

# Measuring the Dependence of Weight on Temperature in the Low Temperature Regime using a Magnetic Suspension Balance

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**Abstract.** A novel setup was developed that allows to cool samples close to liquid helium temperatures, measure their exact temperature and determine their weight in a buoyancy-free environment along a wide temperature range using a magnetic suspension balance. This allows for the first time to accurately determine the weight of both high- $T_c$  (BSCCO and YBCO) and low- $T_c$  (Nb) superconductors during their phase transition. Our data allows to put limits on possible weight changes over temperature ( $\alpha < 2 \times 10^{-8} \text{ K}^{-1}$  for copper) as well as violations of the weak equivalence principle for superconductors while passing their critical temperature ( $\eta < 2 \times 10^{-3}$ ).

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*Keywords:* Magnetic Suspension Balance, Superconductivity, Weak Equivalence Principle, Gravitational Constant

## 1. Introduction

The dependence of the gravitational attraction on temperature was studied about 100 years ago by Poynting and Phillips [1], Southern [2] and later by Shaw and Devy [3]. In the approach from Poynting and Phillips [1], a common balance was used in a vacuum and the samples were heated by water steam or cooled by liquid air through a surrounding jacket. They claimed a null result with a sensitivity of  $2 \times 10^{-10}$  per  $^{\circ}\text{C}$ . This result seems quite doubtful, since the actual temperature of the weight was not measured but it was only assumed that its temperature would be the same as the one from the surrounding jacket without taking radiation losses or heat conductivity from the wire connecting the weight sample to the balance at room temperature into account. Southern [2] had a much better temperature control as his weight sample was attached to a calorimeter. However, he varied the temperature only between 11.8 and 32.5  $^{\circ}\text{C}$ . He achieved a null result with a resolution of  $1 \times 10^{-8}$  per  $^{\circ}\text{C}$ .

Two decades later, Shaw and Devy [3] performed torsion balance measurements and excluded variations of the gravitational attraction down to  $2 \times 10^{-6}$  per  $^{\circ}\text{C}$  for temperatures from 18 to 250  $^{\circ}\text{C}$ . No other investigations on that topic were reported for the next 80 years. Recently, Dmitriev et al [4, 5] re-analysed their data and performed additional measurements claiming a temperature relationship for weight (mass) of  $\Delta m/m_0 = \alpha \cdot \Delta T$  with  $\alpha = 6.5 \times 10^{-6} \text{ K}^{-1}$  for copper among other materials. Classical physics predicts only a coupling factor of  $\alpha \simeq 10^{-14}$  using Einstein's  $E = m \cdot c^2$  relationship [6]. A weight variation could be related to a possible variation of the gravitational constant  $G$  with temperature. Since  $G$  is still the fundamental constant with the largest uncertainty [7], it seemed worthwhile to re-examine the relationship of the gravitational attraction with temperature.

We chose to extend the range of temperature tested towards the low temperature regime triggered by reported weight anomalies as high as 1% for high- $T_c$  superconductors [8]. Reiss used a balance which was directly connected to the samples floating in gaseous and liquid nitrogen which required large buoyancy corrections ‡. We briefly assessed this claim previously in a similar setup finding no anomalies but we also used large buoyancy corrections which was somewhat unsatisfactory [9]. On a related subject, Tate et al [10, 11] reported a net mass excess of about 0.01% for Cooper-pairs in niobium superconductors which still remains unexplained so far [12] and may be interpreted as a violation of the Weak Equivalence Principle (WEP) for quantum objects as recently proposed by a number of authors [13, 14, 15]. Jain et al [16] attempted to test the strong equivalence principle for superconductors and ruled out violations at a level of 4% which is not sufficient to rule out the other claims.

We designed and built a novel setup that allows to precisely measure the weight and temperature of a sample from room temperature down to nearly liquid helium temperatures in a buoyancy-free environment. Our setup used a high precision magnetic

‡ he suggested to repeat the measurements with a magnetic suspension balance at the end of his paper

**Table 1.** Experimental Results

Sample	Sample Mass $m_0$ [g]	Eötvös Factor $m_g$ Variation $\eta_g$	Eötvös Factor $m_i$ Variation $\eta_i$	Temp. Infl. on Grav. Attraction $\alpha$ [ $K^{-1}$ ]	Critical Temp. $T_c$ [K]	Temp. Range [K]
Copper	15.572074	-	-	$8.5 \pm 5.1 \times 10^{-9}$	-	84 - 231
BSCCO (2223)	6.256984	$0.2 \pm 1 \times 10^{-6}$	$1.3 \pm 6.5 \times 10^{-4}$	$3.8 \pm 3.4 \times 10^{-8}$	$\sim 108$	85 - 135
YBCO	6.534149	$0.6 \pm 7.3 \times 10^{-7}$	$0.4 \pm 4.7 \times 10^{-4}$	$-2.5 \pm 3.9 \times 10^{-8}$	$\sim 92$	85 - 117
Niobium	15.572074	$2.0 \pm 4.0 \times 10^{-7}$	$1.3 \pm 2.6 \times 10^{-4}$	$1.2 \pm 0.6 \times 10^{-6}$	9.2	8 - 13

**Table 2.** Summary of Claims in the Literature

Sample	Claim	References	Limits from Our Work ( $3\sigma$ )
Copper	$\alpha = 6.5 \times 10^{-6} K^{-1}$	Dmitriev [4, 5]	$\alpha < 2.4^{-8} K^{-1}$
BSCCO (2212)	$\Delta m/m_0 = 0.001 - 0.01$	Reiss [8]	$\Delta m/m_0 < 3.2 \times 10^{-6}$
YBCO	$\Delta m/m_0 = 0.007$	Reiss [8]	$\Delta m/m_0 < 2.2 \times 10^{-6}$

suspension balance from Rubotherm § that allows to mechanically de-couple the test sample from the balance using feedback-controlled magnetic levitation. This is necessary since our samples had to be cooled down to near liquid helium temperatures while the balance with its electronics had to remain at room temperature. We developed a cooling and temperature measurement method that allows to first cool down the sample and continue to measure its weight under vacuum conditions and therefore buoyancy free, while still remaining the capability of monitoring the samples temperature. This allowed to accurately track the weight of superconductors during their phase transition. Since we performed weight measurements, composed of the gravitational attraction and the centrifugal force from the Earth’s rotation, our results can be used to put first limits on possible WEP violations for low and high- $T_c$  superconductors. This extends the range of WEP tests from Baryon number, iso-spin charge [17, 18], rotating [19], spin-polarized sources [20, 21] and quantum particles [22] to macroscopic quantum objects.

Our measurements did not show any significant weight changes within our resolution and time-scales for either superconductors during their phase transition or for a copper sample along a wide temperature range. Our results are summarized in Table 1 together with a comparison of previous claims and the limits obtained by us in Table 2.

## 2. Experimental Setup

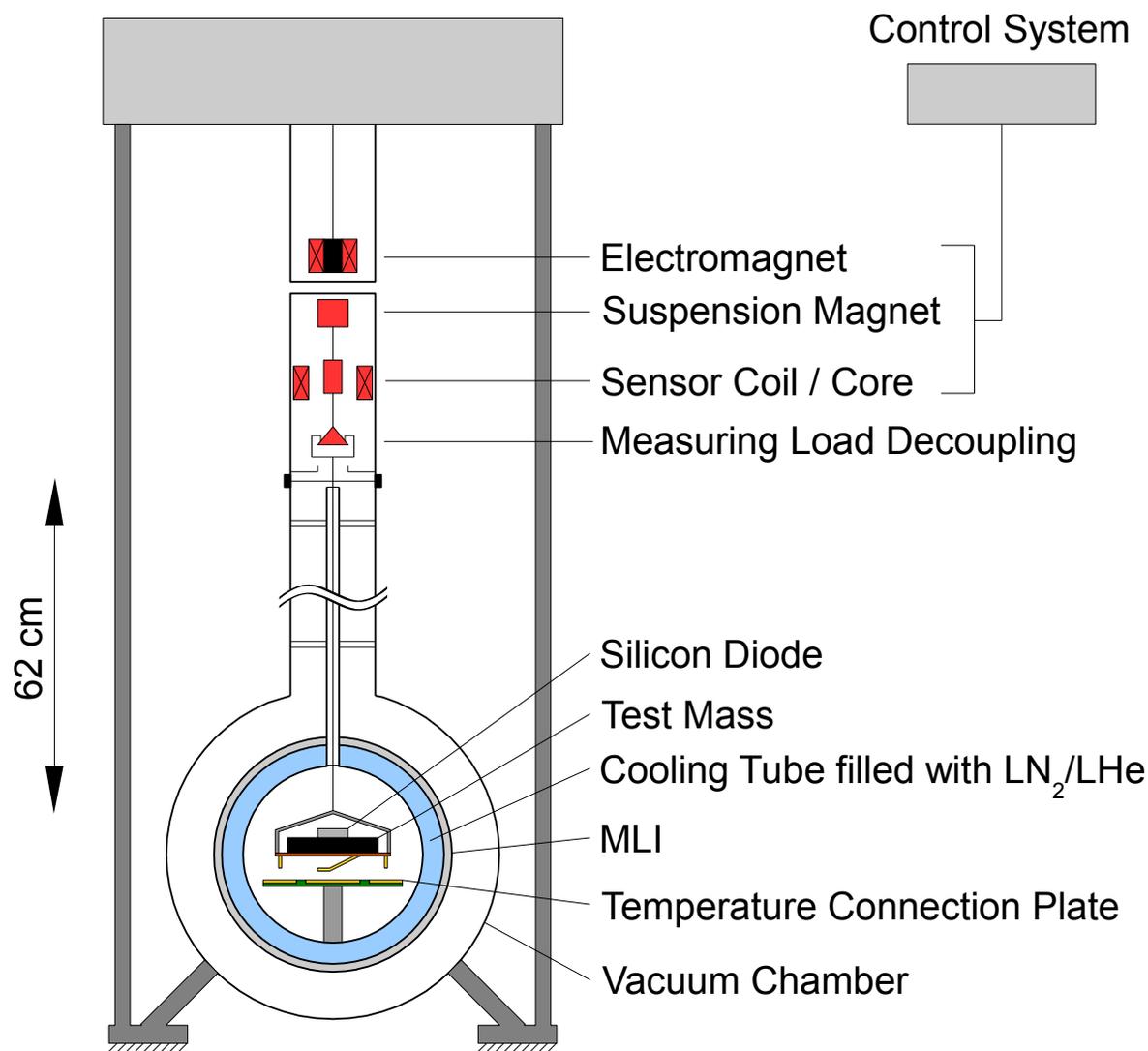
The overall setup is illustrated in Fig. 1. We used a magnetic suspension balance (MSB) from Rubotherm (microgram version) which is connected to a Sartorius CC111 electronic mass comparator. Such a balance allows to weigh a sample contactless under nearly all environments (vacuum, high pressure, wide temperature ranges). Instead

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of hanging directly at the balance, the sample to be investigated is linked to a so-called suspension magnet which consists of a permanent magnet, a sensor core and a device for decoupling the measuring load (sample). An electromagnet, which is attached to the underfloor weighing hook of a balance, maintains a freely suspended state of the suspension magnet via an electronic control unit. Using this magnetic suspension coupling, the measuring force is transmitted contactlessly from the measuring chamber to the microbalance, which is located outside the chamber under ambient atmospheric conditions. Consequently, this arrangement eliminates almost all restrictions which are inherent to conventional gravimetric measuring instruments and makes it possible to measure the weight of a sample at different temperatures while residing inside a vacuum chamber.

Samples up to 30 g can be measured with a resolution down to 1  $\mu\text{g}$  and a reproducibility of  $\pm 4 \mu\text{g}$ . On the bottom of the magnetic coupling part, we attached a vacuum chamber which contained a cooling tube (see Fig. 2). This tube is made out of copper and has a diameter of 50 mm and a length of 92 mm. It is wrapped with copper tubes which can be connected either to a liquid helium or nitrogen dewar outside the vacuum chamber. Multilayer Isolation (MLI) is used around the tubes in order to provide good thermal isolation. The front part of the cooling tube can be opened in order to attach samples to a wire (1.4304 stainless steel, diameter 0.125 mm) which in turn is connected to the magnetic coupling pick up hook about 62 cm above. All samples are pellet shaped with a diameter of about 25 mm and a thickness of 3 mm. Each one of them are put on a sample holder with a silicon diode (Lakeshore DT-670B-SD, temperature tolerance  $\pm 0.5$  K) glued on top of them (see Fig. 3). We used a STYCAST 2850FT/LV24 low temperature epoxy which has a high thermal conductivity to ensure an excellent thermal contact. Another silicon diode is glued on the inside surface of the cooling tube.

Usually, the temperature of the sample is measured by a thermocouple in close vicinity to the sample since most measurements with magnetic suspension balances are done in a gas atmosphere which ensures thermal conduction between sample and probe. In our case, this was not possible as we are performing measurements in vacuum in order to neglect buoyancy effects and to obtain good thermal isolation. We implemented another method using one of the operational characteristics of the MSB. The balance was operated with a measurement frequency of 0.025 Hz allowing 40 s of data averaging per measurement point. Every three measurements points, the balance performs a zero point measurement in order to compensate drifts. In this mode, the balance lowers the sample by about 3 mm until the sample is not levitating any more and only the weight of the levitating magnet and its support structure is measured. After the zero-point evaluation, the sample is again levitated and measurement points are acquired. In addition, an automatic balance calibration is performed after every 15 zero-point measurements. This constant re-calibration and drift compensation eliminates all external influences that build up in conventional weighing schemes such as thermal expansion or tilts.

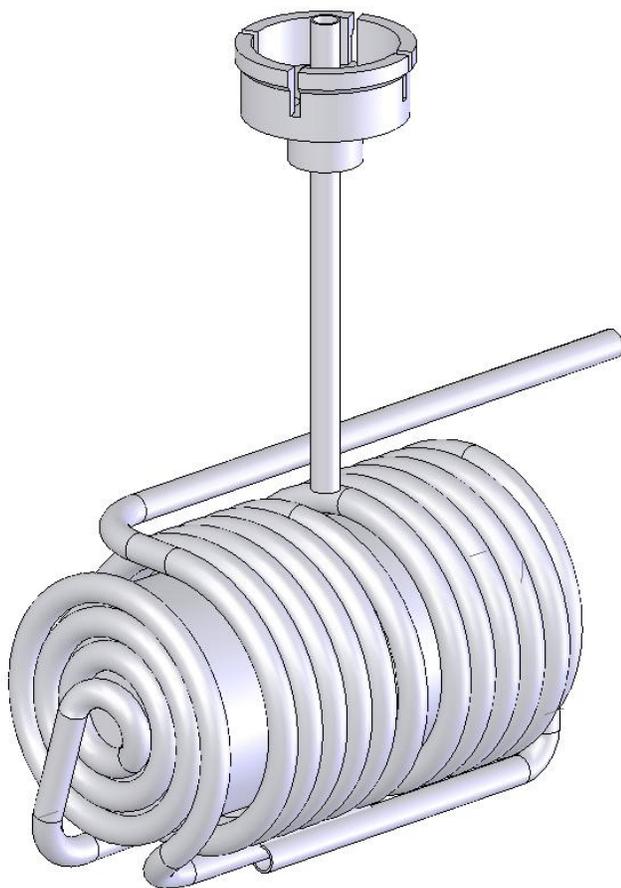


**Figure 1.** Experimental Setup

Our sample holder contains 4 pins on its edges as well as a spring connector in the middle (see Fig. 3) to which the silicon diode is connected. During the zero-point operation, the sample holder is now lowered by 3 mm and the temperature read-out circuit is closed. We can therefore measure the actual sample temperature during the zero-points and interpolate the values during the measurement point operation. Since temperature changes were observed to be small (usually 0.1 - 0.3 K / measurement point), this method is sufficient to reliably obtain the sample's temperature. The only other method we are aware of would be fluorescence temperature sensing requiring special sample coatings, laser and frequency analysis which is expensive and resource intensive [23].

The following procedure proved most effective to cool the sample:

- (i) Evacuate chamber until a vacuum level of  $10^{-5}$  mbar is reached. Heating belts

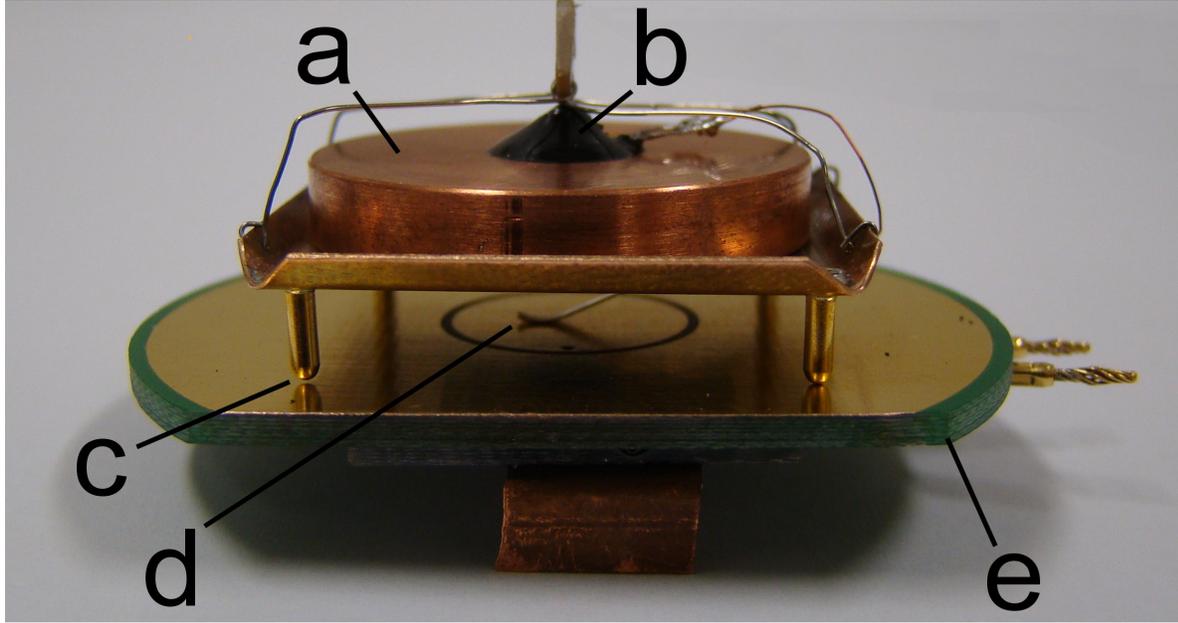


**Figure 2.** Cooling Tube

outside the vacuum chamber were also used to facilitate good outgassing inside the chamber.

- (ii) Venting of chamber with helium gas up to a pressure of 1 mbar to provide thermal contact between cooling tube and sample.
- (iii) Initiate flow of liquid nitrogen/helium through copper tubes.
- (iv) Wait until sample temperature is close to tube temperature (usually 30 minutes).
- (v) Evacuate chamber to a vacuum level better than  $10^{-5}$  mbar (reached within minutes).
- (vi) Stop liquid nitrogen/helium flow and allow the sample to slowly warm up.

Without the helium gas contact, the sample cools down to only  $-100^{\circ}\text{C}$ . We chose a pressure of 1 mbar since we wanted to have a pressure as low as possible and this was close to the minimum pressure required for heat conductivity based on the mean free path of helium and the dimensions of our sample/tube configuration (helium mean free path is 3.5 mm at 1 mbar and 77 K).



**Figure 3.** Details of the Sample Holder with Copper Sample (a ... Sample, b ... Silicon Diode, c ... Pin, d ... Spring Connector, e ... Connecting Plate)

## 2.1. Systematic Effects

*2.1.1. Buoyancy Contribution* The volume of the suspension magnet, sample holder and sample is about  $2 \text{ cm}^3$ . It therefore requires a pressure of about  $10^{-2}$  mbar of air or  $10^{-1}$  mbar of helium in order to create a buoyancy effect larger than our  $1 \mu\text{g}$  balance resolution (confirmed experimentally). Since the measurements are performed at a minimum pressure of  $10^{-5}$  mbar, we can safely neglect buoyancy effects in our measurements.

*2.1.2. Meissner Effect* When a superconductor is cooled below its transition point, it expels all external magnetic fields from its interior. Our experiment was not shielded against the Earth's magnetic field so that this so-called Meissner effect may create disturbing forces on our setup. At the latitude of our laboratory ( $47.97^\circ$ ), the Earth's magnetic field strength is about  $B_{Earth} = 48 \mu\text{T}$ . Given our sample dimensions, a maximum shielding current can be estimate as  $I = 1 \text{ A}$  assuming a single loop coil that has to counterbalance the Earth's magnetic field and we can compute a worst-case magnetic moment of our superconducting sample as  $\mu_{SC} \sim 4.5 \times 10^{-4} \text{ J/T}$ . Since the Earth's magnetic field at our sample's location can be considered homogenous, there is no force directly acting on the sample's magnetic moment. However, there is a dipole-dipole interaction with the Earth's magnetic moment creating a torque on the sample. The maximum force due to this torque can be expressed as

$$F_{torque} = \frac{3\mu_0}{2\pi} \cdot \frac{\mu_{SC} \cdot \mu_{Earth}}{r^4} \quad (1)$$

With  $\mu_{Earth} \sim 10^{22} J/T$  and  $r = 6.4 \times 10^6$ , we get a maximum disturbance force of  $F_{torque} \sim 10^{-14} N$  which is about 6 orders of magnitude below our balance's resolution.

*2.1.3. Sample Temperature* The temperature of the sample is measured on the surface with the silicon diode. If there would be a temperature difference with respect to the sample's core, maybe only part of the sample would be in a superconducting state. The homogeneity of the temperature inside the sample may be computed by considering

- (i) thermal conductivity of the sample given by

$$\dot{q}_{conductivity} = \frac{\lambda}{l} A \Delta T \quad (2)$$

where  $\dot{q}$  is the rate of heat flow,  $\lambda$  the sample's thermal conductivity,  $l$  the length of the sample,  $A$  the area and  $\Delta T$  the temperature difference within the sample,

- (ii) free convective heat transfer on the sample-He gas interface, which is given by

$$\dot{q}_{convection} = h A \Delta T \quad (3)$$

where  $h$  is the free convective heat transfer coefficient,

- (iii) and the radiation heat transfer between the sample and the cooling tube, which is given by

$$\dot{q}_{radiation} = \sigma \epsilon A (T_{Tube}^4 - T_{Sample}^4) \quad (4)$$

where  $\sigma$  is the Stefan-Boltzmann constant and  $\epsilon$  the emissivity of the sample.

During cooling, we can neglect the radiation term. Here we get a homogeneous temperature distribution inside the sample if  $\dot{q}_{conductivity} > \dot{q}_{convection}$  which translates into  $\frac{\lambda}{l} > h$ . For the high- $T_c$  superconductors, the lowest heat conductivity at 77 K is about  $\lambda_{high-T_c} \sim 10 W/mK$  [24] and for niobium at 9 K  $\lambda_{high-T_c} \sim 22 W/mK$  [25]. With  $l \sim 0.01 m$  we get  $\frac{\lambda}{l} \sim 1000 W/m^2K$ . According to [26], the free convective heat transfer coefficient for low temperature helium gas is  $h < 100 W/m^2K$ . So we see that the heat transfer inside the sample is always at least an order of magnitude more efficient than the cooling from convection and therefore the sample's temperature on the surface as measured by the diode will be very close to the overall sample temperature. We performed a numerical simulation using ANSYS fitting  $h$  to our observed cooling times. Our result was that  $h \sim 7 W/m^2K$  and the maximum temperature variation inside the sample was on the order of 0.01 K which is much less than the tolerance of  $\pm 0.5 K$  of the temperature sensor.

A similar analysis can be done for the warming up phase where we have now to neglect the convection term due to the vacuum environment and include the radiation heat transfer. In order to get a homogenous temperature distribution inside the sample we need  $\dot{q}_{conductivity} > \dot{q}_{radiation}$ . This can be expressed as

$$\frac{\dot{q}_{radiation}}{\dot{q}_{conductivity}} = \frac{\sigma \epsilon l (T_{Tube}^4 - T_{Sample}^4)}{\lambda \Delta T} \cong \frac{0.1}{\Delta T} < 1 \quad (5)$$

where we assumed an emissivity of  $\epsilon = 1$  and a worst case ( $T_{Tube}^4 - T_{Sample}^4$ ) based on the data in Fig. 4. We see that even under these conditions, the maximum allowed temperature difference within the sample is  $\Delta T = 0.1 K$  and hence the temperature distribution within the sample is very homogeneous.

*2.1.4. Summary* All systematic effects analyzed are well below the balance and temperature sensor resolution. Therefore, our measurement resolution is only limited by the balance's measurement reproducibility of  $\pm 4 \mu\text{g}$  and the long-term drift of  $< 1 \mu\text{g/h}$  only as well as the temperature tolerance of  $\pm 0.5 K$  from the Lakeshore silicon diode.

### 3. Experimental Results

#### 3.1. Test with Copper Sample and $\text{LN}_2$

The copper sample (machinable copper alloy according to standard DIN 1751) was tested in order to investigate the sample's weight stability over an extended temperature range and measurement time using liquid nitrogen cooling. The sample and tube temperature as well as the pressure inside the vacuum chamber are shown in Fig. 4 respectively. Both sample and tube do not fully acquire the  $\text{LN}_2$  temperature but stabilize a few degrees above at around 84 K. The cold tube acts as a cryopump improving the vacuum down to low  $10^{-6}$  mbar compared to the  $10^{-5}$  mbar level produced by our turbopump. The tube temperature rises more quickly compared to the sample temperature since the sample is levitating in vacuum whereas the tube is mechanically connected to the chamber walls. As the temperature is rising, the pressure is rising too as the cryopump effect decreases.

Fig. 5 shows the mass change and the chamber pressure measured by the balance as a function of the sample's temperature. Up to about 140 K, the mass reading was stable. Then the balance measured a mass increases of about  $3 \times 10^{-5}$  g up to a temperature of 170 K and sharply falls down again close to its initial value. This rise in weight is similar in shape to the increase in pressure. We believe that this phenomenon occurs due to the warming up of the cold tube, where trapped condensed gas residuals are released and drop down on the sample holder where they slowly evaporate. If the chamber is evacuated only a few hours before liquid nitrogen flows through the copper tubes, this peak is even higher. Long evacuation (several days) and the heating belts around the chamber helped to reduce the peak down to the value reported in Fig. 5. For the superconductor investigation, this peak is not important as we can focus on the  $< 140$  K regime.

We can now evaluate the possible influence of temperature on gravitational attraction by performing a linear regression analysis. Although the balance in fact performs a force measurement, it uses a fixed value for the gravitational acceleration to output its values in units of mass. Any measured mass change is therefore directly related to a weight change. Performing a linear regression of the mass change over

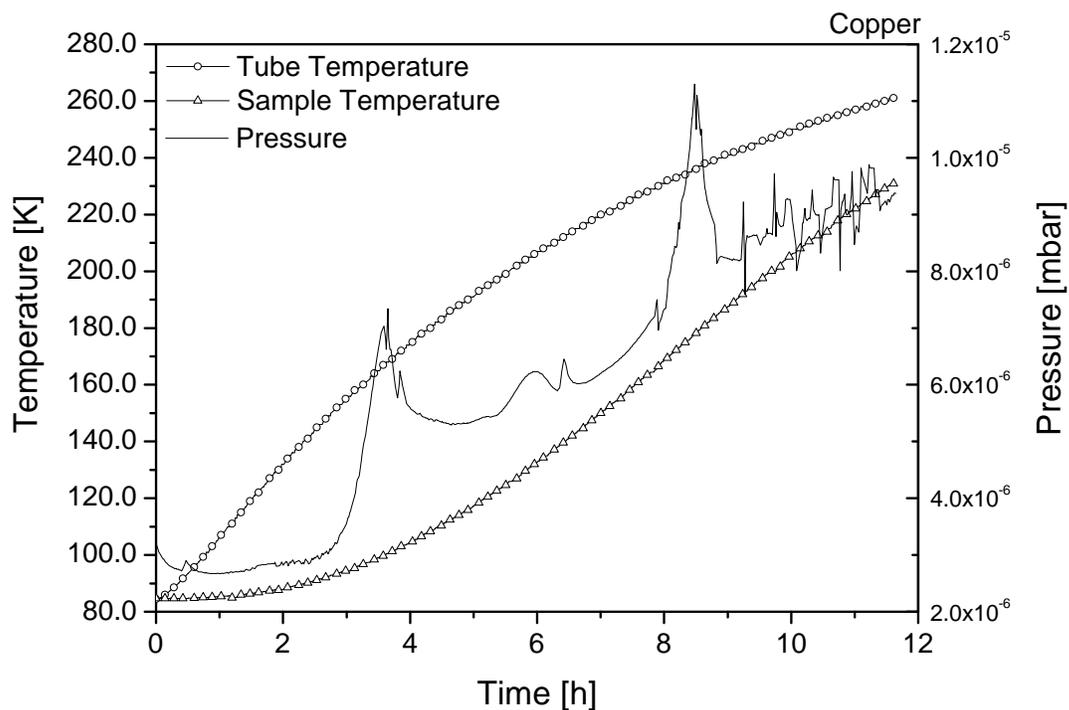


Figure 4. Temperatures and Pressure Evolution over Time for Copper Sample

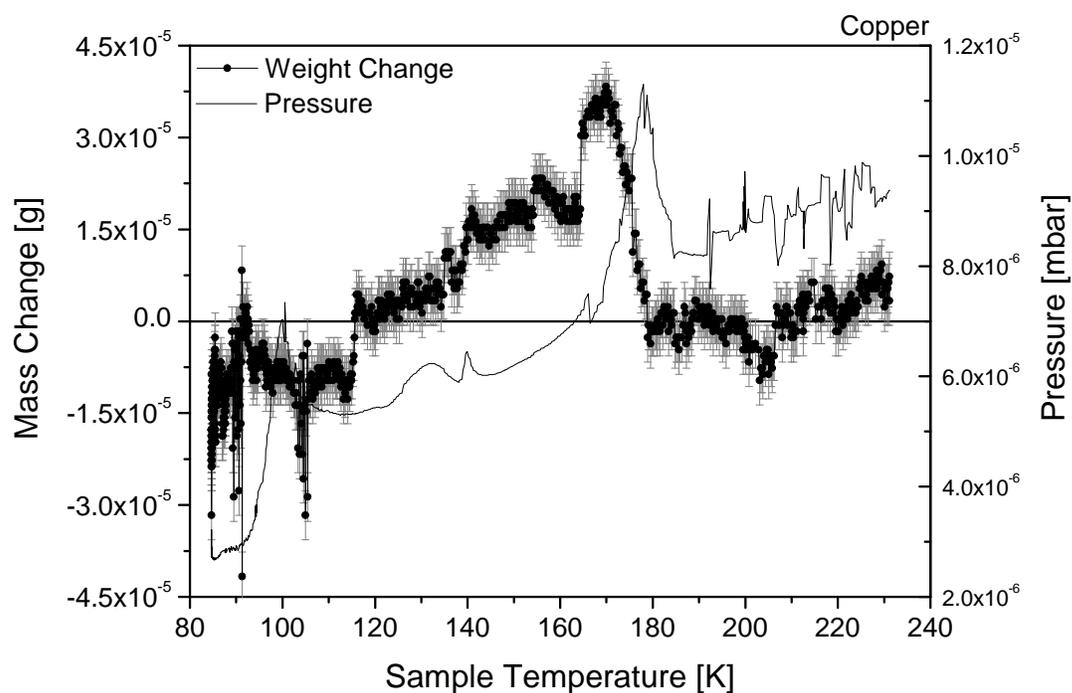


Figure 5. Mass Changes and Pressure over Temperature for Copper Sample

temperature in Fig. 5, we get  $\alpha = 8.5 \pm 0.5 \times 10^{-9} \text{ K}^{-1}$  (neglecting the peak between 140-180 K gives  $\alpha = 6.3 \pm 0.3 \times 10^{-9} \text{ K}^{-1}$ ). Although we subtract drifts using the regular zero-point measurements, the calibration performed every 15 zero points introduces another drift which is specified as  $< 1 \mu\text{g/h}$ . Over the nearly 12 hours of measurement time, this results in a maximum error for the temperature coefficient of  $< 5.1 \times 10^{-9} \text{ K}^{-1}$ , which is an order of magnitude higher than the error bar of the linear regression analysis. Using this upper bound, we find  $\alpha = 8.5 \pm 5.1 \times 10^{-9} \text{ K}^{-1}$  for copper, which is close to three orders of magnitude below the value reported by Dmitriev et al [4, 5] (measured at a higher temperature range of 300-350 K). Given the error bars, we conclude that no change in weight of our copper sample was observable within our resolution between a temperature range of 84-230 K. A similar analysis was done for the superconductor samples but with less accuracy which is summarized in Table 1.

### 3.2. Test with High- $T_c$ Superconductors and $\text{LN}_2$

We used BSCCO (2223) and YBCO high- $T_c$  superconductor samples produced by Colorado Superconductors. Their critical temperature is about 108 K and 92 K respectively according to literature and manufacturer specification. We verified their superconducting properties by cooling them with liquid nitrogen and observing the Meissner effect using a permanent magnet before mounting them in our balance setup. The mass change versus sample temperature for both samples is shown in Fig. 6 and Fig. 7 together with an indication of their respective critical temperatures. No anomalous weight (mass) changes can be seen. We can use this data to put an upper limit on a possible WEP violation during the phase transition from the superconducting to the normal conducting regime. As the balance measures the sum of the vertical contribution of the gravitational attraction and the centrifugal force from the Earth's rotation,

$$F = m_g g_0 - m_i \omega^2 r \cos^2 \phi \quad (6)$$

where  $\omega$ ,  $r$  and  $\phi$  are the Earth's angular velocity, radius and latitude of our laboratory, we have to split our analysis into two cases:

- (i) WEP violation due to variation of  $m_g$  with  $m_i$  constant

Since the balance uses a fixed value for the gravitational acceleration  $g_0$ , we can write the Eötvös parameter as

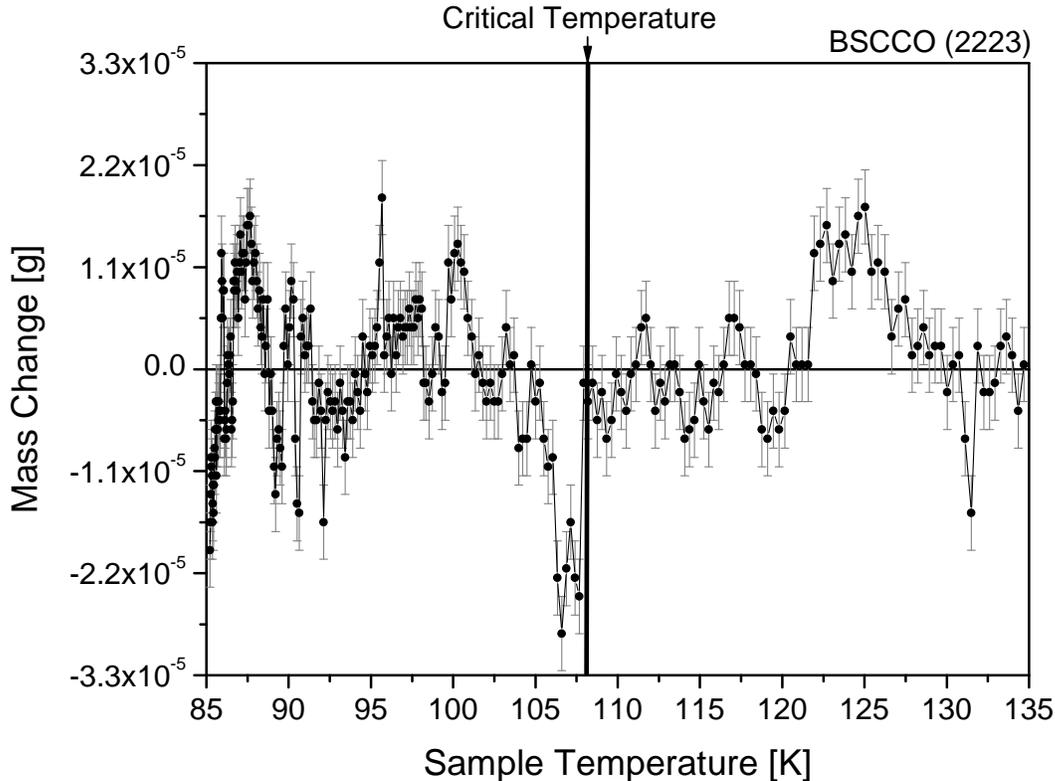
$$\eta_g = \frac{\Delta(F_{SC} - F_{NC})}{F_0} = \frac{\Delta(m_{SC} - m_{NC})}{m_0} \quad (7)$$

evaluating the difference between the superconducting (SC) and normal-conducting (NC) regime.

- (ii) WEP violation due to variation of  $m_i$  with  $m_g$  constant

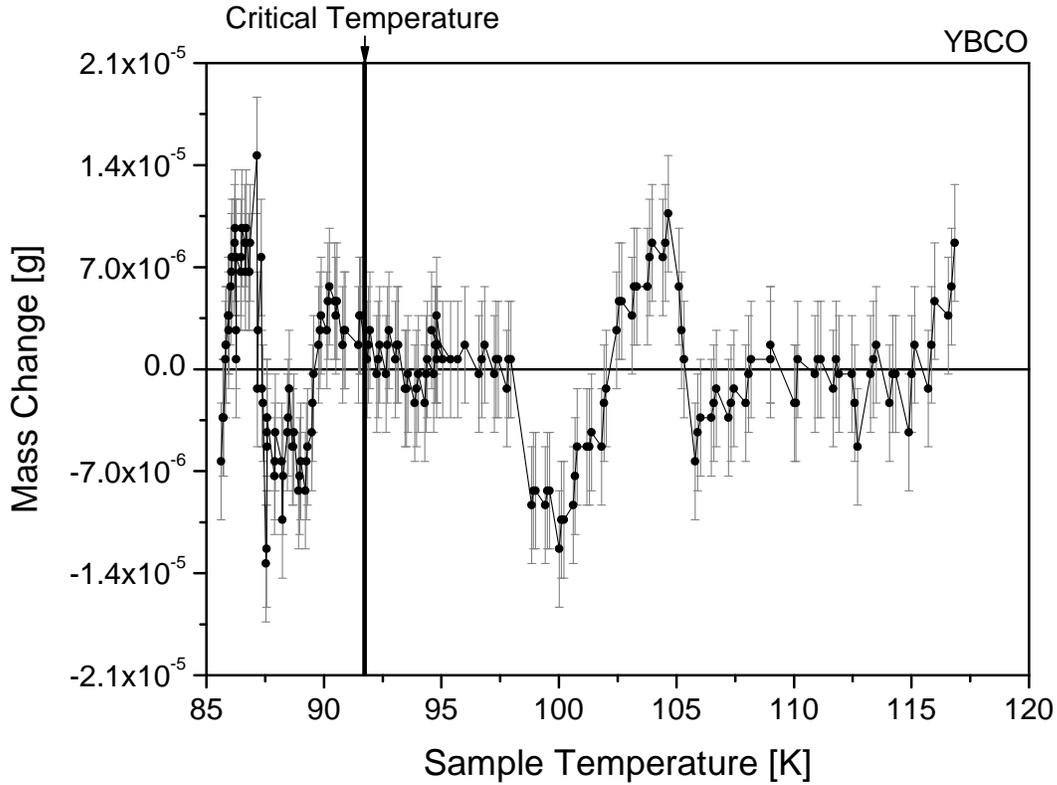
Here, the Eötvös parameter is less sensitive due to the weaker contribution from the inertial mass component and is given by

$$\eta_i = \eta_g \cdot \frac{g_0}{\omega^2 r \cos^2 \phi} = \eta_g \cdot 649.6 \quad (8)$$



**Figure 6.** Mass Changes along Critical Temperature for BSCCO (2223) Sample

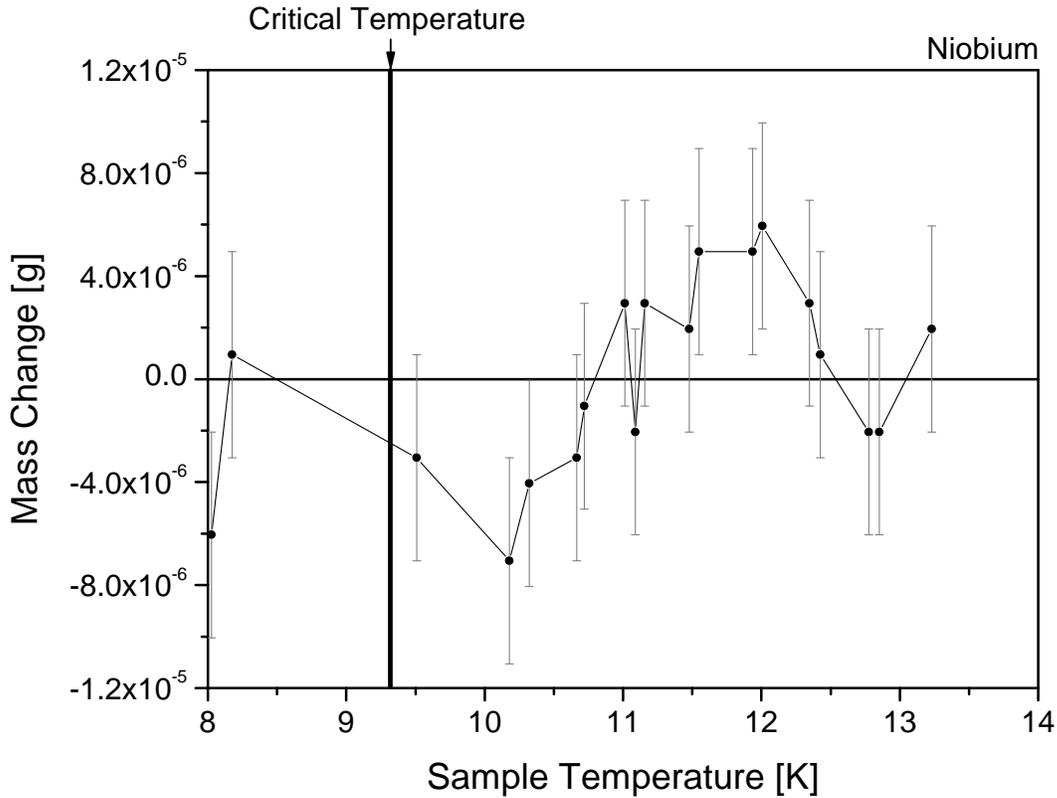
using our laboratory conditions ( $r = 6.37 \times 10^6$  m,  $\phi = 47.97^\circ$ ). In our analysis, we left a two Kelvin margin above and below the critical temperature to account for a broad critical temperature interval common to high- $T_c$  superconductors. The Eötvös parameters for a gravitational mass variation for both samples are  $\eta_{g,BSCCO} = 0.2 \pm 1 \times 10^{-6}$  and  $\eta_{g,YBCO} = 0.6 \pm 7.3 \times 10^{-7}$  and for an inertial mass variation they are given by  $\eta_{i,BSCCO} = 1.3 \pm 6.5 \times 10^{-4}$  and  $\eta_{i,YBCO} = 0.4 \pm 4.7 \times 10^{-4}$  which is the upper limit that we can give. The  $\eta_g$  values are slightly above the resolution obtained from a recent atomic-level WEP test [22] ( $\eta_{Rb} = 0.4 \pm 1.2 \times 10^{-7}$ ). Our values represent the first bounds for high- $T_c$  superconductors. We can rule out reported weight (mass) anomalies from Reiss [8] by some 4 orders of magnitude confirming our previous assessment [9]. Most likely, the buoyancy effects of the nitrogen gas were underestimated which are absent in our measurement. Although Reiss also used YBCO, another type of BSCCO (2223 instead of 2212) was used in our assessment.



**Figure 7.** Mass Changes along Critical Temperature for YBCO Sample

### 3.3. Test with Low- $T_c$ Superconductor and LHe

We tested niobium as a classical low- $T_c$  superconductor due to its relatively high critical temperature of 9.2 K as well as we specifically wanted to link our results to assess the Tate Cooper-pair mass anomaly measurement which was also done with niobium. Cooling down to liquid helium temperatures was much harder compared to the liquid nitrogen cooling as the sample only reached about 25 K with a usual helium gas pressure of 0.1 bar from the dewar. Only after increasing the gas pressure to 0.3-0.5 bar, the sample gradually reached 7 K which was the lowest temperature we could obtain. The temperature stability was just enough to cool down in the helium gas atmosphere, evacuate the chamber, perform a zero-point and afterwards two measurement points below the niobium's critical temperature as shown in Fig. 8. We were still more than one Kelvin away from  $T_c$  which is enough confidence that the sample was actually superconducting. Also here, we observed no anomalous weight (mass) changes up to a temperature of about 13 K. The Eötvös parameters are  $\eta_{g,Nb} = 2.0 \pm 4.0 \times 10^{-7}$  and  $\eta_{i,Nb} = 1.3 \pm 2.6 \times 10^{-4}$ . Our resolution is at least some 4 orders of magnitude too coarse to observe a Cooper-pair mass change on the level as reported by Tate et al [10, 11] (an Eötvös parameter of  $\eta_{Nb} \cong 7 \times 10^{-11}$  is predicted). This value could be tested using a



**Figure 8.** Mass Changes along Critical Temperature for Niobium Sample

cryogenic torsion balance as described by Newman et al [27] that has a design resolution of  $\eta_{Nb} \cong 10^{-14}$ . We believe that this could be an interesting experiment since there is an actual measurement (Cooper-pair excess mass) that warrants confirmation after some 20 years after publication.

The error bar on the mass change over temperature is two orders of magnitude higher compared to our copper and high- $T_c$  superconductor samples due to the much shorter temperature range of 8-13 K. The linear regression analysis yields  $\alpha = 1.2 \pm 0.6 \times 10^{-6} \text{ K}^{-1}$ . Due to the shorter time available for cooling and performing the balance calibration compared to our  $\text{LN}_2$  measurements, it could well be that the helium pressure close to the sample was higher (e.g. due to outgassing from the MLI) than at the pressure gauge position close to the vacuum chamber walls. This could have introduced a larger than expected buoyancy effect and therefore a drift in our measurement. We therefore consider this  $2\sigma$  effect insignificant. As for the evaluation of temperature variation on the gravitational attraction, testing of the copper sample along a wide temperature interval provided the most accurate assessment.

#### 4. Conclusion

An experiment was set up to measure the weight and temperature of samples in vacuum at high precision over a large temperature range using a magnetic suspension balance and a Sartorius electronic mass comparator. A long term test with copper put a new limit on possible variations of weight on temperature of  $\alpha < 2 \times 10^{-8} \text{ K}^{-1}$  with a  $3\sigma$  confidence. This extends previous room-temperature measurements [2] to the low temperature regime as well as rules out various claims from the literature as summarized in Table 2. Assuming that there is no measurable influence of temperature on the intrinsic mass then our experiment also provides bounds on a possible temperature influence of the gravitational constant. By observing the weight during the normal and superconducting regime, we could establish first limits of  $\eta < 2 \times 10^{-3}$  with a  $3\sigma$  confidence on possible violations of the weak equivalence principle (WEP) on both high- $T_c$  (BSCCO and YBCO) and low- $T_c$  (Nb) superconductors which is an order of magnitude better than the only other assessment performed so far which tested the strong equivalence principle of superconductors [16].

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