voltage across the potentiometer. The box will separate both the AC and DC components from the signal returned by the potentiometer, using a 1 Hz low-pass filter.

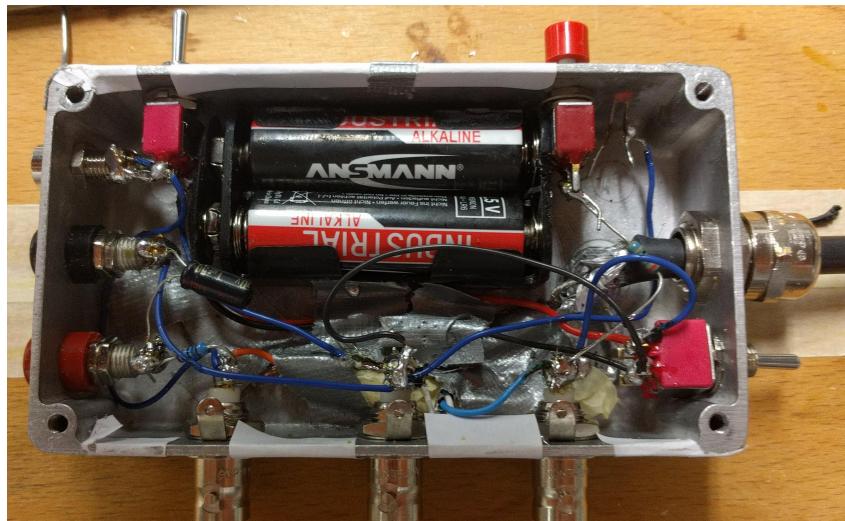


Figure 4: The box that supplies the potentiometer with power (2x1.5 V), and outputs the AC and DC component of the returned signal.

Because the battery depletes over time, we record the current battery voltage on a Keithley 2000 multimeter. Dividing the AC and DC components by this voltage then makes the AC and DC signals dimensionless and compensates for battery drain. We can then use the potentiometer as a reference by dividing the VSM's signal by the potentiometer's signal. Because both signals will reflect the changed amplitude in the same way, dividing them like this cancels out the reduction in amplitude, thus eliminating the effects of the position dependent friction, as has been done in Figure 1 and Figure 3.

4 Calibration

Now the sample motion can be controlled, and the response signal from the pickup coils and the potentiometer can be measured. However, before any measurement can be done, the VSM first needs to be calibrated. First the potentiometer's DC component must be calibrated, in order to know the position of the sample at any given time. Next we need to find the position that leaves the sample exactly between the pickup coils, the flat area in the position loop plot (Figure 2). And finally the VSM's signal has to be calibrated with a sample of known magnetization.

4.1 Calibrating the potentiometer

To calibrate the potentiometer's DC component, set the VSM to vibrate at some position and read off the DC voltage on a multimeter or the computer. Do this for 4-5 points, and make a linear fit through these points, this will yield parameters 'a' and 'b' ($position = a * \frac{pot_meter_{DC}}{battery_{DC}} + b$). These parameters will be used in the next section, to convert the recorded DC component back to a position and plot the VSM's signal as a function of this position (Figure 3). Note that this calibration needs to be redone every time the sample changes or the motor/connector piece/potentiometer is adjusted, because new sample is not going to be in exactly the same position after adjustments.

4.2 Centering the sample

Next we must make sure that the tip of the VSM (coils + sample) is approximately in the center of the external magnetic field. This can be done by using the given specifications of the magnet [2], and the known lengths of the VSM parts (as found in Appendix C). The VSM can be moved up/down as needed with the screw shown in Figure 20. Furthermore, to get a nice sinusoidal signal from the VSM it is even more important that the sample is set to vibrate exactly between the upper and lower pickup coils (the flat area in Figure 3). To accomplish this we need to measure the VSM signal as a function of the sample's position. For this purpose the VSM's control program has a 'position loop' option. This will set the VSM to vibrate at a set begin position for a set amount of time. After which it will move a set distance down, and vibrate for that same amount of time at that position, and so on.

When analyzing the VSM's signal on the lock-in we will see that most of the signal is imaginary since the coils' impedance is mostly inductive. We can use the lock-in's "autophase" function to phase shift the signal such that almost all of the signal is real. We can then use the real part in our response versus position plots, since any change in the imaginary part is just noise.

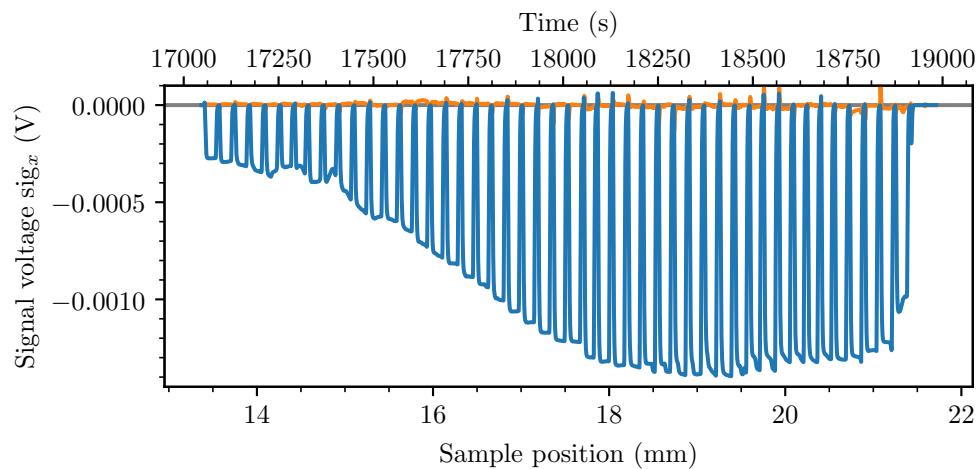


Figure 5: The response of the combination of all four pickup coils, designated '9-10' (18.1 Hz, 1.08 mm) to nickel (42.5 mg), at 2 T (cell 3). This figure contains the unprocessed data used to create Figure 3. The blue curve is the real part, while the orange curve represents the imaginary part.

Doing such a position loop for nickel will yield a graph as shown in Figure 5. In this figure we see that the resistive part (imaginary after "autophase") will fluctuate a bit as well. We can ignore this because we are only interested in the inductive part when looking for the flat area.

If we then divide Figure 5 by the reference we get Figure 6. The advantage of dividing by the reference is immediately clear: Not only does the curve look smoother, it also compensates for the reduced amplitude of the VSM's motor (Section 3.3). This is particularly visible in the right end of the plot, where in Figure 5 there is a significant reduction in amplitude. That is not visible at all in Figure 6.

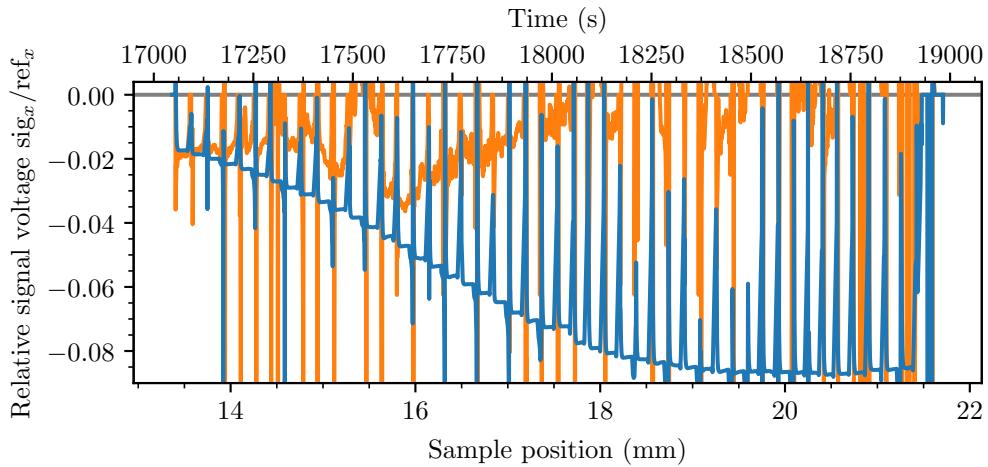


Figure 6: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 1.08 mm) to nickel (42.5 mg), at 2 T (cell 3). This figure is Figure 5 divided by the reference. The blue curve is the real part, while the orange curve represents the imaginary part.

However there is also a disadvantage to dividing by the reference, demonstrated by the spikes in Figure 6. These spikes are caused by points where the reference becomes almost 0 for a brief time, dividing by these near-zero numbers will cause very large positive or negative points in the plot. This is unavoidable since the motor has to stop before it can move to the next position. However, if we identify where each peak occurs in the data file before dividing by the reference, and use only those points after the division, we circumvent the divide-by-zero problem.

To automate this process we have made a simple python script, which can be found here [1]. Which takes data from any raw data file (e.g. Figure 5), locates each peak, divides by the reference, and then calculates the average and standard deviation of each peak. It then takes these data points and plots them against the position, converted from the DC component of the potentiometer’s signal, and if able fits Equation 7 through the data (Figure 1 and Figure 3). More detailed instructions on how to use this can be found in the script’s readme file, and on github [1]. Now that we have such a plot, we can easily identify the flat area and choose the sample’s vibration position accordingly.

4.3 Calibrating with nickel

To measure the unknown magnetization of a sample, we must first calibrate the VSM's signal using a sample with a known magnetization, such as nickel. The saturation magnetization of nickel has been measured to be (58.57 ± 0.03) emu/g [7]. Therefore our sample of 42.5 mg should have a magnetization of (2.489 ± 0.002) emu

By setting the VSM to vibrate within the flat area and changing the strength of the external magnetic field, we obtain a plot as in Figure 7. Here we can see that nickel saturates at about 1 T. We can now calculate the average and standard deviation of all points from the saturation point onwards. We find that our saturated nickel sample ((2.489 ± 0.002) emu) gives an absolute relative signal of 0.0848 ± 0.0002 . Now using the fact that 0 magnetization should give 0 signal, we can make a linear fit. This gives a calibration constant of $c = (29.34 \pm 0.06)$ emu ($\text{magnetization} = c * \text{signal}$)

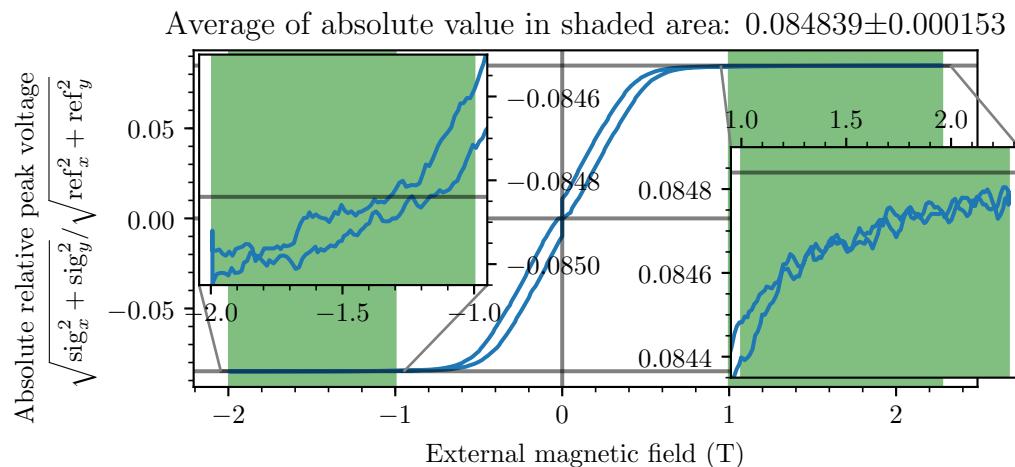


Figure 7: The response of the combination of all four pickup coils, designated '9-10' (18.1 Hz, 1.08 mm) to nickel (42.5 mg), at room temperature (cell 3, 20 mT s^{-1}).

5 Sensitivity of the VSM

Now that the VSM is calibrated, we can measure the unknown magnetization of any sample. However, first we analyze the capabilities of our VSM. We do this by measuring a sample with a very small magnetization and by doing a measurement of an empty sample holder.

5.1 Measuring the saturation magnetization of $\text{CuSO}_4 \cdot 5 \text{ H}_2\text{O}$

Copper sulfate pentahydrate has a very small magnetization, since it consists mostly of diamagnetic atoms, thus enabling us to test how sensitive the VSM is. In fact, at room temperature the magnetization of this material is so small that it is near impossible to distinguish from an empty sample holder (Figure 8). Additionally, at room temperature copper sulfate will not saturate within the range of the cell 3 bitter magnet (max 33 T).

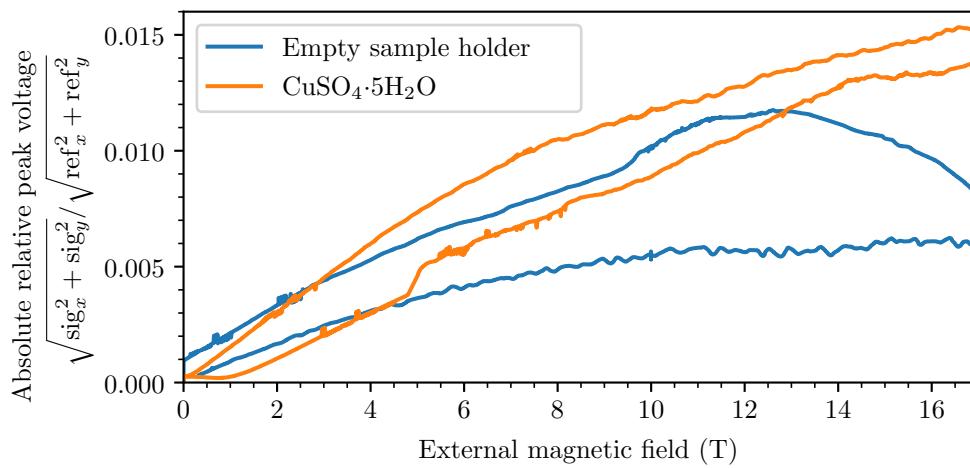


Figure 8: The response of the combination of all four pickup coils, designated '9-10' (18.1 Hz, 1.08 mm) to $\text{CuSO}_4 \cdot 5 \text{ H}_2\text{O}$ (143 mg), at room temperature (cell 3, 10 mT s^{-1}). Compared to the same experiment with an empty sample holder.

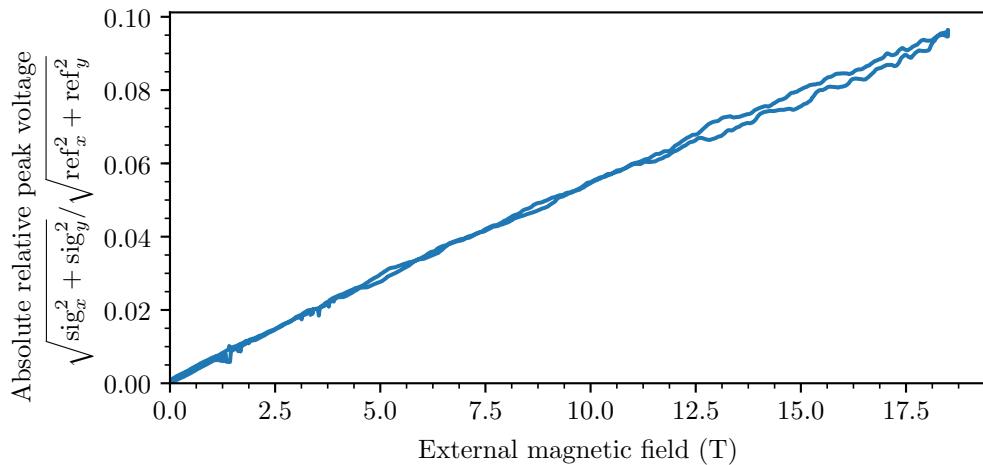


Figure 9: The response of the combination of all four pickup coils, designated '9-10' (18.1 Hz, 1.08 mm) to $\text{CuSO}_4 \cdot 5 \text{ H}_2\text{O}$ (143 mg), at 15 K (cell 3, 10 mT s^{-1}).

To still be able to measure the saturation magnetization, we need to cool the sample down using a cryostat (cryostat 2) [2]. Because thermal energy causes randomization of the direction of the magnetic moments, lower temperatures will make it easier to induce magnetization. The cryostat's thermometers

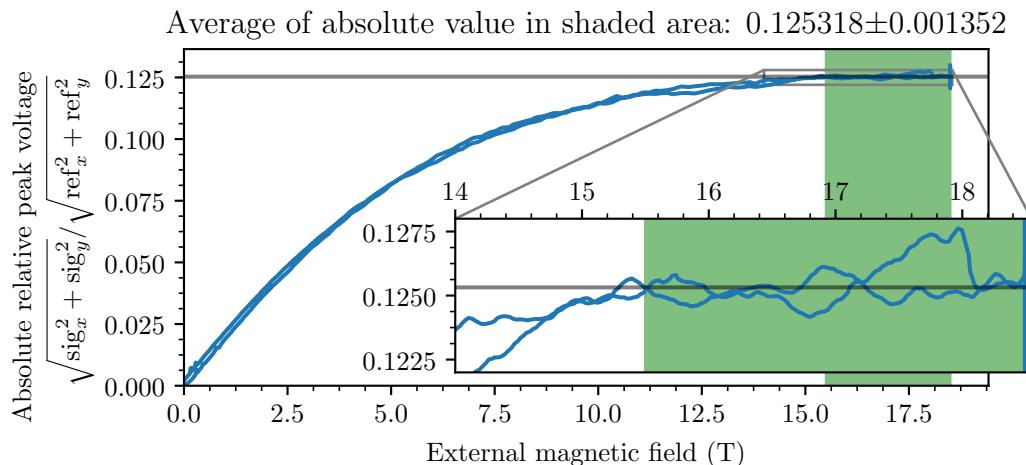


Figure 10: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 1.08 mm) to $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ (143 mg), at 4 K (cell 3, 10mT s^{-1}).

(46682) and heater as well as the VSM’s internal thermometer (x07377) are connected to a LakeShore 350 which controls the cryostat’s heater and measures the temperature in the cryostat and the VSM.

In Figure 9 we see that copper sulfate does not yet saturate at 15 K, thus we need to go down further to 4 K (Figure 10) where saturation occurs at about 15.5 T. The calibration of the potentiometer was very different between the nickel and copper sulfate measurements ($y = 4287.347911 * x + 736.918625$ compared to $y = 8830.788615 * x + 238.4372817$, see section 7). This means that if we divide each measurement by their respective reference we cannot do a direct comparison between the two. However, since the calibration parameters for both measurements are known, we can easily compensate for this by using the ratio of the slopes (a_1/a_2) of both calibrations:

After also compensating for amplification on the VSM’s signal by the filter we can now divide the signal by the corrected reference and plot this (Figure 10). We see that at saturation we get a absolute relative signal of 0.125 ± 0.002 . Using the calibration parameter ‘c’ obtained in Section 4.3 we obtain a saturation magnetization of (3.67 ± 0.04) emu, which gives (25.7 ± 0.3) emu/g.

Sanity Check: In copper sulfate pentahydrate most of the magnetization is due to the Cu^{++} ions. These ions, being a spin $1/2$ system, have a spin magnetic moment of $2\sqrt{s(s+1)} = \sqrt{3} \mu_B$. Using the atomic mass given in table 1, we get a magnetization of 39 emu/g. This is the same order of magnitude as the measured magnetization, but not exactly equal. This is because we have only taken into account the magnetization of the copper ions, however, the sulfate ions and water molecules add a diamagnetic contribution lowering the overall magnetization.

Element	Atomic mass (u)
Cu	63.546
S	32.065
9 O	9×15.9994
10 H	10×1.00794
$\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$	249.684

Table 1

5.2 Measuring background signal of the sample holder

To further analyze the sensitivity of the VSM we measure the VSM's response to an empty sample holder and compare these results to that of 1.2 mg of nickel. The data of this comparison is shown in Figure 11.

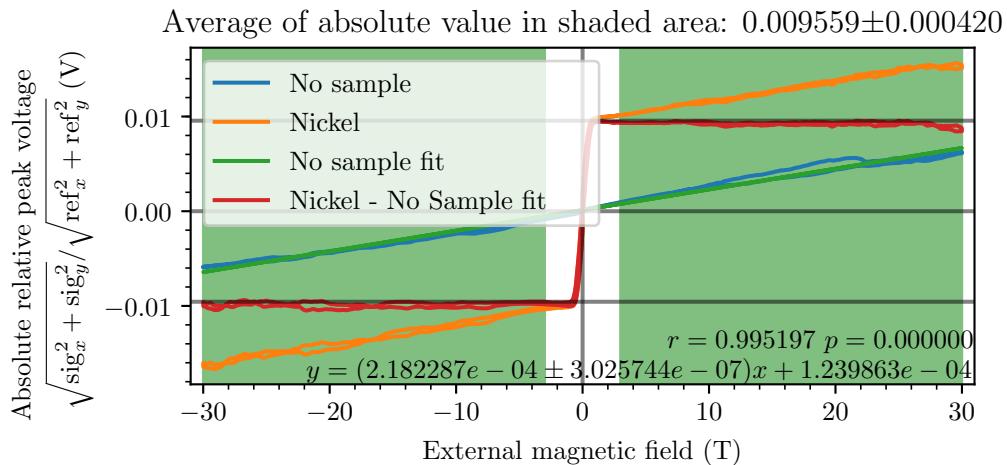


Figure 11: The response of the combination of all four pickup coils, designated '9-10' (18.1 Hz, 2.40 mm) to nickel (1.2 mg) and an empty sample holder, at room temperature (cell 5, 33.33 mT s⁻¹).

Figure 11 shows that the empty sample holder has a linear response within the range of the bitter magnets. After subtracting a linear fit of the sample holder's background from the nickel curve it becomes flat, as it should be.

The data shown in Figure 11 is the response of the sample holders used in all previous measurements. However a new type of sample holder (Figure 12) has been developed using a 3D-printer. It is hexagonal and therefore has superior helium flow along its six sides compared to the circular sample holder. The response of this new sample holder is shown in Figure 13 (room temperature) and Figure 14 (190 K).

Note that during the measurements in Figure 13 and Figure 14 the potentiometer had reached the end of its lifetime. The wiper kept losing contact which caused significant noise in the reference signal, which increased over time. This caused the lock-in to register a decreasing reference time, making the reference signal unusable as a reference. Despite the lack of a good reference, it is still possible to get a correct order of magnitude in the plot while preserving the shape of the curve. This has been done by dividing the signal in Figure 13 by a constant instead. This constant was chosen such that the average of points beyond saturation (shown in the plot's title) is the same in Figure 13 and Figure 11. By doing so Figure 13 and Figure 14 are correct in shape only, the numbers in these figures are limited to educated guesses. That being said, it is still clear that the slope from the new sample holder is smaller than that of the old sample holder. Thus, the new sample holder produces slightly less background noise, and is therefore recommended over the old sample holder for future measurements. Furthermore, from Figure 14 we can conclude that the temperature dependence of the sample holder's background noise is negligible. Following these measurements the potentiometer was replaced with a new one of the same model, a quick check showed that this immediately fixed the problems with the reference.

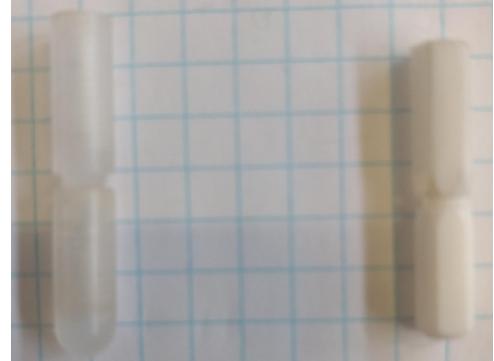


Figure 12: Old sample holder (left) and new sample holder (right).

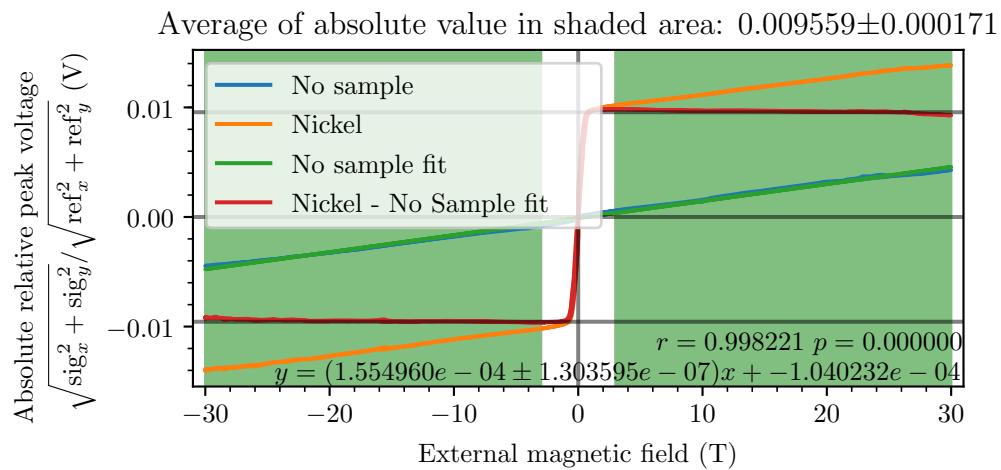


Figure 13: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to nickel (1.2 mg) and a empty sample holder (new version), at room temperature (cell 5, 33.33 mT s^{-1}).

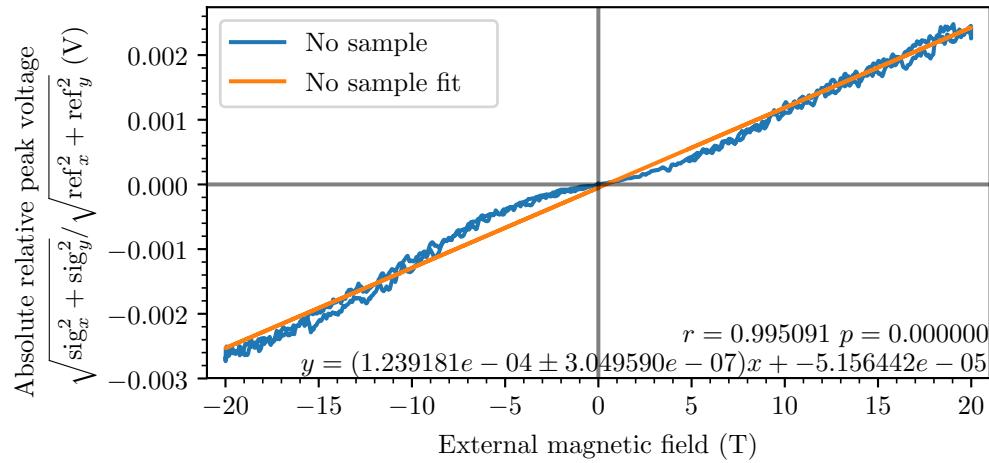


Figure 14: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to nickel (1.2 mg) and a empty sample holder (new version), at 190 K (cell 5, 33.33 mT s^{-1}).

6 Measurements

In the previous sections we have successfully found the saturation magnetization of $\text{CuSO}_4 \cdot 5 \text{ H}_2\text{O}$ and have concluded that the VSM is sensitive enough to do measurements of this order of magnitude (Section 5.1). We have also found that the sample holder(s) themselves introduce a small background signal (Section 5.2). We can now measure the magnetization of some more interesting samples.

6.1 Determining the phase transition point of $\text{KEr}(\text{MoO}_4)_2$

As we shall see $\text{KEr}(\text{MoO}_4)_2$ has a magnetic phase transition at around 20 T. The sample was provided by dr. Dmytro Kamenskyi and Bence Bernáth, who were researching this material. To ensure the best results, the first step was to again measure the background for this specific sample holder, because the data in Figure 13 and 14 is not reliable (see Section 5.2). This background signal as a function of magnetic field can be obtained directly from a nickel calibration. Because nickel's magnetization does not change after saturation, any change in signal beyond the saturation point is background noise.

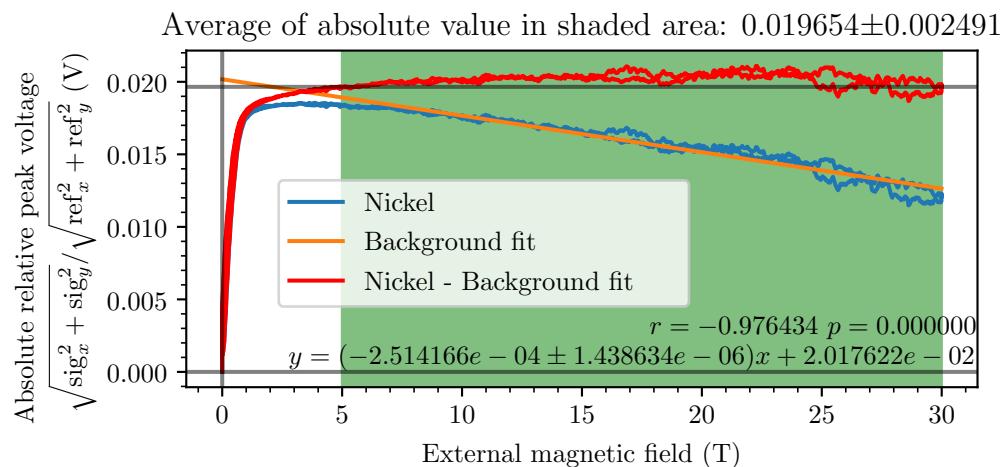


Figure 15: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to nickel (1.2 mg), at 1.3 K (new sample holder, cell 5, 66.67 mT s^{-1}).

Figure 15 shows the response of a small nickel sample in a new sample holder is shown. A line is fitted through the data from 5 T to 30 T, this fit represents the background noise and should therefore be subtracted from data obtained from measuring $\text{KEr}(\text{MoO}_4)_2$. The red curve shows the data after subtracting the slope of the fit times the magnetic field. Using the magnetization and mass of the sample ((58.57 ± 0.03) emu/g [7] and 1.2 mg) and the average signal at saturation ((0.020 ± 0.003) V), we obtain the calibration parameter $c = (3.6 \pm 0.2)$ emu/V. Note that the unit of this calibration parameter differs from the one found in Section 4.3 because for these measurements the box (Figure 4) was used. Thus the reference has been made dimensionless by dividing it by the battery voltage. And therefore the signal keeps the unit Volt after dividing by the reference, hence the calibration parameter now has the unit emu per Volt.

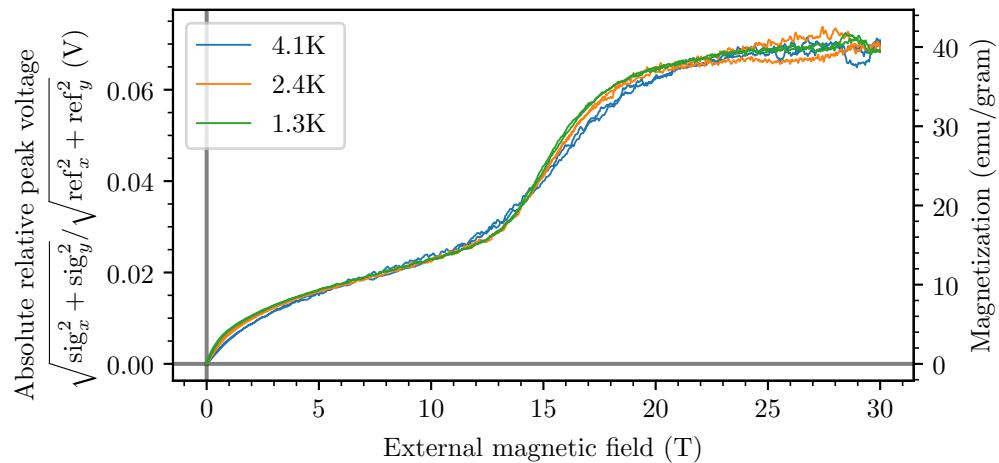


Figure 16: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to $\text{KEr}(\text{MoO}_4)_2$ (6.2 mg), at 1.3 K (new sample holder, cell 5, 33.33 mT s^{-1}). The background signal obtained from Figure 15 has been subtracted from the data.

Figure 16 shows the response of $\text{KEr}(\text{MoO}_4)_2$, using the calibration parameter obtained from Figure 15 the magnetization has been plotted on the secondary axis. A phase transition is seen from 12 T to 25 T. We also see that the curve in Figure 16 becomes sharper as the temperature decreases. This makes sense because as the magnetic field moves the phases’ energy levels closer together, there will be less thermal excitation to the higher states for lower temperatures. This mimics a Fermi-distribution function becoming sharper as the temperature decreases.

The 2.4 K curve in Figure 16 shows some hysteresis at high magnetic fields. This illustrates an experimental problem called the “Helium bubble issue”. When the magnetic field is high, diamagnetic repulsion will push helium gas to the field center, causing helium gas to accumulate in the cryostat at the center of the magnet. This locally reduces the thermal isolation, and thus increases the temperature slightly, which reduces the magnetization. This helium bubble issue is not present in the 1.3 K curve because at this temperature helium is superfluid, giving it very high thermal conductivity, and precluding the formation of bubbles.

6.2 Analyzing phase transitions of $\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$ and $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$

The following section briefly describes the research by dr. Patricia Lázpita from the University of Basque Country and Anabel Pérez Checa from BCMaterials using the VSM at HFML. More information on this will be available in their paper. This research is aimed at the materials $\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$ and $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$ which are Heusler alloys. These alloys exhibit a phase transition from an austenite phase (face-centered cubic) to a martensite phase (strained body-centered tetragonal) [5]. Before measuring the magnetization of these samples, we first do another nickel calibration.

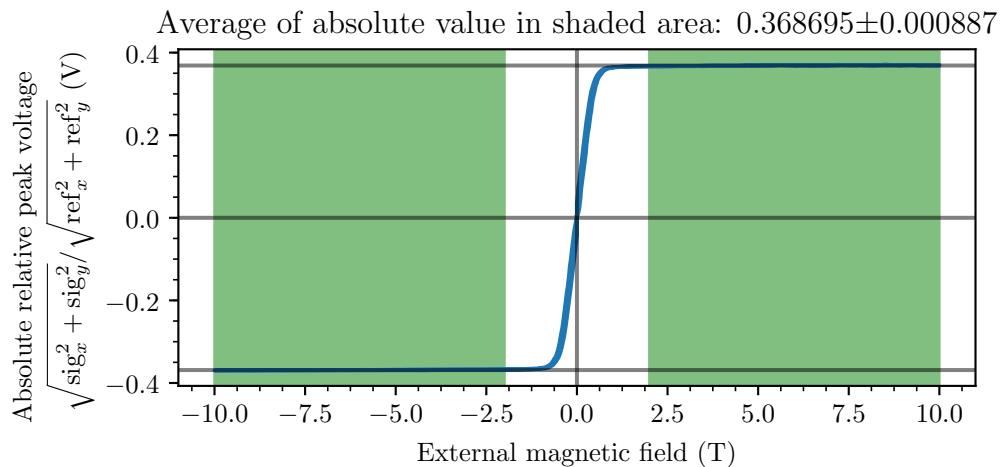


Figure 17: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to nickel (49.5 mg), at 100 K (new sample holder, cell 5, 33.33 mT s^{-1}).

Using the magnetization and mass of the sample ($(58.57 \pm 0.03) \text{ emu/g}$ [7] and 49.5 mg) and the average signal at saturation ($(0.3687 \pm 0.0009) \text{ V}$), we obtain the calibration parameter $c = (7.8635 \pm 0.0004) \text{ emu/V}$. This was used in Figure 18 and 19 to calculate the magnetization. Note that this calibration parameter is about twice that of the calibration parameter in the previous section. An explanation for this difference is given in Section 7.3.

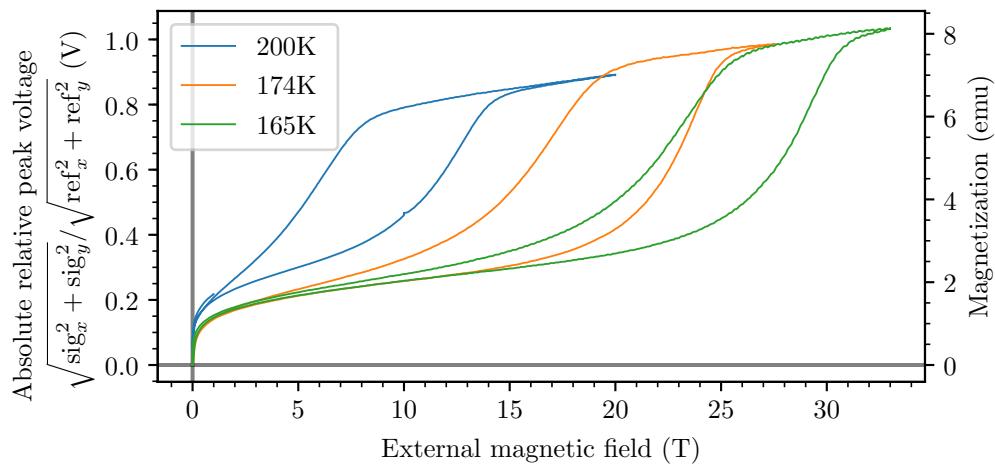


Figure 18: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to $\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$ (new sample holder, cell 5, 33.33 mT s^{-1}).

The results of the first sample ($\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$) are plotted in Figure 18. The phase transition shows a strong temperature dependence, illustrated by the phase transition moving to higher magnetic fields for lower temperatures. This is because lower temperatures favor the martensite phase [5].

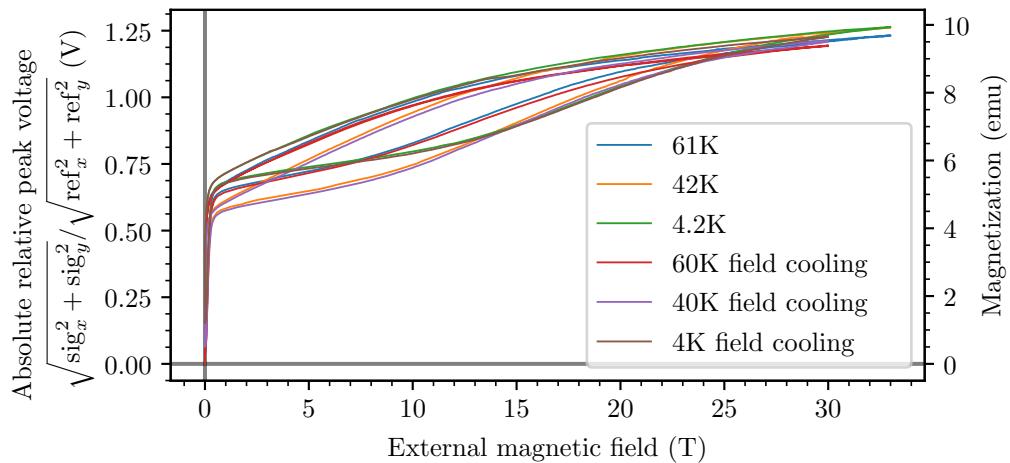


Figure 19: The response of the combination of all four pickup coils, designated ‘9-10’ (18.1 Hz, 2.40 mm) to $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$ (new sample holder, cell 5, 66.67 mT s^{-1}).

The second sample $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$ has one less nickel and one more iron. Its phase transition (Figure 19) shows less temperature dependence than the previous sample. However Figure 19 shows an interesting finding, suggesting that the phase transition point depends on the sample’s history. There is a visible difference between first cooling the sample then sweeping the field up and down, and first sweeping up, cooling down and then sweeping down (field cooling). Figure 19 illustrates this by comparing the ‘field cooling’ and non ‘field cooling’ lines at the same temperature. For small magnetic fields the curves are similar, however at high fields the magnetization is lower for the ‘field cooling’ curves.

7 Discussion

7.1 Different potentiometer power supplies

The large difference in potentiometer calibration outlined in Section 5.1 arises from the fact that the potentiometer's power supply has been replaced between measurements. Initially, a Keithley 236 current source was used. However, this (very expensive) piece of equipment was considered excessive for the mere purpose of supplying power to the potentiometer. Therefore, the plan was to construct a box (Figure 4) that could supply this voltage instead, and which also includes a 1 Hz low-pass filter to separate the DC component.

We first tested the feasibility of this new box by using a simpler voltage source (Delta elektronika power supply E015-2) during the copper sulfate experiments. However, the voltage on this source was set higher than when using the Keithley 236 (during the nickel calibration). This was not thought to be a problem initially. However, this lead to a significant undesired difference between the 'a' parameters of the two measurements.

A solution for this was discussed in Section 5.1. However, using the ratio of the calibration parameters 'a' as a correction factor means that now the errors in these parameters are relevant. The parameters were obtained using linear regression, this also yielded the following coefficients of determination for nickel and copper sulfate respectively:

- 0.9999985027
- 0.9998095329

This shows that the error in the linear fit is sufficiently small to not effect the measurements.

Note that when using the batteries of the box as the power supply for the potentiometer, it is still required to correct the reference if there is a large time delay between measurements. This is due to the battery slowly depleting over time. However, if one records the current battery voltage for each measurement one can easily divide the reference signal by this voltage. This compensates for the battery drain and makes the reference dimensionless. This has been done for all measurements after Section 5.1.

7.2 Magnetization at B is not the same as at -B?

The attentive reader might have noticed that in Figure 7 at saturation the signal for positive B is smaller than average-line, while for negative B it is always larger. This appears to suggest that the saturation magnetization of the nickel sample is larger for negative magnetic field and smaller for positive magnetic field. This is of course impossible, something else must be going on!

A possible explanation for this aberration would be a constant negative DC offset in the VSM's signal. However, the signal goes through a 4 Hz high pass so any DC offsets should have been filtered out. Therefore this cannot be a valid explanation of this difference.

Another potential explanation is that there is some small contribution to the overall magnetization originating from the sample holder or from impurities left behind by previous samples in the holder. Such a explanation would be consistent with the measurements of the empty sample holders in Section 5.2. We indeed see in Figures 11, 13 and 14 that there is a non-zero 'b' parameter in the linear fits of the empty sample holders. However, this just changes the question to, "why is the 'b' parameter non-zero for the linear fit of the sample holders?" A satisfying explanation for this strange behavior has yet to be found.

Note that the difference between signals for positive and negative fields is about 0.2% of the average. Since our VSM's flat area has a deviation of 0.1%[4] (Section 2.3), this problem might very well just be caused by the limit of our VSM's sensitivity. To account for this problem, the difference between the contributions at negative and positive field is included in the error by calculating the standard deviation.

7.3 Changed calibration parameters?

In Sections 4.3, 6.1 and 6.2 different VSM calibration parameters were found:

- Section 4.3: $c = (29.34 \pm 0.06)$ emu
- Section 6.1: $c = (3.6 \pm 0.2)$ emu/V
- Section 6.2: $c = (7.8635 \pm 0.0004)$ emu/V

For Section 4.3 this is because the amplitude of the vibration was different compared to the later measurements and because this calibration did not use a dimensionless reference (Section 7.1).

The difference in VSM calibrations in Section 6.1 and Section 6.2 is explained by the fact that during the measurements in Section 6.2 the sample was not correctly centered in between the coils. Instead it was unintentionally in the local minimum of the lower coils (Figure 2). Therefore the sample was not in the flat area (Figure 2) and the VSM's sensitivity was halved (only half the coils are contributing). The reason the sample was not in the center is because when finding the center the local minimum was mistaken for the global maximum. To prevent this problem from re-occurring in the future, one should always check that the found maximum is wide enough to be the global maximum (flat area Figure 2). This can also be checked by measuring the contributions of individual pickup coils, if all of the signal is on the upper or lower pickup coils the sample is not in the global maximum but in a local minimum. Whilst it is not optimal to have the sample in a incorrect position, it is not a big problem for $\text{Ni}_{38}\text{Mn}_{49}\text{Sn}_9\text{Fe}_4$ and $\text{Ni}_{37}\text{Mn}_{49}\text{Sn}_9\text{Fe}_5$ because they have relatively large responses. The calibration parameter in Section 6.1 however is correct, since the sample was correctly in the center (flat area Figure 2) in this case.

Acknowledgments

None of this would have been possible without the help of Prof. Dr. Uli Zeitler, who gave me the opportunity to work with the VSM at HFML, and was always there to answer my many questions and to explain how all the things worked; the VSM, the lock-in amplifiers, the cryostat, the helium pump, and of course the bitter magnets. My sincerest thanks.

My thanks also to Hung van Luong, who taught me how to use the VSM control program, showed me how all the devices (lock-ins, filters) should be connected to each other, and helped me whenever I had problems with the motor or the computer.

Furthermore, my thanks go out to Lijnis Nelemans, Michel Peters and Edwin van Leverink, who set up the VSM, helped me to create the potentiometer's box and provided me with technical advice and equipment.

To dr. Patricia Lázpita from the University of Basque Country and Anabel Pérez Checa from BCMaterials, thanks for providing a very interesting sample, and explaining the physics behind the martensite/austenite phase transitions.

Also thanks to dr. Dmytro Kamenskyi and Bence Bernáth for providing another interesting sample.

I also would like to thank the rest of the amazing staff and students at HFML, who have been very kind and helpful in showing me around and helping me find all the things in the lab.

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A Manual for the VSM motor controller program

Warning: If you disconnect the motor's powersupply, the motor will have to be reinitialized ("Init"). This means that the motor will have to be disconnected from the VSM, which usually means that both the potentiometer's calibration and the VSM's center will change. Hence a full recalibration is required.

To start motor:

1. Press "Connect to VSM", usually the VSM resides on COMM 3, and the default settings are fine.
2. Press "Ok".
3. If not already on, press "Motor on".
4. If this is first time the motor is used after it has been plugged in: press "Init". The motor will move as far up as it can go, and use that as the zero position. For this to work, the motor has to be disconnected from the rest of the VSM, and the potentiometer must be moved all the way up.
5. Set the desired frequency, displacement (2x amplitude), and begin position (the motor will vibrate between 'begin position' and 'begin position' + 'displacement').
6. Click "Calculate freq code", this will convert the frequency and displacement into a single code that the motor can understand.
7. Click "Send calc freq code", which will send the frequency code to the motor.
8. Click "Send string", this sends the string that contains the 'begin position' parameter to the motor.
9. Click "Start motor", to stop the motor click "Stop motor".

Starting a position loop:

1. Complete the above first, but do not yet start the motor.
2. Set the desired step size, number of steps and step duration.
3. Click "Start position loop".
4. When the loop has been completed, the motor will continue to vibrate at the last position, to stop the motor click "Motor on" immediately followed by "Motor off".

Troubleshooting:

1. Motor does not respond:

The motor stores commands in finite volatile memory. When it is full the motor cannot receive any further commands from the controller program. To clear the memory, press the buttons "Flush input queue", "Flush output queue", and "Reset macro".

2. Motor does not start:

If the motor does not start after it has been disconnected from the power, press "Init" to reinitialize the motor. Because the motor uses volatile memory, it will forget everything once the power has been disconnected.

3. Motor still does not start:

Try to start the motor without anything attached to it. If it works now, it is because there was too much friction. Try adjusting the potentiometer, or turn the connector piece a bit.

4. The motor stops halfway a position loop:

Beyond this point the motor encounters too much friction to continue, sometimes the friction can be reduced by turning the connector piece a bit for optimal alignment.

B VSM measurement step-by-step

Initial setup

- Insert cryostat in magnet, and put VSM inside cryostat.
- Adjust VSM height (Figure 20) such that the pickup coils are in the center of the external magnet (Measure the distance from top of cryostat to the center of the magnet, and use schematics in appendix C).
- Connect lock-ins/amplifiers etc. as shown schematically on page 30.
- Connect cryostat to the lakeshore 350 and to the helium pump. Connect the VSM's thermometer to the lakeshore as well.
- Set the calibration curves the lakeshore should use for the thermometers (VSM:x07377, cryostat2:46682).
- Vacuum pump the cryostat's vacuum chamber.
- Setup measurement program to record signal, AC-reference, DC-reference, temperature and magnetic field
- Move potentiometer all the way up.
- Switch the motor on, and initialize (Appendix A), if not already initialized.
- Switch the motor off.

Putting a sample in the VSM

- Insert calibration sample in sample holder, and screw it onto the sample stick (Figure 21b).
- Screw the sample stick onto the connector piece (Figure 21a), and put the stick in the VSM.
- Connect the motor to the connector piece.
- Adjust the connector piece such that the motor can move freely (Try screwing the connector piece tighter/looser), when done correctly the motor will fall down when pushed up and released.
- Adjust potentiometer such that the motor can make use of the full range of the connector piece.
- Turn motor on
- Locate the smallest and largest possible motor position, by choosing some position and visually checking the connector piece.
- Calibrate the potentiometer, by setting the motor to a couple of different begin positions and recording the corresponding DC component of the reference, and making a linear fit.

Cooling the sample

- Fill cryostat with liquid nitrogen followed by liquid helium, depending on desired temperature.
- If required, set desired temperature in the lakeshore and enabled the heater.
- Once desired temperature has been reached turn on the motor and the bitter magnet.

Doing a measurement

- Set the motor to vibrate at some position, and set the bitter magnet to some field where there is a clear signal, adjust signal/reference amplification if required.
- Use the lock-in's "autophase" function
- Set the VSM to do a position loop from the smallest to the largest position. To save time it might be a good idea to first do a rough position loop with large steps, and finding where the center is approximately. Followed by a second more detailed position loop, with more steps, using an educated guess as begin position. Use coilprofileplotter.py to help find the center, detailed instructions are available in the script's readme file [1].
- Double check that the found center is in fact correct, by checking that both the upper and lower coils give equal contributions to the signal. If the lower or upper coils do not give any signal the position is incorrect, shorten/lengthen the sample stick as required.
- Adjust the motor's begin position and displacement such that the sample will vibrate exactly in between the pickup coils.
- Measure as a function of the magnetic field.

Replacing the sample

- Switch off the motor, and disconnect it from the connector piece.
- Create a over-pressure in the cryostat, unscrew the connector piece and remove the sample stick, then put the connector piece back on the VSM.
- Replace sample in sample holder.
- Unscrew the connector piece and reattach the sample stick, screw the connector piece back on the VSM and reconnect the motor.
- Wait for sample to reach desired temperature.
- Re-calibrate the potentiometer, and do a new position loop.

Calibrating VSM

- Steps "Putting a sample in the VSM" and "Cooling sample" for calibration sample.
- Steps "Doing a measurement".
- For better calibration: Steps "Replacing Sample" with second calibration sample, or measure the empty sample holder.
- For better calibration: Steps "Doing a measurement".
- Identify the saturation point(s) and find the relative absolute signal(s) at saturation.
- Use the known magnetization of the calibration sample(s) to obtain a calibration 'c*x' or 'c*x+b'.

Measuring sample with unknown magnetization

- Steps "Replacing sample" for target sample.
- Steps "Doing a measurement"
- Identify saturation point and find the relative absolute signal at saturation, make sure to correct the reference first using the ratio of the battery voltages (if changed).
- Use calibration to find magnetization and magnetization per gram.

C Schematics and Pictures

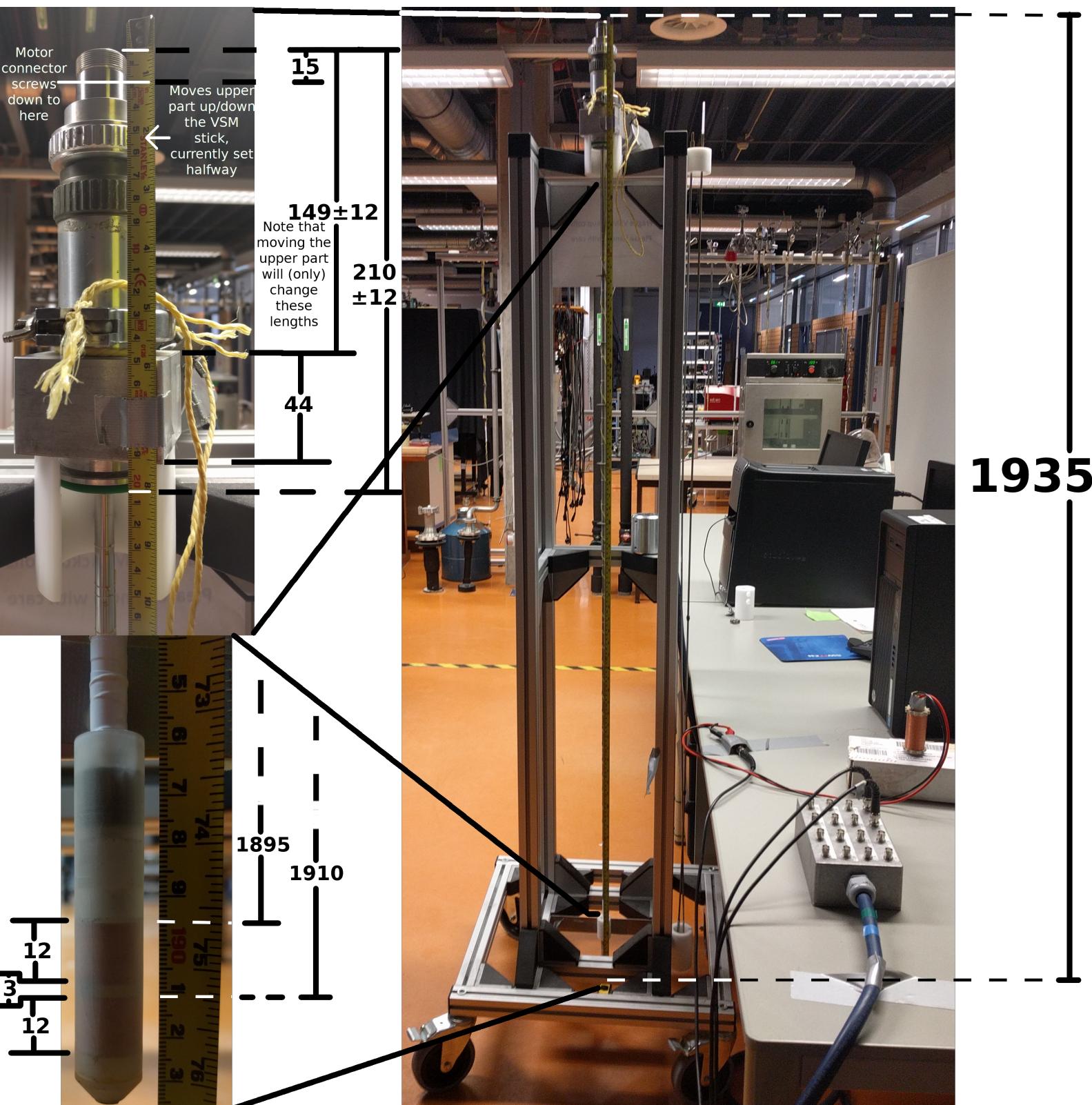
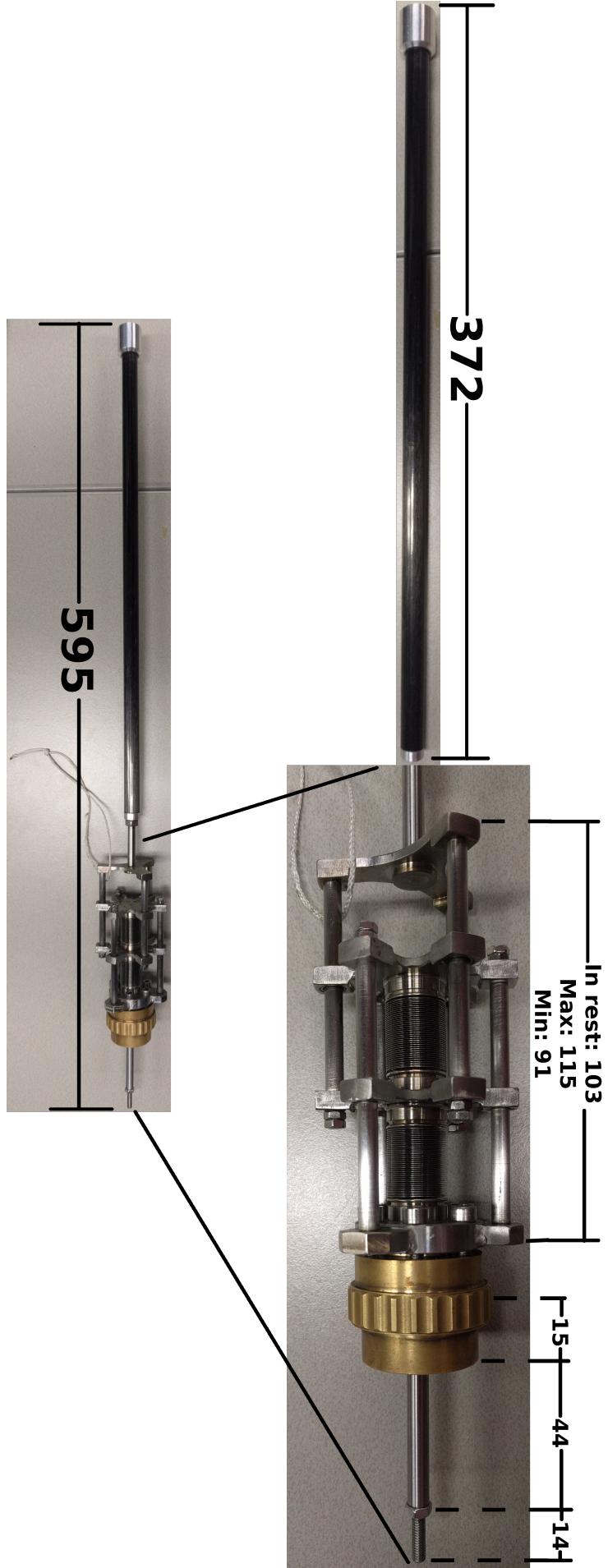
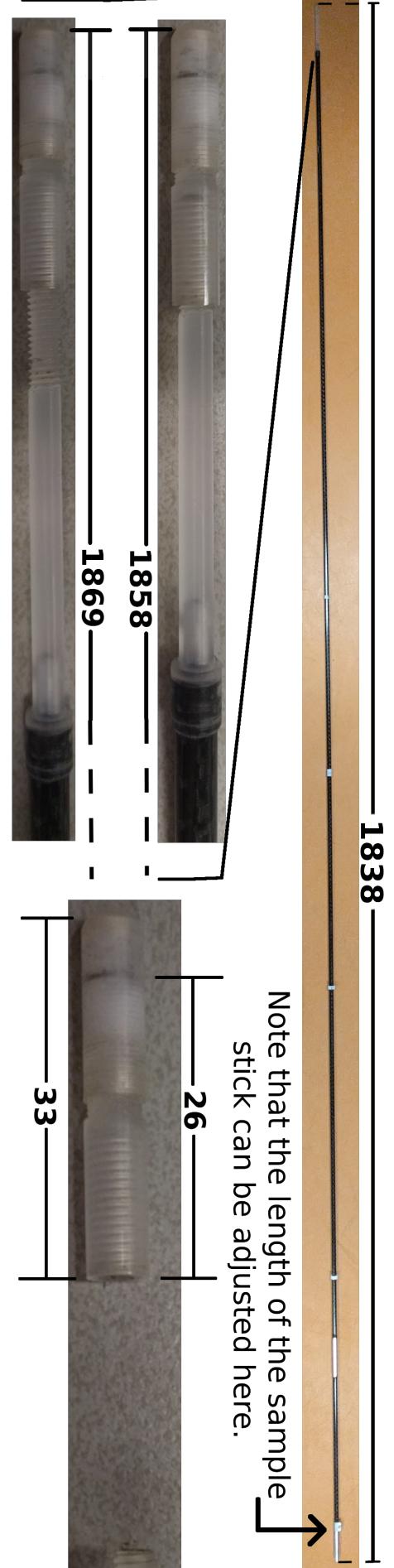


Figure 20: VSM at room temperature, lengths (mm) will depend on temperature. Lengths to coils are taken from the top of the VSM to the top of the coils. The VSM can move up/down in order to center the coils in field center of the bitter magnet. The distance from the top of the cryostat to the field center is 1420 mm[2]



(a) Connection piece for connecting the motor to the sample stick and VSM.



(b) The sample stick, its length can be adjusted. The length of the sample stick shown here is without a sample holder (right) and with sample holder (left).

Figure 21

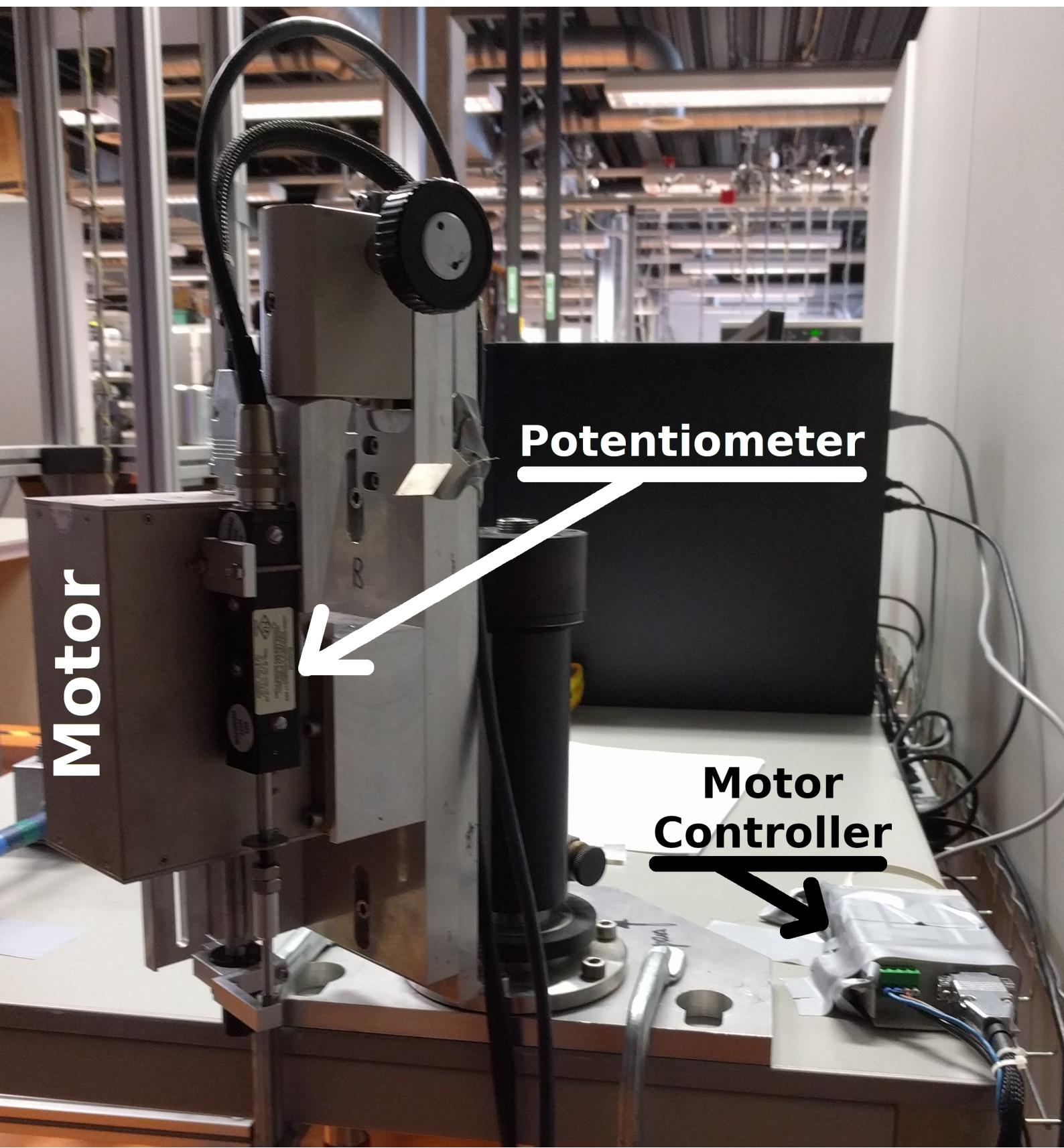


Figure 22: The motor and potentiometer.

