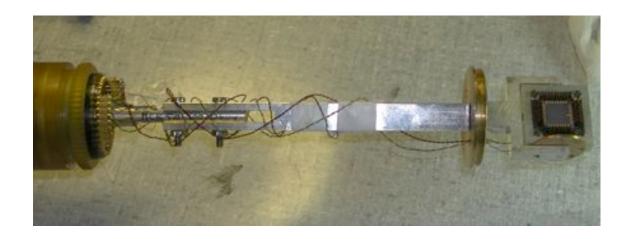
Institut für Physik Universität Basel

Dilution Refrigerator: Setup and Sample Holder Design



A Semester Project by Florian Dettwiler

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Supervised by Prof. Dr. Dominik Zumbühl

Abstract

One way to reach temperatures of a few millikelvin (mK) is the usage of a ${}^{3}\text{He}/{}^{4}\text{He}$ dilution refrigerator (DR). The newly established *Quantum Coherence Group* at the University of Basel uses such equipment for various measurements of their samples. During this semester project a new MCK 50-100 TOF DR was put into operation. This included the design and assembly of a sample holder unit which can be attached to the DR in order to cool down chips containing quantum dots or similar nanostructures.

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1 Introduction

Humankind has always been interested in achieving low temperatures - especially on a stable level - mostly to cool groceries and therefore prolong their storage lifetime. The first technical refrigerator was invented in the middle of the 18th century. Commercially available fridges were first constructed in the 1830s and technical improvements are made ever since, however most of them are still based on the same principle of a vapor compression cycle, which is more or less the inversion of a heat engine.

In science low temperatures are also of great importance and there is a multitude of applications, not only in physics but also in biology and medicine. In a scientific context the term low often has a different meaning. If a physicist is talking about cooling something down, he most probably refers to temperatures below 100 K. The whole field of physics that deals with experimental methods and material behavior at such low temperatures is called cryogenics. Permanent gases like nitrogen, oxygen, helium, neon and hydrogen have a boiling point below -180°C or 93.15 K. And exactly these gases or better their liquefaction play an important role in achieving such low temperatures. Just by dipping something into a cryogenic liquid, it already cools down to the temperature of the liquid, which is its boiling point under normal conditions. Sir James Dewar, the inventor of the dewar flask and the first to liquefy hydrogen, and Heike Kamerlingh-Onnes, the first to liquefy helium, are two of the main contributors to the progress in cryogenics. By a coincidence Kamerlingh-Onnes found also the superconductivity (of mercury, but for the first time ever) just by performing some electronic measurements in liquid $helium^{(1)}$.

What is temperature and why do physicists need low temperatures? Temperature is an intensive thermodynamic state function of a system which is closely related to energy. The temperature is basically the average of the kinetic energy of a system. This means it is the average over all microscopic motions, which include translational, rotational and vibrational energies of gas molecules but also lattice vibrations in solids. At absolute zero at 0 K (-273.17°C), which can not be achieved in practice, only the quantum mechanical zero-point energy is left, all other motions are frozen out. This concept of microscopic motion, but also the much simpler ideal gas law and considerations about the maximum efficiency of a heat engine led to this absolute temperature scale of Kelvin. In the case of the Quantum Coherence Lab of Dominik Zumbühl at the University of Basel low

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temperatures are necessary to reduce noise as much as possible. As it is the goal to measure for example single electrons tunneling between quantum dots in semi-conducting structures and related experiments, no thermal motion of bulk atoms should disturb these measurements. For this purpose the group uses dilution refrigerators, which are able to achieve temperatures in the low millikelvin range. There are different methods, that enable scientists to cool down small samples below 300 mK. Dilution refrigerators (DR) which are discussed in this report have the advantage of a quite stable operation and a relatively easy way of controlling the temperature. The principle was first described by Heinz London in $1951^{(2)}$. Since then, a lot of progress has been made on this field. An important contributor is Giorgo Frossati⁽³⁾. His research led to many improvements in the construction of DRs.

² He/⁴He Dilution Refrigerator

2.1 Cryogenic Liquids

For the understanding of the function of the dilution refrigerator, it is important to know some properties of cryogenic liquids. While liquid nitrogen (LN_2) only plays a rather secondary role as a pre-cooling liquid to bring the whole setup to 77 K it is much more important to know and understand the behavior of liquid helium, especially how its isotopes behave upon mixing. First of all the two isotopes are discussed separately in the next two sections and the following paragraph is about their mixture.

In 1908 H. Kamerlingh-Onnes achieved the liquefaction of helium for the first time. Since then its importance for low temperature experiments grew until nowadays it is the most important cryogenic liquid for all experiments performed at temperatures below 10 K. An important application can also be found in medical diagnostics, e.g. the cooling of the coils used to achieve large magnetic fields for magnetic resonance imaging (MRI).

2.1.1 ⁴He

The bosonic atom 4He is the most stable helium isotope and the only one which occurs naturally (on earth and its atmosphere) in a reasonable and technically usable amount. Even though it is the second most common element in the universe, helium is quite rare on earth and is obtained from helium-rich natural-gas sources. As the number 4 in the name already reveals, the atomic nucleus consists of two protons and two neutrons. The importance as a cryogenic liquid is attributed to the low boiling point of 4.21 K and the fact, that it is impossible to have it in a solid phase, at least not under its own vapor pressure, even if the temperature approaches zero. The reason for this can be found in the weak van der Waals forces which act as attractive forces. The closed s shell (helium is a noble gas) and the low polarizability are the reasons for this. In addition, there is a relatively large zero-point energy⁽⁴⁾

$$E_0 = \frac{h^2}{8ma^2} \tag{2.1}$$

even at $T \to 0$. In this formula m denotes the mass of the atom and a its radius. The quantum mechanical energy E_0 can also be seen as a zero-point vibrational amplitude. This amplitude is only slightly smaller (factor 3) than the actual separation of the atoms in a theoretical solid phase. Therefore the freezing point is very low and He cannot be solidified (only under high pressure), see phase diagram (figure 2.1). The large E_0 arises from the small mass and radius and therefore liquid helium (LHe) is also called a quantum liquid.

An additional phase transition occurs, if the temperature is further decreased: At

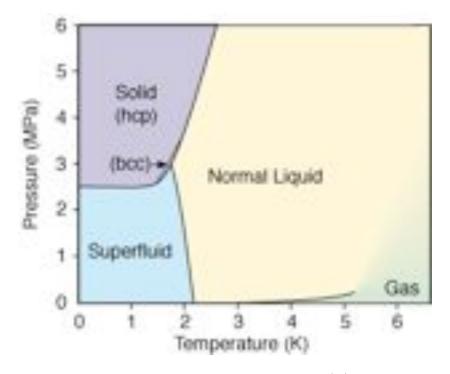


Figure 2.1: phase diagram of ${}^{4}He^{(12)}$

the λ -point $T_{\lambda}=2.17K$ the liquid begins to form a superfluid phase 4HeII (figure 2.1). In this phase the viscosity and most important the entropy both vanish. The specific heat and the thermal conductivity increase at the transition temperature. Superfluid 4HeII does not boil anymore and only atoms at the surface can go into vapor phase.

$2.1.2^{-3}$ He

 ${}^{3}He$ is obtained as a byproduct of tritium manufacture in nuclear reactors. A lot of properties of liquid ${}^{4}He$ seem to be similar for the other isotope ${}^{3}He$, although the

origin lies somewhere else. ${}^{3}He$ also has two protons and two electrons but only one neutron. Therefore it is a fermionic atom that follows Fermi-Dirac statistics. This becomes important when the entropy is considered. From figure 2.2 one can clearly see, that the transition to a superfluid phase occurs at much lower temperatures than ${}^{4}He$. Like in a superconductor fermions have to build bosonic pairs to form a superfluid phase, that's why the temperature for this phase transition is so low. The boiling point is also a bit lower at 3.19 K.

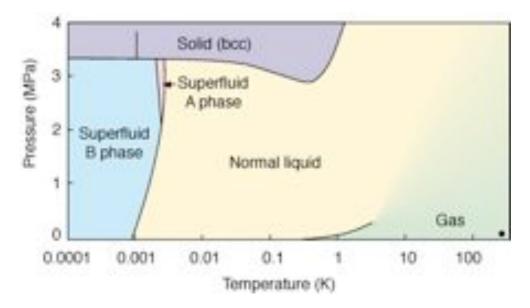


Figure 2.2: phase diagram of ${}^{3}He$, notice the logarithmic temperature scale⁽¹²⁾

2.1.3 3 He/ 4 He -Mixture

As the phase diagram of the mixture (figure 2.3) shows, the miscibility of the two isotopes is very limited. Between 2.87 K and 0.867 K a superfluid can only exist under a maximal amount of ${}^{3}He$ which increases with decreasing temperature. But below 0.867 K an interesting effect leads to a split up into two phases. The superfluid phase of ${}^{4}He$ contains about 6 % of the lighter isotope and is therefore called diluted phase (left side of figure 2.3). It can be compared to a saturated solution. The ${}^{3}He$ almost behaves like a perfect gas in a mechanical vacuum (basically no mutual friction between the atoms) and therefore is sensitive to pressure. On the other hand (and right side of figure 2.3) a concentrated phase of almost pure ${}^{3}He$ forms. The composition (mole fractions) within the two phases is defined

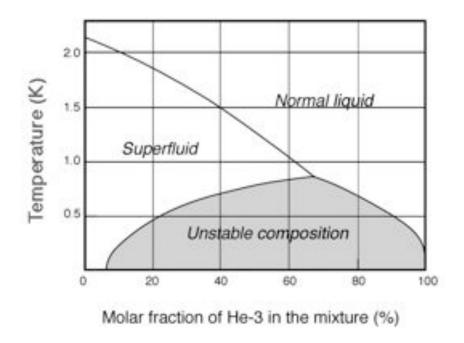


Figure 2.3: phase diagram for the mixture of ${}^{3}He$ and ${}^{4}He^{(13)}$

by temperature and can be calculated by the following formulas⁽⁴⁾:

$$x_3 = 0.0648(1 + 8.4T^2 + 9.4T^3) (2.2)$$

$$x_4 = 0.85T^{2/3}exp(-\frac{0.56}{T}) (2.3)$$

As an effect of different densities the concentrated phase floats on top of the diluted phase. Additionally the ${}^{3}He$ atoms in the concentrated phase have a lower enthalpy than in the diluted phase. But if a ${}^{3}He$ crosses the phase boundary to the diluted phase, its enthalpy must increase. This energy can be taken from a surrounding system and is the basic cooling principle in dilution refrigerators, which will be discussed in the next sections.

2.2 Setup

The diluted and concentrated phase normally are in equilibrium, which means that there has to be a constant amount of ${}^{3}He$ (for constant T) in the diluted phase. But ${}^{3}He$ atoms can be forced to cross the phase boundary by pumping the lighter isotope out of the superfluid. For the design of a dilution refrigerator (figure 2.4),

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this means that the sample should be located at the place where the cooling occurs and this is the phase boundary. Therefore it is called mixing chamber (MC). Then there is a chamber where liquid ${}^{3}He$ is boiled and pumped out which is denoted as still (from distiller). The heat for boiling is supplied by a conventional heater. For a continuous operation, vapor phase ${}^{3}He$ is pumped back and condensed again. For this purpose it is precooled to 1 K at the 1K-pot (missing in figure 2.4) and exchanges thermal energy over several heat exchangers to bring it as closely as possible to the temperature of the mixture. In order to condense it, one ore more flow impedances are introduced.

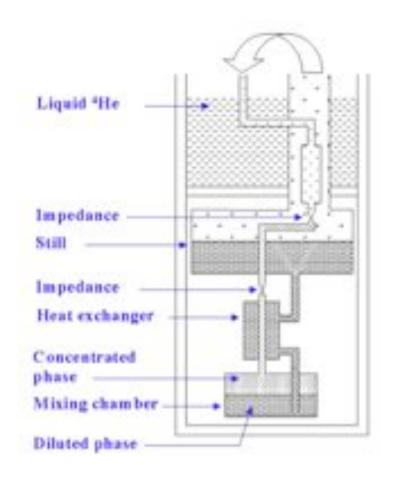


Figure 2.4: simplified dilution fridge⁽¹⁴⁾

The dilution refrigerator used in this work is a MCK 50-100 TOF by Leiden $Cryogenics^{(8)}$. It consists of three major parts (figure 2.5):

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- A big dewar flask: The first cooling step is to bring everything to 4.2 K. This is provided by a LHe bath in a dewar. The inner helium bath is surrounded by a vacuum chamber called outer vacuum chamber (OVC) and a reservoir of LN_2 .
- The dilution unit itself: The DR is actually a insert to the dewar flask. All the parts discussed before and everything visible on figure 2.4 is inside this unit.
- A gas handling system (GHS): This system includes the controls of all the valves, the vacuum pumps needed, and the helium storage dumps. The fridge, which is connected to the GHS by numerous cables and tubes, is actually controlled by this unit.

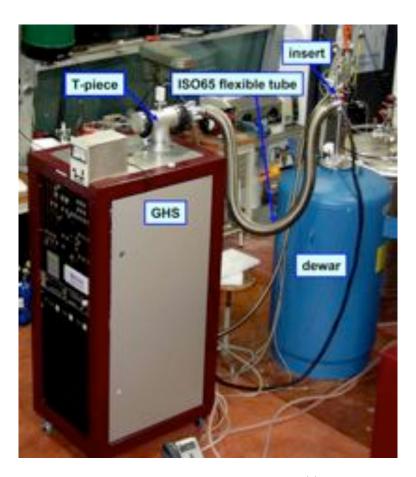


Figure 2.5: $MCK \ 50-100 \ TOF^{(7)}$

2.3 Cooling Power

The cooling power \dot{Q} at the mixing chamber is strongly connected with the number of ${}^{3}He$ atoms crossing the phase boundary or rather the rate $\dot{n_{3}}$ of the crossing. And every atom cools the system by the difference in enthalpy or the enthalpy of mixing ΔH :

$$\dot{Q} = \dot{n_3} \Delta H \tag{2.4}$$

More detailed calculations can be found in Pobell⁽⁴⁾ and in Frossati's review⁽³⁾. Both derived after some slightly different considerations that:

$$\dot{Q} \propto T^2 \tag{2.5}$$

$$Pobell: \dot{Q} = 84\dot{n}_3 T^2 \tag{2.6}$$

$$Frossati: \dot{Q} = 82\dot{n}_3 T^2 \tag{2.7}$$

The main statement can be found in formula (2.5): The cooling power at 10 mK for example, is 100 times smaller than at 100 mK.

The base temperature which can be reached with a certain fridge is the equilibrium at the point, where all the heat leaks can be compensated by the provided cooling power. An example to illustrate this statement: If the heating power in the still is increased in order to get a larger i_3 , the cooling power increases. On the other hand this produces a heat load onto the MC. Therefore an ideal heating power for the heater in the still exists for every fridge.

3 Cold Finger Design

As already mentioned, the DR was fabricated by *Leiden Cryogenics* and is a standard model. In order to use it for experiments, e.g. to cool down and measure quantum dots, the sample somehow has to be inserted in a way, that is probably different in every research group. For this purpose a cold finger elongation with a socket holder had to be constructed. The design is very modular and consists of four main parts (figure 3.1):

- 1: an elongation from the cold finger
- 2: a thermal shield consisting of a cap (2a) and a canister (2b)
- 3: a socket holder
- 4: a socket (not numbered in figure 3.1)

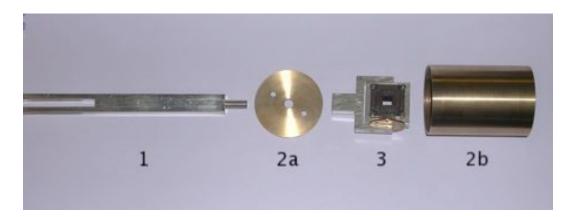


Figure 3.1: disassembled cold finger

In the following paragraphs these parts are discussed in the order of importance of the design process which is exactly opposite to the list above. All the parts except for the socket were manufactured by the in-house workshop of the Institute of Physics. The actual construction blueprints can be found in chapter 8 Appendix.

3.1 Socket

The starting point was a commercially available 32-pin socket manufactured by $Plastronics^{(9)}$ with a slight modification. It differs from the ordinary one in the catalogue by the pin material, which has to be non-magnetic because of experiments with magnetic fields of some tesla. Therefore the pins are of pure copper-berilium

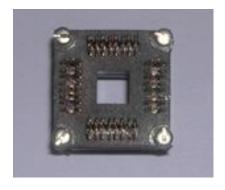


Figure 3.2: top view of the socket after milling



Figure 3.3: side view of the socket after milling

without the gold over nickel contact plating. In the hole in the middle a LED could be installed for experiments with photons. Because of limited space available in the DR insert (IVC d=5 cm) the socket was reduced in its dimension. On each side 1.4 mm of the 60% glass-filled polyphenylene sulfid (PPS⁽¹⁰⁾) was milled and a final width of around 18 mm was achieved. To not destroy the special (and expensive) pins they have been taken out and were reinserted after the milling.

3.2 Socket Holder

The main idea of the socket holder was the possibility to adjust the angle of the sample. As the quantum dot samples are basically heterostructures producing a 2-dimensional electron gas (2DEG) this becomes important for experiments in magnetic fields. Therefore the basic design is a two armed fork which is holding the framed socket. The frame is attached at one axis and can be turned more or less freely. To adjust the angle, a thin plate was mounted between the fork and the frame, which allows to fix certain angles (45° and 90° up to now). Figures 3.4 and 3.5 show a turn by 90°.

For the fork and the frame ultra-pure silver was used, mainly because silver is a good thermal conductor even at low temperature. The thermal conductivity is closely related to the electrical conductivity, because in the low temperature region all the lattice vibrations freeze out and the only channel of conduction are

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Figure 3.4: socket holder (with socket)



Figure 3.5: socket holder turned by 90°

the electrons. But nonetheless the thermal conductivity becomes very weak as it is proportional to T in the low temperature range. Because the socket is a polymer, it means that it is also a bad thermal conductor. The silver frame around it surely helps to cool it down fast from room temperature to around 1 K. But the remaining part of the sample cooling is mainly done through the copper wires connecting the sample.

The plate to fix the angle is made of brass, which is also non magnetic and not as soft as silver which is important when the thickness of only 1 mm is considered. The socket is being held in the frame by the heads of the screws on one side (figure 3.6) and a drop of *Stycast 1266* on the other side.

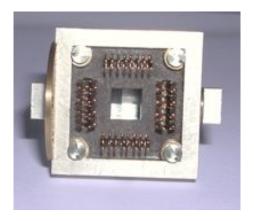


Figure 3.6: socket mounted to the frame

3.3 Thermal Shield

The DR insert is dipped into a bath of liquid helium. But the MC and the sample are in a vacuum chamber called inner vacuum chamber (IVC). The IVC is in direct thermal contact with LHe. Therefore there is a thermal radiation inside the IVC whose energy is according to the Stefan-Boltzman-law⁽⁴⁾

$$Q \propto T^4 \tag{3.1}$$

In order to reduce this heat load a brass shield covers the sample. Its design is a simple can that can be screwed on its cap (figures 3.7 and 3.8). There are two holes for the connecting wires. It is important to close them as well before cool down or at least cover them to prevent direct radiation. A second radiation shield around the whole cold finger sample holder part has been discussed but not yet realized, also because of the very limiting space available. It could reduce the heat load produced by thermal radiation further on.

3.4 Cold Finger Elongation

This part connects the fork and cold finger of the MC by a screw thread. It is also made of ultra-pure silver. One can imagine that a lot of eddy currents could be produced in a magnetic field. Another option discussed was to use a copper tube with a slit, where the slit would reduce eddy currents. But with the silver rod screwed to the fork, a better thermal conductivity and also a good stability can be achieved, although the mass is quite large. It is also easier to connect the rod to the cold finger part by a simple slit and two brass bolts and nuts. As brass has a larger thermal expansion coefficient as copper it contracts more than the silver-plated copper of the cold finger and therefore it tightens upon cooling. The position of the sample can also be adjusted a little, because of the slits. For a setup with a longer elongation part needed, a second rod can be made and added between first rod and fork (see chapter 8 Appendix). The length of the existing part is designed to hold the sample in the lowest position possible in the existing IVC.

3.5 Improvement Ideas

After assembling of the parts for the first time it became clear, that the sample holder can be used as it is. However, improvements could be done on this or on future parts inspired by this design.

The brass shield has a screw thread and can be opened and closed by simply



Figure 3.7: assembled unit with closed shield



Figure 3.8: assembled unit with open shield

3 Cold Finger Design

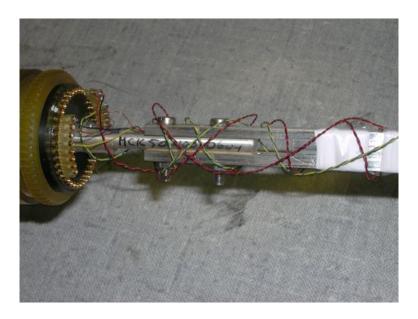


Figure 3.9: joint of cold finger and silver rod

screwing it on the cap. However, the cap is not fixed and can also rotate freely around the central axis. Especially the opening of the shield could become difficult. Therefore a fixation of the cap e.g. with a small (non magnetic) bolt or screw could provide a possible solution.

The second thing, which is not totally satisfying is the attachment of the chip carrier to the silver frame. Inspired by the dipstick design, an attachment to four small lugs in each corner of the frame also with screws is perhaps more stable than the design mentioned in 3.2.

4 Test Runs

4.1 Cool Down Procedure

The exact cool down procedure is fully described in the MCK operation manual⁽⁷⁾. But for a better understanding, the main points will be quickly discussed here:

- Before anything can be cooled down, it is important to check some things: The vacuum of the outer vaccum chamber (OVC) of the dewar should be as low as possible. This is important, only when a new dewar flask is used. Otherwise this vacuum should be stable for years. A leak check of the vacuum parts of the DR is a good idea.
- The first real cooling step is to fill up the inner and outer reservoir with LN_2 and let the system cool down to 77 K for a few hours (or overnight).
- After that, the remaining LN_2 in the inner LHe-reservoir can be pushed into the outer LN_2 -reservoir by applying some pressure with He gas. No air should enter the inner reservoir because of ice-formation. When completely empty, LHe can be transferred to the inner reservoir, which should now be connected to the recovery system. The reservoir should soon reach 4.2 K.
- Before inserting the DR into the dewar, the IVC has to be placed on the conical seal and pre-pumped below 1 mbar. The rest of the system should be pumped according to the step-to-step manual until $p_{IVC} < 10^{-3} mbar$. Then a small amount of exchange gas can be put in. As the name says, this increases the thermal coupling inside the DR in order to let the system equilibrate faster.
- With the help of the crane the DR can then be inserted slowly into the dewar. Calibrated resistors in the still and the mixing chamber monitor the temperature.
- With everything at 4 K the exchange gas can be pumped back and condensing of ${}^{3}He$ can be started. After complete condensation ${}^{4}He$ is condensed. The liquid level inside the Still is monitored by a capacitor and should increase by about 1pF. This value is depending on wether the cold finger unit is

inside or not. Ideal mixture ratio and heating power of the heaters have to be evaluated in numerous tests.

4.2 First Cool Down

The whole first test run was done manually (no automatic mode was used) as described in the manual. No problems occurred during the cool down procedure. Although no temperature curve of the actual cool down was recorded, the values of the RuO₂ resistors were observed regularly and were in the expected range. Figure 4.1 shows the temperature progression over several hours (the DR ran overnight) of the RuO₂ thermometer in the MC. It is clearly visible that the DR was running continuously and stable at around 22 mK. All temperature curves were recorded using *IGOR pro* by *Wave Metrics*⁽¹¹⁾. The interesting part of this cool down was,

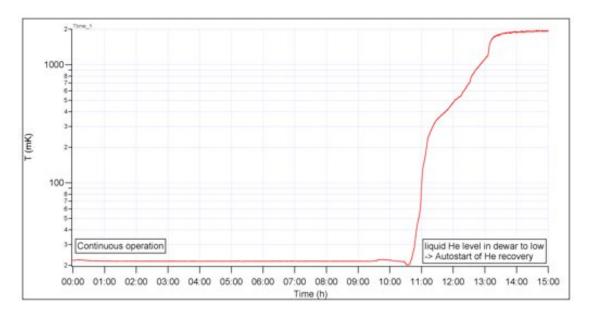


Figure 4.1: temperature recording of the "accidental" single shot during the first test run

that it revealed an error in the blueprints of the dewar. From the measured He level inside the reservoir it has been calculated that the LHe consumption was at around $10 - 13 \frac{l}{day}$ during operation. According to the blueprints of the dewar, the 1K pot should have run out of LHe at around 560 mm. But the 1K pot got dry at a level of around 700 mm and an unexpected *single shot* procedure followed by an automatically started recovery was initiated. A single shot measurement is basically an operation where a fixed reservoir of 3He is pumped through a 4He

phase without being circulated. As no warm liquid is flowing back to the mixing chamber the achievable base temperature is lower than for continuous operation. If the 1K pot of the MCK gets dry (out of 4He) the continuous flow is being interrupted and the systems starts recovery of the mixture. As it begins with pumping out 3He this event is similar to the single shot system described before and therefore also called singe shot measurement. The 3He flow can of course also be interrupted manually, which is normally the case when talking about a single shot. In our accidental single shot a temperature of 20.6 mK was achieved.

4.3 Diagnostic Test Runs

4.3.1 Test Runs with Cold Finger

As the first cool down was successful the fridge was assumed to be working properly. Therefore a further test with the new sample holder unit was the next step on the way to use the DR for real experiments. To attach the sample holder the plastic plug on the mixing chamber was exchanged by the cold finger unit delivered with the fridge. This unit changes the volume of the MC because of the sintered silver part that acts as a heat exchanger. The system was cooled down as described in previous sections. Somehow the expected base temperature could not be reached anymore. The lowest temperature achieved was now at about 130 mK, over 100 mK higher than expected. This result showed up in various cooldowns, therefore several sources of error came into focus. In the useful experimental oriented book by Richardson and Smith⁽⁵⁾ possible reasons are listed:

- Level and composition of the He-mixture (mash)
- Touch
- Plug
- Leak

As the flow meter was still working as before, a plug somewhere in the system could be excluded. A major leak in the vacuum parts was very improbable, because the pressure in the IVC seemed to be OK. Since a leak inside the DR can be a very difficult and time consuming thing to localize, the other two possibilities were favored in the search. Additionally, the only thing changed was the sample holder, therefore assuming a touch in the tight IVC would be a good starting point. But as the fridge already was running, some test with the mash was done first. As mentioned before, the volume of the MC is different with the cold finger attached. This has also an impact on the mixture. Figure 4.2 shows the temperature of

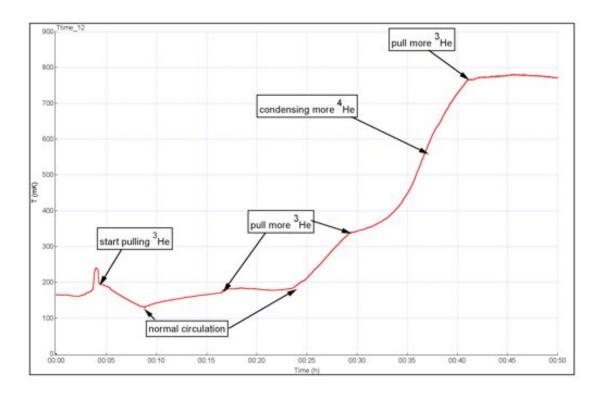


Figure 4.2: temperature recording while changing He-mixture composition

the MC while changing the amount of ${}^{3}He$ and ${}^{4}He$ starting at a point, were all the ${}^{4}He$ was condensed and the capacitor was displaying the Still to be full. The amount of ${}^{3}He$ was reduced twice. During pulling of ${}^{3}He$ the single shot situation led to a small decrease in temperature but during normal circulation the system equilibrated at a higher temperature both times. When the capacitance indicated an empty Still, more ${}^{4}He$ from the dump was condensed resulting in an even higher temperature. Note that this procedure can also be done to do a fine adjustment of the mash and its level in the MC, but in this case, it was done in large steps just to see qualitatively if the phase boundary was in the MC (and therefore of maximal area) and wether the ratio of isotopes was completely wrong or not. Both turned out not to be the limiting factors in order to reach a base temperature of around 20 mK.

Figure 4.3 is an example of a run overnight. It is clearly visible that at some points the system seemed to go to a lower temperature but then somehow equilibrated back to 175 mK. This behavior was a further indicator for a possible touch due to some thermal bending of the silver rod. In order to verify this assumption, the DR was stopped and several sensors were attached to the shield of the sample holder.

These sensors were simple stripes of copper tape, which were isolated from the brass shield by kapton tape and distributed at regular distances around the shield. A breakout box was used to measure the resistance, which should be indefinitely large, if no touch occurs. Although a resistance could be measured at some points while cooling down, it was clear that these touches could also have been caused by the tapes that unglued. It turned out, that the sensors did not adhere strong enough. There were also completely different sensors indicating a touch during a single cool down.

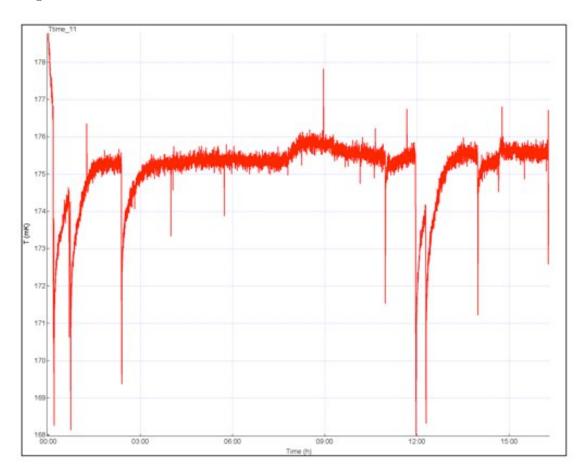


Figure 4.3: temperature recording of the MC overnight

4.3.2 Test Runs without Cold Finger

As the possibility of a touch could neither be excluded nor be proven, the sample holder was removed again, leaving the DR just with the cold finger unit. The following tests should be able to show, that there was a problem somewhere in the DR insert itself, most probably a leak.

In the first test no temperature below 100 mK could be reached, although various isotope ratios have been tested similar to the procedure described before. As the He consumption of the Presicion Cryogenics dewar was too large, an additional heat leak in the dewar was expected. This didn't have any impact on the base temperature of the DR but as LHe is quite expensive, the dewar was exchanged. An additional heat can be produced by ground loops. The only electrical parts connected were a Picowatt AVS-47 resistance bridge and an Anderson-Hagerling capacitance bridge. Therefore the AVS-47 was taken out of the GHS and all the cables have been disconnected. The fisher-plug to measure the resistance of the thermometers was only connected for short periods to measure T_{MC} . As this gave no different result, the resistance was also measured with different equipment leading to the same result. Therefore a ground loop as a heat source could be excluded.

Since there seemed to be a severe leak somewhere inside the fridge which was impossible to be located in our lab, the mixing chamber was being detached and shipped to *Leiden Cryogenics*. The tests in Leiden showed a broken capillary of the ³He impedance (figure 4.4). This problem was also new to the experienced researchers at Leiden. Even after the exchange of the broken capillary the lowest temperature achieved was 75 mK, which is better than the 100 mK in the test runs previously mentioned. After a complete opening of the MC unit, a constriction in one of the heat exchangers could be located. Such a constriction can cause viscous heating, which has a rather big impact if located near the MC. The MC unit therefore was exchanged by a new one.

4.3.3 Test Run with new Mixing Chamber unit

After the new MC unit had been reattached, further test runs were done. The cooldown was successful and a temperature of around 30 mK was reached without major adjustments in mash composition and phase boundary level. Figure 4.5 shows the whole cooldown from LHe temperature down to base temperature. From the beginning of the condensation to a stable base temperature approximately 3 hours are needed. Helium condensation begins at the first peak and is followed by normal circulation at around 300 mK in figure 4.5. By changing some parameters a stable base temperature of 21.1 mK was reached. This value didn't change during the overnight run (figure 4.6) and fluctuations are less than ± 0.1 mK.

Figure 4.7 represents the simultaneous temperature measurement in the MC (upper) and the still (lower). The changes in temperature after changes in the current



Figure 4.4: broken capillary of the 3He impedance

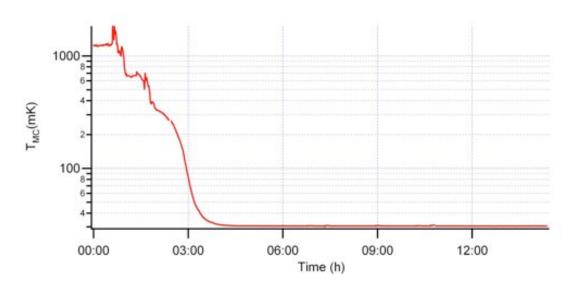


Figure 4.5: $T_{\rm MC}$ of the cooldown with the new MC unit

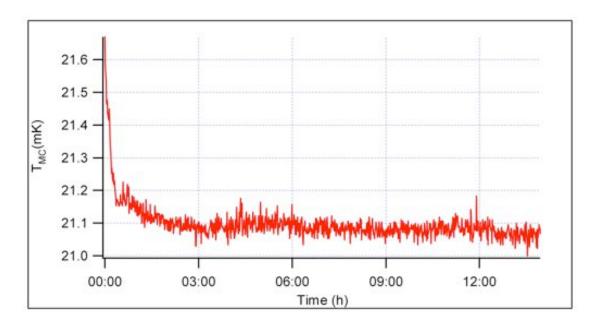


Figure 4.6: T_{MC} overnight

through the heater are much larger in the still, as the heater is located there. Such a test has to be done in order to find the ideal heating power applied to the still. The second part of this curve shows a $single\ shot$ test. The 3He was not re-condensed but pumped back to its dump. Therefore the heat load of the circulating helium could be avoided, but the MC was empty after a short time. By this method the lowest temperatures are reached for a short time. After a single shot the 3He has to be re-condensed and this procedure takes some time.

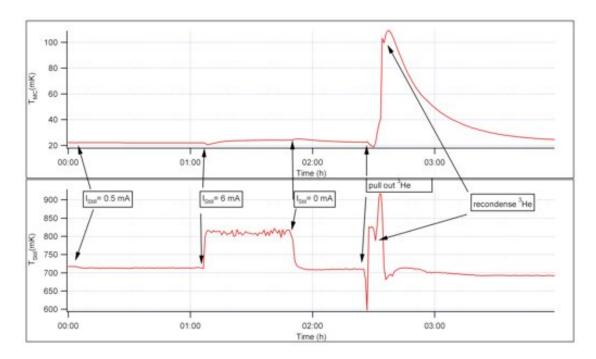


Figure 4.7: Behavior of $T_{\rm MC}$ and $T_{\rm Still}$ upon changing of $I_{\rm Still}$ and single shot situation

5 Conclusion and Outlook

A dilution refrigerator is a strong and useful tool for experimental techniques in low temperature physics. Although it needs a lot of additional equipment e.g. vacuum pumps, measurement and analyzation tools, etc. and also quite a lot of space, its operation is relatively simple and in the case of the *MCK 50-100 TOF* samples can be cooled down in only a few hours. But as the experience in the lab proved, a search for some error or defect cannot be done in a few hours. It is a very time consuming task, requiring a lot of patience, experience and knowledge of all the parts and procedures in the fridge.

Another important requirement is a profound background of condensed matter physics, especially in the low temperature range, where a lot of well known (room temperature) properties change. The right choice of material is sometimes also time consuming and expensive. Deep considerations before the actual experiment however can prevent a lot of trouble and disappointment afterwards. The design and construction of the sample holder will hopefully satisfy the requirements for a lot of future projects and experiments performed in the small MCK fridge. As it is a very modular design, modification or exchange of single parts should be no problem. A LED for example can be added easily. The length and therefore the position of the sample can be adjusted by an additional part and the angle can also be chosen freely. This will become especially important, when a superconducting magnet producing large magnetic fields, which are homogenous only at specific areas, will be added. Some improvement ideas are already given in section 3.5. Other ideas will certainly come up just by designing new experiment concepts or changing the setup.

Additionally it will be of great importance to get more familiar with the fridge: Setting up all the electronic measurement equipment, calibrating the various temperature sensors, measuring the cooling power, etc. Even small adjustments in mash composition, phase boundary level, heating power and other factors can have an impact on various things. A lot of this has now already been done and the fridge is ready for its purpose: delivering the right environment for experiments in the millikelvin range.

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6.4.1 Images from:

- [12] http://ltl.tkk.fi/research/theory/helium.html Figures 2.1 and 2.2
- [13] http://ltl.tkk.fi/research/theory/mixture.html Figure 2.3
- [14] http://www.cryoconcept.fr/FILES/CRYOCONCEPT-2005-01.pdf Figure 2.4

7 Acknowledgment

For a master student with little experimental experience, support of experienced scientists is a very crucial thing. I therefore would like to thank the people who provided this support and took time in helping me and answering my questions. I would like to thank Prof. Dominik Zumbühl for making this project possible and for his big encouragement during the whole project. Especially during the time the DR was not working, he always came up with new ideas which were very inspiring.

But I also would like to thank the whole group for providing an interesting and enjoyable working surrounding on a high scientific level. A special thanks goes to Kai Schwarzwälder who was a big help in setting up the lab.

The workshop team and their experience in fabricating such special equipment we use in the cryolab was also very inspiring. Thank you.

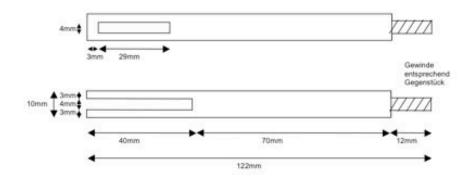
Blueprints

Cold finger Sample holder MCK 50 Verlängerungsstab Silber

Verlängerungsstab:

Profilstange aus Silber: 10 x 10 mm Länge total: 122 mm





Zusätzliche Verlängerung:

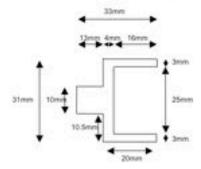


Institut für Physik, Universität Basel Quantum Coherence Lab Florian Dettwiler, Office 1.10, Tel. +41 (0)61 267 3786 florian.dettwiler@stud.unibas.ch

Cold finger Sample holder MCK 50 Silbergabel

Gabel:

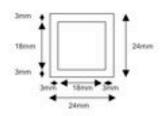
Aus Silberplatte: 50 x 50 mm Dicke überall : 6 mm



Umrahmung Socket:

Ebenfalls aus Silberplatte

Dicke: 6 mm Breite Aussen: 24 mm



Messingscheibe:

Dicke: 1mm Radius: 20mm

Wird an einer Seite des Socketholders befestigt:

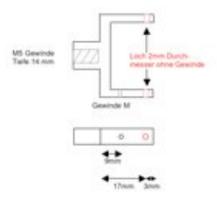


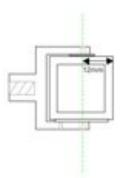




Institut für Physik, Universität Basel Quantum Coherence Lab Florian Dettwiler, Office 1.10, Tel. +41 (0)61 267 3786 florian dettwiler⊜stud unibas.ch

Zusammenbau:





Socketrahmen wird mittels Silberstiften (Durchmesser 2mm) an der Gabel befestigt. Drehachse für den Socket muss in der Mitte sein (12mm).

sein (12mm).

Auf der Seite ohne Messingscheibe wird ein Abstandhalter angebracht -> der Socketholder sollte sich nur mit (kleinem) Kraftaufwand drehen lassen.

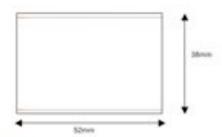
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Cold Finger Sample Holder MCK 50 Messing-Schild

Messingrohr:

Innerer Durchmesser: 35 mm Äusserer Durchmesser: 38 mm Länge: 52 mm



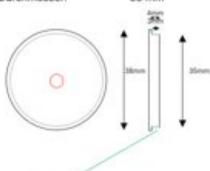


An einem Ende Innengewinde (min 3 mm)

Das andere Ende mit Messingblech (1mm dick) verschliessen

Deckel:

Rundes Messingblech Dicke: 4 mm Durchmesser: 38 mm



Gewinde, so dass der Deckel auf das Rohr geschraubt werden kann, ca. 3mm Loch in der Mitte, Durchmesser 5 mm, ohne Gewinde

Boden:

Rundes Messingblech

Dicke: 1 mm Durchmesser: 38 mm

Ans Rohr gelötet, als Boden

Institut für Physik, Universität Basel Quantum Coherence Lab

Florian Dettwier, florian.dettwier@stud.unibas.ch