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ADIABATIC DEMAGNETIZATION CRYOSTAT

MASTER

FOR

THERMAL EQUILIBRIUM NUCLEAR ORIENTATION

C. H. Smith and W. Weyhmann

Solid State and Low Temperature Physics Group

SCHOOL OF PHYSICS AND ASTRONOMY

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ABSTRACT

We have designed and constructed an adiabatic demagnetization cryostat for thermal equilibrium nuclear orientation. Using a slurry of chrome alum, glycerin, and water, we have cooled a sample containing 8 μCi of ^{60}Co diffused into iron to 7 m°K. The temperature was calculated from the anisotropy of the γ radiation. The warm-up rate of the sample after demagnetization was 0.1 m°K/hr at 10 m°K.

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CHAPTER I

INTRODUCTION

The difficulty of construction and high cost of experimental apparatus to orient nuclei has been richly rewarded in the past. The experiment by C. S. Wu and a group from the National Bureau of Standards which provided the evidence for the overthrow of parity conservation in weak interactions is possibly the most famous experiment using nuclear orientation.¹ The lowest temperatures attained have been achieved by adiabatic demagnetization of a system of oriented copper nuclei to a temperature of 1.2×10^{-6} °K.²

The various methods of nuclear orientation attempt to achieve an anisotropic angular distribution of the nuclear spins in a sample. An isotropic angular distribution of spins averages out any properties which are related to the direction of spin. In an even partially oriented sample, spin dependent effects can be observed.

Nuclear orientation can be used in nuclear physics to obtain information concerning multipolarity of transitions, decay schemes, nuclear spins and moments, parity, and spin dependence of nuclear forces. In solid state physics nuclear orientation provides, among other things, a method of measuring the magnitude and sign of the effective field with which the nuclear magnetic

moment interacts. These two measurements are important for an understanding of local moments, spin compensated states, and other phenomena related to hyperfine interactions. It was for such work that the apparatus described in this thesis was built.

Nuclear orientation was suggested as early as 1934 by Gorter³ and by Kurti and Simon.⁴ However, the first successful attempts were not made until 1951 by Daniels.⁵ Advances in low temperature techniques and in an understanding of hyperfine interactions in paramagnetic salts led to the successes of several methods of nuclear orientation in the early 1950's. The development of ultra-low temperature techniques and high field superconducting magnets along with the utilization of the Samoilov method⁶ have greatly extended the range of applicability of nuclear orientation in the 1960's. Samoilov discovered that most nuclei dissolved in a ferromagnetic host experience large induced hyperfine fields. By 1966 of the first one hundred elements 43 had been oriented by equilibrium methods with at least 40 more presently possible.⁷ An additional 10 elements which have not been oriented by equilibrium methods have been oriented using dynamic methods.^{8,9} The wide applicability of nuclear orientation and the amount of work yet to be done are reasons for constructing an apparatus for nuclear orientation.

In this thesis I will describe the design and operation of an apparatus for thermal equilibrium nuclear orientation. I will also discuss the performance and capabilities of the apparatus as

indicated by test runs performed with a sample of cobalt-60 dissolved in iron for which the nuclear parameters are well-known.¹⁰

CHAPTER II

THEORY

Nuclear Parameters

Consider an isolated nucleus with spin $\vec{I}$ and magnetic dipole moment $\vec{\mu} = g_N \mu_N \vec{I}$ where g_N is the nuclear g-factor and μ_N is the nuclear magneton. If we choose some arbitrary z axis, the projection I_z of the spin on that axis is quantized and characterized by the $2I+1$ values of the magnetic quantum number m . Here I is the maximum projection of $\vec{I}$ on the z axis. Similarly μ_z has $2I+1$ possible values from $-g_N \mu_N I$ to $+g_N \mu_N I$. In the absence of any interaction with an external magnetic field the $2I+1$ quantum states are degenerate in energy. An ensemble of such nuclei in thermal equilibrium has equal numbers of nuclei in each quantum state.

In the presence of a magnetic field $\vec{H}$ the degeneracy is removed. The z axis can now be taken as the direction of the applied field, and the energy of interaction, $-\vec{\mu} \cdot \vec{H}$, can be written $\mu_z H = g_N \mu_N m H$ where m takes values $-I, -I+1, \dots, I-1, I$. In an ensemble of nuclei in thermal equilibrium the populations of the quantum states are determined by the Boltzmann distribution:

$$N_1/N_2 = \exp (-\Delta/kT). \quad \text{II-1}$$

N_1 and N_2 are the populations of two states, Δ is the energy difference between the states, k is Boltzmann's constant, and T is the absolute temperature. If a_m and a_{-m} are the

populations of quantum states characterized by the magnetic quantum numbers m and m' respectively, then

$$a_m/a_{m'} = \exp [H g_N \mu_N (m-m')/kT]. \quad \text{II-2}$$

The orientation of an ensemble of nuclei can be uniquely specified by the $2I+1$ a_m 's corresponding to the $2I+1$ values of m . If we include the normalization condition $\sum_m a_m = 1$ there remain $2I$ independent a_m 's. In calculations expressions of the form $\sum_m m^n a_m$ called the n th moments of the a_m 's occur; therefore, $2I$ independent linear combinations of the moments are introduced. These orientation parameters designated $f_k(I)$ completely specify the orientation of the nuclei. They are defined by the equations

$$f_k(I) = \sum_{n=0}^k c_{k,n} \sum_m m^n a_m, \quad \text{II-3}$$

$$f_k(I) = 0 \text{ if } a_m = \sum_{p=0}^{k-1} c_p m^p \text{ for arbitrary } c_p, \quad \text{II-4}$$

$$\text{and } c_{k,k} = I^{-k} \quad (\text{normalization}).^{11} \quad \text{II-5}$$

By letting $a_m = m^q$ ($q = 0, \dots, k-1$) in II-4 we obtain from II-3 k linearly independent equations in the $k+1$ coefficients $c_{k,0}, \dots, c_{k,k}$. With the additional condition in II-5, the $c_{k,n}$'s are completely determined. For an arbitrary orientation the dependence of a_m on m can be expressed in the form

$$a_m = \sum_{p=0}^{2I} c_p m^p. \quad \text{II-6}$$

Therefore, it is obvious from II-4 that the f_k 's are identically

zero for $k \geq 2I + 1$. The orientation parameters are all zero for a system in which the a_m 's are equal. Such a system is said to have no orientation. This property is easily verified for the first two orientation parameters

$$f_1 = I^{-1} \sum_m a_m m \quad \text{II-7}$$

$$\text{and} \quad f_2 = I^{-2} \sum_m a_m [m^2 - I(I+1)/3]. \quad \text{II-8}$$

In analyzing data we also use the orientation parameter of order four:

$$f_4 = I^{-4} \left[\sum_m m^4 a_m - \frac{(6I^2 + 6I - 5)}{7} \sum_m m^2 a_m + \frac{3}{35} I(I-1)(I+1)(I+2) \right]. \quad \text{II-9}$$

A general form for $f_k(I)$ can be derived from II-3, II-4, and II-5:

$$f_k(I) = \left(\frac{2k}{k} \right)^{-1} I^{-k} \sum_m \sum_{q=0}^k \frac{(-1)^q}{q!} \frac{(I-m)!(I+m)!}{(I-m-q)!(I+m-k+q)!} \left(\frac{k}{q} \right)^2 a_m. \quad \text{II-10}$$

Orientation can be further classified as polarization or alignment. If one or more of the "odd" $f_k(I)$'s are nonzero, the system is said to be polarized. If one or more of the "even" $f_k(I)$'s are nonzero while all of the "odd" $f_k(I)$'s are zero, the system is said to be aligned. Polarization occurs when the nuclear spins tend to be in the same direction as some axis. Alignment occurs when the nuclear spins tend to be along an axis but are equally likely to be parallel or antiparallel to it. In an aligned system $a_m = a_{-m}$.

Production of Oriented Nuclei

To orient the nuclei in a sample one must establish differences between the populations of the magnetic sublevels. An appreciable difference between two a_m 's is reached in thermal equilibrium when the absolute value of the exponent in the Boltzmann equation is of the order of 10^{-1} or greater. With equation II-2 this condition requires for two states with m 's differing by one that $g_N \mu_N H / kT \geq 10^{-1}$. Since g_N is of the order of unity, $\mu_N = 5.05 \times 10^{-24}$ ergs/gauss, and $k = 1.38 \times 10^{-16}$ ergs/deg, the condition becomes $H/T \geq 3 \times 10^6$ gauss/deg. In other words, a temperature of 0.01 °K would require a field greater than 3×10^4 gauss to produce a 10% population difference between adjacent energy levels.

The "brute force" method of nuclear orientation requires temperatures below 0.01 °K and large applied fields to achieve orientation. Most equilibrium methods, however, depend upon interactions other than the direct coupling of an external field to the nuclear magnetic moment. The various methods of nuclear orientation and the interactions which contribute to them are discussed in several review articles and books.^{12, 13, 14, 15} I will only discuss the particular method used in our experiment, that of orientation of nuclei dissolved in a ferromagnetic host.

The local magnetic field $\vec{H}_\ell$ within a domain of a ferromagnetic material can be written

$$\vec{H}_\ell = \vec{H}_0 + 4\pi\vec{M}/3 - D\vec{M} + \vec{H}' \quad \text{II-11}$$

where $\vec{H}_0$ is the applied field, $4\pi\vec{M}/3$ is the Lorentz field, $-D\vec{M}$ is

the demagnetization field, and $\vec{H}'$ is the field from the dipoles in the Lorentz cavity. For our sample, a thin foil parallel to the applied field, the demagnetization factor is zero,¹⁶ and $\vec{H}'$ is zero as it is for any cubic crystalline structure.

A ferromagnetic material is characterized by domains within which there exists a spontaneous magnetization even in the absence of an applied field. In iron the "easy" directions of magnetization and hence the directions of spontaneous magnetization within the domains are along the [100], [010], and [001] directions. Except for hysteresis effects the net magnetization of a multiple domain sample of iron averages to zero in the absence of an applied field. A very pure single crystal of iron cut into a long cylinder with its axis along an "easy" axis of the crystal can reach its saturation magnetization of 1740 gauss at 0° K¹⁷ with a field of a few gauss applied parallel to the axis. The domains with spontaneous magnetization parallel to the applied field grow at the expense of the other domains until the sample becomes a single domain. A polycrystalline sample, however, requires an applied field on the order of a thousand gauss to reach its saturation magnetization. At first the most favorably oriented domains grow at the expense of less favorably oriented domains. Finally these domains rotate until they are parallel to the applied field. Therefore, with a polycrystalline sample as used in this experiment, the local field is not much larger than the applied field.

A large increase in the splitting between energy levels with different values of m comes from the interactions of the nuclear spin with the spins of electrons of its own atom and with the spins of the itinerant electrons of the host. The d electrons of the solute are polarized by exchange interaction with the electrons of the ferromagnetic host. The core s electrons of the solute, polarized by exchange interaction with the d electrons of the solute, orient the solute nuclei by means of the Fermi contact interaction. The itinerant s electrons of the host are polarized by exchange interaction with the ferromagnetically polarized d electrons localized on the host sites. The polarized s electrons can migrate to solute sites and interact with the solute nuclei -- again by means of the Fermi contact interaction. These interactions are known as hyperfine interactions and are discussed in detail elsewhere.¹⁸ The energy of interaction can be written as the interaction of a hyperfine magnetic field with the nuclear magnetic moment. The total effective magnetic field that a nucleus experiences in a ferromagnet is

$$\vec{H}_{\text{eff}} = \vec{H}_{\text{hfc}} + \vec{H}_l. \quad \text{II-12}$$

$\vec{H}_{\text{hfc}}$ is the "corrected" hyperfine field representing only the hyperfine interaction. The hyperfine field usually reported in the literature is

$$\vec{H}_{\text{hf}} = \vec{H}_{\text{hfc}} + 4\pi\vec{M}/3 + \vec{H}', \quad \text{II-13}$$

as $\vec{H}_{\text{hfc}}$, $\vec{M}$, and $\vec{H}'$ are essentially parallel or antiparallel.¹⁹

The effective field is then written

$$\vec{H}_{\text{eff}} = \vec{H}_{\text{hf}} + \vec{H}_0 - D\vec{M}. \quad \text{II-14}$$

For our experimental situation we can write the Hamiltonian for the magnetic energy levels as

$$\mathcal{H} = -\vec{\mu} \cdot \vec{H}_{\text{eff}} \quad \text{II-15}$$

or
$$\mathcal{H} = -g_N \mu_N m (H_{\text{hf}} + H_0) \quad \text{II-16}$$

where the z axis is taken along $\vec{H}_0$ as usual. The value of g_N is given for the solute nucleus, and H_{hf} must be measured experimentally for the particular combination of solute and host.

Angular Distribution of Radiation from Oriented Nuclei

To determine the degree of orientation achieved in a sample, we use a gamma emitting isotope as the nucleus to be oriented and observe the angular distribution of the gamma radiation. The angular distribution of γ radiation from a sample with all its spins parallel is the same as the angular probability distribution for emission of a γ from a single nucleus. Both distributions are determined by the nuclear transition producing the γ radiation. For example a quadrupole transition in which I changes by 2 and m changes by ± 2 will result in the radiation pattern

$$W(\theta) = 5(1 - \cos^4\theta)/4, \quad \text{II-17}$$

where $W(\theta)$ is proportional to the intensity in the direction θ

from the z axis. The pattern is rotationally symmetric about the z axis. $W(\theta)$ is normalized so that $\int W(\theta) d\Omega = 4\pi$ where $d\Omega$ is an element of solid angle.

The radiation pattern from a sample with arbitrary nuclear orientation can be expanded in terms of the Legendre polynomials $P_k(\cos \theta)$:

$$W(\theta) = \sum_k D_k f_k(I) P_k(\cos \theta). \quad \text{II-18}$$

The "amount" of orientation is specified by the factors $f_k(I)$ which have been previously defined. The angular momentum properties of the transition are included in the D_k 's. In the limit of no orientation $f_k(I) = \delta_{0,k}$, so $W(\theta) = D_0$. Since the D_k 's are not functions of θ , to preserve normalization $D_0 = 1$; therefore, $W(\theta) = 1$. With no orientation there is no preferred direction, and the radiation is isotropic.

Figure 1 shows the angular distribution of γ radiation appropriate for oriented ^{60}Co at various temperatures. The intensity at angle θ is proportional to the length of an arrow from the origin to the edge of the pattern inclined at an angle θ from the z axis.

Half-lives for γ emission in nuclei used for nuclear orientation are usually very short--less than 10^{-12} seconds. To achieve thermal equilibrium, however, several seconds are necessary. To install a sample in our apparatus and cool it from room temperature to the millidegree region requires several hours. Hence, β -radioactive parent nuclei which have sufficiently

Figure 1. Angular distribution of γ radiation from oriented ^{60}Co .
(Temperatures and f_k 's for ^{60}Co in Fe.)

long half lives for handling and orientation are used. The oriented nucleus β decays to a short-lived state which in turn decays by the desired γ transition or possibly decays by a cascade of γ transitions, one or more of which are used for detection of orientation. Figure 2 shows one such decay scheme.

Figure 2. Decay scheme of ^{60}Co . (Weak β and γ transitions omitted.)

The two γ transitions of the excited states of ^{60}Ni , $I_i=4 \rightarrow I_f=2$ and $I'_i=2 \rightarrow I'_f=0$, following the β transition from ^{60}Co are quadrupole. For a pure quadrupole transition in which $I_f=I_i-2$,

$$W(\theta) = 1 - \frac{15}{7} N_2(I_i) f_2(I_i) P_2(\cos \theta) - 5 N_4(I_i) f_4(I_i) P_4(\cos \theta),^{11} \quad \text{II-19}$$

Here we have used N_k to represent the particular nuclear parameters D_k which apply to transitions in which the spin

decreases:

$$N_2(I) = I/(2I-1) \quad \text{II-20}$$

and
$$N_4(I) = I^3/(I-1)(2I-1)(2I-3). \quad \text{II-21}$$

There is not sufficient time for the short-lived state following the β transition to reach thermal equilibrium before it γ decays. The populations of its magnetic sublevels are determined by the partial transition probabilities for the transitions $(I_0, m_0) \rightarrow (I_1, m_1)$. We can use these probabilities to relate the a_{m_1} 's to the a_{m_0} 's and hence the $f_k(I_1)$'s to the thermal equilibrium $f_k(I_0)$'s before the β transition. Fortunately the relationship is particularly simple for ^{60}Co . For a transition $I_0 \rightarrow I_1 = I_0 - L$ where L is the angular momentum carried away by the radiation,

$$N_k(I_1)f_k(I_1) = N_k(I_0)f_k(I_0). \quad \text{II-22}$$

The particles from the β transition carry away one unit of angular momentum ($L=1$), and as mentioned before both γ transitions are quadrupole ($L=2$). Looking at Figure 2, it is easy to verify that $I_1 = I_0 - L$ and $I_1' = I_0' - L$. Therefore,

$$N_k(I_1')f_k(I_1') = N_k(I_0')f_k(I_0') \quad \text{II-23}$$

and
$$N_k(I_1)f_k(I_1) = N_k(I_0)f_k(I_0). \quad \text{II-24}$$

But I_1 and I_0 are the same, so either of the γ transitions can be described by using $N_k(I_0)f_k(I_0)$ in equation II-19. Using the expressions for $N_k(I)$ from II-20 and II-21 with $I_0=5$ in

II-19, we get

$$W(\theta) = 1 - \frac{25}{21} f_2(I_0) P_2(\cos \theta) - \frac{625}{252} f_4(I_0) P_4(\cos \theta). \quad \text{II-25}$$

Equation II-25 gives the radiation pattern for either of the γ 's from a ^{60}Co sample.

CHAPTER III

APPARATUS

Design Considerations

Equilibrium nuclear orientation requires a cryostat which can maintain a sample at temperatures in the millidegree region long enough to insure that the sample is in thermal equilibrium during the time in which each measurement is taken. Adiabatic demagnetization of a paramagnetic salt is a well known technique for producing the required temperatures. Careful design to minimize possible heat leaks and the use of a relatively large salt pill insure a slow warm-up rate.

The utilization of the negative heat of solution of liquid ^3He in liquid ^4He has provided another method of reaching millidegree temperatures. The ability of a ^3He - ^4He dilution refrigerator to handle large heat loads and to provide steady state operation at low temperatures promises to be of great use in nuclear orientation experiments. In the fall of 1966, however, when the apparatus described in this thesis was designed and construction was started, dilution refrigerators were in their brief infancy. Only recently have they become capable of reaching temperatures below $10\text{m}^\circ\text{K}$.^{2b}

When a magnetic field is applied to a paramagnetic salt, the spins tend to align themselves with the field. This increase in order corresponds to a decrease in the entropy of the spin

system. If the magnetic field is then reduced adiabatically, the entropy of the spin system will correspond to a temperature lower than the one at which it started.

The entropy of a system of N spins in $2S+1$ magnetic sublevels can be written

$$S = k(N \log N - \sum_{i=1}^{2S+1} n_i \log n_i) \quad \text{III-1}$$

where n_i is the population of the i th magnetic sublevel. The populations of the sublevels are determined by the Boltzmann distribution; therefore, the entropy removed is a function of H/T . For a paramagnetic salt with spin $3/2$, the entropy of the spin system is reduced to 20% of its maximum value of $Nk \log 4$ if H/T is 20kG/deg and to 10% if H/T is 30kG/deg. Superconducting magnets providing 20kG over a volume of 1000 cm³ are easily available. To remove 80% of the entropy from the paramagnetic salt before demagnetization, temperatures close to 1°K are desirable. To remove even more of the entropy by increasing H/T , it is easier and less expensive to reduce the temperature than to utilize even more powerful magnets; therefore, we chose to pump on liquid ³He rather than liquid ⁴He to cool the paramagnetic salt. Evaporators using liquid ³He can reach 0.3°K compared with about 1°K for ⁴He evaporators. The higher vapor pressure of liquid ³He and the absence of a superfluid transition -- at least in the temperature range of an evaporator -- are reasons why ³He can be pumped to a lower temperature than ⁴He.

Another reason to use the lower temperature of a ^3He evaporator is to reduce the heat leak to the paramagnetic salt after demagnetization. The largest heat leak to the salt pill is due to thermal conduction through the superconducting lead heat switch between the salt pill and the evaporator. The thermal conductivity of lead in its superconducting state goes as T^3 at low temperatures. The amount of heat conducted from the evaporator to the salt pill, whose temperature can be taken as zero, will go as the fourth power of the temperature of the evaporator. Reduction of the temperature of the evaporator from 1 to 0.3°K will decrease the heat leak by a factor of one hundred. The heat leak due to thermal radiation can be reduced by enclosing the salt pill and sample in a radiation shield connected to the evaporator. The radiation heat leak will then also go as the fourth power of the temperature of the evaporator. The lower temperature of a ^3He refrigerator is well worth its extra effort and cost.

Helium-3 at present costs about \$100 per STP liter. Therefore, a sealed system must be used in order to recover and recirculate the ^3He . Whereas ^4He is transferred into the apparatus as a liquid, ^3He must be liquified within the cryostat. The ^3He is liquified by compressing it to about 25 Torr and cooling it to below 2°K by thermal contact with a ^4He evaporator. Both evaporators are isolated from an outer liquid helium bath by a vacuum space. The entire liquid helium bath could be pumped to about 1°K, but the use of a ^4He evaporator separated from the main bath by a vacuum space enables us to refill the helium dewar without disturbing the ^3He refrigerator.

Figure 3 is a schematic representation of the apparatus showing the temperatures attained by various parts. The individual parts will be discussed in subsequent sections.

Stand and Dewars

The cryostat is shielded from the outside world by a concentric pair of glass dewars filled respectively with liquid nitrogen and liquid helium. Both liquids are boiling at atmospheric pressure. The dewars are suspended from a tubular aluminum stand. The legs and cross braces of the stand are filled with sand to damp vibrations. The inner dewar, 4 in. i.d. $\times$ 60 in. long, is sealed to the head block by a standard glass pipe joint. The outer dewar is suspended by a styrofoam padded plywood ring at the point where its diameter decreases. On the top of the dewar stand several valves and two diffusions pumps are mounted.

As the total height of the dewar stand is over ten feet, access to the cryostat is achieved by lowering the dewars with a system of pulleys and ropes into a cart and rolling the cart out of the way.

Cryostat

A schematic diagram of the cryostat is shown in Figure 4. For clarity the liquid nitrogen dewar is not included. The superconducting magnets for polarizing the sample and for saturating the paramagnetic salt are located in the liquid helium bath at 4.2°K. A vacuum can isolates the low temperature parts of

Figure 3. Block diagram of apparatus.

Figure 4. Schematic of cryostat.

the cryostat from the 4.2°K bath. Returning ^3He is liquified by a condenser in thermal contact with a ^4He evaporator at 1°K. The ^4He evaporator is filled from the 4.2°K bath through a needle valve and capillary. The pressure drop in the ^3He return line from 10 Torr at 1°K in the condenser to 10 mTorr at 1/3°K in the ^3He evaporator is provided by a length of capillary which serves as an impedance. Most of the capillary is in thermal contact with the ^3He evaporator. A radiation shield enclosing the salt pill and sample is attached to the ^3He evaporator. This shield at 1/3°K reduces the heat leak to the salt pill due to thermal radiation. The salt pill is thermally connected to the ^3He evaporator by a lead heat switch located in the fringing field of the demagnetizing magnet. The sample is soldered to copper fins imbedded in the salt pill. The distance from the sample to the salt pill insures that the polarizing magnet does not magnetize the salt pill and increase its temperature.

^4He evaporator. -- Figure 5 shows the ^4He evaporator in a cutaway view. It is machined from copper with a 1/4-in. pitch spiral groove 1/16 in. deep cut in its outside surface to accept the condenser tube. The volume of the evaporator is 77 cm³. Use of a reentrant bottom increases the length of the thermal conduction path along the pumping line between the ^3He and ^4He evaporators. Filling is regulated by a needle valve located in the 4.2°K liquid helium bath. The valve is connected to the evaporator by a 0.024-in. i.d. stainless steel capillary. The needle valve utilizes the valve stem from a Hoke 1325M4B valve.

Figure 5. Cutaway diagram of evaporators
and ^3He pumping line.

This valve stem with 56 turns per inch and a 3° taper on the needle allows very fine adjustment. The valve body is machined from brass and is soft soldered to the top of the vacuum can. A diagram of the valve is shown in Figure 6. The valve is operated by a 1/8-in. o.d. $\times$ 0.020-in. wall stainless steel tube soldered to the valve stem. The tube, which extends through a Veeco C-12 quick vacuum coupling on the cryostat flange, is turned by a knurled handle which can be reached from the top of the dewar stand.

A brass pumping orifice 0.1 cm in diameter by 0.5 cm long is soldered in the ^4He pumping line just above the evaporator to reduce superfluid film flow.

Condenser and impedance. -- The condenser consists of 60 inches of 3/32-in. o.d. $\times$ 1/32-in. wall copper tube. The total volume of the condenser is 0.8 cm³. The tube is wrapped into the spiral groove around the ^4He evaporator and soldered into it with cadmium-bismuth solder.²¹ The Cd-Bi solder has been found to have much better thermal conductivity at low temperatures than the usual tin-lead soft solders.²²

The impedance is furnished by 10 in. (25 cm) of 0.018-in o.d. $\times$ 0.004-in. wall stainless steel capillary followed by 20 in. (50 cm) of 0.008-in. o.d. $\times$ 0.002-in. wall stainless steel capillary with a 0.002-in. tungsten wire inside it. The smaller capillary is wrapped around the ^3He evaporator.

Figure 6. ⁴He needle valve.

Figure 7. Transfer tube tip and extension.

^3He evaporator. -- The ^3He evaporator shown in Figure 5 is also machined from copper. It has an integral flange on its bottom which has threaded connections for both the graphite support rod and for the radiation shield. Inside the evaporator a spiral of 12 turns of 1/64-in. copper sheet is soldered to the bottom. The copper spiral provides a vertical thermal path to establish thermal equilibrium within the liquid ^3He . With the copper spiral, the available volume in the evaporator is about 7 cm³.

Pumping lines. -- The pumping line for ^3He is shown in Figure 5. The tube size increases from 3/8-in. at the coldest part to 2-in. at room temperature. Sizes and lengths of the tubes are given in the figure. The ^4He pumping line is similar to the one shown except the tube from the ^4He evaporator to the top of the vacuum can is 2 in. of 1/4-in. o.d. x 0.010-in. wall stainless steel tube.

Both lines are optically baffled at the top of the vacuum can and at the point where the lines increase from 3/4 to 1-1/2 inches in diameter. The highest baffles are at the top of the liquid nitrogen dewar. The purpose of the baffles is to reduce the thermal radiation down the pumping lines.

Radiation shield. -- The radiation shield consists of two pieces of Textolite tubing ²³ 3/4 and 2-3/16 inches in diameter epoxied to each other and to a copper collar with Epibond 100-A.²⁴ The collar threads onto the flange at the bottom of the ^3He evaporator. The outer surface of the tubes is covered with

32 gauge Formvar coated copper wires. The wires are soldered to the collar with Cd-Bi solder and epoxied to the shell with Epibond 100-A and Epibond 121. The wires are arranged on the cylinder in a single dense longitudinal layer for minimal thermal path length. Two thirds of the wires end at the junction of the tubes. The remaining wires form a dense single layer on the smaller tube.

Heat switch. -- The thermal link or heat switch between the salt pill and the ^3He evaporator is made from 99.9999% lead rolled to 0.013 cm thickness. A piece 0.4 cm wide by 2.5 cm long is attached with Cd-Bi solder horizontally between extensions from the ^3He evaporator and from the salt pill. The switch is located in the fringing field of the demagnetizing magnet. When the magnet is at full field the lead is driven normal and conducts heat. When the magnet current is reduced below $3/4$ maximum, the lead becomes superconducting and a poor conductor of heat due to the pairing of electrons with positive and negative momenta.

We found that by tilting the lead switch so that its width was not quite vertical we could reduce the thermal conductivity in the off state. An angle of about 30° from vertical seemed to provide enough transverse component of the magnetic field to prevent magnetic flux from being trapped in the lead switch. Trapped magnetic flux leads to areas of normal conductivity and hence higher thermal conductivity when the switch is off.

Salt pill. -- The shell of the salt pill is also made from Textolite tubing and Epibond 100-A. A diagram is shown in Figure 8. Two sets of copper fins are spaced by blocks of Textolite and epoxied into the tubes. One set of fins with a surface area of 570 cm^2 in contact with the paramagnetic salt is gathered at the top of the salt pill, soldered together with Cd-Bi solder, and soldered to the heat switch. A second set of fins which do not make electrical contact with the first set serves as thermal contact between the salt and sample. These fins have a contact area with the salt of 880 cm^2 . Below the larger tube this set of fins are reduced in width, gathered, and extended through the smaller tube of the shell. The sample is soldered to the fins below the bottom of the small tube.

The salt pill is supported from the ^3He evaporator by a graphite support rod. The high thermal conductivity of pitch bonded graphite above liquid nitrogen temperatures rapidly decreases at lower temperatures.²³ Below 1°K it provides very good thermal isolation. The large high-temperature conductivity of graphite decreases the time necessary for precooling, and its rigidity reduces vibrational heating. Pitch bonded graphite also machines very easily.

Centering of the salt pill and radiation shield within the vacuum can is accomplished by wrapping a short two layer open spiral of string around the bottom of the smaller tube of each unit. The string wrapped unit then fits snugly inside the next concentric layer. In the case of the salt pill, an extra

Figure 8. Salt pill shell.

layer, a short section of 9/16-in. Textolite tube wrapped with string, was necessary to fill in between the 7/16-in. diameter salt pill and 3/4-in. diameter shield. The string with its many interfaces provides a poor thermal path and small contact area between layers. A snug fit damps out vibrations.

The salt pill is filled with a slurry of powdered chrome alum ($\text{KCr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) mixed with a solution of half glycerin and half saturated aqueous solution of chrome alum. A mixture of 4 parts solution to 17 parts powdered chrome alum by weight provides a fairly thick slurry which can be stuffed into the salt pill and tamped down. The volume of the salt pill is about 300 cm^3 , and the total amount of chrome alum it contains when filled with slurry is 0.8 gram ions.

Sample. -- The sample used for calibration of the apparatus is a 0.8 cm square of iron foil 0.013 cm thick with about 10 μCi of ^{60}Co diffused into it. For details of sample preparation see Appendix A. The sample is soldered with indium to one of the copper fins which protrude from the bottom of the salt pill. The remaining copper fins are then greased with Apeizon N grease ²⁶ and tied together. An earlier attempt using only N grease to hold the sample to the copper fins did not reach as low temperatures as later attempts with the sample soldered on. We believe that the heat from absorption within the sample of β particles was not conducted away as well by contact with only N grease resulting in a thermal gradient between the sample and the salt pill.

Magnets. -- The magnet used for saturating the paramagnetic salt was made by Westinghouse. It has a 2.50-in. bore and is 9-1/4 in. long with 16,102 turns of superconducting wire. It has a rated field of 15 kilogauss at 18.4 amperes; however, we usually operate at 18 kilogauss with 22 amperes current. The magnet is equipped with a persistent current switch consisting of a length of superconducting wire connected across the magnet coil and a heater in close proximity to the wire. The heater allows the switch to be driven normal so the current in the magnet can be increased or decreased. If the heater is turned off, the switch becomes superconducting and the current in the magnet flows through a continuous superconducting circuit. The power supply can then be turned off or removed.

To increase the efficiency of cooling the demagnetizing magnet from liquid nitrogen temperature to liquid helium temperature, the end of the liquid helium transfer tube is below the magnet. In this way the enthalpy of the gas as well as the latent heat of evaporation of the liquid is used to cool the magnet. A conical Teflon tip with a 20° taper is threaded on the end of the vacuum jacketed transfer tube. This tip seals against a matching tapered socket at the end of the guide tube in the cryostat to allow the liquid helium to be forced through a tube which extends to the bottom of the magnet. Figure 7 shows a view of the tip and socket. Earlier attempts with a 45° tapered tip on the transfer tube were not as successful.

The magnet used for polarizing the sample is a 1-in. bore split solenoid wound in this laboratory. A split solenoid with the sample centered between its halves is used to minimize attenuation and scattering of the γ rays seen by the detector perpendicular to the axis of the magnet. The geometry of our solenoid provides a "window" within 21° of a line through the center of the solenoid perpendicular to its axis. With 25 amperes current (the maximum from our power supply) in the 910 turns of insulated copper coated 0.010-in. niobium-zirconium wire, the calculated field at the center of the magnet is 4.3 kilogauss.

The polarizing magnet is also equipped with a persistent current switch. A 120 cm length of bare Ni-Zr wire is wound into a spiral groove in a 0.9 cm diameter solid rod of Textolite. The pitch of the spiral allows space for 120 cm of Formex coated 36 gauge Evanohm wire²⁷ wound between the turns of the superconductor wire. The room temperature resistance of the heater wire is 160 ohms. Epibond 100-A secures the wires to the rod and acts as insulation. Eight layers of masking tape provide enough additional insulation to allow the switch to be driven normal in liquid helium by 30 mA heater current or 108 mW power dissipated. The ends of the superconducting wire are spot welded to the leads for the polarizing magnet. The heater leads are connected to copper wires leading to one of the feedthroughs on the cryostat flange.

The demagnetizing magnet is supported from the cryostat head by three 1/4-in. o.d. x 0.010-in. wall stainless steel tubes with threaded end plugs. The polarizing magnet is suspended from the bottom of the demagnetizing magnet by three 1/4-in. aluminum rods. A layer of tape around the bottom of the vacuum can insures a snug fit between can and polarizing magnet to prevent vibration of the magnets and the associated eddy current heating of the sample.

The current for the superconducting magnets is supplied by a Westinghouse 503B magnet controller. This transistorized unit provides up to 25 amperes current with a RMS current ripple of less than 0.01%. It also supplies power for the persistent current switch heaters. The current can be adjusted manually or can be increased or decreased linearly by a motor. The speed of the motor is variable between 2-1/2 and 20 minutes for full increase or decrease of the magnet current.

The current to the magnets is supplied through a 3 wire 10 gauge cable to the cryostat head. Inside the cryostat each current lead consists of 8 insulated 30 gauge wires 1.4 meters long. Since the magnets are in the persistent mode most of the time, the leads are smaller than the optimal size for minimum helium loss during continuous operation.

Heaters. -- Two 100 ohm heaters of 36 gauge Evanohm wire wound and epoxied on copper cylinders are located on the ^3He evaporator and on one of the copper fins of the salt pill. The heaters are used to boil off ^3He when necessary and to increase the temperature of the salt pill. Power to the heaters is supplied

by two separate 0-15 volt 250 mA power supplies. A third 500 ohm heater wound around the bottom of the polarizing magnet is used to boil off the liquid helium in the 4.2°K bath at the end of a run. It is supplied by a transformer.

Gas Handling System

Figure 9 is a schematic of the gas handling system. The pumping system for the ⁴He evaporator and the gas handling system for the ³He refrigerator are shown except for parts in the cryostat itself. For ease of operation much of the gas handling system is mounted on a 1/4-in. aluminum plate attached to the laboratory wall. The diffusion pumps and associated valves are mounted on top of the dewar stand. Ports are provided on both of the pumping lines for attaching a mass spectrometer leak detector.

The diffusion pumps in both systems are Consolidated Vacuum Corporation (CVC) PMSC-2C 2 in. oil diffusion pumps. A CVC BCR-21A 2 in. air-cooled chevron baffle and a CVC VCS 22-A 2 in. gate valve are mounted above each diffusion pump.

The backing pump for the diffusion pump in the ⁴He system is a Precision Scientific Company D-150 mechanical pump with a nominal pumping speed of 2-1/2 liters/sec. The mechanical pump is usually used alone on the ⁴He evaporator. The ⁴He diffusion pump is used to evacuate the ⁴He and ³He systems within the cryostat before each run. A cross connection between the two systems is provided for this operation.

Figure 9. External Gas Handling System.

The diffusion pump in the ^3He system is backed by a Welch 1402 KBG shaft sealed mechanical pump. This nominally 2 liters/sec pump has a special oil sealed shaft to prevent air from leaking into the exhaust side of the pump. The ^3He pumped from the evaporator is compressed by the pump and passes through a fiberglass filter in the exhaust line to trap pump oil and into a 2 liter ballast tank. The ballast tank together with the dead volume of the pump have sufficient volume to contain all the ^3He in the system without exceeding one atmosphere should the return line be plugged in the cryostat. A cold trap filled with CVC Sorbent A and immersed in liquid nitrogen removes impurities from the ^3He before it is returned to the condenser. Valves on the dewar stand allow ^3He to be returned either to the condenser or directly to the evaporator via the pumping line.

An interconnection between the ^3He pumping and return lines on the board allows the ^3He to be circulated in a closed loop on the board through the cold trap to purify the ^3He . After each run the cold trap is valved off from the rest of the ^3He system, a valve to the ^3He pumping line is opened, and the cold trap is heated and pumped out to remove collected impurities. An automatic Toepler pump is used to pump out the ^3He remaining in the exhaust side of the mechanical pump and associated lines before changing oil or repairing the pump. Sufficient valves are provided to return the ^3He to the storage tank or to add additional ^3He from a new tank without allowing air into the system. The system is presently filled with 2-1/2 STP liters of ^3He .

The pressure in various parts of the ^3He and ^4He systems is monitored by Pirani and thermocouple gauges. The amount of ^3He in the ballast tank is indicated by a vacuum gauge reading 0-30 in. Hg. The condenser pressure is monitored by a Heraeus 0-600 Torr diaphragm gauge.

Thermometry

The primary thermometry for a sample mounted in the cryostat utilizes the anisotropy of the γ rays from a known sample. Resistance thermometry is used to monitor the temperature of various parts of the apparatus. The behavior of Speer carbon resistors at low temperatures is well-known.²⁶ The reproducibility of their behavior after temperature recycling is good enough for our purposes to use the resistors without recalibrating them for each run.

An Allen Bradley 470 ohm resistor is located in the liquid helium bath for use during precooling. Two Speer 1002 grade 470 ohm resistors are mounted in copper blocks with Apezion N grease for thermal contact. The blocks in turn are mounted with N grease on the ^4He and ^3He evaporators. A 1002 grade 100 ohm Speer resistor is wrapped and epoxied in a copper sheet soldered with Cd-Bi solder to a fin in the salt pill. A 560 pf silver-mica capacitor is connected in parallel with each resistor to shunt any rf power picked up by the twisted pair leads. The leads are filtered by LC filters just below the cryostat flange and just above the feedthroughs into the vacuum can. The filters below the cryostat flange are 1/2 MHz low pass filters. A circuit diagram is shown in Figure 10.

Figure 10. 1/2 MHz LC filter.

The ferrite bead (Ferroxcube #56 390 31/4B)²⁹ slipped over the wires acts as a series inductance and resistance at high frequencies. The filters above the vacuum can are 10 MHz low pass filters. A circuit diagram is shown in Figure 11.

Figure 11. 10 MHz LC filter.

Miniature silver mica capacitors are used in the 10 MHz filter because they have a very low temperature coefficient. The filters at the vacuum can are immersed in liquid helium.

All leads enter the vacuum can through feedthroughs made by casting Epibond 100-A about wires held in place by a Teflon plug in a brass tube. The brass tube assemblies are soldered into fittings on top of the vacuum can.

The resistance of the carbon resistors is measured with a 33 Hz ac bridge designed and built by Professor Weyhmann. Resistances can be easily determined to within 1/2% by this bridge with as little as 10^{-14} watts of power dissipated in the resistor. A block diagram of the bridge is given in Appendix B.

The resistors on the ^3He and ^4He evaporators were calibrated against the vapor pressure of ^3He and ^4He respectively. The vapor pressure was measured with a McLeod gauge, and thermomechanical corrections were made to the pressure readings. The resistor on the salt pill was calibrated against the temperature of the ^3He evaporator as measured by its resistor. Recently a new salt pill resistor was temporarily soldered to the ^3He evaporator and calibrated against the vapor pressure of ^3He .

The low temperature behavior of a Speer resistor between 2.5 and 0.15°K has been given by Beal and Hatton as

$$T = A \log R / (B - \log R)^2,^{30} \quad \text{III-1}$$

For temperatures below about 1/2°K this expression can be approximated by

$$1/T = C + D \log R. \quad \text{III-2}$$

Therefore, for each resistor we fit a line through the calibration values of $\log R$ vs. $1/T$ and extrapolate it to lower temperatures as a straight line. The extrapolation is needed only for the salt pill resistor thermometer. The other resistors

are calibrated over their operating range. Temperatures for a given resistance can be easily read from these graphs.

Resistance thermometry cannot be relied on below 0.2°K in our apparatus. We have no reason to believe that equation III-1 is valid outside the temperature range used by Beal and Hatton. Calibration to lower temperatures would be necessary, because extrapolation is notoriously inaccurate.

A second source of error at low temperatures is thermal equilibrium--or rather a lack thereof. We have noticed a lack of thermal equilibrium between sample and resistor after demagnetization. Several hours after demagnetization, while the salt and sample are definitely warming, the resistor is still cooling. It would also be possible at the lowest temperatures to dissipate enough power in the resistor from the resistance bridge to cause the resistor and sample not to be in equilibrium.

Gamma-Ray Detection

The anisotropy of the distribution of γ rays from the sample is measured by two scintillation detectors aligned with the sample, one parallel to the polarizing field and one perpendicular to it. These detectors are called the π and σ detectors respectively after spectroscopic notation. The detectors are Marshaw 12S12 integral line scintillation detectors with 3x3-in. NaI(Tl) crystals and 3-in. RCA 8054 photomultiplier tubes. The pulse height resolution of the detectors is better than 7-1/2% at 662 KeV.

The high voltage for the photomultipliers is supplied by a Fluke model 402M power supply and two model 2901 photomultiplier voltage dividers made by Sturup Nuclear Division of Canberra Industries.

The photomultiplier tubes are magnetically shielded to protect them from the field of the polarizing magnet. An inner shield of μ -metal was supplied with each detector. In addition two extra layers of magnetic shielding were added.³¹ The detectors, shielding, and voltage dividers are assembled as shown in Figure 12. The front portions of the detector mounts are machined from magnesium to reduce the scattering mass near the crystals. The detector mounts are bolted between aluminum rails attached to the dewar stand for proper alignment of the detectors with the sample. The distance of the detectors from the sample can be adjusted by 1/2 inch increments.

A block diagram of the data handling electronics is shown in Figure 13. The photomultipliers are connected to two Sturup model 1405 scintillation preamplifiers. The pulses from these charge sensitive preamplifiers are amplified and shaped by two Sturup model 1415 RC amplifiers. The outputs of the amplifiers are connected both to a common Sturup model 1465 summing amplifier and to separate Sturup model 1432 integral discriminators. The summing amplifier is connected to the low voltage input of a Victoreen SCIPP 400 multichannel pulse height analyzer. The integral discriminators provide routing pulses to the pulse height analyzer.

Figure 12. Scintillation detector and shielding.

Figure 13. Block diagram of electronics.

When the pulse height analyzer receives a pulse from the summing amplifier and a routing pulse from the discriminator connected to the σ detector perpendicular to the polarizing field, the pulse is analyzed and the count stored in the first two hundred channels of the memory. Similarly a routing pulse from the other discriminator received simultaneously with a pulse from the summing amplifier causes the analyzer to analyze the signal pulse and store the count in the second two hundred channels of the memory. Two routing pulses received by the analyzer at the same time cause the analyzer to ignore the data pulse received with the routing pulses.

For visual examination the data accumulated by the pulse height analyzer can be displayed on a cathode-ray tube in the analyzer and photographed. The data can also be printed out and punched on tape by a Victoreen modified model 33 Teletype page printer and tape punch. Due to the three to four minutes necessary to type out one data record of 400 six place numbers, much of our data is recorded on magnetic tape and not examined until after computer processing. An interface designed and built in the department's electronic shop converts the output from the analyzer into computer compatible language which is recorded on a Kennedy 1500 incremental tape recorder. The interface also adds immediately preceding the spectra in each data record an automatically advanced file number and two six place numbers which are set manually. Thus additional information such as field strength and length of counting period can be supplied to the computer with each data record.

CHAPTER IV

OPERATION

Assembly

Due to the many vacuum lines connected to the cryostat head, the apparatus was designed for bottom access to the sample space. The cryostat head is not removed from the dewar stand except for repairs and modifications. The pumping lines and evaporators are kept under a vacuum between runs. Periodic checks of the ^3He and ^4He systems are made by attaching a leak detector to the ports on the pumping lines.

Once a sample has been prepared and soldered to the salt pill and the salt pill has been filled with slurry, the apparatus is assembled in place in the dewar stand. First the salt pill is suspended by a graphite rod from the ^3He evaporator, and leads are connected to the thermometer resistor and heater mounted on the salt pill. The lead heat switch is also soldered in place. At this time all heaters and thermometers are checked with an ohmmeter for continuity and resistance at the feedthroughs on the cryostat flange. Next the spacer is positioned near the bottom of the salt pill, and the radiation shield is installed. For better thermal contact a small amount of Apiezon N grease is put on the threads of the evaporator flange before the radiation shield is threaded on. After the shield is in place, the grease is heated with a heat gun until it flows. The vacuum can

is installed with a new indium O ring each time. The overlapping ends of the 0.031-in. indium wire are crossed before the O ring is evenly compressed by a 12 bolt circle of brass machine screws. Next the magnet support rods are screwed into the cryostat flange, and the magnets are attached as a unit. After the magnet leads are plugged into the magnet assembly, they too are checked at the feedthrough for continuity and for isolation from ground. Finally the dewars and detectors are bolted in place. The detectors are positioned so that their counting rates in the energy peak from the sample are approximately equal. Before the apparatus is cooled, the ^3He and ^4He evaporators and the condenser are pumped on with the ^4He diffusion pump to remove any gas remaining in them.

Precooling

Cooling of the apparatus and sample proceeds in several stages to the starting temperatures for adiabatic demagnetization. After the inner dewar has been flushed and filled with nitrogen gas, the outer dewar is filled with liquid nitrogen. The nitrogen in the inner dewar serves as an exchange gas to cool the cryostat. It also insures that no water vapor condenses in the dewar to obscure vision through the clear strips in the dewar silvering. The apparatus is allowed to stand overnight and cool to 77°K.

At liquid nitrogen temperature the salt pill is well-frozen, and the vacuum can can be pumped out without fear of dehydrating the slurry or allowing entrapped air bubbles to expand forcing the slurry out of the top of the salt pill shell. The vacuum can

can now be checked for leaks by attaching the leak detector to the vacuum can pump line while helium is admitted to the inner dewar. The vacuum can is then filled to a pressure of about 5 Torr with hydrogen as an exchange gas, and the valve in the vacuum can pumping line on top of the dewar stand is closed.

After the inner dewar has been flushed and filled with helium gas to prevent nitrogen snow from obscuring the view or clogging the needle valve, liquid helium is transferred into the inner dewar. The transfer is started very slowly to prevent rapid cooling of the vacuum can and premature freezing of the hydrogen exchange gas. Once the hydrogen freezes, the parts inside the vacuum can are thermally isolated from the helium bath. We have found the ^3He evaporator to be at as high a temperature as 50°K after a rapid transfer. By adjusting the rate of the transfer we can cool the evaporators and salt pill to below 15°K before the hydrogen freezes out. We use the pressure of the hydrogen in the vacuum can as an indication of how the transfer is proceeding. If the pressure starts to rise, the transfer rate is too slow and the vacuum can is warming. If the pressure drops rapidly, the transfer rate is too fast and the hydrogen is beginning to freeze out. We attempt to achieve a slow decrease in pressure. Occasionally we stabilize at a constant pressure to allow the components inside the vacuum can to come into equilibrium with the vacuum can. The temperature, meanwhile, is monitored by the resistors on the ^3He evaporator and the salt pill. Once the temperature of the evaporator is about 15°K , usually about an

hour after the transfer is started, we increase the rate of transfer to allow liquid helium to start collecting in the dewar.

The magnets are energized during the transfer after they are covered with liquid helium. By energizing the magnets during transfer we are able to replace the liquid helium boiled off by Joule heating in the magnet leads before the transfer is completed. An additional reason for energizing the magnets at this time is that the lead heat switch is cooling to its superconducting transition temperature. The demagnetizing magnet must be at its rated field to keep the switch from becoming superconducting and isolating the salt pill from the ^3He evaporator.

The ^4He evaporator is filled by opening the needle valve on the vacuum can while the evaporator is being pumped on by the mechanical pump. We adjust the rate of filling so that the temperature of the evaporator is about 2.1°K . By staying below the λ point while filling, we do not need to pump the ^4He through its superfluid transition once the evaporator is full. We, therefore, reach the operating temperature of the ^4He evaporator while it is still almost full rather than half empty. Indication that the evaporator is full is given by an increase in its temperature as the ^4He starts to fill the pumping line towards the 4.2°K bath. The needle valve is then closed before the temperature rises above the λ point. Once

the entire apparatus is cold, the ^4He evaporator will operate for 35 to 40 hours between fillings. During cooling more frequent fillings are necessary.

The ^3He evaporator is cooled by thermal conduction along its pumping line and by conduction and convection of ^3He gas in the pumping line. After the ^4He evaporator is filled and after the ^3He has been circulated through the cold trap, we admit ^3He gas to the evaporator through the pumping line. Approximately two hours after the initial filling of the ^4He evaporator, the ^3He evaporator has cooled sufficiently to collect liquid ^3He . We then start to pump on the ^3He evaporator while returning ^3He gas to the condenser. The salt pill, meanwhile, is cooled by thermal contact with the ^3He evaporator through the lead heat switch. The heat switch is in its normal or high thermal conductivity state due to the field of the demagnetizing magnet. The ^3He refrigerator is usually left in the recirculating configuration overnight while the salt pill cools to about 0.5°K . The following morning the return line valve is closed, the condenser is pumped out, and the diffusion pump is turned on. Without the heat leak from the returning liquid ^3He and with the diffusion pump operating, the ^3He evaporator and salt pill reach 0.3°K . The residual heat leak to the ^3He evaporator once the salt pill has cooled to the same temperature as the evaporator is $30\ \mu\text{W}$. The present 2.5 liters of ^3He in the refrigerator allows operation for over 20 hours with this heat leak before refilling. The evaporator is refilled by returning to the recirculating mode.

During a calibration run without the salt pill or radiation shield, the heat leak to the evaporator was 5 μW and its lowest temperature was 0.25°K.

The performance of the ^3He refrigerator in the recirculating mode is very dependent upon the heat load. At relatively high heat loads such as 200 μW the refrigerator reaches a steady state recirculating configuration at about 0.5°K. Enough liquid is returned to the evaporator from the condenser to compensate for the liquid that is evaporated from the evaporator. The volume of the condenser was designed to be smaller than the total volume of liquid ^3He in the system. Therefore, as the amount of liquid in the condenser increases, liquid ^3He starts to back up in the return line towards the 4.2°K bath. The resulting increase in pressure above the liquid in the condenser forces the ^3He through the impedance at a greater rate thus keeping the ^3He evaporator from becoming empty.

At low heat loads the refrigerator is not as successful at regulating itself. During one run with a heat load of 9 μW the refrigerator cooled to 0.45°K before the level of the liquid in the condenser dropped below the ^3He evaporator. The temperature then increased to a stable 0.56°K. In this configuration not enough ^3He is returned to the condenser to maintain a pressure great enough to liquify the ^3He . The extra thermal load of liquifying the ^3He is placed upon the ^3He evaporator, and the

temperature of the evaporator increases. We usually close the return line to the condenser before it becomes empty and operate the refrigerator without recirculation.

The failure of the ^3He refrigerator to operate in the recirculating mode with small heat loads is not really serious to us. As was mentioned earlier, with our typical heat load of 30 μW the refrigerator will operate more than 20 hours without refilling.

Demagnetization

The salt pill is usually cooled to 0.3°K by the ^3He refrigerator before demagnetization. We have made some demagnetizations from higher temperatures. From this initial temperature the salt pill and sample are cooled to their final temperature by reducing the field of the demagnetizing magnet. The program of reducing the magnet current which we usually use is to reduce the current from 22 to 11 amperes at 2.0 amps/min, remain at 11 amperes for 5 minutes, then reduce from 11 to 0 amperes at 1.2 amps/min. During the first reduction of current the heat switch becomes superconducting and therefore a poor thermal conductor isolating the salt pill from the ^3He refrigerator. We have achieved temperatures below 10 m°K by this method. The performance of the salt pill will be described in the following section.

Data is collected while the sample gradually warms up due to the residual heat leak to the salt pill. The temperature of the salt pill can be raised more rapidly by applying power

to the wire wound heater attached to the salt pill. After applying enough power to raise the temperature a millidegree, however, several hours are required before the salt pill and sample are in equilibrium. The temperature of the sample overshoots the temperature of the salt pill then decreases until it reaches equilibrium with the salt pill. An example of the behavior of the temperature of the sample after heating is shown in the next section.

The salt pill can be demagnetized as often as necessary during a run. Before we remagnetize the salt pill, we turn off the ^3He diffusion pump and allow it to cool. The heat of magnetization is often enough to raise the temperature of the salt pill and evaporator to a point where the vapor pressure of ^3He is greater than the diffusion pump can handle. The current in the demagnetizing magnet is slowly increased to 22 amperes. Several hours are required to cool the salt pill to 0.3°K before another demagnetization. The demagnetization proceeds as before.

Liquid helium can be transferred into the dewar at any time. Usually data is not taken during the transfer due to vibrational heating associated with the transfer. The ^4He and ^3He evaporators can be refilled without overly disturbing the temperature of the sample once the salt has been demagnetized.

Collection of Data

At some time after the electronics have stabilized the energy peaks in the two halves of the memory must be made to coincide by adjusting the gain of the linear amplifiers.

This adjustment is usually made while checking the electronics before the apparatus is cooled.

To measure the temperature of a sample we need only to measure the intensity of the γ rays at one angle. This measurement when divided by the intensity at the same angle at infinite temperature (anything greater than 0.3°K for our purposes) is the properly normalized $W(\theta)$ discussed in Chapter II. The temperature is found on a table of $W(\theta)$ as a function of temperature for a given θ and for the particular materials in the sample. A detector parallel to the polarizing field, $\theta=0$, experiences the largest change in intensity and is, therefore, most sensitive to temperature.

The use of a second detector at a different angle provides a method of checking data. Not only can an independent temperature be determined from it, but the coefficients of $P_2(\cos \theta)$ and $P_4(\cos \theta)$ in the expansion of $W(\theta)$ can also be determined. Our data consists of simultaneous spectra taken by the π and σ detectors.

Once the paramagnetic salt has been demagnetized and the sample cooled, the acquisition of data can be accomplished automatically. The length of a counting period is set on a timer in the pulse height analyzer. Usually periods of between five and thirty minutes are used. At the end of a counting period the data is recorded on magnetic tape, the memory of the analyzer is erased, and another counting period begun automatically. This cycle can be interrupted manually to change the length of the count or to print out or photograph a data record. The apparatus

can collect data overnight while the temperature is gradually increased by the residual heat leak to the salt pill.

Warm spectra of zero anisotropy are collected during a run by heating the salt pill to at least a third of a degree. Usually several warm spectra are taken at various times during a run.

Termination of Run

At the end of a run the ^3He in the evaporator is boiled off with the aid of the heater and pumped back into the ballast tank. The diffusion pump is used to reduce the pressure in the evaporator to less than 10^{-3} Torr. The gate valve above the diffusion pump is then closed, and the diffusion pump is turned off. The ^4He evaporator is also pumped out. The current in the magnets must be reduced to zero before the liquid helium in the bath is boiled off. The heater wound around the bottom of the polarizing magnet allows the helium in the bath to be boiled off in about two hours. Once the entire apparatus has warmed to 77°K, the inner dewar is pumped out, flushed, and filled with nitrogen gas to prevent helium from diffusing into the dewar vacuum space as the temperature increases. The nitrogen dewar is then lowered into the cart. A Bunsen valve protects the inner dewar from overpressure as its temperature increases to room temperature.

If the salt pill is to be preserved for another run, the apparatus is disassembled while still somewhat cold. The salt pill is stored in liquid nitrogen. Storage of the salt pill at room temperature will lead to dehydration of the salt by the glycerin in the slurry.

CHAPTER V

RESULTS

Data Processing

As the reader will recall, the orientation of the nuclei of cobalt-60 in an iron host and, therefore, the angular distribution of the γ radiation from these nuclei are temperature dependent. Figure 1 illustrated the temperature dependence of this radiation pattern. It is easy to see from Figure 1 that the counting rate of a detector perpendicular to the polarizing field along the z axis will increase with decreasing temperatures while the counting rate of a detector parallel to the field will decrease. This effect is also illustrated by Figure 14 which shows photographs of data displayed on the cathode-ray tube of the pulse height analyzer. Each photograph contains two spectra. The left half of each photograph is the spectrum from the detector perpendicular to the polarizing field. The right half is from the detector parallel to the field. Each spectrum shows the number of counts on the vertical scale as a function of the energy on the horizontal scale. The two peaks near the right or high energy side of each spectrum result from the two γ rays at 1.33 and 1.17 MeV from ^{60}Co . These two peaks called photopeaks represent events in which the entire energy of the γ ray was absorbed in the scintillation crystal. They exhibit the temperature dependent

σ π $T > 1^\circ\text{K}$ σ π $T = 7.65 \text{ m}^\circ\text{K}$

Figure 14. Typical spectra of ^{60}Co displayed on pulse height analyzer.

anisotropy in which we are interested. The remainder of the spectrum comes from background and from events in which only part of the energy of a γ ray was absorbed in the detector. These parts of the spectrum do not necessarily exhibit the temperature dependent anisotropy associated with the γ rays from the sample. They have contributions from γ 's which did not start out in the direction of the detector but were scattered into it. We will, therefore, ignore all of the spectrum save the photopeak.

The γ rays emitted by the sample are monoenergetic since they result from transitions between nuclear energy levels. Processes in the scintillation crystal and in the photomultiplier, however, result in a distribution of pulse heights received by the analyzer. The spectra show photopeaks of finite width. The processes which widen the spectral line involve the statistics of large numbers. For each γ detected by the scintillation crystal a large number of photons are emitted. For each photon detected by the photomultiplier a large number of electrons contribute to the gain. One might expect the shape of the photopeak to be Gaussian from statistical considerations. A detailed analysis by Breitenberger shows that the distribution of pulse heights from monoenergetic γ rays is very nearly Gaussian in the vicinity of its peak.³² Therefore, we use a computer to fit a Gaussian curve to the top of the 1.33 MeV photopeak in our data. The higher energy peak is used because the lower energy peak has more distortion due to larger background and due to contributions from the higher peak.

Each data file taken by the pulse height analyzer consists of 400 six place numbers. These numbers comprise two spectra from the two detectors. The magnetic tape with the data is fed into the computer together with the approximate channel numbers of the tops of the 1.33 MeV photopeaks in both spectra. The computer fits a Gaussian curve to the data in the eleven channels centered on the predicted peak in the first 200 channels. A fit is achieved by varying the three parameters in the Gaussian curve

$$f(x) = b \exp [-(x-x_0)^2/a^2], \quad V=1$$

The deviation of the eleven data points from the fitted Gaussian is also calculated. The computer then repeats the procedure with the data in the eleven channels centered one channel higher than the predicted peak. A total of six fits are made to each spectrum by shifting the center of the data used one channel each time. The integral under the Gaussian, $\sqrt{\pi} b/a$, is calculated from the fit with minimum deviation. The entire procedure is repeated for the peak in the second 200 channels and then for each succeeding data file. The best fit is usually found for the data in channel peak minus three through channel peak plus seven. This displacement to the right is not surprising. The low energy side of the 1.33 MeV peak overlaps the high energy side of the 1.17 MeV peak; therefore, the data in the channels above the peak channel for the 1.33 MeV photopeak are closer to a Gaussian than those below the peak channel. A program can be written to separate the peaks, but as they have the same anisotropy it is not necessary.

Shifting the channels to which the Gaussian is fitted allows the computer to follow any gain shift which might move the peak position during a run.

As was mentioned in the last chapter, the temperature of a sample can be found from the ratio of the γ intensity at the temperature in question to the γ intensity at high temperature. The counting rate of a detector includes parts of the spectrum which do not have the proper anisotropy; therefore, we need the counting rate of a detector restricted to the photopeak. This counting rate can be calculated from the integral under the peak calculated by the computer and the length of the counting period.

To check whether the radiation pattern is indeed the quadrupole pattern which we expect, we must calculate the expansion coefficients of $W(\theta)$. The expansion of $W(\theta)$ in II-25 can be rewritten

$$W(\theta) = 1 - \epsilon_2 P_2(\cos \theta) - \epsilon_4 P_4(\cos \theta). \quad V-2$$

The ϵ 's are merely the product of the appropriate coefficients of the Legendre polynomials. If we designate the intensity of the γ rays detected at angle θ while the sample is cold as $I_c(\theta)$ and the intensity while the sample is warmed to zero anisotropy as $I_w(\theta)$, we can write

$$W(\theta) = I_c(\theta)/I_w(\theta). \quad V-3$$

Using the values of the Legendre polynomials at the angles

corresponding to our detectors we have

$$I_c(0)/I_w(0) = 1 - \epsilon_2 - \epsilon_4, \quad V-4$$

$$\text{and } I_c(90)/I_w(90) = 1 + \epsilon_2/2 - 3\epsilon_4/8. \quad V-5$$

These two equations can be solved for the temperature dependent coefficients ϵ_2 and ϵ_4 :

$$\epsilon_2 = \frac{3}{7} \frac{I_h(0) - I_c(0)}{I_h(0)} - \frac{8}{7} \frac{I_h(90) - I_c(90)}{I_h(90)}, \quad V-6$$

$$\text{and } \epsilon_4 = \frac{4}{7} \frac{I_h(0) - I_c(0)}{I_h(0)} + \frac{8}{7} \frac{I_h(90) - I_c(90)}{I_h(90)}. \quad V-7$$

Temperatures can also be obtained from the ϵ_2 's and ϵ_4 's; however, the error in the temperatures evaluated from the ϵ 's is much greater than the error in temperatures evaluated from $W(0)$. The coefficients ϵ_2 and ϵ_4 result from differences between quantities with statistical errors and, therefore, have larger errors than the quantities themselves.

We have, with the aid of a computer, generated a table of $W(0)$, $W(90)$, $W(90)/W(0)$, ϵ_2 , and ϵ_4 for a sample of ^{60}Co in iron at temperatures from 5 to 100 m°K and with applied fields of 0 and 3000 gauss. Graphs plotted from this table enable us to easily find the temperature of our sample once we have one of the listed parameters. The ratio of the integrals in the same data file will give $W(90)/W(0)$ even if the counting period is not known. The ratio must, of course, be multiplied by $W(0)/W(90)$ at high temperature for normalization.

Correction Factors and Error

The presence of an isotropic background will reduce the anisotropy measured in an experiment. The temperature indicated will, therefore, be higher than the actual temperature of the sample. We have found in our laboratory not only the usual uniform background which decreases at high energies but also several energy peaks. There is a peak at 1.46 MeV which we have tentatively identified as ^{40}K . There are also peaks at 0.58 and 2.62 MeV which we believe to be ^{208}Tl -- possibly from thoriated sands. The background does not appear to be localized in the laboratory. There is even some evidence that the activity may be in the concrete. Fortunately the activity is quite small. The height of the 1.46 MeV photopeak is a factor of 25 smaller than the 1.33 MeV photopeak from our 8 μCi ^{60}Co sample at zero anisotropy.

For future work the computer program will include a step to subtract a sample background from each spectrum. An approximate correction was made to our present data by subtracting from each integral the sum of the background in 12 channels which include the 11 channels to which the Gaussian was fitted.

Another correction which must be made to the data is due to the finite size of the detectors. A detector of finite size accepts radiation leaving the sample within a certain solid angle determined by the geometry. All of the radiation within this solid angle will not have the same anisotropy; therefore, the anisotropy will be somewhat "smeared out". Because our detectors are positioned at the angles where the anisotropy is greatest,

the effect of a finite detector size will be to lower the anisotropy measured. For cylindrical detectors in an axial geometry the effect of detector size can be expressed by inserting attenuation factors in the expression for $W(\theta)$. Equation V-2 becomes

$$W(\theta) = 1 - Q_2 \epsilon_2 P_2(\cos \theta) - Q_4 \epsilon_4 P_4(\cos \theta), \quad V-8$$

where the Q 's are the attenuation coefficients. Tables by Yates give values for Q_2 and Q_4 for distances up to 14 cm for 3×3 -in. crystals.³³ I have extrapolated these values to our distance of 20 cm and estimate that for a 1.3 MeV photopeak $Q_2 = 0.98$ and $Q_4 = 0.93$. Temperatures obtained from a graph of $W(0)$ as a function of temperature with $W(0)$ corrected for solid angle effects are approximately 0.3 m°K lower at 10 m°K than those obtained if $W(0)$ is not corrected.

A possible source of error is the positioning of the sample and the detector. The largest error in alignment of the sample with the π detector axis is about $\pm 3^\circ$. This misalignment could increase $W(0)$ by 1% at 10 m°K causing the temperature measured to be 0.15 m°K higher than the actual temperature. Notice that systematic errors tend to reduce the measured anisotropy causing the anisotropy to indicate a temperature higher than the temperature that the sample actually reached.

The statistical error associated with counting N random events in a given time is taken to be $\pm \sqrt{N}$. The integrals evaluated for our photopeaks represent the total number of counts

in the Gaussian. However, possibly a more accurate estimate of the percentage error would be $\sqrt{N}/N$ where N is the sum of the counts in the channels to which the Gaussian was fitted. The background subtracted from each integral is also subject to statistical error. Error bars assigned to points on the graph in the next section have been calculated from statistical errors only. The errors in temperature were found by determining on the plot of $W(0)$ vs. T what error in T is caused by a given error in $W(0)$.

Performance

The performance of the salt pill after a typical demagnetization is shown in Figure 15. The temperature of the sample calculated from $W(0)$ is plotted as a function of the time after demagnetization. Temperatures have been corrected for background and for the solid angle subtended by the detector. The center of the salt pill was demagnetized from $H/T = 57$ kG/deg. The initial temperature was 0.31°K and the field was 17.7 kilogauss at the center of the magnet. The minimum temperature the sample reached was 7.0 m $^\circ\text{K}$ about 15 minutes after the end of the demagnetization. The initial warm-up rate was rather high because the salt was probably in the region in which its specific heat decreases rapidly below its magnetic ordering temperature. The ordering temperature of chrome alum is 0.011°K ;³⁴ however, as some of the chrome alum in our salt pill is diluted, its ordering temperature may be lower due to the increased distance between chromium ions reducing the exchange interaction.

Figure 15. Temperature of sample after demagnetization.

After two hours the warm-up rate became an approximately constant $0.15 \text{ m}^\circ\text{K/hr.}$ At the end of the sixth hour 2.5×10^5 ergs were applied to the heater attached to the salt pill. By extrapolating the warm-up rate of the sample after it had returned to equilibrium with the salt pill back to the time of the heat pulse, we can measure the temperature difference of $0.9 \text{ m}^\circ\text{K}$ caused by the heat pulse. The heat capacity of the salt pill calculated from this value is $2.8 \times 10^4 \text{ ergs/m}^\circ\text{K}$. Other measurements we have made yield values of $2.2\text{--}3.1 \times 10^4 \text{ ergs/m}^\circ\text{K}$. We have been unable to find published measurements of the specific heat of chrome alum below $15 \text{ m}^\circ\text{K}$. Vilches and Wheatley show a nearly constant specific heat for chrome alum between 17 and $40 \text{ m}^\circ\text{K}$ of $2.5 \times 10^7 \text{ ergs/mole/}^\circ\text{K}$.³⁵ This value would correspond to a heat capacity of $2 \times 10^4 \text{ ergs/m}^\circ\text{K}$ for our salt pill which contains 0.8 gram ions of chrome alum.

The break in data between 9 and 11 hours after demagnetization occurred while we retransferred liquid helium, refilled the outer dewar with liquid nitrogen, and refilled the evaporators. The entire operation appears to have added approximately 5×10^3 ergs to the salt pill. The warm-up rate during the next several hours was $0.10 \text{ m}^\circ\text{K/hr.}$ This warm-up rate multiplied by our measured heat capacity of the salt pill indicates a heat leak to the salt pill of $3/4 \text{ erg/sec.}$

A portion of the heat leak to the salt pill can be accounted for by the β particles emitted by the ^{60}Co in the sample. These β 's are absorbed within the sample. Assuming an activity of 8 μCi , the heat from 1/3 MeV β 's totally absorbed in the sample accounts for 9 ergs/min of the heat leak to the salt pill.

The consistency of the data with itself was quite good. Temperatures calculated from ϵ_2 and $W(90)/W(0)$ are within statistical error of the temperatures calculated from $W(0)$. Temperatures from the former quantities and even more markedly from $W(90)$ have more scatter than do the temperatures from $W(0)$. This scatter is to be expected due to the relative sensitivities of $W(0)$ and $W(90)$ to temperature and due to the differences between statistical quantities which enter into ϵ_2 .

The consistency of the data is also supported by the values of ϵ_4 . The errors from statistics in ϵ_4 are very large because ϵ_4 is evaluated from differences between quantities. In this run the values of ϵ_4 calculated from the data scattered about the values of ϵ_4 appropriate for the corresponding temperatures calculated from ϵ_2 . A previous run resulted in values for ϵ_4 calculated from the data which were consistently smaller than the ϵ_4 's appropriate for the corresponding temperatures. The only modification in the apparatus between runs was the removal of the portion of the copper fins from the salt pill which extended below the sample. This portion of the fins constituted a scattering mass between the sample and the π detector which was not between the sample and the σ detector.

CHAPTER VI

CONCLUSION

The apparatus as it now stands is capable of cooling samples for nuclear orientation to temperatures below 10 m°K. It has a reasonable working time at temperatures as low as 8 m°K. With its warm-up rate in this temperature range of less than 0.2 m°K/hr the change in temperature of a sample during a thirty minute counting period is less than 0.1 m°K or 1%.

Recent modifications by Mr. R. J. Holliday have provided the possibility of applying radio frequency power to the sample while observing the anisotropy of the radiation pattern. Resonant destruction of the anisotropy offers a very precise method of directly measuring the energy splitting between magnetic sublevels.

Other possible future additions are a more powerful polarizing magnet and a solid state detector. A larger polarizing field would increase the number of possible samples. The resolution of a solid state detector would allow investigation of otherwise unusable isotopes with γ -ray energies which are not resolved by a scintillation crystal. The lower efficiency of a solid state detector requires the use of longer counting periods than a scintillation detector. The temperature stability of our apparatus is ideally suited to such detectors.

APPENDIX A

SAMPLE PREPARATION

The sample of cobalt-60 in iron was prepared by electroplating ^{60}Co onto an iron foil and diffusing the activity into the iron at high temperature.

The iron foil was degreased in acetone and etched in 1 to 1 HCl and water solution. It was then mounted in an electrolytic cell designed after one described in an article by Dézsi and Molnár.³⁶ The cell was filled with one ml of the electrolyte described below, one ml of ^{60}Co solution, and ammonium hydroxide to raise the pH to 10. The ^{60}Co was received as CoCl_2 in HCl which we diluted with HCl and water to facilitate measuring out small quantities of the ^{60}Co . One ml of the ^{60}Co solution contained about 30 μCi of activity. The electrolyte, modified from the Dézsi and Molnár article, contained

2.5 gm ammonium citrate
2.5 gm hydrazine hydrate
50 ml distilled water
ammonium hydroxide to adjust pH to 10-11.

A platinum foil was used as the anode. The ^{60}Co was electroplated onto the iron foil for 10 hours at 0.7 mA. Every hour a drop of hydrazine hydrate was added to reduce any oxidation products at the anode. The sample was removed from the cell, washed with water, and diffused at 1050°C for 24 hours. The

sample remained in the oven as the oven cooled. An atmosphere of 5% hydrogen-95% argon was passed over the sample while it was in the oven to prevent oxidation of the iron. The argon in the mixture eliminates any explosion hazard. After etching the sample with HCl until its surface was bright, the activity of the sample was about 9 μ Ci.

APPENDIX B

RESISTANCE BRIDGE

Figure 16 is a block diagram of the 33 Hz ac resistance bridge used to measure the resistance of carbon resistors used for thermometry. It consists of an ac bridge powered by a 33 Hz tuning fork oscillator. The power delivered to the thermometer resistor can be adjusted by the power level set. The imbalance of the bridge is amplified by a tuned operational amplifier with a FET input. The amplifier is tuned by a 33 Hz twin tee oscillator. The output of the tuned amplifier is fed into a second operational amplifier whose gain is controlled by a photoresistor activated by the output of the final amplifier. This feedback loop produces a nonlinear output such that the circuit and meter are most sensitive near balance. The in-phase component of the signal is selected by a phase detector. The in-phase component represents only the purely resistive part of the impedance. The in-phase signal is integrated, amplified by the final operational amplifier, and displayed on a microammeter. A phase shifter allows compensation for the capacitance of the leads to the thermometer resistor.

Great care has been taken to isolate the bridge circuit from both rf and 60 Hz pickup. An isolation transformer decouples the circuit from the power line. This transformer and the Burr Brown #501 dc power supply are wrapped in Conetic AA foil to

Figure 16. Block diagram of 33 Hz resistance bridge.

reduce magnetic coupling to the bridge. All lines into the bridge section contain rf filters. The 60 Hz and 120 Hz power delivered to a resistor at the bridge output is less than 10^{-15} watts.

FOOTNOTES

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