A Versatile Nuclear Demagnetization Cryostat for Ultralow Temperature Research

Weijun Yao

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Abstract

A new cascade nuclear demagnetization cryostat has been designed and constructed. Our aim was to build a versatile, modular cryostat with a large experimental space providing an excellent platform for various types of ultra-low temperature measurements. A powerful dilution refrigerator, combined with a massive copper nuclear cooling stage, will enable us to reach the low temperatures necessary in studies of nuclear magnetic ordering. First experiments will include nuclear magnetism in Rh and a search for superfluidity of $^3$He in the dilute $^3$He/$^4$He mixture. Details of the design and results of the performance tests will be presented.

The suitability of normal metal tunnel junction chains for primary thermometry at mK temperatures has been studied. Preparations of experiments for studies of nuclear magnetic ordering in rhodium will be described as well.

To Yanli and Cecilia
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1 Introduction

Modern nuclear cooling extends the lowest temperature that has been reached in a laboratory ten orders of magnitude below 2.7 K, the temperature of the cosmic microwave background. Traditionally, there are two major fields of research below millikelvin temperatures. The first is related to $^3$He, $^4$He, and their mixtures in the liquid and solid states. At such low temperatures, the richness of phenomena in these systems is caused by quantum mechanical effects, originating from the large zero point energy of the helium atoms. The second field of research is connected with magnetic ordering in nuclear spin systems. Owing to the extremely weak interactions between nuclear spins, these phenomena are observable only when the disturbing thermal vibrations die out at very low temperatures. Recently, the research on the Bose-Einstein condensation of some gaseous alkaline atoms has entered this temperature regime (Anderson, Ensher, Matthews, Wieman, and Cornell, 1995; Davis, Andrews, Van Druten, Durfee, Kurn, and Ketterle, 1995).

The idea of demagnetizing nuclear spins emerged in the 1930’s (Gorter, 1934; Kurti and Simon, 1935), soon after electron paramagnets had been employed in magnetic cooling (Giauque and MacDougall, 1933; de Haas, Wiersma, and Kramers, 1933). The first experimental realization of the idea was made at Oxford University by Kurti, Robinson, Simon, and Spohr (1956). The team managed to reduce the nuclear spin temperature of copper to about 1 $\mu$K. In their experiment, the nuclear spins were at record low temperature for several minutes while the conduction electrons and the crystal lattice remained at the starting temperature of 12 mK. A major step forward in the development of the nuclear cooling technique was the successful use of a dilution refrigerator and a superconducting magnet to obtain improved starting conditions in the early 1970’s, first in Helsinki (Berglund, Ehnholm, Gylling, and Lounasmaa, 1970).

Soon after this, two new phases in liquid $^3$He were discovered at Cornell University (Osheroff, Gully, Richardson, and Lee, 1972a; Osheroff, Richardson, and Lee, 1972b). In the original experiment, the transitions were reached, at a temperature slightly below 3 mK, with the aid of an experimental setup combining dilution refrigeration and Pomeranchuk cooling (see for example, Lounasmaa, 1974). The lower limit of the latter method is about 2 mK. Soon afterwards, experiments in Helsinki, using a vibrating wire viscometer, confirmed the new phases and showed that they were, indeed, the superfluid states which had been searched for several years (Alvesalo, Anufriev, Collan, Lounasmaa, and Wennerström, 1973). These discoveries strongly promoted development of the nuclear cooling technique which was then turned into a sophisticated tool to reach temperatures far below 1 mK. Much of this work has been done in our laboratory (Andres and Lounasmaa, 1982).

In this thesis, I will describe the construction and performance of a new versatile cryostat which was designed for facilitating experimental work on condensed
matter at ultralow temperatures.

The principle of demagnetization cooling and the related formulae are presented in § 2 in order to ease the understanding of the terminology and expressions used in the rest of the thesis. At the end of this chapter, a mathematical model to simulate nuclear cooling numerically is introduced.

The project to build the new YKI cryostat has greatly benefited from the long tradition of ultralow temperature research and the wealth of experience accumulated by the use of several generations of nuclear cooling apparatus in our laboratory. Of course, the new design was influenced by the preceding YKI cryostat. In § 3.1, the old apparatus and a cryostat built for neutron diffraction work in Berlin are described. During the designing stage, I was inspired by the know-how of similar facilities in other laboratories which I visited. In the rest of the chapter, some of them are discussed briefly to give a broad picture of the state-of-the-art of nuclear refrigeration at present.

Later chapters are devoted to description of the new cryostat, its design, construction, and performance, and to thermometry, especially the Coulomb blockade thermometer, as well as to some preliminary experiments on rhodium.

The new facility is a result of team work which concerned many people who contributed significantly in different stages. I took the main responsibility throughout the whole procedure of designing, constructing and running the new machine, and was involved in all details related to the cryostat. The work on Coulomb blockade thermometer (§ 5.4) was a collaboration between our group and the group in Jyväskylä University.
2 Adiabatic nuclear demagnetization

2.1 Basic equations

Cooling by demagnetization of noninteracting magnetic dipoles can be understood with the aid of Fig. 1, which shows the entropy vs. temperature curves in various magnetic fields. Although the absolute values are valid just for copper nuclei, the shape of the curves apply for all systems with noninteracting magnetic dipoles. From A to B, the dipoles are magnetized by a 9-T field. More entropy is then removed from the system by external cooling in a constant field, corresponding to the path from B to C. At C, the thermal contact to the precooling device is cut off. When the external field is reduced, the system cools adiabatically from C to D. The temperature decreases in proportion to the field while the entropy of the system stays constant. Warming up will then start at D and the system returns back towards the original point A by absorbing heat.

![Graph showing entropy vs. temperature for different magnetic fields.]

Figure 1: Molar entropy $S_n$ of copper nuclei in different magnetic fields as a function of temperature. The maximum entropy $S_{\text{max}} = R \ln 4$ for copper with spin $3/2$, where $R$ is the gas constant.

Magnetic dipoles can be either of electronic origin, as in several paramagnetic salts, or due to nuclei with non-zero spins. In both systems, the minimum temperature in a cooling process can be estimated from the energy of the dipolar interactions, \textit{viz}.,

$$E_{\text{dip}} = \frac{\mu_0}{4\pi} \left( \frac{\bar{\mu}_i \cdot \bar{\mu}_j}{r_{ij}^3} - \frac{3(\bar{\mu}_i \cdot \bar{r}_{ij})(\bar{\mu}_j \cdot \bar{r}_{ij})}{r_{ij}^5} \right)$$  \hspace{1cm} (2.1)
if no other significant coupling mechanisms between the moments are present. Spontaneous ordering of magnetic dipoles occurs when the interaction energy is comparable to the thermal energy $k_B T$.

The difference between the two systems is due to the fact that the magnetic moments of nuclear spins are about 2000 times smaller than their electronic counterparts. Owing to the weaker interactions between the nuclei, much lower temperatures can be reached. In electronic systems, the ordering temperature ranges from room temperature and above, as in metallic iron, down to several millikelvins as in cerium magnesium nitrate (CMN), a paramagnetic salt commonly used for electronic magnetic cooling. In nuclear spin systems, the interaction energy is usually below 1 $\mu$K and can be in the sub-nanokelvin range as in rhodium. In this thesis, I will mainly discuss nuclear magnetism. When the term “spin” is used it refers to nuclear spins if not specified otherwise.

The statistics for the two systems is identical, but here we write the formulae for nuclear dipoles. All the required thermodynamic quantities can be derived from the partition function of $n$ moles of noninteracting dipoles in an external field, viz.,

$$Z = \left[ \sum_{m=-J}^{m=J} \exp(-m\mu_n g_n B/k_B T) \right]^{nN_0},$$  

(2.2)

where $N_0$ is Avogadro’s number, $B$ is the external magnetic field, $g_n$ is the nuclear $g$-factor, and $\mu_n$ is the nuclear magneton. For example, the entropy, magnetization and heat capacity of the system can be written as,

$$S_n = k_B \partial (T \ln Z)/\partial T$$
$$= nR \left\{ \frac{x}{2J} \coth \frac{x}{2J} - \frac{(2J+1)x}{2J} \coth \frac{(2J+1)x}{2J} \right\}$$
$$+ \ln \left[ \frac{\sinh \frac{(2J+1)x}{2J}}{\sinh \frac{x}{2J}} \right]$$

(2.3)

$$= S_n \left( \frac{B}{T} \right), \quad \text{with } x = \mu_n g_n JB/k_B T,$$

$$M_n = k_B T \partial \ln Z/\partial B|_T,$$  

(2.4)

$$C_n = k_B T \partial^2 (T \ln Z)/\partial T^2|_B.$$  

(2.5)

In the temperature region where magnetic cooling is used the condition $\mu_n g_n B \ll k_B T$ is usually valid. In this high temperature approximation all the above formulae have rather simple forms, viz.

$$S_n = nR \ln(2J+1) - \Lambda B^2/2T^2,$$  

(2.6)
\[ M_n = \Lambda B / T , \quad (2.7) \]

\[ C_n = \Lambda B^2 / T^2 , \quad (2.8) \]

where the Curie constant \( \Lambda = n \lambda = n(N_0 J(J + 1) \mu_n^2 g_n^2 / 3k_B) \).

If a system starts from an initial temperature \( T_i \) and a field \( B_i \), and is demagnetized to \( B_f \), the final temperature will be

\[ T_f = T_i \frac{B_f}{B_i} . \quad (2.9) \]

This simple formula arises because the entropy depends only on the ratio \( B/T \) (see Eq. 2.3). For small \( B_f \), the internal field \( B_{\text{int}} \) arising from the interactions between the magnetic moments becomes important and one has to replace \( B_f \) with \( \sqrt{B_f^2 + B_{\text{int}}^2} \), giving

\[ T_f = T_i \frac{\sqrt{B_f^2 + B_{\text{int}}^2}}{B_i} . \quad (2.10) \]

From Eq. (2.6) one can see that in order to reach the same amount of entropy reduction at the starting point \( C \) in Fig. 1, in a nuclear system the \( B/T \) ratio should be about 2000 larger than in an electronic system. For example, at 10 mK, the entropy reduction in copper is only 5\% in a field of 6 T. Even this rather modest initial condition could not be achieved until a dilution refrigerator and a superconducting magnet were combined (Gylling, 1971). This happened almost forty years after the first successful experiment using a paramagnetic salt and the emergence of the idea of using nuclear spins as a refrigerant. During the last decades this combination has been the standard technique to reach temperatures below 1 mK. Nuclear cooling and relative techniques are explained thoroughly in two textbooks (Lourensmaa, 1974; Pobell, 1992).

### 2.2 Influence of heat leaks

The final temperature is not only a function of \( B_f \), but depends also on the procedure used during demagnetization, which is not strictly adiabatic owing to heat flow into the system. In this section the effects of the external heat leak and eddy current heating, caused by the changing magnetic field, are discussed. An optimal scheme for demagnetization is required in order to minimize the losses of entropy (Gylling, 1971; Gachchiladze, Pavlov, and Pantsulaya, 1986; Veuro, 1978).

Assuming that the total heat leak \( \dot{Q} \) entering the nuclear spin system consists of a constant external term \( \dot{q} \) and eddy current heating, proportional to \( \dot{B}^2 \), we have

\[ \dot{Q} = \dot{q} + \gamma \dot{B}^2 , \quad (2.11) \]
where

$$
\gamma = \begin{cases} 
\frac{r^2 V}{8 \rho_0} \frac{\text{RRR}}{d^2 k^2} & \text{for a cylinder of radius } r , \\
\frac{V}{161 + k^2 \rho_0} \text{RRR} & \text{for a rectangle of width } w \text{ and} \\
\text{thickness } d, \text{ where } k = w/d ,
\end{cases}
$$

(2.12)

$\rho_0$ is the resistivity at room temperature, $V$ is the volume of the sample, and $\text{RRR} = \rho_{300K}/\rho_{4.2K}$ is the residual resistivity ratio (Pobell, 1992).

Starting from

$$
TdS = \dot{Q} \, dt
$$

(2.13)

and from Eq. (2.6), we obtain a measure of non-adiabaticity, \textit{viz}.

$$
\Delta \left( \frac{B}{T} \right) = \left( \frac{B_{t}}{T_{t}} \right) - \left( \frac{B_{i}}{T_{i}} \right)
$$

$$
= \int_{0}^{t_{f}} \frac{\dot{Q}(t) \, dt}{\Lambda B(t)} .
$$

(2.14)

Inserting Eq. (2.11) yields

$$
\Delta(B/T) = a \int_{0}^{t_{f}} \frac{\dot{B}(t)^2 + \alpha}{B(t)} \, dt
$$

(2.15)

where $a = \gamma/\Lambda$ and $\alpha = \dot{q}/\gamma$. The magnetic field $B(t)$ minimizing this functional must satisfy the Euler equation

$$
2B\dot{B} - \dot{B}^2 + \alpha = 0
$$

(2.16)

with the boundary conditions

$$
B(0) = B_{i}, \quad B(t_{f}) = B_{t} .
$$

(2.17)

When $t_{f} < \theta_{2}$ ($\theta_{2}$ is defined in Eq. 2.22), a solution exits, \textit{viz}.

$$
B(t) = B_{i} - 2 \frac{t}{t_{f}} D(t_{f}) + \left( \frac{t}{t_{f}} \right)^2 C(t_{f}) .
$$

(2.18)

$D(t_{f})$ and $C(t_{f})$ are defined as

$$
D(t_{f}) = B_{i} - \frac{1}{2} \sqrt{\alpha (\theta_{2}^2 + t_{f}^2)} ,
$$

(2.19)
\[ C(t_f) = B_i + B_f - \frac{1}{2} \sqrt{\alpha (\theta_1^2 + t_f^2)} , \quad (2.20) \]

\[ \theta_1 = 2 \sqrt{\frac{B_i B_f}{\alpha}} , \quad (2.21) \]

\[ \theta_2 = 2 \sqrt{\frac{B_i^2 - B_i B_f}{\alpha}} . \quad (2.22) \]

The smallest non-adiabaticity resulting from demagnetization can be obtained by inserting Eq. (2.18) into Eq. (2.15), giving

\[ \Delta(B/T)_{opt} = 4a \left[ \frac{C(t_f)}{t_f} + \sqrt{\alpha} \ \text{arcsinh} \left( \frac{t_f}{\theta_1} \right) \right] . \quad (2.23) \]

When \( t_f > \theta_2 \), there is no optimal demagnetization equation which would simultaneously satisfy the boundary conditions and the requirement \( 0 < B(t) \leq B_i \) at \( 0 \leq t \leq t_f \).

Differentiating Eq. (2.23) with respect to \( t_f \), and equating the derivative to zero, one finds the optimal demagnetization time, \( t_{f,opt} \), from \( B_i \) to \( B_f \), viz.

\[ t_{f,opt} = (B_i - B_f)/\sqrt{\alpha} . \quad (2.24) \]

Substitution of \( t_{f,opt} \) into Eq. (2.18), gives the surprisingly simple result that the magnetic field should be changed linearly with time, viz.

\[ B_{opt}(t) = B_i - \sqrt{a/\gamma} \ t . \quad (2.25) \]

When demagnetization is carried out according to Eq. (2.25), eddy current heating and background heat leak are equal and

\[ \Delta(B/T)_{min} = 2a \sqrt{\alpha} \ln \frac{B_i}{B_f} . \quad (2.26) \]

### 2.3 Spin lattice relaxation

In the discussion above, the spin system is assumed to be isolated, except for an external heat leak which is introduced with the simplifying assumption that the energy flows directly into the spin system. In a real situation, it is meaningful to distinguish between energy reservoirs for the nuclei, the conduction electrons, and the lattice.

Electrons and nuclei in metals are coupled via the hyperfine interaction. If the thermal relaxation rate within the nuclear spin system is much faster than that between electrons and nuclei, one can introduce separate temperatures for electrons, \( T_e \), and for nuclei, \( T_n \).
Well below the Debye temperature, the specific heat of the lattice decreases rapidly as $T^3$. Owing to their small heat capacity, phonons will follow the electronic temperature with a very short relaxation time, and one can assume that the lattice and the conduction electrons are in thermal equilibrium at all times.

The spin lattice relaxation process can be considered with the help of Fig. 2. We assume that the nuclear spin system at left is connected through a relaxation processes to an infinite thermal reservoir at right. For simplicity, we consider a system with only two energy levels, which corresponds to the situation of spin 1/2. $N_1$ and $N_2$ denote the occupation numbers of states (1) and (2). For a transition to occur in the spin system, there should be two states (a) and (b) in the reservoir which have the same energy separation as that between (1) and (2). This requirement ensures that the energy released by a jump from state (1) to (2) will be absorbed by a transition from (b) to (a). $N_a$ and $N_b$ denote the occupation numbers of states (a) and (b), respectively. Denoting the probability for such double transitions per unit time by $W_{2a \rightarrow 1b}$, $W_{1b \rightarrow 2a}$, we obtain

$$\frac{dN_1}{dt} = N_2 N_a W_{2a \rightarrow 1b} - N_1 N_b W_{1b \rightarrow 2a}, \quad (2.27)$$

$$\frac{dN_2}{dt} = N_1 N_b W_{1b \rightarrow 2a} - N_2 N_a W_{2a \rightarrow 1b}. \quad (2.28)$$

According to quantum mechanics,

$$W_{1b \rightarrow 2a} = W_{2a \rightarrow 1b}. \quad (2.29)$$
Then,

$$\frac{d(N_2 - N_1)}{dt} = (N_a + N_b)(N_1 + N_2) \left( \frac{N_b - N_a}{N_b + N_a} - \frac{N_2 - N_1}{N_2 + N_1} \right) W_{1b \rightarrow 2a} .$$  

(2.30)

We can define $\tau_1$, which has the dimension of time, as

$$\frac{1}{\tau_1} = (N_a + N_b)W_{1b \rightarrow 2a} ,$$  

(2.31)

and introduce

$$p_n = \frac{N_2 - N_1}{N_2 + N_1}, \quad p_{eq} = \frac{N_b - N_a}{N_b + N_a} .$$  

(2.32)

Eq. (2.30) then has a very simple form, viz.

$$\frac{dp_n}{dt} = -\frac{1}{\tau_1}(p_n - p_{eq}) ,$$  

(2.33)

where $p_n$ is the polarization of the spin system and $p_{eq}$ denotes the value of polarization when the spins are in thermal equilibrium with the lattice. The solution of Eq. (2.33) shows that the spin system approaches equilibrium exponentially, with a time constant $\tau_1$, the so-called spin lattice relaxation time. Using the high temperature approximation from Eq. (2.7) and

$$p = \frac{M_n}{M_{sat}} ,$$  

(2.34)

where $M_{sat}$ is the saturation magnetization of the nuclei, we arrive at the well known relaxation equation,

$$\frac{d}{dt} \left( \frac{1}{T_n} \right) = -\frac{1}{\tau_1} \left( \frac{1}{T_n} - \frac{1}{T_e} \right) .$$  

(2.35)

In metals, only electrons near the Fermi surface can contribute to spin lattice relaxation. Their number is proportional to $T_e$. This leads to the Korringa relation

$$\tau_1 T_e = \kappa ,$$  

(2.36)

where $\kappa$ is the Korringa constant of the material.

The spin system reaches internal thermal equilibrium with a time constant $\tau_2$, the so-called spin-spin relaxation time. In metals, usually $\tau_2 \ll \tau_1$, which is the condition for the existence of a separate nuclear spin temperature.
Using Eq. (2.33), the heat flow between the spin system and the electronic heat reservoir can be written as

\[ \dot{Q} = -B \frac{dM_n}{dt} = \frac{B}{\tau_i} (M_n - M_{eq}) . \]  

(2.37)

Using the high temperature approximation of Eq. (2.7) for nuclear magnetization, we obtain

\[ \dot{Q} = \frac{\Lambda B^2}{\kappa} \left( \frac{T_e - T_n}{T_n} \right) , \]  

(2.38)

where the Korringa relation Eq. (2.36) has been used. The rate of temperature rise in the spin system caused by a heat leak \( \dot{Q} \), then becomes

\[ \frac{dT_n}{dt} = \frac{\dot{Q}}{C_n} = \frac{\dot{Q} T_n^2}{\Lambda B^2} . \]  

(2.39)

From Eq. (2.38), the electronic temperature can also be solved, giving

\[ T_e = \left( \frac{\kappa \dot{Q} + \Lambda B^2}{\Lambda B^2} \right) T_n . \]  

(2.40)

It is obvious from this relation, that an external heat leak sustains a temperature difference between the spins and the electronic system. The influence of the Korringa constant can be seen from this equation as well.

### 2.4 Mathematical model for nuclear refrigeration

Following the discussion above, a simple mathematical model for nuclear refrigeration of a metallic specimen is presented here (see Fig. 3). In this model, the sample to be cooled is connected through a metallic thermal link to the copper nuclear stage. Both the copper refrigerant and the sample are divided into two parts: nuclei and electrons. The heat capacity resides in the nuclei and the interactions with the environment occur via the electronic system. Electronic heat capacities are very small and can be neglected. The temperatures of different assemblies and the heat flow between them are shown in Fig. 3. \( \dot{Q}_{1e} \), \( \dot{Q}_{1ec} \), \( \dot{Q}_{2e} \), and \( \dot{Q}_{2ec} \) represent the external heat leaks and the heat from eddy currents.

The thermal model in Fig. 3 gives rise to the following equations, \textit{viz}.

\[ \dot{T}_{n1} = T_{n1} \frac{\dot{B}_1}{B_1} + \frac{\dot{Q}_1}{C_{n1}} , \]  

(2.41)

\[ \dot{Q}_{1e} + \dot{Q}_{1ec} + \dot{Q}_{12} - \dot{Q}_1 = 0 , \]  

(2.42)
Figure 3: Temperature distribution in a nuclear cooling system

\[ \dot{Q}_{2e} + \dot{Q}_{2ec} + \dot{Q}_2 - \dot{Q}_{12} = 0 , \]  
(2.43)

\[ \dot{T}_{n2} = T_{n1} \frac{\dot{B}_2}{B_2} - \frac{\dot{Q}_2}{C_{n2}} , \]  
(2.44)

\[ \dot{p}_2 = \frac{T_{e2}}{2\kappa_2} \left( 1 - p_2 \coth \frac{T_{e2}}{2T_{e2}} \right) , \]  
(2.45)

where \( T_{e2} \) depends on \( B_2 \) (see definition in Appendix A). And,

\[ \dot{Q}_1 = -B_1 M_{s1} \dot{p}_1 , \]  
(2.46)

\[ \dot{Q}_2 = B_2 M_{e2} \dot{p}_2 , \]  
(2.47)

\[ \dot{Q}_{12} = \frac{1}{2R_{\text{link}}} (T_{e2}^2 - T_{e1}^2) , \]  
(2.48)
where $M_{e1}$ and $M_{e2}$ denote the saturation magnetizations of the materials. Eqs. (2.41) – (2.45) consist of three differential equations and two non-linear algebraic equations. The quantities to be solved are $T_{1e}$, $T_{1n}$, $T_{2e}$, $T_{2n}$, and the polarization of the sample, $p_2$. The numerical calculation sequence is the following:

1. Solve Eq. (2.41) to advance $T_{n1}$ one step to $T_{n1}(t + h)$.

2. Solve $T_{e1}$ and $T_{e2}$ from Eqs. (2.42) – (2.43) by using $T_{n1}(t + h)$ to get $T_{e1}(t + h)$ and $T_{e2}(t + h)$.

3. Solve Eq. (2.44) to advance $T_{n2}$ one step resulting in $T_{n2}(t + h)$ by using $T_{n1}$, $T_{e1}$, and $T_{e2}$ at $t + h$.

4. Solve Eq. (2.45) to obtain $p_2(t + h)$.

5. Repeat steps 1 – 4.

In the calculation, the high temperature approximation $k_B T \gg \mu_n g_n B$ is not valid, especially for the sample. The exact expressions for heat capacity and polarization are listed in Appendix A. Some constants needed in the calculation are also listed there.

This simple model was successfully used in the thermal analysis of the neutron diffraction experiments on $^{109}$Ag (see § 3.1) and has now been used to simulate the cooling of Rh (see § 7).

### 2.5 Choice of refrigerant

A material which is suitable for nuclear cooling has to satisfy several criteria. Its microscopic properties, such as the nuclear spin, the lattice structure, and the interaction between the spins and the lattice are reflected in the macroscopic performance such as the minimum temperature, the time constant to establish equilibrium, cooling power, etc. For an ideal refrigerant, the following properties are desirable: i) small internal field, ii) small Korringa constant, iii) good thermal conductivity, iv) large $\lambda / v_m$, where $v_m$ is the molar volume.

The first requirement follows from Eq. (2.10); the lowest temperature obtainable in a system is affected by the internal field. $B_{int}$ is the net effect from interactions listed below (Pickett, 1988):

- A magnetically ordered host lattice will produce a field at the site of the nuclei.

- Nuclei with non-zero electric quadrupole moment can become ordered by the electric field gradient of the host lattice. In systems with spin 1/2 or in a cubic lattice, the quadrupolar interaction is zero, either because the quadrupole moment is zero, or because the electric field gradient vanishes owing to lattice symmetry, respectively.
• The indirect exchange interaction mediated by conduction electrons (the so-called RKKY interaction) contributes to $B_{\text{int}}$. The direct exchange interaction between nuclear spins is very weak because the overlap of wave functions is small owing to the comparatively large distance between the nuclei.

• The dipolar interaction, which is always present between magnetic nuclei, sets the lower limit for the local field, if other interactions are absent or very small.

Therefore, a possible candidate for nuclear refrigerant should have a nonmagnetic host lattice, and it should possess either a spin $1/2$ or a cubic structure. The RKKY interaction must be small as well.

The Korringa constant is a measure of the spin-lattice relaxation. A small $\kappa$ is preferred for quick attainment of temperature equilibrium between the spins and the lattice. Also, from Eq. (2.40) it can be seen that a lower $\kappa$ will result in a lower electronic temperature in the presence of an external heat leak.

Spin diffusion obeys the equation

$$D\nabla^2 p = \frac{\partial p}{\partial t},$$

(2.49)

with $D \approx 10^{-17}$ m$^2$/s (Veuro, 1978) in a typical solid, where $p$ is the spin polarization. This process is rather slow in transferring polarization over macroscopic distances and it is useful only in establishing local thermal equilibrium. Temperature equilibrium between different locations in the nuclear stage is established through conduction electrons. Therefore, the refrigerant should be metallic with good thermal conductivity. Superconductors are excluded owing to their poor thermal conductivity and the slow spin lattice relaxation in the superconducting state.

From a practical point of view, a larger $\lambda/\nu_m$ provides a higher cooling capacity with a same volume of the refrigerant.

All requirements cannot be fulfilled simultaneously, and some of them are actually conflicting. Table 1 lists several metals with non-zero nuclear spins. Many of them have been tried for demagnetization. So far the most commonly used material is copper. Some research has been done on indium (Symko, 1969) and on thallium (Angerer and Eska, 1984). More recently, TiH$_2$ (Li, 1996), aluminum (Wendler, 1996), and AuIn$_2$ (Herrmannsdörfer and Pobell, 1995) have been investigated to see whether they would be suitable for working materials of nuclear cooling.
### Table 1: Properties of metals suitable for nuclear refrigeration

<table>
<thead>
<tr>
<th>Metal</th>
<th>I</th>
<th>Abundance (%)</th>
<th>$\mu_0\lambda/\nu_m$ (µK)</th>
<th>$B_c$ (mT)</th>
<th>$\kappa$ (Ks)</th>
<th>$\tau_2$ (ms)</th>
<th>Lattice structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{27}$Al</td>
<td>5/2</td>
<td>100</td>
<td>0.87</td>
<td>10.5</td>
<td>1.8</td>
<td>0.03</td>
<td>fcc</td>
</tr>
<tr>
<td>$^{63,65}$Cu</td>
<td>3/2</td>
<td>69.1, 30.9</td>
<td>0.57</td>
<td>1.27, 1.09</td>
<td>0.15</td>
<td>fcc</td>
<td></td>
</tr>
<tr>
<td>$^{107,109}$Ag</td>
<td>1/2</td>
<td>51.8, 48.2</td>
<td>0.002</td>
<td>12, 10</td>
<td>10</td>
<td>fcc</td>
<td></td>
</tr>
<tr>
<td>$^{197}$Au</td>
<td>3/2</td>
<td>100</td>
<td>0.0016</td>
<td>4.6</td>
<td></td>
<td>fcc</td>
<td></td>
</tr>
<tr>
<td>$^{113,115}$In</td>
<td>9/2</td>
<td>4.3, 95.7</td>
<td>1.1</td>
<td>28</td>
<td>0.09</td>
<td>0.1</td>
<td>tetr</td>
</tr>
<tr>
<td>$^{93}$Nb</td>
<td>9/2</td>
<td>100</td>
<td>1.99</td>
<td>200</td>
<td>0.36</td>
<td>0.02</td>
<td>bcc</td>
</tr>
<tr>
<td>$^{203,205}$Tl</td>
<td>1/2</td>
<td>29.5, 70.5</td>
<td>0.207</td>
<td>18</td>
<td>0.004</td>
<td>0.035</td>
<td>hcp</td>
</tr>
<tr>
<td>$^{195}$Pt</td>
<td>1/2</td>
<td>33.80</td>
<td>0.019$^a$</td>
<td>0.03</td>
<td>1.0</td>
<td></td>
<td>fcc</td>
</tr>
<tr>
<td>$^{103}$Rh</td>
<td>1/2</td>
<td>100</td>
<td>0.0013</td>
<td>0.05</td>
<td>10.0</td>
<td>10.5</td>
<td>fcc</td>
</tr>
</tbody>
</table>

$^a$natural isotopes
3 Nuclear demagnetization refrigerators

3.1 YKI cryostat in Helsinki and NYKI cryostat in Berlin

Built in the end of the 1970's (Ehnholm, Ekström, Jacquinot, Loponen, Lounasmaa, and Soini, 1980), the YKI cryostat, with two nuclear stages in cascade, was the first refrigerator of this type. It was intended for studies of the very weak interactions between nuclei in the nanokelvin range (see Fig. 4).

Although a dilution refrigerator and a superconducting magnet substantially improved the initial conditions for nuclear cooling, starting from 10 mK and 9 T is not good enough to attain nanokelvin temperatures. Cascade nuclear refrigeration was used to overcome this problem. The Helsinki cryostat was first employed to study the magnetism of copper nuclei. A close-up of the nuclear stages is shown in Fig. 5.

The dilution refrigerator was home made (Ehnholm et al., 1980). In its design, special care was taken to ensure a rigid structure. The machine reached 7 mK without an external heat load. The whole cryostat was located on top of a concrete block which was cast directly on the bedrock and separated from the rest of the building. The thermal link between the dilution refrigerator and the copper nuclear stage was by means of a heat switch made out of a piece of bulk tin.

The cascade setup consisted of two nuclear stages, each polarized by a superconducting solenoid. The magnets were compensated at both ends, so that sweeping the field of one solenoid did not affect the field inside the other. The first nuclear stage was made of copper and served as an intermediate step. After having been cooled by the dilution refrigerator to an initial temperature of 10 – 12 mK in an 8-T field, it was demagnetized to 0.1 T, cooling to about 0.2 mK. At the same time the second nuclear stage, which was also the specimen to be investigated, was magnetized to 7 T by another solenoid.

The sample was made of copper foils (Huiku, Jyrkkiö, Kyynäräinen, Loponen, Lounasmaa, and Oja, 1986) of mass less than 1% of the first stage. Consequently, the heat produced by magnetization of the sample could be absorbed by the first stage without significantly raising its temperature. Therefore, in experiments for studying nuclear ordering phenomena, the first nuclear stage does not have to be very large, as long as it can absorb the heat leak from the environment during the precooling period. After the field of the second stage magnet had been swept up, it took 1.5 h to reach thermal equilibrium between the sample and the first stage. During this period nuclear spins of the sample were polarized almost to 100% and the spin entropy was reduced close to zero. The first stage was then demagnetized to 20 mT, producing a 0.05 – 0.1 mK background temperature in the conduction electron system of both nuclear stages since there was no heat switch in between. Next, the second stage was demagnetized from 7 T to zero in 20 min. The spin system of the sample was thereby cooled to below 50 nK.
Figure 4: YKI cascade nuclear demagnetization cryostat in Helsinki (Ehnholm et al., 1980).
The first nuclear stage in Fig. 5 had the by-now-standard solid-block design (Pekola, Simola, and Nummila, 1984); its final version, after a development process of 15 years, is shown in the figure (Ehnholm et al., 1980; Huiku et al., 1986) When the cryostat was built in the late 1970's (Ehnholm et al., 1980), the first nuclear stage was made of 10 moles of 0.5 mm diameter copper wire, insulated with fiberglass to suppress eddy current heating. With this construction, a temperature of 250 \( \mu \)K could be reached. Over the years, the disadvantages of the wire design became obvious, namely:

- The construction is soft and thus susceptible to vibrations, giving rise to heating.

- It is difficult to ensure that all wires are properly connected to the top flange of the bundle. Loose and thus warm wires lead to a large heat leak at low temperatures.
• Good thermal contact between the sample and the copper wires of the first stage is difficult to achieve. This makes precooling of the sample less successful.

• The insulating material of the wires severely restrains the heat treatment procedure aiming at an improved conductivity.

In later versions, rigid copper rods were used. Essential improvements were then observed. The conduction electron temperature decreased from 250 to 50 μK and the total heat leak to the nuclear stage was reduced from 200 pW/mole (Ehnholm et al., 1980) to 28 pW/mole (Huikku et al., 1986).

In the cascade setup, the sample was always thermally connected to the first stage, and therefore, its electronic temperature, $T_e$, could not be lowered below that of the first nuclear stage at 50 to 100 μK. After demagnetization to a low field, the spin system of the sample is at a much lower temperature than the lattice. The nuclear spin temperature $T_n$ then begins to relax towards $T_e$ at a rate determined by $\tau_0$. The long spin lattice relaxation time at low temperatures decouples the nuclei from the conduction electrons and helps to keep the spins cold.

At a temperature around 100 μK, the heat capacity of electrons is small. A tiny heat leak to the sample is always present. A poor thermal contact then leads to a large temperature difference between the electrons of the sample and those of the first nuclear stage, thus speeding the warming up of the specimen. The methods for increasing the thermal conductivity of the metallic link will be discussed in § 7.3.

The longitudinal and transverse susceptibilities of the samples were measured. In the YKI cryostat, copper nuclei were first cooled below 50 nK, and spontaneous nuclear ordering was observed at 58 nK (Huikku et al., 1986). Later, with similar setups, the magnetism in silver and rhodium nuclei was studied (Hakonen, Nummila, Vuorinen, and Lounasmaa, 1992; Vuorinen, Hakonen, Yao, and Lounasmaa, 1995). In rhodium, the spin temperature was lowered to 280 pK, which is, at present the lowest temperature ever reached.

In the middle of 1980's, a similar facility was built to conduct neutron diffraction studies of nuclear magnetic ordering in metals (Jyrkkö, Huikku, Siemensmeyer, and Clausen, 1989). A powerful dilution refrigerator and a solid-block nuclear stage were used in this machine. In addition, the cryostat was especially tailored to the needs of neutron diffraction measurements. For example, the polarizing field on the sample was generated by a split pair coil allowing passage of the neutron beam.

The NYKI cryostat was first operated at the Risø National Laboratory in Denmark, where research on copper was done. After a complete renovation, the apparatus is now working at the Hahn-Meitner-Institut in Berlin. Fig. 6 shows a schematic view of the cryostat (Nummila, 1996). Using this facility, antiferromagnetic nuclear order in silver at picokelvin temperatures was revealed by
Figure 6: Schematic view of the nuclear demagnetization cryostat in Berlin for neutron scattering experiments (Nummila, 1996). $R_T$ denotes the coefficient in the thermal conductivity for a metal, $\lambda = T/R_T$. 
neutron diffraction experiments (Tuoriniemi, Nummila, Vuorinen, Lounasmaa, Metz, Siemensmeyer, Steiner, Lefmann, Clausen, and Rasmussen, 1995).

The cooling method using nuclear stages in cascade was mainly developed on the basis of these two machines. In the design of the new cryostat, many features to be discussed in the following chapters, can be attributed to inventions and practical experience gained with the earlier machines.

3.2 Cryo-II apparatus in Leiden

In Leiden, a very powerful dilution refrigerator has been used to cool a copper nuclear stage in a 6 T field to 7 mK (Van de Haar, 1991).

The setup has some special features (Fig. 7) which differ from arrangements used elsewhere. Normally, the nuclear stage hangs below the mixing chamber. The cryostat as a whole then becomes rather long, especially if there are more than one nuclear stages. Such a structure can be susceptible to vibrations which introduce heat leaks. In a long cryostat, the alignment of the various parts has to be more accurate, in order to avoid touches between the radiation shields anchored to different temperatures.

The Leiden design is very compact. It makes use of a special feature of their dilution refrigerator: there is a large cylindrical space inside the heat exchangers. The copper nuclear stage, attached to the bottom of the mixing chamber, extends upwards into this space. The magnet is hung below the still and is working at 0.7 K. In principle this could raise the critical field, but in practice the magnet, located in vacuum, does not work as well as in liquid helium. A 6-T field can be produced over the nuclear stage.

Lead was used as the heat switch material because it may have a better switching ratio $\lambda_n/\lambda_s$ (ratio of thermal conductivity in the normal state to that in the superconducting state) (Odoni, Fuchs, and Ott, 1983), than the nowadays commonly used aluminum (Mueller, Buchal, Oversluizen, and Pobell, 1978). The heat switch was made of three $1 \times 12 \times 40 \, \text{mm}^3$ strips with a 6N purity. The ends of the strips were molten against copper pieces which were screwed to the nuclear stage and to the cold plate of the mixing chamber. Without annealing, the RRR of the lead strips was 4000.

The home made dilution refrigerator is the most powerful unit ever built when characterized by the cooling power below 10 mK (Fig. 8). Without thermal load, the lowest temperature is 3.2 mK. With a circulation of 4 mmole/s, the cooling power inside the mixing chamber is 10 $\mu$W at 7 mK.

The nuclear stage, consisting of 30 moles of copper, can be cooled to 10 mK (7 mK) in 10 h (40 h). Although the precooling time was quite short, compared to similar machines in other laboratories, the capacity of the dilution refrigerator cannot be fully utilized. The nuclear stage could have been cooled to 5 mK in 20 h if the thermal resistance to the cold plate of the mixing chamber had been negligible. It is difficult to make a heat switch with a sufficient conductivity
Figure 7: The nuclear stage and the dilution refrigerator of the Leiden cryostat (Van de Haar, 1991).
Figure 8: Cooling power of the Leiden dilution refrigerator (Van de Haar, 1991).

to be compatible with such a powerful dilution unit. The lowest temperature measured with this cryostat is 115 $\mu$K. The facility has been used in research on solid helium at submillikelvin temperatures (Van de Haar, 1991).

The compact structure of the Leiden machine was adapted into the design of our new nuclear refrigerator (see § 4). The Leiden know-how in the construction of powerful dilution refrigerators also appears in our machine (see § 4.3), whose dilution unit was purchased from Leiden Cryogenics, Ltd. The superconducting magnet, operating in vacuum and producing a field of 6 T, was the first of this type. Its performance gave us a good reference for the design of our second stage magnet (see § 4.6).

3.3 Lancaster cryostat

The setup used in Lancaster is somewhat different from the previously described apparata (Bradley, Guénaunt, Keith, Kennedy, Miller, Musset, Pickett, and Pratt Jr., 1984). Instead of a bulky copper bundle, the group employs a few moles of copper flakes, mainly for cooling liquid $^3$He or $^3$He/$^4$He mixtures.

A typical setup consists of two cells (see Fig. 9). The outer cell with thick epoxy walls is filled with copper flakes working as the refrigerant for $^3$He. The liquid is precooled by several silver pads with their surfaces covered with sintered silver powder. Ag wires of 1 mm in diameter were spot-welded to the pads and emerge from the cell through the epoxy wall. The other ends of the wires are connected to an aluminum block which works as a heat switch between the nuclear stage and the mixing chamber. The inner cell, with thin epoxy walls, thermally isolates a small volume of $^3$He from the outer cell. Copper plates
covered with silver sinter work as the refrigerant in the inner volume. The plates are connected to the aluminum heat switch by silver wires (see Fig. 10).

A 6.4-T magnet is used to polarize the copper nuclei. In an experiment, $^3\text{He}$ in both cells is cooled by the dilution refrigerator to 8 mK in 5 days. During demagnetization, at first $^3\text{He}$ in both chambers cools simultaneously. The liquid in the outer cell reaches about 0.5 mK while the temperature in the inner cell decreases further. The copper flakes have a much larger internal field of 300 mT than the 0.3 mT in bulk copper. Owing to this, demagnetization of the flakes much below 300 mT does not produce further cooling. But the high internal field assures a large heat capacity, working as a good thermal sink which prevents the external heat leak from reaching the inner cell. The temperature of the copper plates, acting just like bulk pieces, decreases much more during demagnetization. Therefore, the liquid in the inner cell can be cooled to a temperature of 100 μK. Thermal isolation of the inner volume relies on the Kapitza resistance, which is rather large between the epoxy wall and liquid $^3\text{He}$ at such low temperatures.

The superconducting heat switch between the mixing chamber and the nuclear stage does not have its own magnet. It is operated by the fringe field of the main demagnetization solenoid. The proper functioning of this scheme relies on good performance of the dilution refrigerator. During demagnetization, before the heat contact is cut off by the superconducting transition in the switch, the
temperature of the dilution refrigerator decreases faster than that of the nuclear stage because the thermal load becomes smaller.

The Lancaster type design is not only very successful in cooling $^3$He to the lowest temperatures, but also rather simple in construction. For each individual experiment, one can make a nuclear stage just for the specific purpose needed. With a slight change in the inner cell, the nuclear spin-lattice relaxation in copper metal was studied below 10 $\mu$K (Enrico, Fisher, Guénault, Miller, and Pickett, 1994). In our planned project of searching for the superfluid transition in $^3$He/$^4$He mixtures, a similar multi-cell setup is being designed (see § 4).

3.4 Other refrigerators

The efficiency of the so-called "brute force" nuclear magnetic cooling, which was described in the previous sections, is determined by the initial ratio of $B/T$. To achieve a higher cooling power and lower final temperatures, a high $B/T$ ratio is needed. When the nuclear demagnetization technique started developing at the beginning of the 1970's, typical starting conditions were 15 mK and 6 T; reduction of the spin entropy was thus less than 5%. Since then, progress has been made in the following areas:

- Better understanding of the factors which influence the performance of a nuclear cooling stage. Among them are the heat treatment methods, the influence of magnetic impurities, thermal conductivity at ultralow temperatures, etc.

- Improvements in the design of the dilution refrigerators, which have resulted in much higher cooling powers.
• Better understanding of the sources of internal and external heat leaks, which earlier prevented the nuclear stage from cooling to lower temperatures.

• Improved knowledge of thermometry at ultralow temperatures.

These developments have substantially improved the performance of nuclear cooling cryostats. Besides the machines already mentioned, several similar cryostats have been built (Gloos, Smeibidl, Kennedy, Singaas, Sekowski, Mueller, and Pobell, 1988; Xu, Avenel, Xia, Xu, Lang, Moyland, Ni, Adams, Ihas, Meisel, Sullivan, and Takano, 1992). Among them, the Bayreuth apparatus has the best performance (Fig. 11). The cryostat has a very large nuclear stage, 275 moles (about 17 kg) of copper with 104 moles in 8-T field. A $^{195}$Pt NMR thermometer, located on the top flange, was cooled to 15 $\mu$K. The nuclear stage can alternatively precool a second stage (2 moles of copper in 9 T). Demagnetization of this stage has resulted in a temperature of 12 $\mu$K, measured by another $^{195}$Pt NMR thermometer.

In our laboratory, there are two nuclear demagnetization cryostats for studies of vortex structures in rotating superfluid $^3$He (Hakonen, Ikkala, Islander, Markkula, Roubeau, Saloheimo, Garibashvili, and Tsakadze, 1983; Salmelin, Berglund, Kyynäräinen, and Pekola, 1989).

Today, for a copper nuclear stage of 100 moles in a field of 9 T, a typical initial temperature is around 10 mK after 2 days of precooling. With decreasing temperature, the magnetic heat capacity of nuclei increases approximately as $T^{-2}$ while the cooling power of a dilution refrigerator decreases as $T^2$. Further decrease of temperature becomes difficult owing to the rapidly vanishing cooling power of the dilution refrigerator. Increasing the magnetic field is limited both financially and experimentally. A magnet made out of NbTi multifilament superconductor can reach 9 T at 4.2 K, and be boosted to 10 T at 2 K. A composite magnet using NbTi and NbSn$_3$ wire can attain a field as high as 20 T. The manufacturing procedure of a NbSn$_3$ magnet is rather complicated and, therefore, the price is an order of magnitude higher. Also, experiments have shown that flux jumps are very large in an NbSn$_3$ magnet. During demagnetization, the temperature of the nuclear stage then increases because of large eddy current heating (Frossati, 1993).
Figure 11: Bayreuth nuclear refrigerator (Gloos et al., 1988).
4 The new apparatus

4.1 Background information

4.1.1 Motivation for a new cryostat

In previous sections, I summarized briefly the work done on the old YKI cryostat. When this apparatus was built, the techniques used were modern and many novel and pioneering studies, both in basic physics and in cryogenics, were made with this refrigerator. During the 15 years when the YKI machine was on duty, many other cryostats were built. Some of the progress in nuclear cooling experiments, both in Helsinki and elsewhere, was reflected in the evolution of the YKI cryostat, such as the nuclear stage made of bulk copper. Some other innovations could not be adopted without major reconstruction. YKI's limited experimental space and the relatively small first nuclear stage prevented experiments using different schemes. These limitations finally led to the decision to build a new apparatus.

During the designing stage, three experiments were proposed for the cryostat:

- Studies of nuclear magnetism in platinum.
- Search for superconductivity in gold.
- Search for the superfluid transition in liquid $^3$He/$^4$He mixtures.

4.1.2 Platinum experiment

Compared with the nuclear spin systems of copper, silver, and rhodium, a special feature of platinum is that a natural specimen consists of 33.8% of $^{195}$Pt with $I = 1/2$, while the rest of the stable isotopes have zero spin. By enriching, one can obtain samples with higher concentrations of the magnetic component. The average distance of nuclei with non-zero spins and, therefore, their interactions can be varied continuously in different samples, without changing the properties of the lattice. Platinum is thus a good system for studying nuclear magnetism as a function of interactions between nuclei and the effects of randomness.

Compared to copper, silver, and rhodium, the Korringa constant $\kappa = 30$ msK in platinum is two orders of magnitude smaller. In the experiments on Cu, Ag, and Rh, the achievement of the very low spin temperatures, in the nK region, relies on the long spin-lattice relaxation time $\tau_1$, which isolates the spins from the lattice (see § 3.1). A typical electronic temperature of 50 $\mu$K was produced by the first stage; $\tau_1$ is 6 and 9 h for copper and silver, respectively. For platinum, $\tau_1 = 10$ min at 50 $\mu$K. Therefore, a small heat leak, to the electronic system, will rapidly flow to the spins and raise their temperature before the heat can be absorbed by the first nuclear stage. Consequently, it is necessary to cool the electrons in Pt well below 10 $\mu$K for sufficient isolation.

To solve this problem, a special scheme, shown in Fig. 12, was suggested.
Figure 12: One possible scheme for the proposed platinum experiment.

Compared with the usual setup, an intermediate stage is introduced. The sample is very close to this additional unit and attached to it with a good thermal link which, in turn, is connected to the first stage through a heat switch. Both the sample and the intermediate stage are cooled by the first stage to below 200 μK in a field produced by the second stage magnet. During demagnetization, the sample and the intermediate stage cool simultaneously. A field of 30 mT is maintained on the intermediate stage by another small magnet, while the large field for the second stage is reduced to zero. The electronic temperature $T_e$ of the combined system is maintained by the large heat capacity of the intermediate stage around 1 μK while the spin system in the sample cools to a much lower temperature by demagnetization to zero field. If we assume a total external heat leak of 5 pW, then the electronic temperature of the combined system will be at 1.5 μK.

Platinum NMR is a commonly used method of thermometry (see § 5.3). A sample of natural Pt has recently been cooled to $T_n \simeq 0.3$ μK and $T_e \simeq 1.5$ μK with no indication for nuclear magnetic ordering (Pobell, Hermannsdörfer, Rehmann, and Wendler, 1996).

4.1.3 Search for superconductivity in Au

The efforts of searching for the superconductivity in gold can be traced back to the time of Kamerlingh Onnes. Whenever a new cryogenic technique decreased the low temperature limit, an attempt to detect superconductivity in noble metals has been made (Buhrmann and Halperin, 1974; Hoyt, Scholz, and Edwards,
1981; Buchal, Mueller, Pobell, Kubota, and Folle, 1982, see also the references therein).

Because of the rather weak electron-phonon interaction in noble metals, theoretical calculations for predicting the transition temperatures are inaccurate. Experimentally, the superconductivity of Cu, Ag, and Au alloyed with elements Ga, Al, In, Sn, or Zn, in concentrations between 3% to 15%, has been investigated (Hoyt and Mota, 1976). Estimates of $T_c$'s for pure noble metals can be extrapolated from the experimental results; $(T_c)_{\text{Au}} \simeq 200 \, \mu K$, $(T_c)_{\text{Ag}} \simeq 4 \, nK$ and $(T_c)_{\text{Cu}} \simeq 0.6 \, nK$. Among the three noble metals, Au seems to be the most promising choice to detect superconductivity.

Similar experiments on $\text{Au}_{1-x}\text{In}_x$ from 38 $\mu K$ to 80 mK, with $x < 0.1$ gave an extrapolated $(T_c)_{\text{Au}} \simeq 100 \, \mu K$ (Buchal et al., 1982). The pairbreaking influence of magnetic impurities in the low ppm range was studied as well. It was concluded that sub-ppm amounts of impurities could have been responsible for the failure to observe superconductivity in Au. Therefore, sample preparation may be the most difficult part in this experiment.

From the point of view of cryogenics, the Au experiment imposes two requirements on the cryostat: low $\mu K$ temperatures and a small magnetic field at the site of the sample. Estimated from the transition temperature of 100 $\mu K$, the critical field in gold at zero temperature is about 2 $\mu T$. Multiple magnetic shields and active compensation must be used to ensure such a low value.

### 4.1.4 Search for superfluidity in $^3\text{He}/^4\text{He}$

A superfluid phase of $^3\text{He}$ as a dilute solution in $^4\text{He}$ was suggested in 1960's (Bardeen, Baym, and Pines, 1967), even before the discovery of superfluidity in pure $^3\text{He}$. One of the most intriguing experimental problems in ultra low temperature physics today is the search for superfluidity in such a system. The theoretically predicted transition temperatures vary from 1 $\mu K$ to 1 mK (Bardeen et al., 1967; Bashkin, 1978; Owen, 1981). Persistent efforts have been made by experimentalists to find the transition (Osheroff and Corruccini, 1981; Guénault, Keith, Kennedy, and Pickett, 1982; Owens-Bradley, Chocobacs, Mueller, Buchal, Kubota, and Pobell, 1983; Ishimoto, Fukuyama, Nishida, Miura, Takano, Fukuda, Tazaki, and Ogawa, 1989; Oh, Ishimoto, Kawae, Nakagawa, Ishikawa, Hata, Kodama, and Ikehata, 1994). So far, the lowest temperature reached in a $^3\text{He}/^4\text{He}$ mixture is 97 $\mu K$ (Oh et al., 1994).

The difficulties which prevent the mixture from reaching even lower temperatures originate from the large thermal boundary resistance, which varies as $T^{-3}$ at temperatures below 10 mK (Cousins, Fisher, Guénault, Pickett, Smith, and Turner, 1994; Voncken, Riese, Roobol, König, and Pobell, 1996). A small heat leak, therefore, will produce a large difference between the liquid temperature
Figure 13: Suggested cell for the $^3$He/$^4$He mixture experiment.
\( T_{\text{liq}} \) and the refrigerant temperature \( T_n \), viz.

\[
T_{\text{liq}}^4 = T_n^4 + 4R_K \dot{Q}/A
\]  \hspace{1cm} (4.1)

where \( R_K \), \( A \), and \( \dot{Q} \) are the coefficient of the Kapitza resistance, the contact surface area to the coolant, and the heat leak, respectively. Increasing \( A \) and decreasing \( \dot{Q} \) are the straightforward approaches for solving the problem. It has been suggested that high pressure and/or high polarization of \(^3\text{He}\) may increase the transition temperature (Roobol, Frossati, Bedell, and Meyerovich, 1995).

From the point of view of cryogenics, this experiment does not require special properties from the cryostat. The problems mentioned are common in almost all ultralow temperature experiments. Fig. 13 shows a Lancaster type design, suggested for the experiment to search for superfluidity in \(^3\text{He}/^4\text{He}\) mixtures. The nuclear cooling stage is similar to the one described in § 3.3 except in the following respects:

- An intermediate cell is added to further separate the inner and outer cells.
- Copper is used to construct the wall of the inner and intermediate cells.
- A liquid helium operated cold valve is employed to block the filling line to the cell.

At the moment, this project is still in its designing stages. I will not discuss it in the rest of my thesis.

4.1.5 Overview of the new cryostat

A general design principle of the new refrigerator was to construct a versatile unit providing a platform for many types of low temperature experiments. Suitability for the previously mentioned experiments was especially considered. A temperature below 100 \( \mu \)K is the common requirement for all of them. The Pt experiment also requires a triple-stage system with a large first stage, to ensure sufficient cooling capacity needed to refrigerate the intermediate stage. Although the other two experiments do not demand any specific new features of the cryostat, a large space was left on top of the first nuclear stage for mounting a gold sample and/or a helium cell.

The first experiment in the new cryostat will be a continued research on nuclear magnetism in rhodium. Fig. 14 shows the general layout of the apparatus with a rhodium sample mounted on top of the first nuclear stage.

The rhodium sample, acting as the second cooling stage, is located in the space inside the heat exchangers of the dilution refrigerator, mounted on top of the first nuclear stage. The sample is magnetized by a 7.5-T magnet which is fixed to the bottom of the still. The first nuclear stage, 170 moles of copper, is magnetized by the 9-T superconducting solenoid. The space between the mixing
Figure 14: Layout of the cryostat.
chamber cold plate and the top flange of nuclear stage is for other experiments which need either mK or μK temperatures. The heat switch and the pulsed NMR thermometer are positioned here as well. The dimensions of this space are 160 mm in diameter and 190 mm in height. The two superconducting magnets are so designed that the field in this region is less than 5 mT at all times.

Unlike the arrangement in a usual double nuclear stage cryostat, with the second stage hanging beneath the first one, we make use of the space inside the heat exchangers. The total length of the cryostat is thereby reduced and the overall rigidity is increased. The first nuclear stage can be dismounted easily by removing the four copper screws which bolt the cold plate to the mixing chamber. A different nuclear stage can then be positioned into this space.

4.2 Peripherals

The new cryostat, inside an electrically shielded room, rests on the bedrock beneath the laboratory building (see Fig. 15). Measurement electronics is located inside the room, while the controlling PCs are outside. Communication between the devices is done through optical cables, adapted to IEEE-488 and RS-232 buses.

Special attention was paid to the construction of the platform in order to isolate the apparatus from vibrational disturbances of the environment. The cryostat is suspended from an aluminum plate A which rests on a wooden block B, supported by four air springs D (Kinetic Systems, Inc.), on top of four stainless steel pillars. The total weight of the platform, including the dewar, is about one ton. The pillars pass through holes in the floor with their lower ends cast inside a concrete block K, which is cast directly on the bedrock beneath and separated from the building. An accelerometer connected to a Fourier analyzer was used to measure vibrations of the platform. With a resolution of $5 \, \mu g \quad (g = 9.8 \, m/s^2)$, no vibrational modes were observed.

All mechanical pumps are located in the basement. The small tubes between the cryostat and the pumps are anchored to the concrete blocks G and K, and to the wooden block B. Soft metallic bellows sections H are used in between. The still pumping line C (15 cm to 25 cm in diameter) for circulation of $^3$He is on the other side of the cryostat, passing through a hole in the floor. It is connected to the booster pump which rests on the concrete block K.

The ends of the elbow I in Fig. 15 are connected to stainless steel bellows. Such a structure provides a flexible section on the stiff pumping line without sacrificing stability under the strong force on the bellows when the tube is evacuated. Two sections of rubber sleeves J in series were added to the main pumping line, acting both as mechanical and electrical isolators. There are similar rubber sleeves in the small pumping lines as well, so that the cryostat is electrically isolated from the environment to facilitate proper grounding of the measurement setups.
Figure 15: Supporting structure of the cryostat. For further explanations, see text.
The $^3$He circulation system consists of an Edwards 18B4 oil booster pump F, which is backed by an EH-1200 mechanical booster and an EH2M80 rotary pump in series. This combination can maintain a $^3$He circulation rate of more than 3000 μmole/s.

4.3 Dilution refrigerator

Dilution refrigeration is the only way to maintain continuously a temperature in the low millikelvin region. Since the mid sixties when the technique was invented, the designs have improved considerably. The most significant step forward was the sintered heat exchanger using fine silver powder, typically with a grain size of 700 Å (Frossati, 1978). Nowadays, the lowest temperature that can be reached is about 2 mK (Vermeulen and Frossati, 1987; Bunkov, Guénault, Hayward, Jackson, Kennedy, Nichols, Miller, Pickett, and Ward, 1991). In commercial machines, 5 mK can usually be guaranteed.

For a dilution refrigerator used as the precooling stage in a nuclear demagnetization cryostat, the important requirement is a high cooling power around 10 mK to remove the nuclear entropy. Attention should be paid also to the overall structure to make it as rigid as possible for steady support of a metallic nuclear stage which can weigh as much as 10 kg.

The dilution refrigerator used in our machine was designed and manufactured by Leiden Cryogenics b.v. (Fig. 16). Most parts, such as the 1-K pot, the still, and the heat exchangers are quite similar to those described earlier (Frossati, 1978; Vermeulen and Frossati, 1987). There are some changes in the construction of the heat exchangers to make them less susceptible to leaks. The mixing chamber is of special design to ensure a high cooling power outside as well. The following summarizes the main features of the dilution refrigerator.

The pot with a volume of 300 cm$^3$ surrounds the still pumping tube (see Fig. 14). It is thermally isolated from the bath by the vacuum enclosure extending above the IVC top flange. There are two filling lines in parallel; the one with a lower impedance can be closed by a needle valve. The two impedances are chosen such that when $^3$He circulation is larger than 200 μmole/s, both filling lines are open. When the circulation is lower than 200 μmole/s, one of the lines is closed to decrease liquid helium consumption. There are two $^3$He condensing lines in parallel, for high and low circulations, respectively. The former can be closed by another low temperature valve. A capacitive level gauge is mounted inside the 1 K pot for monitoring the liquid level.

The still is hung from the low end of its pumping line. In the original design, the whole device below the still, which weighs about 20 kg, was suspended on the $\phi$ 20 × 0.7 mm diameter stainless steel tube. This section at first was the most flexible part of the apparatus and the parts below this point could easily swing. After four extra supports were added between the still and the 4-K flange, the situation was much improved. There is a capacitive helium level gauge in the
Figure 16: Dilution refrigerator (Leiden Cryogenics b.v.).
still as well. This is useful for determining the right amount of liquid inside the dilution machine.

The construction of the heat exchangers is the critical part of a dilution refrigerator. There are quite some publications on their design (Frossati, 1978; Vermeulen and Frossati, 1987; Bunkov et al., 1991; Oda, Fujii, and Nagano, 1978; Oda, Fujii, Ono, and Nagano, 1983). In our machine, there are eight heat exchangers below the 50 mK plate. On the concentrated side, there is 800 g of silver powder, and on the dilute side 1200 g, which correspond to surface areas of 1600 m$^2$ and 2400 m$^2$, respectively.

![Diagram of Mixing Chamber Design]

Figure 17: Mixing chamber design.

The structure of the mixing chamber is shown in Fig. 17. The copper body, including the four copper feet, was machined of a single piece of metal. Slots cut inside were covered with 900 g of sintered silver powder to increase the contact area between the liquid and the copper body. The mixing chamber has two feedthroughs for placing devices inside the liquid directly. A 10 mm thread was tapped in each foot for fixing the cold plate.

The dewar (Precision Cryogenic Systems, Inc.), thermally isolated with a multi-layer radiation shield, does not use liquid N$_2$. Its large liquid helium belly has a volume of about 100 liters. The holding time for liquid depends on the circulation rate in the dilution refrigerator. At 200 $\mu$mole/s, 100 liters of helium lasts for 4 days. This long time interval reduces the disturbances coming from
helium transfers, especially while the second stage is being precooled in a high magnetic field.

The performance of the dilution refrigerator can be judged from the minimum temperature and from the cooling power (see Fig. 18). The main part of the figure shows the minimum temperature as a function of the circulation rate. The lowest temperature measured inside the mixing chamber by the vibrating wire viscometer (see § 5.1) is 2.8 mK. The inset in Fig. 18 shows the temperature with external heating. At 10 mK, the cooling power is 10 μW inside the mixing chamber at the circulation rate of 1500 μmole/s. Considering the very high cooling power at 10 mK, our dilution refrigerator is one of the most powerful machines ever built.

Figure 18: Lowest temperature of the mixture inside the mixing chamber as a function of the $^3$He circulation rate, with and without external heating.

4.4 Nuclear stage

The first nuclear stage is made of 170 moles of high purity copper. It consists of three parts: the copper rod, the supporting tube, and the top flange (see Fig. 19). The pieces were connected by electron beam welding. Next, 22 slits, 0.4 mm wide, were cut in parallel along the axis by a spark cutter. The slits started 2 mm above the bottom of the nuclear stage and stopped below the top flange, so that the copper plates were connected at both ends. In the lower end, another ten grooves were cut perpendicular to the main slits, extending upward
for 10 mm. This scheme ensures that no plate is thicker than 3 mm in the high field region. Copper used for the main part of the nuclear stage was from Hitachi Cable, Ltd. with an analyzed purity of 4N+. The material for the top flange and the supporting tube was from Outokumpu Poricopper Oy., specified as cryogenic copper of high conductivity.

![Diagram of nuclear stage]

Figure 19: Composition of the nuclear stage.

The reason for such a design of the nuclear stage is to ensure efficient cooling of the second stage, to be located on top of the middle part of the main nuclear stage, as an extension of the copper rod (see Fig. 14). Above the electron-beam welded joint of the rod and the tube, these two parts do not touch each other. The copper tube thus works as an intermediate stage between the top flange and the main nuclear stage. The heat load from the parts attached to the flange, i.e. thermometers, supporting tubes, and the heat switch, is absorbed by the heat capacity of the copper tube before reaching the main part of the nuclear stage. The temperature at the site where the sample is connected thus may be lower than at the top flange.

The whole nuclear stage was annealed at 960 °C in a vacuum tube oven for 100 h with air at $1 \times 10^{-3}$ mbar flowing through during the heat treatment. A test piece, which was cut from the main rod, was put inside the oven, too. An
Table 2: Impurity concentration of the copper rod according to the manufacturer’s analyses (Hitachi Cable, Ltd.)

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Bi</th>
<th>Cd</th>
<th>Pb</th>
<th>O₂</th>
<th>P</th>
<th>Se</th>
<th>S</th>
<th>Te</th>
<th>Zn</th>
<th>Sb, As, Mn, Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>ppm</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
<td>4</td>
<td>3</td>
<td>2</td>
<td>&lt; 1</td>
<td>9</td>
<td>&lt; 1</td>
<td>&lt; 1</td>
<td>9</td>
</tr>
</tbody>
</table>

$RRR$ measurement gave a value of $1500 \pm 100$ after annealing. Some pieces of Outokumpu copper were also annealed in a similar way; their $RRR$ rose to 500.

Before the heat treatment, $\text{Al}_2\text{O}_3$ wool was put inside the slits to prevent the copper plates from sticking together during the high temperature annealing process. The wool, being suspected of becoming a source of time dependent heat leaks at $\mu$K temperatures, was removed after several cooling cycles of the nuclear stage.

4.5 Heat switch

Superconducting heat switches are used exclusively at temperatures below 0.3 K (Lounasmaa, 1974). The performance of such a switch relies on the high ratio of thermal conductivity between the normal and the superconducting states, $\lambda_n/\lambda_s$. Commonly used materials are aluminum (Mueller et al., 1978; Lawson, 1982; Bunkov, 1989), tin (Ehnholm et al., 1980), zinc (Krusius, Paulson, and Wheatley, 1978), and lead (Van de Haar, 1991). Among them, Al has the largest $\lambda_n/\lambda_s = 1600 T^{-2}$ (Mueller et al., 1978). Aluminum also has the smallest critical field of 10 mT for the superconducting transition.

There is an easy way to estimate the conductance of a heat switch in its normal state without doing the actual measurement in the low mK region. The thermal conductivity $\lambda$ of a metal and its electrical conductivity $\sigma$ follow the Wiedemann-Franz law,

$$\lambda/\sigma = L_0 T,$$  \hspace{1cm} (4.2)

where the Lorentz number $L_0 = 2.45 \times 10^{-8}$ $\text{W} \Omega/\text{K}^2$ is a universal constant. Therefore, by measuring the residual resistance at 4.2 K, one can deduce the thermal conductance from Eq. (4.2). For a heat switch, $\lambda$ should be compatible with the size of the nuclear stage and the cooling power of the dilution refrigerator. In our case, the cooling power at 10 mK is about $9 \mu\text{W}$. If we assume 1 mK for the temperature difference across the switch, the thermal conductance should be on the order of $0.9 T W/\text{K}^2$ and the residual resistance $R$ should be around 30 n$\Omega$.

A low temperature resistance in the n$\Omega$ region is not difficult to achieve with either Cu or Al pieces of good purity and proper annealing. The technical difficulty in constructing an aluminum heat switch is that a good contact between Cu and Al is hard to achieve because of the oxide layer on aluminum.
Diffusion welding was used in our construction of the switch (see Fig. 20). First, all copper and aluminum pieces were annealed in a vacuum oven. The $RRR$ of the aluminum foils and copper pieces were then 1600 and 500, respectively.

Figure 20: Construction of the heat switch. (A) Diffusion joint between copper and aluminum was made in a vacuum oven. A 5400 N force was applied onto the copper rod through anvils. (B) Ten copper plates were electron beam welded onto the copper rods.

Eight pieces of 0.25 mm thick aluminum foils were fitted inside the slits cut into the copper pieces. Diffusion welding was done in a special vacuum oven. During welding, a force of 5400 N was put onto the copper pieces through anvils to squeeze copper and aluminum together. The diffusion process took place at a temperature of 550 °C for 30 min. A larger force could have resulted in a better contact, but the copper pieces were so soft after annealing that they could not sustain more force.

The mechanical properties of the weld would also benefit from a longer diffusion time and higher temperature. But in our special case, this would have increased the contact resistance because of the Cu-Al alloy formed at the interface of the joint. Some tests showed that at a temperature of 550 °C, the process should not be much longer than 15 min.

The residual resistance of our switch is 80 nΩ, which corresponds to a thermal conductance of 0.3 $T$ W/K$^2$. This high value still prevents the large cooling power of the dilution refrigerator from being fully realized, although our switch has the best conductance ever reported.
There are other ways to join aluminum with copper or silver. Mueller et al. (1978) described a method where the oxide layer is first removed and then prevented from rebuilding by gold plating. Then, copper plates and the gold plated aluminum foils are pressed together by bolts. A conductance of 0.1 T W/K² was achieved. A similar method has been used in constructing the heat switches of the Bayreuth machines (see § 3.4). Lawson (1982) described a way to squeeze silver wire into a monolithic aluminum block by current (see Fig. 10). A typical resistance of such a joint is 0.5 μΩ which is equivalent to a thermal conductance of 0.05 T W/K². Lancaster scientists use this method for their heat switches (see § 3.3).

Impact welding has been reported for joining copper and aluminum (see Willekers, Bosch, Mathu, Meijer, and Postma, 1989). If the thermal conductance of such a joint can be scaled to the geometry of our switch, it has the value of 6.1 T W/K². So far, this method has not been reported in actual use to construct a real heat switch.

4.6 Superconducting magnets

4.6.1 Magnet for the first nuclear stage

Our first stage magnet was manufactured by Cryomagnetics, Inc. It has an 82 mm bore with a full field of $B_0 = 9$ T. The upper part of the coil has compensation windings which make the field above the top flange of the nuclear stage smaller than 5 mT at full current. An accessory coil can be added to the lower end of the main solenoid to compensate the field in this part as well if some experiments have to be installed below the first stage. Of the 170 moles of copper in the nuclear stage, the effective mass is 97 moles calculated from the formula

$$n_{\text{eff}} = \int_z \frac{B(z)^2}{B_0^2} n(z) A(z) \, dz,$$

where $n(z)$ is the molar density and $A(z)$ is the cross-sectional area of the nuclear stage at $z$. The magnet can be persisted.

4.6.2 Magnet for the second nuclear stage

Our second stage magnet is home made. It is located inside the vacuum jacket (see Fig. 14), hanging below the still. In the design stages of the cryostat, the performance of such a magnet was rather uncertain because it was intended to work in vacuum without stabilization by liquid helium.

Brass was used for the coil former. The magnet has 51 layers of windings in the main solenoid and several compensation windings in its lower part. The solenoid was wound using a single piece of superconducting wire (Vacuumschmelze GmbH), 0.5 mm in diameter with 54 NbTi filaments inside the copper matrix.
Figure 21: Scheme for vacuum impregnation of a magnet by epoxy.

After winding, the coil was put inside the device shown in Fig. 21 for impregnation with epoxy. The resin was first mixed in vessel B. The tube connecting A and B could be lifted above the epoxy level, therefore both containers could be evacuated by a vacuum pump at the same time. After the air dissolved in the epoxy had come out, tube in B was pushed down into the resin. Air was let into B so that atmospheric pressure would drive the epoxy into A. When the magnet was completely immersed in resin, air was let into A as well. Atmospheric pressure then forced the epoxy into the very small voids between the windings.

Vessel A, with the coil inside, was then put into an oven, heated up to 50 °C in order to decrease the viscosity of the resin and to speed up the impregnation process. After 5 h, the coil was lifted out, with the extra resin dripping down. The coil was then put back into the oven and the epoxy was hardened and cured at a temperature of 140 °C for 8 h. The coil was very tightly wound to avoid large amounts of epoxy between wires. The recipe for mixing the epoxy is given in Table 3 (Paloniemi). It is not specially intended for low temperature use but for gluing the windings of conventional electric motors. Compared with Stycast 1266, which is commonly employed at low temperatures, the epoxy we used has a much longer casting time.

The magnet was tested both in liquid helium and in vacuum before it was fixed into place. In 4.2 K, 78 A were fed into the coil, producing a field of 8.3 T without going normal. In vacuum, the magnet quenched when the current was 73 A. No training effects were observed despite several superconducting to normal
Table 3: Recipe for mixing the epoxy

<table>
<thead>
<tr>
<th>Component</th>
<th>Viscosity (25°C) (mPa·s)</th>
<th>Viscosity (60°C) (mPa·s)</th>
<th>percent (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resina</td>
<td>5000 ± 500</td>
<td></td>
<td>52.3</td>
</tr>
<tr>
<td>Hardenerb</td>
<td>55 ± 10</td>
<td>10 ± 2</td>
<td>47.2</td>
</tr>
<tr>
<td>Acceleratort</td>
<td></td>
<td></td>
<td>0.5</td>
</tr>
</tbody>
</table>

a Rütapox-Epoxy, Resin 0162, Bakelite GmbH  
b Rütapox-Epoxy, Härter HM, Bakelite GmbH  
t DMP-30, Rohm and Haas, Philadelphia

transitions. Thermometers fixed at various positions on the magnet showed that the current leads warmed up quickly just before quenches. The transition at a lower current in vacuum might have been triggered by the rather poorly connected current leads in the test setup. When the magnet was in its place, we never put more than 70 A into it to keep some safety margin from a quench. In the center of the magnet, the field is 7.5 T with 70 A in the coils.

The current can be persisted by a switch which is located in the ⁴He bath. The scheme for making the switch is similar to that described by Uchiyama and Mamiya (1987), except that superconducting wire with low conducting CuNi matrix is used, so that it was not necessary to remove the matrix material. The two superconducting joints, connecting the persistent switch and the magnet, were made by compression and are located inside the vacuum can. The performance of the joints and the switch was tested by keeping the magnet in the persistent mode with a 70 A current for two days. No decay was observed during this time.
5 Thermometry

When a new cryostat is being built, many sensors are required to measure the temperatures at various places in order to diagnose the problems and to test the performance. In our refrigerator, we have to measure the temperature from 300 K down into the μK region. Fig. 22 summarizes the methods of thermometry we use.

![Thermometry Diagram]

**Figure 22:** Methods for temperature measurement in the new cryostat.

A commercial platinum resistance thermometer, Pt-100 is employed to measure the temperature of the cryostat above 10 K. Its resistance decreases almost linearly from 300 K to about 50 K. The resistances at 273 K, 77 K, and 4.2 K are 100 Ω, 20 Ω, and 1.2 Ω, respectively. A digital multimeter is employed to measure the resistance, using the 4-wire technique. Pt-100 is a cheap and convenient thermometer for monitoring precooling from room to liquid helium temperature.

Sliced Speer carbon resistors were fixed to the 1 K pot, still, 50 mK plate, and the mixing chamber. They were compared against a commercial germanium thermometer (Lake Shore Ltd.) which had been calibrated between 10 K and 50 mK by the manufacturer. Below 50 mK the resistance increases very quickly to 1 MΩ and can no longer be measured accurately. Sliced carbon resistors work quite well to 10 mK if they are carefully shielded against rf disturbances. A special low temperature filter for this purpose was designed. The resistance of
a germanium thermometer is sensitive to magnetic field, so the sensor is protected by a superconducting Nb shield. The influence of magnetic field to the performance of a carbon resistor is negligible (Sample and Rubin, 1977).

In this chapter, I will discuss four thermometric methods in detail, i.e., the vibrating wire thermometer, the nuclear orientation thermometer (NO), the platinum pulsed NMR thermometer (PLM), and Coulomb blockade thermometer. A vibrating wire, placed inside the mixing chamber, measures the temperature of the helium liquid directly and is very useful in diagnosing problems encountered in performance tests of a dilution refrigerator. The NO thermometer, used as a primary standard, was employed to calibrate the Pt NMR thermometer which was exclusively used to measure temperatures below 1 mK. The Coulomb blockade thermometer, using nanoscale tunnel junctions, is recently under developing. It is a promising method which covers a large temperature range from 40 K to 20 mK.

5.1 Vibrating wire viscometer

About 20 years ago, measurements by a vibrating wire made it clear that the newly discovered phases in $^3$He were superfluid states (Alvesalo et al., 1973). The viscosity of Fermi liquids, i.e., pure $^3$He or $^3$He/$^4$He mixtures, have well defined temperature dependences in the degenerate low temperature limit. A vibrating wire viscometer can thus work as a thermometer. This method was mainly developed by the group in Lancaster (Bradley and Oswald, 1990, and the references therein).

We installed two wires inside the mixing chamber for monitoring the performance of the dilution refrigerator. Fig. 23 shows the two resonators and the measurement setup. The viscometers were constructed by bending a 0.125 mm diameter tantalum wire into a U-shaped loop with its two ends glued to the holders by epoxy. Voltage and current leads were connected to the tantalum wire by pressed joints. A superconducting magnet produces a 50 mT field parallel to the plane of the loop. When a small alternating current is passed through the wire, the Lorentz force excites a mechanical flopping mode, with the induced voltage across the loop being proportional to the speed of oscillation. The in-phase component shows a Lorentzian lineshape (see Fig. 24), while the linewidth of the resonant curve is a measure of damping on the wire. The Q-value of the resonator is determined by energy dissipation which mainly arises from friction with the viscous fluid around the wire.

By sweeping the frequency of the AC current slowly through the resonance, one can measure typical curves shown in Fig. 24. The half width of the in-phase signal yields the viscosity and consequently the temperature of the liquid. In principle, the quadrature signal could be used as well to deduce the same information.

A vibrating wire thermometer has several favorable properties.
Figure 23: Setup for our vibrating wire viscometer.

- It probes excitations in the liquid, thus measuring the temperature directly, thereby avoiding problems caused by the Kapitza resistance.

- Viscosity measurements are insensitive to magnetic field, which is an obvious advantage in a nuclear demagnetization cryostat. This feature is due to the fact that damping by magnetic field, which is caused by flux jumps in the superconductive tantalum wire, is rather small and can be neglected at temperatures below 100 mK.

- The construction of a resonator is very simple. The resonant frequency of a resonator is determined by the properties of the wire, the shape and the diameter of loop, and also slightly influenced by the viscosity of the liquid. But none of these factors affects the accuracy in the half width measurement.

- Time constant for the measurement is short.

- Energy dissipation during measurements can be as low as picowatts. In the mixing chamber this has a negligible effect on the temperature.
Figure 24: In-phase and quadrature signals of a vibrating wire. The solid line in (a) is a Lorentzian fit to the data.

The theory of an infinitely long cylinder moving in a viscous liquid is due to Stokes (Stokes, 1901; Retsina, Richardson, and Wakeham, 1987). In the viscous regime, the mean free path \( l \) of the quasiparticles is much shorter than the wire radius \( r_w \), and the description of motion is based purely on hydrodynamics. When \( r_w \) is much smaller than the radius of the loop, the infinite length approximation is valid. The motion of such a wire is described by the equation of a driven damped harmonic oscillator, \( \text{viz.} \)

\[
 m\ddot{x} + \Gamma \dot{x} + m\omega_0^2 x = F_0 e^{i\omega t} + F_{\text{visc}},
\]

(5.1)

where \( m \) is the mass per unit length of the cylinder, \( \Gamma \) describes the intrinsic damping, \( m\omega_0^2 x \) is the restoring force due to the elasticity of the wire, \( F_0 e^{i\omega t} \) is the driving force per unit length, and \( F_{\text{visc}} \) is the viscous damping force per unit length. In our temperature range, the intrinsic damping is much smaller.
than that caused by the viscous liquid. In the following discussion we neglect the former effect.

The term $F_{\text{visc}}$ is related to the viscosity of the liquid. Stokes' result can be written (Stokes, 1901; Retsina et al., 1987)

$$F_{\text{visc}} = -\omega m'[k'(\gamma) + ik(\gamma)]\dot{x}$$

$$\equiv -m(\lambda_2 + i\lambda_1)\dot{x}, \tag{5.2}$$

where $m' = \rho_n \pi r_w^2$ is the mass of the fluid replaced by unit length of the wire and $\rho_n$ is the density of the liquid. The dimensionless parameter $\gamma$ is the ratio of the wire radius and the viscous penetration depth $\delta$, $\gamma = r_w/2\delta$, and

$$\delta = \left(\frac{\eta}{\rho_n \omega}\right)^{1/2}. \tag{5.3}$$

For steady state motion, the velocity of the wire has the form

$$v(t) = \dot{x} = v_0 e^{i\omega t}. \tag{5.4}$$

Substituting this into Eq. (5.1), gives

$$v_0 = C \left(\frac{i\omega}{\omega_0^2 - \omega^2 - \omega\lambda_1 + i\omega\lambda_2}\right), \tag{5.5}$$

where $C$ is a constant which depends on $F_0$ and $m$. The in-phase and out-of-phase components correspond to the two curves in Fig. 24. The former has the same phase as the driving force, hence it causes energy dissipation. The quadrature component represents the inertia of the liquid. When $\omega_0 \gg \lambda_1, \lambda_2$, the real component has the simple form

$$\text{Re}(v_0) \simeq \frac{\lambda_2}{\lambda_2^2 + 4(w - w_0)^2}, \tag{5.6}$$

where the half width of the Lorentzian curve $\Delta w = \lambda_2$. And, according to Eq. (5.2),

$$\Delta f = \frac{\Delta w}{2\pi} = \frac{\lambda_2}{2\pi} = \frac{w}{2\pi m} k'(\gamma) \simeq f_0 \frac{\rho_n k'}{\rho_w}. \tag{5.7}$$

If higher-order terms are included into this equation, one obtains (Black, Hall, and Thompson, 1970),

$$\Delta f = f_0 \frac{\rho_n k'}{\rho_w} \left(1 - \frac{3}{2} \frac{\rho_n k'}{\rho_w}\right) \left(1 - \frac{1}{2} \frac{\Delta f}{k'} \frac{\partial k'}{\partial f} + \cdots\right), \tag{5.8}$$
where the terms in the second set of parentheses allow for a change of \( k \) over a broad resonance. Expanding to the first order in \( k \) gives

\[
\Delta f = f_0 \frac{\rho_n k'}{\rho_w} (1 - 1.14 \frac{\rho_n k}{\rho_w}) .
\]

A fit of the in-phase curve by the Lorentzian function of Eq. (5.6) then yields \( f_0 \) and \( \Delta f \). By using Eq. (5.9) iteratively, one can obtain \( k(\gamma) \) and \( k'(\gamma) \). The corresponding \( \gamma \) is then found from a table calculated by Stokes (1901). The viscosity \( \eta \) is obtained from Eq. (5.3). For a saturated solution, \( \eta \) is given below 80 mK by the relationship

\[
\eta T^2 = a + bT ,
\]

with \( a = (2.7 \pm 0.2) \cdot 10^{-8} \text{ Pa} \cdot \text{s} \cdot \text{K}^2 \) and \( b = (34 \pm 3) \cdot 10^{-8} \text{ Pa} \cdot \text{s} \cdot \text{K} \) (Zeegers, de Waele, and Gijsman, 1991). Thus the temperature of the liquid can be calculated.

When the wire moves in a \(^3\text{He}/^4\text{He} \) mixture, \( \rho_n \) is the fluid density of \(^3\text{He} \), because the superfluid \(^4\text{He} \) does not interact with the wire. In a saturated solution, the concentration of the normal \(^3\text{He} \) is given by

\[
x_s = 0.066 + \sum_{i=2}^{5} D_i T^i ,
\]

where \( D_i \)'s are constants (Kuerten, Castelijns, de Waele, and Gijsman, 1985). At typical mixing chamber temperatures, the density of \(^3\text{He} \) is (Zeegers et al., 1991)

\[
\rho_3 = 0.1437 + 104.5 \ x_s \ (\text{kg/m}^3) .
\]

With the effective mass \( m^* = 2.46 \ m_3 \), where \( m_3 \) is the mass of a \(^3\text{He} \) atom,

\[
\rho_n = 2.46 \ \rho_3 .
\]

For a Fermi liquid, \( l \) increases at low temperatures as \( T^{-2} \). In the ballistic regime where \( l \gg r_w \), the relations based on local hydrodynamics break down. In a saturated solution, the transition from viscous to ballistic regime starts around 3 mK, and below 0.3 mK the liquid is purely ballistic (Guénault, Keith, Kennedy, and Pickett, 1983; Guénault, Keith, Kennedy, Mussett, and Pickett, 1986). For our dilution refrigerator, the minimum temperature is slightly below 3 mK, so a small correction to the Stokes’ theory is needed at the lower end of temperatures.

In between the viscous and ballistic regimes, “slip” of the fluid at the surface of the wire can be accounted for by redefining \( k \) and \( k' \) as functions of the original
Figure 25: Comparison between the magnetization $M_n$ recorded by a pulsed Pt NMR thermometer and $1/T_{v,w}$ measured by the viscometer.

$k_0$ and $k'_0$ (Højgaard Jensen, Smith, Wölflé, Nagai, and Maack Bisgaard, 1980) as

$$k = 1 + \frac{k_0 - 1}{(1 + \gamma^2 \beta k'_0)^2 + \gamma^4 \beta^2 (k_0 - 1)^2} ,$$

(5.14)

$$k' = \frac{k'_0 + \gamma^2 \beta [(k_0 - 1)^2 + k'_0^2]}{(1 + \gamma^2 \beta k'_0)^2 + \gamma^4 \beta^2 (k_0 - 1)^2} ,$$

(5.15)

where

$$\beta = \frac{0.579 \bar{l}}{0.579 \bar{l} + \bar{r}_w} .$$

(5.16)

It has been observed that a modification to this somewhat empirical definition of $\beta$ will make the fit in the transition region even better (Guénault et al., 1983; Guénault et al., 1986; Careless, Hall, and Hook, 1983). In our calculation, we use the form suggested by Guénault et al. (1983), viz.

$$\beta = \frac{0.579 \bar{l} + 10 \alpha d/r_w}{r_w \bar{l} + 1 + 10 \bar{l}/r_w} ,$$

(5.17)

with the "fudge" factor $\alpha = 2.2.$
Figure 26: Comparison of signals when the wire was inside the $^3$He dilute or concentrated phase. The curves were measured around 10 mK.

The vibrating wire thermometer was compared with the calibrated germanium resistor. Between 70 and 50 mK, the agreement was better than 5%. The wire may be used at higher temperatures as well, up to 200 mK, where the temperature dependence of viscosity is known. Between 100 and 200 mK, the half width of the Lorentzian curve becomes so small that the wire’s intrinsic damping has to be taken into account. Flux jumps in the superconducting tantalum wire may increase the half width as well. These effects are independent of temperature and should be subtracted. At temperatures below 70 mK, they can be neglected.

In the low temperature limit, the vibrating wire and the Pt NMR thermometer were compared. Fig. 25 shows the linear relationship of the measured magnetization $M_n$ of platinum and the inverse temperature, $1/T_{v,w}$, measured by the wire.

A vibrating wire can additionally be used to locate the position of the phase boundary inside the mixing chamber. This information is very useful for tuning a dilution refrigerator. The viscosity of pure $^3$He is much larger than that of the dilute mixture at temperatures below 50 mK. A sudden increase in the half width can be seen at the moment when the upper vibrating wire in Fig. 23 gets inside the $^3$He rich phase while $^3$He is being added into the circulating gas (see Fig. 26).
5.2 $^{60}$Co nuclear orientation thermometer

The nuclear orientation (NO) thermometer is a primary standard (Berglund, Collan, Ehnholm, Gylling, and Lounasmaa, 1972; Soulen and Marshak, 1980; Marshak, 1986). It makes use of the spatial anisotropy of $\gamma$-ray emission from polarized nuclei to indicate the temperature of the host lattice. For the $^{60}$Co single crystal probe we used, the emitted $\gamma$-ray intensity in the direction of polarization, as a function of temperature, can be expressed by

\[
W(0, T) = 1 - 0.047619Q_2 \left[ \sum_{m=-I}^{I} m^2 P(m) - 10 \right] \\
- 0.003968Q_4 \left[ \sum_{m=-I}^{I} m^4 P(m) - 25 \sum_{m=-I}^{I} m^2 P(m) + 72 \right]
\]

(5.18)

where $Q_2$ and $Q_4$ are the solid angle corrections due to the finite size of the detector, and $P(m)$ is the Boltzmann relative population for the polarized nuclei, viz.

\[
P(m) = \frac{\exp(-E_m/k_B T)}{\sum_{m=-I}^{I} \exp(-E_m/k_B T)}.
\]

(5.19)

The internal field of the hcp ferromagnetic host is −21.90 T in a needle-shaped single crystal, measured by the NMR technique (Hagn, 1982). Thus the splitting of the Zeeman levels is 6.07 mK in this system. The physical size of the probe is $7 \times 1 \times 0.7$ mm$^3$, with its longest dimension along the crystalline c-axis (Oxford Instruments, Ltd.). The half life of $^{60}$Co is 5 years. When it was received in 1995, the strength of our source was 5 $\mu$Ci. The probe is mounted in the experimental region, where the field is smaller than 5 mT, in order to avoid reorientation of the magnetic domains. The single crystal was soldered onto a copper plate, which in turn, was screwed to the place where temperature was to be measured. A low temperature solder was used to ensure that the temperature of the crystal was never raised above 250 °C. At 390 °C the crystal undergoes a phase transition which changes its symmetry from hcp to fcc, thereby changing the magnetization.

A block diagram of the measuring instruments is shown in Fig. 27. The scintillation detector is a $3'' \times 3''$ NaI(Tl) crystal, located outside the dewar, 22 cm away from the source. The signal is fed into the computer through the plug-in multichannel analyzer card which utilizes gain stabilization to compensate for small drifts during a measurement. This can be done by specifying a peak in the previously measured spectrum. A new recording then always tries to fix the specified peak to the same position. Fig. 28 shows a typical spectrum. The raw
Figure 27: Experimental setup for the $^{60}\text{CoCo}$ nuclear orientation thermometer.

data are fitted with two Gaussians corresponding to the two $\gamma$-ray energies. A linear background correction is applied. The fitting function has the form

$$C(E) = aE + b + \frac{A_1}{\Gamma_1 \sqrt{\pi}/2} \exp \left[ -\frac{2(E - E_1)^2}{\Gamma_1^2} \right] + \frac{A_2}{\Gamma_2 \sqrt{\pi}/2} \exp \left[ -\frac{2(E - E_2)^2}{\Gamma_2^2} \right].$$

(5.20)

The temperature can then be deduced from

$$W(0, T) = \frac{C - C_b}{C' - C'_b} = \frac{A_1 + A_2}{A'_1 + A'_2},$$

(5.21)

where $C_b$ denotes background counts and the symbols with primes correspond to the high temperature values when the $\gamma$-ray distribution is isotropic. The solid angle correction factors for our geometry are $Q_2 = 0.982$ and $Q_4 = 0.945$ (Berglund et al., 1972). For a $^{60}\text{CoCo}$ probe, the thermometer has the highest sensitivity around 7 mK. The usable region for temperature measurements is between 3 and 30 mK with reasonable counting times.

At low temperatures, the heating induced by radioactivity limits the use of the NO thermometer. A weak source reduces this problem but the counting time then becomes longer to suppress the statistical error. For a 5 $\mu$Ci source, the rate is about 100 counts/s. One measurement usually takes 10 to 30 minutes.

Heating originates from two sources, $\dot{Q}_\beta$ and $\dot{Q}_\gamma$. The first term is due to electrons from $\beta$-decay, which have a penetration depth of 0.1 mm in a metal and which are completely absorbed by the crystal. With an average $\beta$-ray energy of 95.8 keV (Marshak, 1986), $\dot{Q}_\beta = 2.9$ nW with a 5 $\mu$Ci source. $\dot{Q}_\gamma$ is due to $\gamma$-rays which are very penetrating and have no heating effect on the source itself. Fractional absorption of $\gamma$-rays may still produce heating in the surroundings.
Figure 28: Measured γ-ray spectrum from $^{60}$Co in hcp cobalt over 15 min.

depending on the geometry. A curve is available to estimate this effect (Marshak, 1986). When the probe is in its usual place on the bottom plate of the mixing chamber, the upper limit of the γ-ray heating effect to the nuclear stage is 1 nW which may be significant.

5.3 Platinum NMR thermometer

NMR is the only method available at present for thermometry below 1 mK. The nuclear magnetization $M_n$ of platinum is measured and the temperature is deduced from Curie's law

$$M_n = \frac{\lambda_n B}{T}.$$  \hspace{1cm} (5.22)

This technique has been described in detail in two books (Lounasmaa, 1974; Pobell, 1992). Usually, $^{195}$Pt is chosen as the probe nuclei for two reasons: platinum has a small Korringa constant, 30 msK, resulting in a quick spin-lattice relaxation, and a relatively long spin-spin relaxation time $\tau_2$, about 1 ms, which makes measurements easier. Our setup for a platinum wire pulsed NMR thermometer is shown in Fig. 29.

The static field is produced by a small superconducting solenoid wound of 0.1 mm diameter wire. The coil former is thermally anchored to the mixing chamber and does not touch the probe. The setup uses two niobium shields.
The outer is a Nb tube, which screens the stray field from the environment, while the inner one is a piece of insulated Nb foil wrapped on the coil former and glued with epoxy underneath the static field coil. Owing to its insulation, the Nb foil acts as an open superconducting shield which homogenizes the static field inside (Hechtfischer, 1987). By using two Nb shields, one can produce a rather homogeneous field at the site of the NMR probe, without paying too much attention to the winding accuracy. The coil is a simple solenoid of six uniform layers without compensation windings. It produces fields up to 28 mT needed for NMR on platinum at a frequency of 250 kHz. The measured time constant \( \tau'_2 \) for the free induction decay is 0.8 ms. The homogeneity of the magnetic field can be estimated from

\[
\frac{1}{\tau'_2} = \frac{1}{\tau_2} + \frac{\gamma_n}{2\pi} \delta B ,
\]

where \( \gamma_n \) is the gyromagnetic ratio of platinum nuclei. We obtain \( \delta B / B = 1 \times 10^{-3} \) over the sample. In order to avoid the influence of possible ripple from the power supply, the current in the solenoid can be persisted.

Two probes were made using 0.025 mm platinum wire from different sources. One was from Reactor Experiments, Inc. with a specified purity of 99.999%. The other, whose supplier is unclear, was from old stock in our laboratory. Both were annealed in air for 15 min, one at 800 °C at ambient pressure and the other at 400 °C with \( P_{\text{air}} = 5 \times 10^{-4} \) mbar, resulting in RRR of 1300 and 270, respectively. However, no essential differences in the performance of the two probes were observed. A brush consisting of 2000 platinum wires was fixed to a silver foot by electron beam welding. The electron beam was focused on silver which melted, with some of the metal flowing onto the platinum brush. Owing to the large difference in the melting temperatures of Pt and Ag, we expect that very little of the Ag-Pt alloy was formed. On the other hand, a small and localized heating may not melt enough silver to make contact to all of the 2000 wires. Loose ends may be poorly linked to the silver piece. This will be discussed in § 6.3.

The silver piece was annealed for 13 hours at 800 °C in 2 \times 10^{-4} \) mbar of O₂, resulting in an \( RRR \) of 1200. The probe was fixed with a 5 mm copper screw to the top flange of the nuclear stage.

The rf excitation coil was made of 25 \( \mu \)m insulated copper wire which was first wound onto a teflon rod of 3 mm in diameter. A drop of dilute GE varnish was used to glue the windings together. After the varnish was dry, the teflon rod was pulled out. The coil usually had about 1000 turns with a total length of 6 mm and an inductance around 800 to 900 \( \mu \)H. It was pushed over the platinum brush and fixed with a tiny amount of diluted GE varnish. The two leads for the coil were twisted together and glued onto the silver foot at several places.

We use the commercial PLM-4 instrument (Picowatt RV-Elekroniikka OY.) whose frequency can be set to 125 kHz, 250 kHz, or 500 kHz, with 125 kHz being
our usual operating frequency. The tank circuit has a ceramic 1 nF capacitor fixed to the mixing chamber plate. The coaxial cable connecting the probe and the preamplifier adds 100 pF of capacitance to the system. The final tuning of the resonant circuit is by means of an adjustable capacitor of 50 pF in the preamplifier of PLM-4. The Q-value of the circuit is 6.

The transmitter pulse heats the probe and thus increases its temperature. At low temperatures the length of the pulse should be shorter. Fortunately, the signal simultaneously becomes larger since $M_n$ increases at lower temperatures and, hence, the signal to noise ratio improves. A pulse length of 0.16 ms was used above 0.5 mK. Below this temperature, the pulse was 8 µs long, which is the shortest length available at 125 kHz using PLM-4. The amplitude of the transmitter pulse was set to 2 V.

The heating due to a pulse may originate from three sources: 1) Joule heating
caused by the finite resistance of the Cu excitation coil, 2) eddy current heating in the Pt wires and other metallic parts nearby, and 3) the NMR absorption.

The Joule heating of the pickup coil turns out to be on the order of 0.2 nJ.

The increase in the spin temperature due to NMR absorption can be estimated from the change of magnetization, \textit{viz.}

\[
Q = \Delta M_n \cdot B \\
= M_n (1 - \cos \theta) B \\
\simeq \frac{1}{2} \theta^2 M_n B .
\]  

(5.24)

The tipping angle \( \theta \) as a function of the transmitter pulse length was measured. In our setup, an 8-\( \mu \)s pulse is equivalent to \( \theta = 0.8^\circ \). The corresponding heat input is rather small, on the order of 40 fJ.

The heating due to eddy currents induced in the platinum wires may be calculated from the relation

\[
P = \frac{V \dot{B}^2}{8\rho_0} RRR .
\]  

(5.25)

This gives a heating of 0.2 nJ for the probe with \( RRR = 270 \). A rough estimate on the heating induced by eddy currents in the silver foot gives 5 nJ per pulse, which is the largest among all heating effects.

The signal in a magnetization measurement is proportional to the tipping angle \( \theta \propto B_{rf} \Delta t \), where \( B_{rf} \) and \( \Delta t \) are the amplitude and the duration of the rf pulse, respectively. The eddy current heating can be written as

\[
Q_{\text{eddy}} \propto \omega^2 B_{rf}^2 \Delta t \propto \omega^2 B_{rf} \theta .
\]  

(5.26)

One can thus reduce \( Q_{\text{eddy}} \) by decreasing the pulse amplitude and increasing the length without deteriorating the signal to noise ratio. The 2 V amplitude we use is the lowest value of the measurement electronics.

In §6.3, a method for measuring the heat from a pulse is discussed. Actually, the heat does not influence the on-going measurement, because the recorded \( M_n \) corresponds to the value just before the pulse. At very low temperatures, the interval of measurements should be long enough for the Pt temperature to relax back to that of the nuclear stage. Below 0.5 mK, we used an interval of 30 min between measurements.

The room temperature electronics is connected to the rf coil through a relay which is closed for about 10 s in each measurement. In this way, some extra heating that might be fed to the platinum probe through the cable can be eliminated.

The PLM was calibrated against the \(^{60}\text{CoCo} \) nuclear orientation thermometer at temperatures between 3 and 30 mK, using 160 \( \mu \)s pulses (see Fig. 30). The temperatures for the last two points on the high temperature side were obtained from the calibrated germanium resistor. Between 1 and 0.5 mK, the temperature readings with the 8 \( \mu \)s pulses were calibrated against 160 \( \mu \)s pulses.
5.4 Coulomb blockade thermometer

5.4.1 Theory

Substantial progress in microfabrication techniques has made it possible to manufacture in a controlled way metallic tunnel junctions with capacitance $C \approx 10^{-15}$ F. In this range, the charging energy of a nanostructure $E_c = e^2/2C$ for a single electron equals to the thermal energy at 1 K. Single electron tunneling effects have been studied intensively in the temperature region where $E_c \gg k_B T$ (Averin and Likharev, 1991; Grabert and Devoret, 1992). In this regime, the electric current is zero below a threshold voltage related to $E_c$; this is the so-called Coulomb blockade. In the opposite limit, where $E_c < k_B T$, the $IV$-characteristics reflect competing thermal and charging effects and can be used to indicate the temperature. This has been studied systematically by Pekola et al. in Jyväskylä (Farhangfar, Hirvi, Kauppinen, Pekola, Toppari, Averin, and Korotkov, 1997, and the references therein). In this section, I will briefly review the theory needed to understand the operation of the thermometer. In the next section, the results of performance tests at low temperatures are presented.

A tunnel junction consists of two electrodes separated by a thin insulating oxide layer for electrons to penetrate. The junction is characterized by a resistance $R$ and a capacitance $C$ in parallel. When several similar junctions are connected in series, they form a one dimensional array. Fig. 31 shows, as the simplest case, an array with two identical junctions. The “island” between them has a ground
The total capacitance of the island is therefore $C_\Sigma = 2C + C_0$. When there are $n$ extra electrons in the island, the potential is

$$\phi(n) = ne/C_\Sigma .$$

(5.27)

Pekola, Hirvi, Kauppinen, and Paalanen (1994) have used the orthodox theory (Averin and Likharev, 1991) to derive the $IV$-characteristics of a double junction in the weak Coulomb blockade regime, where $E_C < k_BT$. In this description, the tunneling rate of an electron is determined by the difference in the free energy of the states before and after a tunneling event, *viz.*

$$\Delta F = \frac{1}{2} e[(\phi_2 + \phi'_2) - (\phi_1 + \phi'_1)],$$

(5.28)

where the subscripts 1 and 2 denote the electrodes from and to which the electron tunnels, and the nonprimed and primed potentials are those before and after the tunneling, respectively. The tunneling rate for a specific event is given by

$$\Gamma = (e^2 R_T)^{-1} \frac{\Delta F}{1 - \exp(-\Delta F/k_BT)} .$$

(5.29)

Then the current through the $i$th junction is

$$I_i = e \sum_{n=-\infty}^{\infty} \sigma(n)[\Gamma_i^+(n) - \Gamma_i^-(n)] ,$$

(5.30)

where $\sigma(n)$ is the probability of finding $n$ extra electrons on the island. The master equation can be used to obtain $\sigma(n)$, *viz.*

$$[\Gamma_1^+(n-1) + \Gamma_2^-(n-1)]\sigma(n-1)$$

$$- [\Gamma_1^+(n) + \Gamma_1^-(n) + \Gamma_2^+(n) + \Gamma_2^-(n)]\sigma(n)$$

$$+ [\Gamma_1^+(n+1) + \Gamma_2^+(n+1)]\sigma(n+1) = 0 .$$

(5.31)
For the particular case when an electron tunnels from left to right in junction 1 (see Fig. 31), \( \Delta F \) can be written as

\[
-\Delta F = \frac{1}{2}e\{[\phi(n) + \phi(n + 1)] - (V/2 + V/2)\}
= \frac{e^2}{C_S} \left( n + \frac{1}{2} \right) - \frac{eV}{2},
\]

and when an electron tunnels from right to left,

\[
-\Delta F = \frac{1}{2}e\{(V/2 + V/2) - [\phi(n) + \phi(n - 1)]\}
= \frac{eV}{2} - \frac{e^2}{C_S} \left( n - \frac{1}{2} \right).
\]

In the high temperature limit, \( u = (e^2/C_S)/k_B T \ll 1 \), an expansion as the function of the small parameter \( u \) yields

\[
\Gamma^+_i(n) - \Gamma^-_i(n) = \left( k_B T / e^2 R_T \right) \times (v + \{[f(v) - f(-v)]/2 - n\}u + \cdots,
\]

where \( f(v) = [1 + \exp(v)(v - 1)]/[1 - \exp(v)]^2 \) and \( v = eV/2k_B T \). The fact that \( \sigma(n) = \sigma(-n) \) leads to \( \sum_{n=-\infty}^{\infty} \sigma(n)n = 0 \) and one obtains the IV-characteristics

\[
I = \left( k_B T / e R_T \right) \{v + u[f(v) - f(-v)]/2\} + \cdots,
\]

where \( I_1 = I_2 = I \), due to charge conservation. Differentiating the equation with respect to \( V \) leads to the conductance \( G = G(V) \),

\[
G/G_T = 1 - ug(v) + \cdots,
\]

where \( g(v) = -f'(v) \) and

\[
g(v) = \frac{v \sinh(v) - 4 \sinh^2 \frac{v}{2}}{8 \sinh^4 \frac{v}{2}}.
\]

At \( V = 0 \),

\[
G(0)/G_T = 1 - u/6 + \cdots,
\]

where \( G_T \) is the asymptotic value of \( G \) as \( V \to \pm \infty \). \( G(V)/G_T \) as a function of bias voltage \( V \) represents a bell shaped dip with the minimum at \( V = 0 \) (see Fig. 34). The ratio between the full width at half minimum, \( V_{1/2} \), and the temperature is a universal constant, \( \text{viz.} \)

\[
V_{1/2} \simeq 5.439 \frac{2k_B T}{e}.
\]
Figure 32: An inhomogeneous one dimensional tunnel junction array.

In such an array of normal metal tunnel junctions, $V_{1/2}$ can be used as a primary thermometer. The depth of the dip at $V = 0$ (see Eq. (5.38)) is inversely proportional to temperature. Therefore, $G(0)/G_T$ is suitable for secondary thermometry and for calibration, one has to determine the capacitance $C_{\Sigma}$ at a known temperature.

In a real array, the junctions are not always identical due to fabrication processes. For the case with $N$ non-identical junctions in series as shown in Fig. 32, Hirvi, Kauppinen, Korotkov, Paalanen, and Pekola (1995) give an analogous result

$$
\frac{G}{G_T} = 1 - 2 \sum_{i=1}^{N} \frac{R_{T,i}}{R_{\Sigma}} \frac{\Delta_i}{k_B T} g \left( \frac{R_{T,i} e V}{R_{\Sigma} k_B T} \right),
$$

(5.40)

using a similar high temperature expansion at $\Delta_i \ll k_B T$. $R_{\Sigma} = \sum_{i=1}^{N} R_{T,i}$ is the total resistance, and $\Delta_i$ describes the Coulomb blockade threshold for the junction $i$, $\Delta_i = (C_{i,i-1}^{-1} + C_{i,i}^{-1} - 2C_{i,i-1}^{-1}) e^2/2$, where $C_{kl}^{-1}$ is the inverse of the capacitance matrix calculated according to the rule described by Ingold and Nazarov (1992). In the fully symmetric situation with $R_{T,i} = R_T$, $C_i = C$, and $C_{0,i} = 0$, we have

$$
\frac{G}{G_T} = 1 - u_N g(v_N),
$$

(5.41)

$$
G(0)/G(T) = 1 - \frac{1}{6} u_N.
$$

(5.42)

Compared to Eqs. (5.36) and (5.38), $u$ and $v$ for the two-junction case are now replaced by $u_N = [(N - 1)/N] e^2/C k_B T$ and $v_N = eV/N k_B T$ for $N$ junctions. Eq. (5.41) describes a similar bell-shaped $G/G_T$ vs. $V$ curve. The full width at half minimum scales by $N$, viz.

$$
V_{1/2} \approx 5.439 \frac{N k_B T}{e},
$$

(5.43)

while the depth of the dip at $V = 0$ scales by $(N - 1)/N$. 
At lower temperatures, where $k_B T \sim \Delta_i$, higher order terms in conductance become noticeable (Farhangfar et al., 1997), viz.

$$
\frac{G}{G_T} = 1 - u_N g(v_N) - \frac{1}{4} u_N^2 [g''(v_N)h(v_N) + g'(v_N)h'(v_N)] \\
- \frac{1}{8} u_N^3 [\frac{1}{4} g'''(v_N)h(v_N)^2 + \frac{1}{3} g''(v_N) + \frac{1}{2} g'''(v_N)h'(v_N)h(v_N)] - \cdots ,
$$

(5.44)

$$
G(0)/G_T = 1 - \frac{1}{6} u_N + \frac{1}{60} u_N^2 - \frac{1}{630} u_N^3 + \frac{1}{5040} u_N^4 - \cdots ,
$$

(5.45)

where $h(v_N) = v_N/\tanh(v_N/2)$. Using terms up to second order in $u_N$, Eq. (5.44) results in a linear correction to the full width at half minimum,

$$
\frac{\Delta V_{1/2}}{V_{1/2,0}} \approx 0.39211 \Delta G/G_T ,
$$

(5.46)

where $V_{1/2,0}$ is the half width given by Eq. (5.43), when only the linear term is considered and $\Delta G/G_T$ is the actual measured depth.

For small variations in junction parameters, there exists a relation (Hirvi, Kauppinen, Korotkov, Paalanen, and Pekola, 1996a)

$$
\frac{V_{1/2}}{V_{1/2,0}} \approx 1 - k[\delta R/R_0]^2_{\text{rms}} ,
$$

(5.47)

where $[\delta R/R_0]^2_{\text{rms}}$ is the deviation of junction resistances from their mean value $R_0 = R_S/N$. The numerical factor has a value $k \approx 0.73 + (N-1)/N$. The formula above is based on the reasonable assumption that all junctions have a uniform oxide thickness and, consequently, $R_{T,i}C_i = \text{constant}$ because $R_{T,i} \propto A_i^{-1}$ and $C_i \propto A_i$, where $A_i$ is the junction area. The quadratic dependence of $V_{1/2}$ on the variations is rather weak.

At low temperatures where $k_B T < E_c$, the $IV$ characteristics are influenced by uncontrollable background charges, which can result from trapping in the tunnel barrier and from trapping states in the substrate near the islands (Averin and Likharev, 1991). Monte-Carlo simulations show that the background charges do not affect $V_{1/2}$ and $\Delta G/G_T$ until $u_N \approx 3$ (Hirvi, Paalanen, and Pekola, 1996b). The temperature at which this occurs depends on the capacitance of the junctions. This will probably be the ultimate low temperature limit for the use of Coulomb blockade thermometers.

In the next section, we will discuss the effect of ohmic heating due to bias currents which can become important at higher temperatures than the background charge effects.
Figure 33: Setup for measuring the resistance of a junction array as a function of bias voltage.

5.4.2 Performance at low temperatures

The specimens were fabricated by electron beam lithography and two or three angle shadow evaporation techniques. Two layers of aluminum, with oxidization in between, were evaporated on a silicon substrate to form an Al/AlOx/Al tunnel barrier (Hirvi et al., 1996a). The capacitance of the junctions was controlled to be within the range from 1 to 40 fF, depending on the temperature regime where the junction arrays were to be used. A typical junction area was several \( \mu \text{m}^2 \). The following results were obtained with a sample having \( C = 26.3 \, \text{fF} \). The probe consisted of four parallel chains, each containing 40 junctions in series. The total resistance \( 1/G_T \approx 300 \, \text{k}\Omega \).

Fig. 33 shows the measurement setup. The junction is biased by a DC current which sweeps slowly through zero. A small AC current, typically 30 Hz, rides on the linear DC sweep in order to measure the differential resistance. The slow DC sweep was provided by a home made unit, based on the ICL 8038 precision waveform generator. The sweep could be varied from several minutes to an hour. The amplitude of the AC excitation was always kept smaller than 5% of \( V_{1/2} \). The DC and AC voltages over the junction array were measured by a voltmeter and a lock-in amplifier, respectively. An LC filter, described in § 5.5, was connected in front of the probe in order to reduce rf noise, which may cause heating of the junctions. The effective circuit diagram for the filter used in our measurement can be reduced to the two capacitors in parallel with the junction array as shown.
in Fig. 33.

Figure 34: Examples of the measured conductance of a junction \((N = 40)\) at 61 and 315 mK.

Fig. 34 shows two typical \(G/G_T\) vs. \(V_{DC}\) curves measured at about 61 and 315 mK, respectively. The solid lines depict fits with Eq. (5.36) based on the high temperature approximation. Eq. (5.36) fits the data for \(T = 315\) mK, where \(u_N = 0.22\), quite well. Whereas it is obvious that without higher order corrections, this equation does not describe the data well at the relatively low temperature, \(T = 61\) mK, where \(u_N = 1.13\). The non-linear fitting involved with Eqs. (5.36) or (5.44) is inherently slow. In the actual analysis, we used a linear method based on a polynomial fitting (Hirvi and Pekola, 1997).

Fig. 35 shows the measured \(V_{1/2}\). The temperatures were measured with a calibrated germanium thermometer, a \(^{60}\)Co NO thermometer (see § 5.2), or a vibrating wire viscometer (see § 5.1). The solid line corresponds to Eq. (5.46). Most of the displayed 29 Hz data were measured with the filter which strongly reduced the dip height. \(\Delta G/G_T\), needed for the correction, was taken from the solid line in Fig. 36. \(V_{1/2}\) follows the expected temperature dependence within 10% down to 20 mK, below which the background charge effects are predicted to show up. We estimated our temperature calibration to be correct within \(\pm 5\%\). We also expect that errors due to fabrication nonidealities and data fitting are less than 5%. The measured \(V_{1/2}\) is slightly below the expected value between 50 - 80 mK, which is the lower end of the working range for our Germanium thermometer. This will be checked in further experiments.
Figure 35: The full width at half minimum, $V_{1/2}$, as a function of the calibration temperature. The measurements were done at two frequencies, 29 Hz ($\Delta$) and 2 Hz ($\odot$). The solid line corresponds to the theoretical result of Eq. (5.46).

Figure 36: $\Delta G/G_T$ vs. the inverse of temperature, measured at 29 Hz ($\Delta$) and 2 Hz ($\odot$, $\Box$). The narrow sweep ($\odot$) is explained in the text. The solid line is calculated from Eq. (5.45) with $C = 26.3$ fF.
The measured depth of the conductance minimum as a function of calibration temperature is shown in Fig. 36. The data agree well with the predicted behavior of Eq. (5.45) depicted by the solid line. A fit of Eq. (5.45) to the high temperature end of the data was used to obtain \( C = 26.3 \, \text{fF} \). After such a calibration, the depth of the conduction dip provides a convenient thermometer. At the low temperature end, the \( \Delta G/G_T \) measurements were made using a very narrow DC sweep, just covering the immediate vicinity of the minimum. \( G_T \) was determined in another sweep over the asymptotic region. The use of a narrow sweep to measure \( \Delta G/G_T \) at low temperatures can be advantageous owing to the reduced ohmic heating caused by the bias voltage.

Fig. 37 shows a comparison between the temperatures deduced from \( V_{1/2} \) measurements and those measured by calibrated thermometers. Good agreement between them extends down to 20 mK. Below 20 mK, the behavior of

![Figure 37: Temperatures indicated by the junction array, \( T_{\text{CBT}} \), vs. temperature measured by other calibrated thermometers, \( T_{\text{Calib}} \) (see text). The solid line depicts ideal behavior.](image)

the present junction thermometer is expected to be affected by the fluctuating background charges. It is possible to push this limit towards lower temperatures by fabricating junctions with larger \( C \) values. This will be a subject for future measurements.

Another potential limiting factor that may set in at low temperatures is the poor thermal coupling between the conduction electrons and the environment together with ohmic heating due to the bias voltage. Fig. 38 shows a thermal
Figure 38: Schematic thermal model of an junction array including the electronic thermal resistance of the junctions $R_{j,i}$, the electron-phonon resistance $R_{el-ph}$, and the Kapitza resistance $R_K$ (Kauppinen and Pekola, 1996).

model of an array. Electrons on an island are connected to the environment through two parallel thermal channels, namely the thermal resistance $R_{j,i}$ of the junction, and the electron-phonon resistance $R_{el-ph}$ and the Kapitza resistance $R_K$ in series. It is believed that the thermal conductivity along the tunnel junctions is very low owing to large $R_{j,i}$, while between $R_K$ and $R_{el-ph}$, the latter is the dominant factor (Kauppinen and Pekola, 1996). The heat transfer between electrons and phonons is described by

$$ P = \Sigma \Omega (T_e^n - T_p^n), $$  \hspace{1cm} (5.48)

where $\Sigma$ is a constant, $\Omega$ is the volume of the island, and $T_e$ and $T_p$ are the temperatures of electrons and phonons, respectively. Kauppinen and Pekola (1996) have studied this effect using samples with different island shape and volume around 200 mK. For a sample with a straight island of $22 \times 0.25 \mu m^2$, they found $n = 5$ and $\Sigma = 0.6$ nW/K$^5\mu m^3$. For samples with extended islands to improve thermalization, the contact was found to be weaker and $n \simeq 3.5$. For our specimen, the island volume is $22.7 \mu m^3$. If we assume the ohmic heating on each junction to be 5 fW, and take somewhat arbitrarily $n = 3.5$ and $\Sigma \sim 1$ nW/K$^{3.5}\mu m^3$, the temperature difference between phonons and electrons can be as large as 5 mK at 11 mK. The above values of $n$ and $\Sigma$ may not be exact, but the calculation does show that the electron phonon coupling can be a limiting factor in the use of junction arrays as thermometers at low temperatures.

One notable feature of the Coulomb blockade thermometer is its insensitivity to magnetic field because the condition $\mu_B B \ll E_F$ is practically always satisfied. Here $E_F$ is the Fermi energy and $\mu_B$ is the Bohr magneton. A small magnetic field is, however, needed to suppress the superconductivity of the Al junctions at temperatures below 1 K.

Measurements in Jyväskylä and our preliminary results show that suitably tailored normal metal tunnel junction arrays can be employed for primary ($V_{1/2}$)
and secondary \((\Delta G/G_T)\) thermometry from 40 K to as low as 20 mK.

5.5 Low temperature filters for carbon resistors

We use sliced Speer carbon resistors (1/2 W, 100 Ω, 220 Ω) to monitor the temperature of the dilution refrigerator in several places. One problem in using them at low temperatures is that the \(R - T\) relation starts to saturate below 50 mK because the rf noise absorbed by the resistor causes self heating. A simple low temperature filter can largely improve the situation (Eska and Neumaier, 1983). We designed two kinds of rf filters to suppress noise through the measurement wires.

Fig. 39 shows the circuit diagrams of an RC filter and an LC filter. Small surface-mount components are used. The whole filter is enclosed in a copper box of size 30 \(\times\) 30 \(\times\) 10 mm\(^3\), which is usually anchored to the mixing chamber or the 50 mK plate.

Figure 39: Low temperature filters. Capacitors: Philips 1812 NPO 22 nF. Inductors: Siemens Simid 03 series 220 \(\mu\)H. Resistors: metal film resistors.

The cutoff frequency of the filters is around 200 kHz. There is no direct prove about the efficiency of the filters, but it can be seen indirectly by comparing the performance of the germanium resistor which is not filtered and the carbon resistor with filter. The former becomes saturated at temperatures around 100 mK.
when the PLM electronics is on, while the latter does not show saturation until 10 mK. On the other hand, it seems that the PLM electronics is quite noisy.
6 Performance of the cryostat

6.1 Starting of the dilution refrigerator

Starting from room temperature, it takes about 8 hours to cool the cryostat to 77 K with liquid N\textsubscript{2} using N\textsubscript{2} at 0.5 bar as exchange gas inside the IVC (inner vacuum can). This procedure can be twice as quick if H\textsubscript{2} is used instead. We try to avoid H\textsubscript{2} when the nuclear stage is fixed inside the machine because small amount of H\textsubscript{2} left on the nuclear stage may introduce a time-dependent heat leak due to the ortho-para transition of H\textsubscript{2} molecules (see § 6.4).

After the leak testing, 1 mbar of \textsuperscript{3}He is admitted into IVC as exchange gas. It takes another 8 to 10 hours to cool the dewar and the first stage magnet to 4.2 K. 60 liters of helium is needed before liquid helium starts to accumulate at the bottom of dewar due to the heat capacity of the magnet. After that, another 100 litres of helium is needed in the initial transfer. Meanwhile, the temperature of the dilution refrigerator inside IVC is monitored by Pt resistance thermometer and carbon resistors. We start to evacuate IVC with a turbomolecular pump when the magnets inside IVC become superconductive at 9 K. After being pumped for 8 hours, \textsuperscript{3}He outgassing level of 5 \times 10^{-7} l-mbar/s can be reached.

The condensation of the 180 l of \textsuperscript{3}He and 800 l of \textsuperscript{4}He into the dilution refrigerator takes 3 to 6 hours depending on the precooling temperature. In order to get a low \textsuperscript{3}He background in the IVC, we pump out the exchange gas quite early when the dilution refrigerator is still at 9 K. This will slow down the condensation in the beginning because the gas condensed at 1 K pot will re-evaporate when it reaches the lower part of the machine where the temperature is still high. After all gas is condensed, the mixing chamber can reach 10 mK within 2 h.

6.2 Precooling of the nuclear stage

The precooling time of the nuclear stage depends on its magnetic heat capacity, on the cooling power of the dilution refrigerator and on the thermal contact between the liquid inside the mixing chamber and the nuclear stage (see Fig. 40). The cooling power of a dilution refrigerator can usually be described by the formula

\[ \dot{Q} = a(T_{mc}^2 - T_0^2) , \]  

(6.1)

where \(a\) and \(T_0\) depend on circulation. When the external heat load \(\dot{Q}\) is zero, \(T_0\) is the lowest temperature reached by the the dilution refrigerator. The values of \(T_0\) and \(a\) were measured with two different circulations. The results are listed in Table 4.
Figure 40: The model for precool of the nuclear stage. $T_{mc}$, $T_{mp}$ and $T_n$ are the temperatures of the liquid in the mixing chamber, the mixing chamber plate, and the nuclear stage, respectively.

Table 4: Performance of the dilution refrigerator at two different circulations.

<table>
<thead>
<tr>
<th>(μmole/s)</th>
<th>a (μW/mK²)</th>
<th>$T_0$ (mK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>750</td>
<td>0.064</td>
<td>4.2</td>
</tr>
<tr>
<td>1500</td>
<td>0.16</td>
<td>6.8</td>
</tr>
</tbody>
</table>

The Kapitza boundary resistance of the dilute mixture has the temperature dependence

$$R_K = RT^{-n}.$$  \hspace{1cm} (6.2)

The measurements of different researchers have given inconsistent data with $n$ varying from 1.5 to 3 (Osheroff and Corruccini, 1981; Franco, Bossy, and Godfrin, 1984; Cousins et al., 1994; Voncken et al., 1996). Systematic studies by Lancaster group show that the temperature dependence of the interfacial boundary resistance itself does obey the theoretical $T^{-3}$ law. The discrepant results come from the different thickness of sinter layers used in the measurements. The length scale in the sinter arises from competition between the boundary and thermal resistances within the sinter and the mixture (for details, see Cousins et al., 1994).
Figure 41: Temperature of the mixture (mc, ▽) and of the mixing chamber plate (mp, △) vs. heating power on the plate. From these measurements, the thermal resistance between the silver sinter and the mixture can be estimated. The inset shows the $\dot{Q} \sim (T_{mp}^{2.5} - T_{mc}^{2.5})$ relation.

We measured the temperature difference between $T_{mc}$ and $T_{mp}$ by heating the mixing chamber plate (see Fig. 41), which gave $n = 1.5$ in Eq. (6.2), approximately. Therefore,

$$\dot{Q} = \frac{0.4}{R} (T_{mp}^{2.5} - T_{mc}^{2.5}) .$$  \hspace{1cm} (6.3)

The thermal conductance of a metal behaves like $\lambda = kT$ as described in § 4.5, and the temperature difference between $T_{mp}$ and the $T_n$ follows the relation,

$$\dot{Q} = \frac{1}{2} k(T_{n}^{2} - T_{mp}^{2}) .$$  \hspace{1cm} (6.4)

In the above equations, if we assume that the contribution of the Kapitza resistance to the temperature gradient between $T_{mc}$ and $T_n$ had the form

$$\dot{Q} = \frac{1}{R^*} (T_{mp}^{2} - T_{mc}^{2}) ,$$  \hspace{1cm} (6.5)

regardless of the actual form of Eq. (6.3), the thermal path can be outlined as three impedances, $1/a$, $R^*$, and $2/k$ in series. The "impedance" $1/a$ specifies the cooling power and it is determined by the circulation rate and the performance of
the dilution refrigerator, while the "impedances" $R^*$ and $2/k$ specify the thermal resistance in the path of heat flow. These three factors should be mutually compatible so that none of them becomes the bottleneck on the path. Table 5 lists the three parameters; $k$ is deduced from the residual resistance measurement described in § 4.5 and $a$ is the measured value when the circulation rate was 1500 $\mu$mole/s. In order to make the comparison, the value of $R^*$ is taken simply from $Q/(T_{mc}^2 - T_{mp}^2)$ at 10 mK. Because of the difference between Eqs. (6.3) and (6.5), $R^*$ is not a constant but decreases with increasing temperature. In this simple model, we can see that within a factor of two, the three parameters are approximately of equal magnitude with $2/k$ being the largest "impedance".

Table 5: Comparison of "impedances" in the thermal path

<table>
<thead>
<tr>
<th>$1/a$ (K$^2$/W)</th>
<th>$R^*$ (K$^2$/W)</th>
<th>$2/k$ (K$^2$/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.5</td>
<td>4.3</td>
<td>6.6</td>
</tr>
</tbody>
</table>

Figure 42: Temperature vs. time during precooling of the nuclear stage. $T_{mc}$ and $T_n$ were measured by vibrating wire and PLM thermometers, respectively. $T_{mp}$ is deduced from $T_{mc}$ through the measured Kapitza resistance with the real temperature dependence given by Eq. (6.3).

Fig. 42 shows the precooling curve of the nuclear stage with a circulation in the dilution refrigerator being 750 $\mu$mole/s. $T_{mc}$ and $T_n$ were measured by using a vibrating wire and Pt NMR thermometers, respectively. $T_{mp}$ was derived from
$T_{mc}$ using Eqs. (6.1) and (6.3). It can be seen that the difference between $T_{mp}$ and $T_n$ is larger than half of the total temperature gradient between $T_{mc}$ and $T_n$. This simplified model gives us the right picture of precooling. As mentioned in § 4.5, the time can still be reduced by improving the heat switch before the Kapitza resistance starts to dominate. At the moment, it takes 50 h to precool the nuclear stage to 10 mK with a circulation of 1000 to 1500 µmole/s. In an ideal situation with no thermal gradient between the nuclear stage and the liquid inside the mixing chamber, it would take only 20 h at a circulation of 1500 µmole/s.

6.3 Demagnetization of the first nuclear stage

Starting from 9 T and 10 mK, demagnetization of the nuclear stage was done in steps. Above 1 T, the sweep rate was 20 mT/min; between 1 T and 100 mT, 4 mT/min; and below 100 mT, 1 mT/min, which is the slowest rate of the power supply. Fig. 43 shows the temperature vs. the magnetic field during demagnetization. The lowest temperature measured by the Pt thermometer, located on the top flange of the nuclear stage, was 67 µK.

![Graph showing temperature vs. magnetic field](image)

Figure 43: Temperature vs. the magnetic field during demagnetization. $T$ was measured by the PLM thermometer, located on the top flange of the nuclear stage. The straight line shows $B/T = B_0/T_0$. The inset illustrates the amount of non-adiabaticity during the cooling process.

From the inset of Fig. 43, it can be seen that above 300 µK the $B/T$ ratio stays constant so that the temperature decreases adiabatically with the field. Below
300 $\mu$K, the decrease of the measured temperature lags behind. Starting from 10 mK at 9 T, the final temperature of the nuclear stage should be 11 $\mu$K, much lower than the measured one. The reason for this difference will be discussed below.

Fig. 44 shows the temperature vs. field dependence during a de- and remagnetization cycle in which the nuclear stage was first demagnetized from 9 T to 10 mT, and then remagnetized to 0.5 T. Based on measurements at 0.5 T after remagnetization, where the thermometer works ideally, we know that the temperature at 10 mT should have been 15 $\mu$K. The minimum temperature at the middle of the nuclear stage was, therefore, somewhere between 11 and 15 $\mu$K. The overall decrease in the $B/T$-ratio is due to eddy currents and background heat leak. The non-ideal behavior is due to the finite thermal conductivity of

Figure 44: Measured temperature vs. field in a de- and remagnetization cycle.

the parts between the Pt wire and the cold Cu nuclei, and various heat inputs onto the systems.

6.4 Sources of heat leak

The overall heat leak to the nuclear stage can be measured directly by monitoring the temperature drift with time (see Fig. 45). These measurements were done at high temperatures where readings of the Pt thermometer still followed the temperature in the middle of the nuclear stage. Fig. 45(a) was measured in zero field, thus the electronic heat capacity of the whole nuclear stage was used in the
Figure 45: Heat leak measurement, (a) in zero external field, (b) in 10 mT field.

calculation. Fig. 45(b) was recorded in a field of 10 mT. The effective magnetic heat capacity of the 97 moles of copper was taken into account.

The heat load on the nuclear stage can be divided into two classes, viz. the external heat leak and the (time dependent) internal heat leak. The former can be further classified into the following seven contributions.

1) The residual gas inside the IVC carries heat from high temperature parts of the apparatus to the nuclear stage, which is proportional to the pressure $P$. The pressure in IVC is monitored by a vacuum gauge at room temperature fixed on top of the IVC pumping line; it usually records below $5 \times 10^{-7}$ mbar. But it is commonly believed that the pressure of the IVC is much lower than that shown by the gauge at room temperature because of gas adsorption by the $^4\text{He}$ pot and by other cold components. Van de Haar (1991) tried to measure the difference of heat leak by fixing a charcoal pump on the still of the Cryo II cryostat (see § 3.2). No obvious change was observed in the total heat leak of 6 nW with or without the charcoal pump inside the IVC. So, it was concluded that the heat leak from the residual gas can be neglected, which may also be the case for our cryostat.

2) An upper limit of the heat flow due to thermal radiation can be estimated by black body radiation,

$$\dot{Q} = \sigma A (T_1^4 - T_2^4) ,$$

(6.6)
where $\sigma = 5.67 \times 10^{-8}\text{Wm}^{-2}\text{K}^{-4}$ is the Stefan-Boltzmann constant. With $T_1$, the temperature of the 50 mK shield, it is smaller than one picowatt, and can be neglected.

3) The nuclear stage is connected through the heat switch, the measurement leads, and the four $\text{Al}_2\text{O}_3$ support tubes to the mixing chamber, which is usually below 4 – 5 mK. In some heat leak measurements, the temperature of the nuclear stage was drifting from below to above the temperature of the mixing chamber, but no difference in the drifting speed could be seen during this procedure. Therefore, the heat flow from the mixing chamber to the nuclear stage contributes insignificantly to the total heat leak.

4) Heating due to the transmitter pulse of the Pt thermometer was discussed in § 5.3. This effect was studied with the set-up shown in Fig. 46. The top flange was heated by frequent pulses from PLM-1, with a time interval of 1 s, to establish a temperature gradient between the middle of the nuclear stage and the top flange, while the temperature of the flange was monitored by PLM-2. The energy from PLM-1 was calibrated against the heater. This gave 2 nJ per 8 $\mu$s pulse. Thus the heating due to temperature measurement at 15 to 30 min intervals is negligible.

![Diagram](image)

**Figure 46:** Temperature and heat leak distribution in the nuclear stage. $\dot{q}$ denotes the heat leak per mole of copper. $N_1, N_2$ are the amounts of copper in supporting tube and in the top flange, respectively. The heater on the top flange was used to calibrate the heating power from the Pt thermometer.

5) The $\gamma$-ray heating of the order of 1 nW from the $^{60}\text{Co}$ source can be a significant contribution as analyzed in § 5.2.
6) The influence of the rf radiation penetrating into the cryostat is unknown. Although the cryostat is located inside the electrically shielded room, the signals of FM radio broadcasts can be detected with a radio. But in general, we believe that the direct disturbance to the nuclear stage is small based on our experience from other cryostats, especially the Berlin cryostat which is not in a shielded room at all.

7) The heat leak was measured in various magnetic field (see Fig. 47). The field dependent heat leak is produced by relative movement of the magnet and the nuclear stage. The vibration of the platform itself is small (see § 4.2), but the vibrations may be induced by evaporation of helium in the bath or by the continuously filled pot. But so far, the reasons for the field dependent heat leak in our cryostat are not clarified.

![Graph of Heat leak vs. magnetic field]

**Figure 47: Measured heat leak vs. magnetic field**

At the time of writing, there are no systematic measurements for the time dependent internal heat leak. The following discussion is based on results from other groups.

1) The ortho-para conversion of H$_2$ molecules at low temperatures releases heat (Koláč, Neganov, and Sahling, 1985; Koláč, Neganov, Sahling, and Sahling, 1986). H$_2$ may be present in copper in the form of bubbles of sub-micron size. After long time annealing at temperatures above 950 °C, in our case 100 h, the concentration of H$_2$ should be lower than 0.1 ppm (Gloos *et al.*, 1988). The heat leak resulting from the ortho-para conversion is

$$Q(t) = U \frac{x_0^2}{(1 + x_0kt)^2}, \quad (6.7)$$
where \( k = 1.9\%/h \) at the melting pressure of solid \( \text{H}_2 \) (Pedroni, Meyer, Weihaus, and Haase, 1974), \( x_0 \) is the initial concentration of ortho-\( \text{H}_2 \) (75\% for the equilibrium mixture at room temperature). From Eq. (6.7), it can be calculated that the total heat leak 10 (20) days after cooldown to 4.2 K is 2.6 (1) nW if we assume 0.1 ppm concentration of \( \text{H}_2 \) in the copper stage.

2) Organic materials, ceramics, glass, and other amorphous substances release heat due to relaxation of two-level states (Zimmermann and Weber, 1981; Zimmermann, 1984; Schwark, Pobell, Kubota, and Mueller, 1985). The generated heating power can be expressed as

\[
\dot{Q} = \frac{\pi^2 V k_B^2 \overline{P}(T_i^2 - T_f^2)}{24 t},
\]

where \( \overline{P} \) is the density of the two level system. Experiments showed that a typical \( \dot{Q} \) is of the order of 0.1 (0.01) nW/g (Schwark et al., 1985), 1 day (week) after cooldown to low temperatures. In our case, the heat release from organic materials is negligible because total amount of these substances on the nuclear stage is less than 0.5 g. \( \text{Al}_2\text{O}_3 \), the material of the supporting tubes, was not observed to give rise to any heat release within the level \( 2 \times 10^{-2} \text{nW/g} \) (Schwark et al., 1985).

3) Experiments (Huiku et al., 1986; Gloos et al., 1988) show that well annealed copper releases heat at very low temperature, attributed to the relaxation of grain boundaries and other two-level systems related to lattice defects such as impurities. The main part of our nuclear stage was made of \( 4\text{N}^+ \) copper. Assumably, the density of the effective impurities is of the order of 1 ppm, after annealing. If we take 30 K to be the energy gap of the two-level system of the lattice defects, the total heat released by the nuclear stage of our size is 40 mJ. The introduced heat leak behaves as

\[
\dot{Q} = \dot{Q}_0 e^{-t/\tau},
\]

with a time constant of several days (Pobell, 1982, and references therein). Taking arbitrarily a time constant of 7 (3.5) days, after 21 days the heating power is 4.2 (0.34) nW.

### 6.5 Analysis of the non-adiabatic behavior

Using the measured heat leak, we can estimate the temperature difference between the center of the nuclear stage and the top plate. We can assume the spin temperature \( T_s \) decrease adiabatically with the demagnetization from 9 T to 10 mT. And if we take 6 nW as the total heat leak, according to Eq. (2.40)

\[
T_s = \left( \frac{\kappa \dot{Q} + \Lambda B^2}{\Lambda B^2} \right) T_n,
\]
the temperature of the lattice should be 14 μK, only slightly higher than that for adiabatic demagnetization, 11 μK. A factor which can produce a substantial temperature difference is the finite thermal conductivity of the copper tube connecting the middle part of the nuclear stage to the top flange (see Fig. 46). Assuming that the heat leak per mole of copper is \( \dot{q} = 35 \) pW, the temperature difference can be expressed as,

\[
T_{\text{top}}^2 = T_{\text{mid}}^2 + \frac{\dot{q}(N_1 + 2N_2)}{k},
\]

(6.10)

in which \( N_1 = 22 \) moles, \( N_2 = 17 \) moles are the amount of copper in the tube and in the top flange, respectively. Coefficient \( k \) in the metallic thermal conductivity has a measured value of 2.56 W/K², corresponding to \( RRR \cong 500 \). The calculated temperature difference is 20 μK, which is still not large enough to explain the measured temperature of 67 μK on the top flange. On the other hand, if we assume that all of the heat leak enters the nuclear stage through the top flange and the support tube, instead of being distributed evenly over the whole nuclear stage, \( \dot{q} = 150 \) pW in the above formula and the temperature difference can be as large as 40 μK.

Another factor can contribute to the poor performance of the Pt thermometer. As mentioned in § 5.3, a loose wire in the probe may result in a much higher measured temperature. The heat leak to the Pt wires, together with a finite conductance of the wire and the joint between the wires and the silver foot, can cause large temperature differences. If we assume a total resistance of the probe to be 1 μΩ, a heat leak in the 50 pW level will be enough to produce a 50 μK temperature difference.

From the point of view of temperature measurement, a thermometer located at the place where the Rh sample is fixed should give a more reliable result for the middle part of the nuclear stage. This is because the main part of the nuclear stage has larger cross section and better \( RRR \), thus \( k \) in (6.10) is 100 times larger. From the point of view of the heat leak, the magnetic field dependent source should still be investigated. So far, the longest period during which the cryostat was in 4.2 K was less than two months. The time dependent heat release thus could have played an important role in the total heat leak.
7 Nuclear magnetic ordering in rhodium

By demagnetization of nuclear spins in metals, it is possible to reach substantially lower temperatures than by any other cooling methods, because of the weak interactions between nuclei. In addition, such systems themselves provide good models to investigate magnetism (see the review papers, Lounasmaa, 1989; Oja, 1991; Hakonen, Lounasmaa, and Oja, 1991; Oja and Lounasmaa, 1997), since the nuclei are well localized and, at low temperatures, the spin assembly is fairly well isolated from other degrees of freedom such as the electrons and the lattice. In many cases, the well known spin interactions make it possible to do first-principles calculations to find the ordered spin structures.

Using the cascade cooling technique, the noble metals copper and silver, and the transition metal rhodium have been studied by means of the YKI cryostat described in § 3.1. By susceptibility measurements, spontaneous nuclear magnetic ordering was observed first in copper at nanokelvin temperatures (Huiku et al., 1986), then in silver at both positive and negative spin temperatures (Hakonen et al., 1992). In rhodium, ordering has not been seen so far (Vuorinen et al., 1995). Neutron scattering experiments have been done on copper and silver (§ 3.1) to investigate the spin structures in their ordered states (Jyrkkio et al., 1989; Tuorinemi et al., 1995).

In this chapter, I will summarize our previous experiments performed on the YKI cryostat and discuss the present status of the Rh experiment on the new cryostat.

7.1 Theoretical background

In copper and silver, the spin Hamiltonian can be written as

\[ \mathcal{H} = \mathcal{H}_{\text{dip}} + \mathcal{H}_{\text{RK}} + \mathcal{H}_Z. \]  (7.1)

The dipolar interaction between spins \( I_i \) and \( I_j \) separated by \( r_{ij} \) is given by

\[ \mathcal{H}_{\text{dip}} = \frac{\mu_0 \hbar^2}{4\pi} \sum_{i<j} \frac{\gamma_i \gamma_j}{r_{ij}^3} \left[ I_i \cdot I_j - \frac{3(I_i \cdot r_{ij})(I_j \cdot r_{ij})}{r_{ij}^2} \right], \]  (7.2)

where \( \gamma_i \) is the gyromagnetic ratio for spin \( I_i \), and \( \mu_0 \) is the permeability in vacuum. The indirect Ruderman-Kittel exchange interaction mediated by conduction electrons is

\[ \mathcal{H}_{\text{RK}} = -\sum_{i<j} J_{ij} I_i \cdot I_j, \]  (7.3)

where \( J_{ij} \) is the coupling constant between spins \( I_i \) and \( I_j \). The Zeeman term has the form

\[ \mathcal{H}_Z = -\hbar B \cdot \sum_i \gamma_i I_i. \]  (7.4)
The dipolar and Zeeman forces are known accurately, while the exchange interaction can be calculated using electronic band structure and wave functions.

In the framework of the mean field theory, the different interactions on a single spin $I_i$ are attributed to an effective field $B_{\text{eff}},$ viz.

$$\mathcal{H} = \hbar B_{\text{eff}} \cdot \sum_i \gamma_i I_i$$  \hspace{1cm} (7.5)

The contributions to $B_{\text{eff}}$ from different interactions can be written as

$$B_{\text{eff}} = B + \mu_0 (R + L - D) pM_{\text{sat}},$$  \hspace{1cm} (7.6)

where $L = 1/3$ is the Lorentz factor due to dipolar interactions, $D$ is due to the shape-dependent demagnetization field (Jackson, 1976), and $p$ is the polarization. $R$ can be deduced by writing the RK interaction for spin $I_i$ as

$$\mathcal{H}_i = -I_i \cdot \sum_j J_{ij} I_j = -\frac{1}{(\hbar \gamma)^2} \mu_i \cdot \sum_j J_{ij} \mu_j$$

$$= -\mu_i \cdot \left[ \left( \frac{\mu_j}{(\hbar \gamma)^2} \sum_j J_{ij} \right) \right]$$

$$= -\mu_i \cdot \left[ \frac{pM_{\text{sat}}}{\rho_0} \frac{1}{(\hbar \gamma)^2} \sum_j J_{ij} \right]$$  \hspace{1cm} (7.7)

$$= -\mu_i \cdot \mu_0 R pM_{\text{sat}},$$

where we define $R = \sum_j J_{ij} / (\mu_0 h^2 \gamma^2 \rho_0),$ $\rho_0$ being the number density of nuclei. $R$ describes the relative strength of the RK and the dipolar forces.

From the Curie law for non-interacting spins

$$\chi_0 = \frac{\mu_0 pM_{\text{sat}}}{B_{\text{eff}}} = \mu_0 \frac{\lambda}{T_n},$$  \hspace{1cm} (7.8)

the static susceptibility in the mean field theory can be written as,

$$\chi'(0) = \frac{\mu_0 pM_{\text{sat}}}{B}$$

$$= \frac{\chi_0}{1 - (R + L - D) \chi_0}$$

$$= \frac{\mu_0 \lambda}{T_n - \theta},$$  \hspace{1cm} (7.9)

where $\theta = \lambda (R + L - D).$
7.2 Some experimental results in Cu, Ag, and Rh

Low frequency SQUID NMR techniques have been used to measure the dynamic susceptibility as a function of frequency, viz.

\[ \chi(f) = \chi'(f) - i\chi''(f) , \]  

(7.10)

Afterwards, the static susceptibility \( \chi'(0) \) was calculated from the Kramers-Kröning relation

\[ \chi'(0) = \frac{2}{\pi} \int_0^{\infty} \frac{\chi''}{f} df . \]  

(7.11)

The measurement coil systems were similar to the one which will be described in § 7.4.

The temperature measurement of the isolated spin system was based on the second law of thermodynamics

\[ T_n = \Delta Q/\Delta S . \]  

(7.12)

At a high external field where the sample was in paramagnetic state, the polarization of the spins was first measured, viz.

\[ p = A \int \chi'' df . \]  

(7.13)

Afterwards, the spins were demagnetized adiabatically to zero field. The heating \( \Delta Q \) was from NMR absorption of rf pulses to the sample,

\[ \Delta Q = \pi f \chi''(B_{zf}^r)^2 \Delta t / \mu_0 . \]  

(7.14)

Then the system was remagnetized back to the high field and the polarization was measured again. The entropy change could be calculated from the polarization, viz. for \( I = 1/2 \) as in Ag and Rh:

\[ \frac{S_n}{\mathcal{R}} = \ln 2 - \frac{1}{2}[(1 + p) \ln(1 + p) + (1 - p) \ln(1 - p)] ; \]  

(7.15)

and for \( I = 3/2 \) as in Cu:

\[ \frac{S_n}{\mathcal{R}} = u(\coth u - 4 \coth 4u) + \ln \frac{\sinh 4u}{\sinh u} , \]  

\[ p = \frac{1}{3}(4 \coth 4u - \coth u) , \]  

(7.16)

where \( u = \gamma hB/2k_BT_n \).

In copper, the first evidence of nuclear magnetic ordering was found from the static susceptibility measurements on a polycrystalline foil sample (Huiiku
and Loponen, 1982). After the specimen had been demagnetized to zero field, $\chi'(0)$ increased initially while the nuclear spin system warmed up owing to the spin-lattice relaxation process. After 5 min, the susceptibility started to decay exponentially, corresponding to the paramagnetic state. Such behavior is quite similar to that observed in electronic antiferromagnetics. Further experiments in a single crystal sample showed unambiguously the first order transition in this system at spin temperature of 58 nK in zero external field (Huiku et al., 1986).

The measured value of $R = -0.42$, shows an antiferromagnetic RK interaction, which is very near the absolute value of the ferromagnetic parameter $L$. The close competition between $L$ and $R$ results in a rich phase diagram with three different antiferromagnetic states (Fig. 48).

![Phase diagram](image)

Figure 48: External magnetic field vs. entropy diagram of copper nuclear spins. The three antiferromagnetic phases are noted by AF1, AF2, and AF3; P stands for the paramagnetic phase and the shaded area indicates regions where first order phase transitions take place (Huiku et al., 1986).

Using similar techniques, silver was studied with polycrystalline foil samples (see the review papers, Oja, Annila, and Takano, 1991; Hakonen et al., 1991). In silver, with $R = -2.5$, the antiferromagnetic RK interaction is dominant (Oja, Annila and Takano, 1990). The transition from paramagnetic to antiferromagnetic state was observed at 560 ± 60 pK, the lowest phase transition temperature ever measured (Hakonen, Yin, and Nummila, 1991).
At positive temperatures, the spins occupy different Zeeman levels according to the Boltzman distribution (see Fig. 49). Negative temperatures can be obtained in such a system by reversing the population of spins within the Zeeman levels. This was achieved in silver by inversion of the external field with a time constant $t \sim 1$ ms, much shorter than the spin-spin relaxation time $\tau_2 = 10$ ms. Because of the quick flip of field, the spins had no chance to redistribute among the energy levels.

At $T_n > 0$, an isolated spin system will minimize its Gibbs free energy $G$ in order to maximize the entropy. In contrast, at $T_n < 0$, $G$ should be maximized. The dominant antiferromagnetic spin interaction which results in antiferromagnetic order at $T_n > 0$, will thus lead to ferromagnetic ordering at $T_n < 0$. Such phenomena were observed in silver. At zero external field, the transition temperature was $-1.9 \text{ nK}$ (Hakonen et al., 1992).

Two polycrystalline Rh samples, made of thin foils, were studied, at both positive and negative temperatures. Unlike copper and silver, Rh has a partially filled $d$-shell. The $d$ electrons, also contributing to the electron mediated spin-spin interaction, cause an anisotropic exchange term in the Hamiltonian, viz.

$$H_{psd} = \frac{1}{2} \sum_{ij} \vec{B}_{ij} [I_i \cdot I_j - 3(I_i \cdot \hat{r}_{ij})(I_j \cdot \hat{r}_{ij})],$$

(7.17)

which can be approximated by the pseudo-dipolar coefficients $\vec{B}_{ij}$ (Narath, Fromhold, and Jones, 1966). The measured $R$ in rhodium is $-1.35$, between the $R$-values for copper and silver (Hakonen et al., 1993). Unlike the situation in silver, susceptibility measurements show that spins have a tendency to order antiferromagnetically on both sides of the absolute zero (see Fig. 50), which means that both the maximum and the minimum energy states in rhodium are antiferromagnetic. So far the ordering transition has not been observed down to temperatures as low as 280 pK.
Figure 50: Absolute value of the inverse static susceptibility $1/|\chi'(0)|$ vs. the absolute temperature at $T_n > 0$ (○) and $T_n < 0$ (●) for Rh nuclear spins. The solid and dashed lines are the antiferro- and ferromagnetic Curie-Weiss laws, calculated from experimental data at small polarizations. The error bars denote the 20% uncertainty in the measurements of temperature (Hakonen et al., 1993).

In order to reach low spin temperatures by adiabatic demagnetization of the sample, a high initial polarization, which is a function of $B/T$, is needed. From the experimental point of view, copper is the easiest material to achieve this goal because its small Korringa constant, $\kappa \approx 1.1$ sK, and relatively large magnetic moment. After 4 h of precooling by the first nuclear stage, the copper sample can be polarized to 99.9% in a field of 7.3 T. In silver, it took 20 h to reach a polarization of 94% in the same field. In rhodium, the highest polarization was 83% after 30 h of precooling in a field of 8.3 T. The difficulties arise from the long spin lattice relaxation time, and the relatively poor thermal conductivity of the rhodium foils.

Some relevant physical properties for Cu, Ag, and Rh are presented in Table 6.

### 7.3 Sample preparation

The new rhodium sample is a $25 \times 5 \times 0.4$ mm$^3$ single crystal (MaTeck Büro für Forschungsmaterialien). Table 7 gives an impurity analysis made by the manufacturer. Besides 15 ppm of Fe, there is about 5 ppm of magnetic impurities of other elements.

Heat treatment at a high temperature with oxygen was commonly used as a means of sample preparation in the previous experiments. Proper oxygen annealing can increase the $RRR$ of Cu and Ag considerably (Fickett, 1974; Ehrlich,
Table 6: Some properties of Cu, Ag, and Rh

<table>
<thead>
<tr>
<th>sample</th>
<th>Cu</th>
<th>Ag</th>
<th>Rh</th>
</tr>
</thead>
<tbody>
<tr>
<td>single crystal</td>
<td>foils</td>
<td>foils</td>
<td></td>
</tr>
<tr>
<td>0.5 × 5 × 20 mm³</td>
<td>25 μm</td>
<td>75 (25) μm</td>
<td></td>
</tr>
<tr>
<td>magnetic impurities</td>
<td>&lt; 1.3 ppm</td>
<td>&lt; 5 ppm</td>
<td>10 (100) ppm</td>
</tr>
<tr>
<td>RRR</td>
<td>8500</td>
<td>900</td>
<td>530 (250)</td>
</tr>
<tr>
<td>γ/2π (MHz/T)</td>
<td>11.3 (⁶³Cu)</td>
<td>1.72 (¹⁰⁷Ag)</td>
<td>1.34</td>
</tr>
<tr>
<td>(MHz/T)</td>
<td>12.1 (⁶⁵Cu)</td>
<td>1.98 (¹⁰⁹Ag)</td>
<td></td>
</tr>
<tr>
<td>τ₁ (at 100 μK)</td>
<td>3.3 h</td>
<td>30 h</td>
<td>28 h</td>
</tr>
<tr>
<td>P_initial</td>
<td>99.9%</td>
<td>94%</td>
<td>83%</td>
</tr>
<tr>
<td>τ₂</td>
<td>0.15 ms</td>
<td>10 ms</td>
<td>10.5 ms</td>
</tr>
<tr>
<td>T_N at 0 T</td>
<td>58 nK</td>
<td>+560 pK</td>
<td></td>
</tr>
<tr>
<td>T_C</td>
<td></td>
<td>−1.9 nK</td>
<td></td>
</tr>
</tbody>
</table>

Table 7: Impurity concentrations in the Rh sample according to the manufacturer’s analysis

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Ag</th>
<th>Al</th>
<th>Au</th>
<th>C</th>
<th>Cl</th>
<th>Co</th>
<th>Cr</th>
<th>Cu</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>(ppm)</td>
<td>&lt; 0.1</td>
<td>2.0</td>
<td>0.9</td>
<td>15</td>
<td>3.0</td>
<td>0.2</td>
<td>2.5</td>
<td>0.4</td>
<td>15</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Impurity</th>
<th>H</th>
<th>Mg</th>
<th>Mn</th>
<th>Mo</th>
<th>N</th>
<th>Na</th>
<th>Ni</th>
<th>Pt</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>(ppm)</td>
<td>&lt; 1.0</td>
<td>0.03</td>
<td>1.0</td>
<td>2.0</td>
<td>&lt; 5.0</td>
<td>0.2</td>
<td>&lt; 1.0</td>
<td>10</td>
<td>6.0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Si</th>
<th>Ti</th>
<th>Ta</th>
<th>V</th>
<th>W</th>
<th>Zr</th>
</tr>
</thead>
<tbody>
<tr>
<td>(ppm)</td>
<td>10</td>
<td>1.0</td>
<td>0.3</td>
<td>0.2</td>
<td>10</td>
<td>2.0</td>
</tr>
</tbody>
</table>

1974), thus improving their thermal conductivities at low temperatures. In a cascade nuclear cooling experiment, a good thermal contact between the sample and a first stage is important to reach sufficient precooling for high initial polarization. Furthermore, it is vital to maintain a low electronic temperature in the sample during the experiment (see § 3.1).

When the external field is on the order of the internal field B_int, the spin-spin interactions will speed up the spin-lattice relaxation. The actual spin-lattice relaxation time τ₁ can be expressed as,

\[
τ₁' = τ₂ \frac{B² + B_{\text{int}}²}{B² + (τ₂/τ_D)B_{\text{int}}²},
\]  

(7.18)

where τ₂ and τ_D are the relaxation times in the Zeeman and dipolar systems, respectively. In high fields, the energy of the spins is in the Zeeman system, whereas when B = 0 the energy is in the spin-spin interactions. In pure metals,
\( r = \tau_2 / \tau_D \) should be between 2 and 3. Early measurements on copper showed that when \( B < 15 \) mT, the magnetic impurities could significantly speed up the spin-lattice relaxation (Huiku, Loponen, Jyrkkö, Kyynäräinen, Oja, and Soini, 1984). A quick spin lattice relaxation prevented the sample from reaching low spin temperatures due to insufficient isolation from the lattice. After the copper sample was annealed at 950°C for 3 h under 0.1 \( \mu \)bar of dry air, a big drop of \( r' = \tau_1(B_{\text{high}}) / \tau_1(0) \), from 100 to 2.6, initiated the final success in observation of the ordered state in the Cu specimen (Huiku et al., 1986), here \( B_{\text{high}} \gg B_{\text{int}} \). For the silver foils, \( RRR \) was 900 and \( r' \) was between 2 and 3 after heat treatment (Hakonen et al., 1991).

---

**Figure 51:** \( RRR \) of the new Rh sample *vs.* time of heat treatment.

In the previous rhodium experiments (Vuorinen et al., 1995), two samples of different purities were used (see Table 6). The foils were heat treated at 1300°C in an oxygen atmosphere of 0.4 \( \mu \)bar for 16 h; \( r' \) was then 170 for the specimen with \( RRR = 230 \) and 50 for the one with \( RRR = 530 \).

The annealing of the single crystal was performed in a vacuum oven with an \( \text{Al}_2\text{O}_3 \) tube, which could be evacuated with a turbomolecular pump to \( 1 \times 10^{-6} \) mbar. The specimen was first cleaned in aqua regia and acetone. \( 5 \times 10^{-4} \) mbar of 99.999% pure \( \text{O}_2 \) was flowing through the tube during the heat treatment. The resulted \( RRR \) *vs.* annealing time is shown in Fig. 51. After 16 days, \( RRR \) had increased from 240 to 760. Further increase in the annealing temperature, which might help to improve the \( RRR \), was limited by the heating element of the oven.
Figure 52: The new rhodium sample, the thermal link, and the location of the measurement coils.

The heat treatment works through selective internal oxidization. In a properly chosen oxygen pressure, the host material will not be oxidized in contrast to magnetic impurities. The effective concentration of magnetic impurities, which are responsible for the scattering of conduction electrons at low temperatures and for speeding up the spin-lattice relaxation, is reduced by either transformation into non-magnetic oxides or formation of large clusters during the annealing process.

Rhodium has a melting temperature of $T_m = 1965$ °C, which is much higher than that for copper (1083°C) or silver (961°C). Typically, the selective oxidation is performed at a temperature $T \approx 0.9 T_m$. The slow diffusion of oxygen in rhodium at $T = 1530$°C = 0.78 $T_m$ might have been the limiting factor in the process. So far, the highest $RRR$ of 2200 in Rh was reported in a sample prepared by the zone melting method (Hornfeldt, Ketterson, and Windmiller, 1969).

A rough estimate for the effective concentration of magnetic impurities, $C_{eff}$, can be made by assuming that at low temperatures the resistance of Rh is totally caused by the scattering of conducting electrons from magnetic impurities, and that $C_{eff} \approx 1/RRR$ (Vuorinen et al., 1995). Compared with a resistance measurement of Rh containing 40 ppm of Fe (Rusby, 1974), $C_{eff}$ in our sample is
about 5 ppm.

Fig. 52 shows the thermal link between the first nuclear stage and the Rh sample. The silver piece was heat treated at 800°C for 48 h at $2 \times 10^{-3}$ mbar of $O_2$, resulting in an $RRR$ of 1500. A silver layer of 0.2 μm thick was evaporated onto the tip of Rh where the joint was to be made. The silver link and the sample were then diffusion welded together at 500 °C for 5 h, using a stainless steel clamp. After that, they were annealed together at 500 °C for 2 h. The contact resistance was then 60 nΩ, which is much lower than that for the earlier foil samples. The joint between the silver and copper links was made by electron beam welding (Yin and Hakonen, 1991). The copper piece was annealed similarly to the nuclear stage.

Comparisons between the better foil sample and the new single crystal specimen are given in Table 8. In the early experiments, an initial polarization of $p = 0.83$ was reached in the foils. The main factors preventing further cooling were the poor thermal conductivity of the sample itself and its joint to the silver link. As can be seen from Table 8, these two parameters have been substantially improved. This together with the longer polarization time, possible because of the long interval between helium transfers ( > 72 h), should be sufficient to reach initial polarizations above 95%.

Table 8: Comparison between the old and new Rh samples

<table>
<thead>
<tr>
<th></th>
<th>Old sample</th>
<th>New sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td>14 foils</td>
<td>single crystal</td>
</tr>
<tr>
<td></td>
<td>2.6 g</td>
<td>0.5 g</td>
</tr>
<tr>
<td></td>
<td>$25 \times 8 \times 0.075$ mm$^3$</td>
<td>$25 \times 5 \times 0.4$ mm$^3$</td>
</tr>
<tr>
<td>$RRR$</td>
<td>530</td>
<td>720</td>
</tr>
<tr>
<td>$R_{4.2K}$</td>
<td>$1.8 \mu\Omega$</td>
<td>$0.4 \mu\Omega$</td>
</tr>
<tr>
<td>Ag-Rh joint</td>
<td>$40$ mm$^2$</td>
<td>$5 \times 5$ mm$^2$</td>
</tr>
<tr>
<td>$R_{4.2K}$</td>
<td>750 nΩ</td>
<td>60 nΩ</td>
</tr>
<tr>
<td>Silver link</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$RRR$</td>
<td>1000</td>
<td>1500</td>
</tr>
<tr>
<td>$R_{4.2K}$</td>
<td>220 nΩ</td>
<td>80 nΩ</td>
</tr>
</tbody>
</table>

7.4 SQUID NMR measurement system

Fig. 53 shows our measurement system for the dynamic susceptibility experiments; it is a variant of the method originally developed by Ehnholm et al. (1980). A solenoid produces a static field in the longitudinal $z$ direction and two saddle coils generate transverse fields in the $x$ and $y$ directions. These three coils
are made of superconducting wires of 100 μm (z direction) and 50 μm diameter (x, y directions), respectively, wound on the mixing chamber radiation shield surrounding the sample. The astatically wound pick-up coil, made of 0.5 mm copper wire, consists of two 5-turn solenoids wound in opposite directions to reduce direct feedthrough of the excitation, produced by the solenoid (0.2 mm copper wire) located symmetrically outside the pickup loop. An auxiliary coil (0.2 mm copper wire) of 4 turns is wound over the upper half of the pickup loop to calibrate the gain and phase of the system. These three coils were cast in a piece of epoxy and glued inside the mixing-chamber radiation shield. There is no contact between the coils at 5 mK and the sample at a much lower temperature.

The signal from the pickup loop is fed into a DC SQUID with an input inductance of 300 nH. The measurement scheme involves a DC SQUID, a dual-phase lock-in amplifier, and a frequency synthesizer. The copper wire of the pickup coil has a resistance of approximately 1 mΩ and works as a high pass filter with a cutoff frequency at 500 Hz. A 3 Ω resistor is connected parallel to the SQUID input coil which suppresses frequencies above 10 MHz.

For low frequency measurements below 100 Hz in Rh, a low resistance pickup loop with a low cutoff frequency is preferred. But the use of superconducting wire is to be avoided. According to our earlier experience, the changes of the flux trapped in superconducting wire were the main source of noise in the measurement.
Figure 53: Arrangement for susceptibility measurements. The static field coils are wound onto the mixing chamber radiation shield. The pickup coil, the calibration coil (not shown), and the excitation coil are located inside the shield.
8 Summary

An experimentalist wishing to pursue research at low temperatures faces four technical difficulties: how to reach the low temperature, how to measure it, how to reduce the external heat leak so that the low temperature can be maintained for a sufficiently long time, and how to transfer cold from one place to another.

The above statement from Olli Lounasmaa’s book (1974) summarizes the four basic tasks for a low temperature experimentalist. In this thesis, they were discussed in details from the point of view of constructing a new cryostat.

The new YKI cryostat started performing steadily in the spring of 1996. This machine, constructed with updated technology, will continue serving research in nuclear magnetism, using double- or even triple-staged demagnetization, with the aid of a large first stage and with a powerful dilution refrigerator.

As a versatile machine, it is flexible enough to be used for other experiments requiring ultralow temperature conditions such as those planned to search for the superfluid transition in $^3$He/$^4$He mixtures.

Research in this area has been accompanied by competition for pursuing ever lower temperatures. Fig. 54 lists the road to absolute zero. Both pure $^3$He and $^3$He/$^4$He mixtures have been cooled to 100 $\mu$K (Carney, Guénault, Pickett, and Spencer, 1989; Oh et al., 1994). In metals, the electrons have been cooled to 1.5 $\mu$K (Pobell et al., 1996), while the nuclei to 280 pK (Hakonen et al., 1993).

Recently, the Bose-Einstein condensation, produced by laser cooling combined with magnetic trapping and evaporation, has shown to be another promising approach to ultralow temperatures (Anderson et al., 1995; Davis et al., 1995). With this method, micron-sized clouds of alkaline atoms can now be cooled to nanokelvin region for several seconds. The progress in this field has been very rapid since the breakthrough.
Figure 54: The temperature scale below 0 °C.
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Scholarships from the Academy of Finland and from the Chinese Academy of Sciences are gratefully acknowledged. A travel grant from the European Science Foundation is also remembered.

Last but not least, I wish to thank my wife, Yanli, for giving me a family full of love, and our daughter, Cecilia, who brings a new world into the family. I also want to thank my parents for their support.

Espoo, June 1997

[Signature]
Weijun Yao
References


Cryomagnetics, Inc. 1006 Alvin Weinsberg Drive, Oak Ridge, Tennessee 37830, USA.


Hitachi Cable, Ltd., Chiyoda Bldg, 2-1-2 Marunouchi, Chiyoda-ku, Tokyo 100, Japan.


Ingold, G. L. and Nazarov, Y. V. (1992). Charge tunneling rates in ultrasmall junctions, in H. Grabert and M. Devoret (eds), Single Charge Tunneling,


Kinetic Systems, Inc., 20 Arboretum Road, PO Box 414, MA 02131, USA.


Lake Shore Ltd., 64 East Walnut St., Westerville Ohio 43081-2399, USA.


Leiden Cryogenics b.v., Galgewater 21, 2311 VZ Leiden, the Netherlands.


MaTeck Büro für Forschungsmaterialien, Karl Heinz-Bechurts-Str. 13, D-52428 Jülich, Germany.


Outokumpu Poricopper Oy, PL 60, 28101 Pori, Finland.


Oxford Instruments, Ltd., Old Station Way, Eynsham OX8 1TL, UK.

Paloniemi, P., ABB Strömberg Drive OY, Private communication.


Picowatt RV-Elektroniikka OY, Veromiehetstie 14, 01510 Vantaa, Finland.


Precision Cryogenic Systems, Inc., 7804 Rockville Rd., Indianapolis, IN 46214, USA.

Reactor Experiments, Inc., 1275 Hammerwood Ave, Sunnyvale, CA 94089-2231, USA.


Soulen, R. J. and Marshak, H. (1980). The establishment of a temperature scale from 0.01 K to 0.05 K using noise and $^{60}$Co $\gamma$-ray anisotropy thermometers, *Cryogenics* **20**: 408.


Vacuumschmelze GmbH, Postfach 2253 D-63412, Hanau, Germany.


Appendix A: Some useful formulae and constants for simulations of nuclear refrigeration

In a system of spin 3/2:

$$\dot{p} = \frac{T_z}{2\kappa} p \left( \coth \frac{T_z}{2T_n} - \coth \frac{T_z}{2T_e} \right)$$  \hspace{1cm} (8.1)

$$p = \frac{4}{3} \coth \frac{2T_z}{T_n} - \frac{1}{3} \coth \frac{T_z}{2T_n}$$  \hspace{1cm} (8.2)

$$C_n = \frac{9}{4} nR \left( \frac{T_z}{T_n} \right)^2 \left( \frac{5}{3} + \frac{1}{9} \coth^2 \frac{T_z}{2T_n} - \frac{16}{9} \coth^2 \frac{2T_z}{T_n} \right)$$  \hspace{1cm} (8.3)

In a system of spin 1/2:

$$\dot{p} = \frac{T_z}{2\kappa} \left( 1 - p \coth \frac{T_z}{2T_e} \right)$$  \hspace{1cm} (8.4)

$$p = \tanh \frac{T_z}{2T_n}$$  \hspace{1cm} (8.5)

$$C_n = \frac{1}{4} nR \left( \frac{T_z}{T_n} \right)^2 \left( 3 + \coth^2 \frac{T_z}{2T_n} - 4 \coth^2 \frac{T_z}{T_n} \right)$$  \hspace{1cm} (8.6)

Table 9: Constants used in numerical simulations of nuclear refrigeration

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<tr>
<th></th>
<th>$^{63}\text{Cu}$</th>
<th>$^{65}\text{Cu}$</th>
<th>$^{107}\text{Ag}$</th>
<th>$^{109}\text{Ag}$</th>
<th>$^{195}\text{Pt}$</th>
<th>$^{103}\text{Rh}$</th>
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</thead>
<tbody>
<tr>
<td>Spin</td>
<td>3/2</td>
<td>3/2</td>
<td>1/2</td>
<td>1/2</td>
<td>1/2</td>
<td>1/2</td>
</tr>
<tr>
<td>$M_s$/mole $\times 10^3$</td>
<td>6.75</td>
<td>7.23</td>
<td>0.344</td>
<td>0.395</td>
<td>1.81</td>
<td>0.267</td>
</tr>
<tr>
<td>$^aT_z \times 10^4$ (K)</td>
<td>5.418$B$</td>
<td>5.804$B$</td>
<td>0.824$B$</td>
<td>0.947$B$</td>
<td>4.367$B$</td>
<td>0.643$B$</td>
</tr>
<tr>
<td>$\kappa$ (sK)</td>
<td>1.27</td>
<td>1.09</td>
<td>12 $\pm$ 1</td>
<td>10 $\pm$ 0.3</td>
<td>0.030</td>
<td>10 $\pm$ 2</td>
</tr>
</tbody>
</table>

$^aT_z = (\gamma \hbar B/k_B)$