

THE FEASIBILITY OF DETECTING INTERACTIONS
WITH ORIENTED NUCLEI IN EMULSION
AT VERY LOW TEMPERATURE

by

Arthur W. Johnson

Submitted in partial fulfilment
of the requirements for the degree of
Master of Science

Department of Physics
Faculty of Pure and Applied Science
The University of Ottawa
Ottawa, Canada



Arthur W. Johnson, 1972

© Arthur W. Johnson 1972

À

CONNIE

MARIE, MARTIN ET ANDREW

ABSTRACT

The very low temperature required by most nuclear orientation methods imposes strict limitations on the detection schemes for the ionizing particle reaction products. Only a few particle detectors of small size and capable of operation at low temperature and pressure may be installed in the cryostat. Nuclear emulsion, however, provides a three-dimensional record of all ionizing particle tracks and may be used in vacuum. An added advantage is that oriented nuclei may be imbedded in the emulsion so that the detector completely surrounds the target.

In this thesis a method is described whereby unsupported nuclear emulsion may be cooled by adiabatic demagnetization of a paramagnetic salt while the emulsion remains in a high magnetic field. The high field is needed by the brute force method of orienting nuclei, although other, less demanding, methods may also be used with emulsion detection. During an experimental run, pellicles placed in a 70 kG magnetic field cooled to 88 mK and remained at $97 \text{ mK} \pm 10\%$ for 106 minutes. If longer irradiation times are necessary, the demagnetizing process may be repeated as needed.

As part of this investigation, the sensitivity of Ilford G5 nuclear emulsion was studied at very low temperature by comparing the

grain density of proton tracks recorded at 0.1 K and at room temperature. It is found that the emulsion retains (74 ± 2) % of its room temperature sensitivity when exposed at 0.1 K and that temperature cycling from 0.1 K to room temperature has no noticeable effect on previously-recorded tracks.

RÉSUMÉ

La très basse température exigée par la plupart des méthodes d'orientation nucléaire limite les moyens de détection des produits ionisants de réaction. Le cryostat peut accommoder seulement quelques détecteurs de particules de faible volume qui doivent être capable de fonctionner à basse température et dans le vide. L'émulsion nucléaire, toutefois, enrégistre en trois dimensions toutes les traces de particules ionisantes et fonctionne dans le vide. De plus, les noyaux peuvent être placés dans l'émulsion de sorte que le détecteur entoure complètement les cibles.

Dans cette thèse, on décrit une méthode de refroidir des émulsions sans support par la désaimantation adiabatique d'un sel paramagnétique pendant que les émulsions restent dans un très fort champ magnétique. La méthode d'orientation directe (brute force) nécessite le champ magnétique, mais l'émulsion peut elle-même servir de détecteur dans d'autres méthodes moins exigeantes. Au cours d'un essai, la température des pellicules placées dans un champ de 70 kG a atteint 88 mK et restaient à 97 mK $\pm 10\%$ pendant 106 minutes. Si on a besoin d'un plus long temps d'irradiation, on peut désaimanter à nouveau.

Une partie intégrante de cette investigation était la détermination de la sensibilité relative de l'émulsion Ilford G5

faite en comparant la densité des grains le long des traces de protons enregistrées à 0.1 K et à la température de la pièce. Une valeur de $(74 \pm 2) \%$ a été obtenu. De plus, il a été observé qu'un refroidissement de l'émulsion jusqu'à 0.1 K n'affecte pas les traces enregistrées antérieurement à l'abaissement de température.

faite en comparant la densité des grains le long des traces de protons enregistrées à 0.1 K et à la température de la pièce. Une valeur de $(74 \pm 2) \%$ a été obtenu. De plus, il a été observé qu'un refroidissement de l'émulsion jusqu'à 0.1 K n'affecte pas les traces enregistrées antérieurement à l'abaissement de température.

ACKNOWLEDGMENTS

I would like to express my gratitude to the following individuals for their contribution to the success of this work:

Dr. J.L.G. Lamarche, my supervisor, for valuable discussions and his continued interest.

Dr. C.J.D. Hébert, for advice in processing and analysing nuclear emulsion, and for making the emulsion sensitivity estimates at 0.1 K.

Prof. J. Hébert, for his enthusiasm in the project and for use of darkroom facilities.

Dr. Y. Hsu and M. M. Boult for help and advice in the early stages of the work.

M. R. Hart for technical assistance.

The staff of the Physics Department Workshop who all helped in the construction of the new cryostat; in particular, Messrs. N. Goodchild, W. Knott, R. Moore, D. Kingswell and W. Laurin.

I am also grateful to the Defence Research Board for educational leave during part of this work.

LIST OF CONTENTS

ABSTRACT	iii
RÉSUMÉ	v
ACKNOWLEDGMENTS	vii
LIST OF FIGURES	x
CHAPTER I INTRODUCTION	1
CHAPTER II THE ORIENTATION OF NUCLEI	
2.1 Introduction	4
2.2 Methods of Nuclear Orientation	5
2.3 The Production of Low Temperature	7
CHAPTER III LOW TEMPERATURE THERMOMETRY	
3.1 Introduction	10
3.2 Experimental	12
3.3 Results	16
3.4 Magnetoresistance	17
CHAPTER IV THE SENSITIVITY OF NUCLEAR EMULSION AT LOW TEMPERATURE	
4.1 Introduction	23
4.2 Processing of Nuclear Emulsion	24
4.3 Low Temperature Sensitivity	26
4.4 Experimental	29
4.5 Results and Discussion	35

CHAPTER V	COOLING OF EMULSION IN A MAGNETIC FIELD	
5.1	Introduction	40
5.2	Experimental	42
5.3	Results and Discussion	48
5.4	Suggestions for Improvement	51
CHAPTER VI	CONCLUSION	53
REFERENCES		55

LIST OF FIGURES

Fig. 1.	Simplified diagram of the calibration cryostat	13
Fig. 2.	The equal-arm Wheatstone bridge	15
Fig. 3.	Calibration curve of R1	18
Fig. 4.	Calibration curve of R1 (detail)	19
Fig. 5.	Simplified diagram of the cryostat	32
Fig. 6.	Arrangement for cooling emulsion in a magnetic field	43
Fig. 7.	The movable stage of the new cryostat	44
Fig. 8.	Wiring diagram for the solenoids	45
Fig. 9.	Temperature curve of pellicles and cooling salt	49
TABLE I		28

CHAPTER I

INTRODUCTION

Nuclear reactions involving oriented targets have a special attraction to workers in the field of nuclear physics. Such reactions often yield information that can only be obtained with difficulty otherwise, or else not at all. Important examples are the discovery of the nonconservation of parity in β -decay and the investigation of internuclear forces. In appropriate reactions, the effects of orienting the target nuclei are evidenced by changes in cross-section for the incident particle or modification of the angular distribution of emitted particles or radiation.

The special conditions needed to obtain the desired orientation are not always easy to achieve and sometimes necessitate mastery of techniques in two or more fields of physics. In many cases, magnetic fields and low temperatures are necessary. Some methods require a temperature of 0.1 K or lower to yield a high orientation.

There is also the problem of the placement of particle detectors at low temperature. Some types of detector may be positioned directly in the cryostat close to the target nuclei, but if many detectors are needed or if they are too bulky to fit in the chamber,

they must be placed outside the dewars. For neutron or γ -radiation emission there may be no difficulty, but charged particles which must pass through the walls of the cryostat may experience a loss in energy and possibly a change in the directional and polarization characteristics. Some particles such as slow protons, electrons and α 's might not be able to reach the outside. By placing the nuclei in emulsion, a record of all ionizing particle tracks may be obtained together with energy and angular information.

In this thesis a description is given of a procedure for cooling nuclear emulsion containing imbedded nuclei to 0.1 K in the presence of a 70 kG magnetic field (Chapter V). These conditions may be considered to be the minimum requirements for polarization by the brute force method.

Chapter II contains an outline of the parameters used to describe the degree of orientation of an assembly of nuclei and a brief review of dynamic and thermal methods of producing nuclear orientation. Means of obtaining the low temperature needed are detailed in section 2.2.

Thermometry at temperatures below 1 K is somewhat of a problem because of the lack of primary thermometers; however, it is possible to calibrate secondary thermometers by extrapolation from an established temperature region. In Chapter III, the calibration of a pair of carbon resistors used to measure the temperature of the cooling salt and of the pellicles is described in detail. Since one of these resistors is employed in a magnetic field, the influence of

the field on temperature readings is explored (section 3.4).

In Chapter IV a description of the experimental arrangement and procedure used to cool the pellicles to 0.1 K is given and the results of the low temperature sensitivity tests are presented.

CHAPTER II

THE ORIENTATION OF NUCLEI

2.1 Introduction

In an assembly of nuclei possessing spin, the spin axes generally assume a random orientation; if a preferred direction is indicated, the system is said to be polarized. The interaction causing the polarization, such as an external magnetic field, quantizes the spin, I , into $2I + 1$ spin states, each with component m along the axis of quantization (z -axis). An expression which provides a measure of the degree of polarization is given by (1):

$$f = \frac{\sum m a_m}{I \sum a_m},$$

where a_m is the population of the state m . The range of f is from -1 to $+1$; $f = 0$ indicates a disoriented system.

When the spins point predominantly in the $+z$ and $-z$ directions with few perpendicular to the z -axis, there is still an orientation even though $a_m = a_{-m}$ and $f = 0$. In this case the nuclei are said to be aligned, to a degree given by the formula (1):

$$g = \frac{\sum a_m [m^2 - I(I + 1)/3]}{I^2 \sum a_m}.$$

2.2 Methods of Nuclear Orientation

(a) Dynamic Methods

The occupation of particular orientation states can be achieved by inducing transitions to them by pumping with optical, radio frequency or microwave radiation at a frequency corresponding to the energy difference of the states concerned. Transient polarization generated by a burst of high intensity radiation can be employed when the spin-lattice relaxation time is long enough to be useful, and continuous polarization is maintained by continuous irradiation. Some experimental arrangements yield quite high degrees of orientation ($f > 0.5$). In these so-called dynamic methods, nuclear magnetic resonance techniques figure prominently.

A review of dynamic methods of orientation is given by Jeffries (2) and Wenckebach et al. (3) describe a more recent application.

(b) Thermal Methods

At very low temperature the lower levels of a hyperfine splitting of a nucleus are selectively populated and the spins become oriented when placed in a magnetic field. If a paramagnetic salt is employed, a field of a few hundred gauss will saturate the electron moments which, because of their own very high magnetic field ($10^5 - 10^6$ gauss), cause a polarization of the nucleus parallel to the external field. By judicious choice of material, it is possible to obtain not only polarization of quite a high order with minimal

magnetic field, but also self cooling if the substance can be grown isomorphously in a cooling salt suitable for adiabatic demagnetization. This has been done for Co^{60} grown in cerium magnesium nitrate (4).

In a very strong external field and at very low temperature, any nucleus with spin and finite magnetic moment can be polarized (5). This brute force method as it is called belies its name since elegant techniques are often needed to attain the optimum conditions. The degree of polarization obtained is given by (6)

$$f = \left(\frac{2I + 1}{2I} \right) \coth \left(\frac{2I + 1}{2I} \cdot \frac{\mu H}{kT} \right) - \frac{1}{2I} \coth \left(\frac{\mu H}{2IkT} \right),$$

where μ is the magnetic moment, k is Boltzmann's constant, H is the magnetic field and T the absolute temperature. When $\frac{\mu H}{kT} \ll 1$, f may be approximated by (7)

$$f = \frac{1}{3} \frac{I + 1}{I} \frac{\mu H}{kT}.$$

The very high magnetic field and very low temperature needed to obtain useful polarization by this method may limit its application; however, valuable results have been obtained with values of $f \approx 0.02$. Stolovy (8), for example, has determined several spin states associated with neutron resonances in ^{115}In polarized by the brute force method. The use of this method in the present context is discussed in Chapter V.

Other methods, such as alignment by the electric hyperfine structure, are described by Daniels (5) to whom the reader is referred for details.

2.3 The Production of Low Temperature

The low temperature required by most nuclear orientation experiments must be maintained throughout the irradiation time otherwise the degree of orientation will decrease as the system warms. Some arrangements permit continuous cooling whereas others such as adiabatic demagnetization require repeated operation to achieve an adequate experimental time.

Specimens may be cooled to about 1 K by placing them in thermal contact with pumped liquid helium in a double dewar. Helium normally boils at 4.2 K, but reducing its vapour pressure with a high-speed vacuum pump to 1 Torr, for example, lowers its boiling point to 1.27 K. The temperature can be maintained as long as helium remains in the bath which may be refilled at low pressure with a specially-designed transfer siphon. Liquid nitrogen is placed in the outer dewar to prevent rapid boil-off of the helium. The method is well established and is easily repeated. If ^3He is used instead of ^4He , the ultimate temperature attainable is about 0.3 K, however a closed cycle system is required because of the high cost of ^3He .

Temperatures below 0.3 K may be attained by employing dilution refrigeration or adiabatic demagnetization of a paramagnetic salt.

Dilution refrigeration is a relatively new method of obtaining very low temperatures and although the technology is fairly well understood (9), use of the refrigerators is not yet widespread. Wheatley et al. (10) describe a unit that will produce continuously a temperature of 10 mK.

Adiabatic demagnetization of a paramagnetic salt is the oldest and most common method of producing very low temperatures. Simply stated, the substance becomes warm when initially magnetized and once this heat of magnetization has dissipated and the substance is thermally isolated, it cools upon being demagnetized. The degree of cooling and rate of rewarming depend upon the choice of salt, its initial temperature T , the magnetic field H , and the thermal isolation. The greater the ratio H/T , the lower will be the final temperature until a lower limit is reached which is approximately equal to the interaction energy of the salt divided by Boltzmann's constant.

The major requirements of a cooling salt are that there should be very little magnetic coupling between the ions (i.e. the salt must be magnetically dilute), the zero-field splitting of its lowest level must be much less than kT at about 1 K, and the next level should be high enough to exert negligible effects (11). Double salts with a large number of waters of hydration are good examples, such as ferric ammonium sulphate $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ and chromium potassium sulphate $[\text{K Cr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$. Cerium magnesium nitrate $[\text{Ce}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24\text{H}_2\text{O}]$ is the most renowned salt for achieving very low temperatures (1 - 2 mK), however its thermal capacity is small. The

salt's main value lies in its unique wide range of linear variation of magnetic susceptibility with inverse temperature (cf. section 3.1).

The cooling salt may be used as a heat sink by the nuclei to be oriented or else the nuclei may form part of the crystal structure for more intimate contact. The salt pill is attached to the walls of the experimental chamber with taut nylon or cotton threads. The threads are poor heat conductors and the tightness minimizes heating by vibration.

The usual experimental procedure is to cool the salt and nuclei to about 1 K by thermal connection to the pumped helium bath - either by employing a heat switch (12) or helium thermal exchange gas at a pressure of 10^{-3} Torr. Higher pressures do not substantially increase the thermal contact but simply make final pumping more difficult. The magnet or solenoid is then energized and when the heat of magnetization has dissipated, the heat switch is opened or the exchange gas is pumped out. At a residual pressure of 10^{-5} Torr or less the salt is slowly demagnetized. If the lowest temperatures are sought, the demagnetizing must occur isentropically or reversibly (13), a process assured by demagnetization long compared to the spin-lattice relaxation time of the cooling salt. A further advantage of slow demagnetization is that eddy-current heating in metal parts is also held to a minimum.

CHAPTER III

LOW TEMPERATURE THERMOMETRY

3.1 Introduction

The principal requirements of a low temperature thermometer besides high sensitivity are small size, low thermal capacity, rapid response and insensitivity to temperature cycling. Several devices are available which meet these criteria and which are useful over different temperature ranges. Junction diodes, for example are usable to about 1 K as are certain thermocouples (14); germanium resistors (15) and Allen Bradley resistors can be used to about 0.3 K although the magnetoresistance of germanium is about ten times that of carbon resistors (14); and Speer resistors have been successfully employed to 10 mK (16). Speer resistors type 1002, 220 ohm, $\frac{1}{2}$ W are the resistor of choice for measuring temperatures below 1 K (17, 18). The outer insulation is usually removed to reduce the thermal relaxation time and the resistors are sometimes ground to flat rectangular sections with the leads still attached. It is recommended that the tin plating and lead-tin solder of the leads be removed if the resistors are to be used at the lowest temperatures in order to minimize the semi-conducting thermal boundary resistance (19).

To calibrate these secondary thermometers it is necessary to establish a temperature scale below 1 K. This can be done by extrapolating some reproducible, single-valued, thermodynamic property of a suitable substance from a known temperature region. It is convenient to extrapolate Curie's Law for magnetic susceptibility from the liquid helium temperature range to temperatures at which the susceptibility is still a smooth function of temperature. The resulting relationship

$$\chi = C/T^*$$

defines a magnetic temperature, T^* , where χ is the susceptibility and C is the Curie constant for the substance used. For a specimen of ellipsoidal shape, the thermodynamic temperature is given by (20)

$$\begin{aligned} T &= T^* + C(N - D) \\ &= T^* + \Delta \end{aligned}$$

where N represents the dipole-dipole interaction, D is a demagnetizing factor and Δ can be considered as a shape-correcting factor.

In the case of a sphere $D = \frac{4}{3} \pi$, and for a cubic array of dipoles, $N = \frac{4}{3} \pi$ and $\Delta = 0$. In this case, the spherical magnetic temperature $T^\otimes = T^* = T$. If $N \neq \frac{4}{3} \pi$,

$$\begin{aligned} T &= T^\otimes + C(N - \frac{4}{3} \pi) \\ &= T^\otimes + \theta, \end{aligned}$$

giving the Curie-Weiss relation

$$\chi(T - \theta) = \text{const.}$$

It is customary to use cerium magnesium nitrate (CMN) as the reference salt because its susceptibility is essentially linear to 6 mK (21) [$\theta = -0.27$ mK (20)], although chromium potassium sulphate has also been used to ~ 11 mK (22) and ferric ammonium sulphate to 33 mK (23).

3.2 Experimental

(a) Apparatus

Two 220 ohm half watt Speer resistors were calibrated to 80 mK by extrapolation of Curie's law for CMN from the liquid helium region. They were cooled by adiabatic demagnetization of about 4 g of ferric ammonium sulphate in the arrangement shown in Fig. 1. (For clarity the mechanical detail, supporting threads, connections and dewars are omitted.) The spherically-shaped CMN reference salt, assembled from four single crystals, was interlaced with the coil foil thermal conductor. Contacting surfaces were thinly smeared with Apiezon N grease and the assembly was held together with nylon thread and a small bakelite clamp. The CMN was oriented so that it did not contribute to the cooling during demagnetization. Indium soldered to the foil were two thin-wall copper tubes in which the resistors were placed. Most of the insulation of the resistors was ground off and a thin coating of

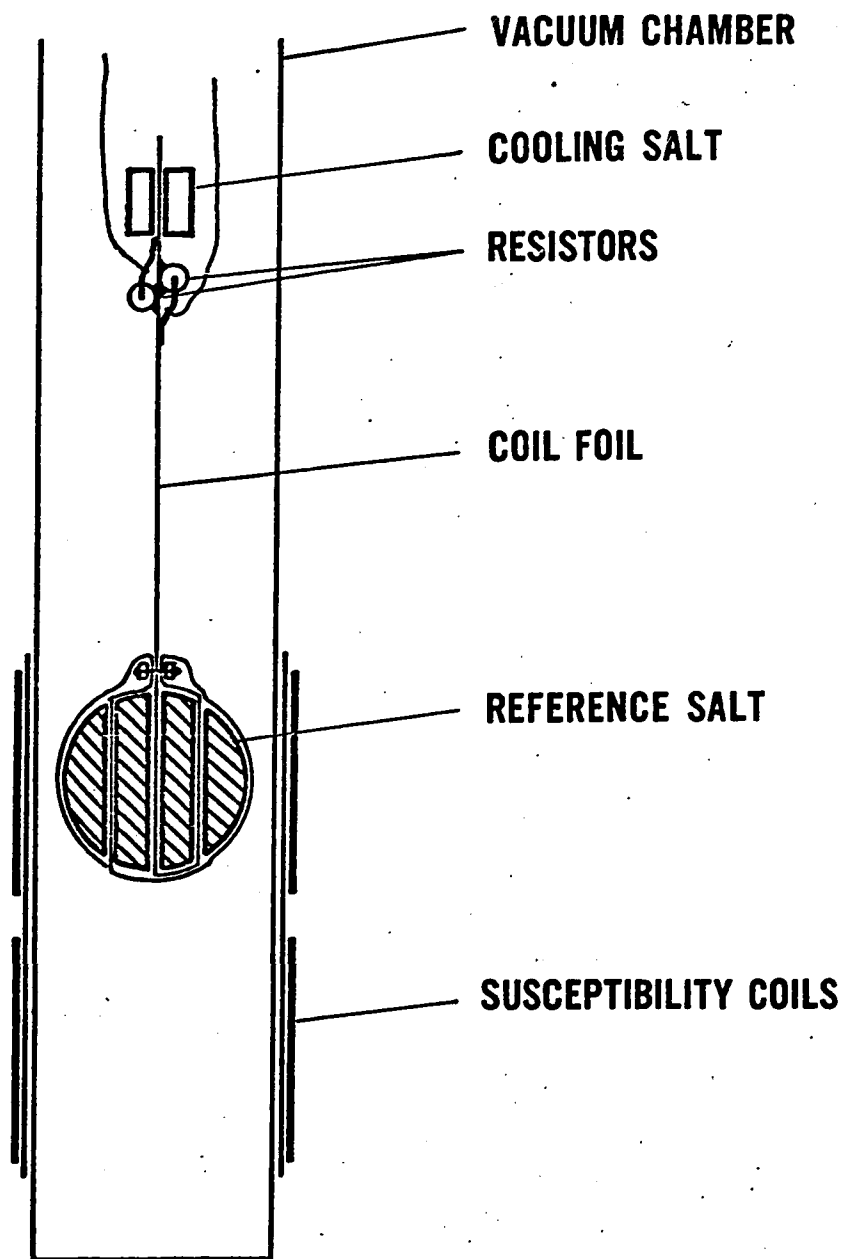


Fig. 1. Simplified diagram of the calibration cryostat

grease was applied prior to assembly. The leads were indium soldered to the foil to improve thermal contact. The cooling salt was also smeared with grease to provide good contact and to prevent decomposition.

The susceptibility of the CMN was measured in a Hartshorn-type mutual inductance bridge employing a ballistic galvanometer detector (24). Current was supplied by a 12 V car battery in series with a rheostat. The resistance of the thermometers was measured to within 0.1% with a three wire, 100 Hz Wheatstone bridge employing synchronous detection. It was previously constructed in the laboratory and consisted of General Radio 510 decades, GR 500J 10 k Ω fixed standards, a Princeton Applied Research model 121 combined audio oscillator and lock-in detector, Hewlett-Packard 403 B transistorized voltmeter and transformer isolation of the oscillator.

To prevent self heating and consequent erroneous temperature readings, the power in the resistance must be limited at low temperature. In the case of the balanced ($R_T = R_V$), equal-arm bridge shown in Fig. 2, half of the applied voltage appears across the thermometer, R_T . Harrison (25) indicates that at 80 mK ($R_T \approx 25 \text{ k}\Omega$), the maximum permissible power is 10^{-12} W . At this level, the voltage across the resistor is $(10^{-12} \times 2.5 \times 10^4)^{0.5} = 1.58 \times 10^{-4} \text{ V}$ and across the bridge the voltage is $3.2 \times 10^{-4} \text{ V}$. If Harrison's data are extrapolated to 1.3 K, the suggested maximum power dissipation is $4 \times 10^{-8} \text{ W}$ or 5.5 mV across 750 ohms; however

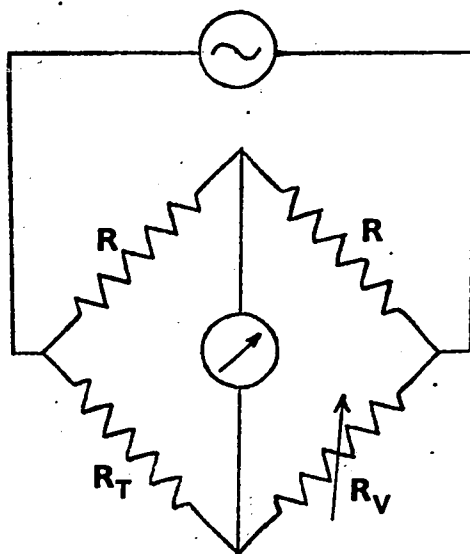


Fig. 2. The equal-arm Wheatstone bridge

slight ($\sim 0.5\%$) heating effects were observed at this level possibly because the thermal contact was not as good as in Harrison's case. It was therefore decided to operate the bridge at 0.1 mV ($8.3 \times 10^{-14} \text{ W}$) at low temperature and 0.2 mV in the $1.3 - 4.2 \text{ K}$ calibration range to improve resolution. Connection to the resistors was made with lead-covered Manganin wire. To minimize heating effects from R.F. pick-up, 1000 pF silver mica capacitors were connected across the resistors.

(b) Procedure

The cryostat was cooled to 77 K in about two hours by pouring liquid nitrogen in the outer dewar. During this time, 15 Torr of nitrogen gas was left in the walls of the inner dewar to maintain thermal contact and 760 Torr of nitrogen exchange gas

remained in the cryostat chamber. The inner dewar was then pumped hard, the chamber evacuated and filled with helium to 50 Torr and liquid helium was transferred into the dewar with a siphon. In the half hour required to complete the transfer, the chamber pressure was reduced to 5 Torr.

Resistance and susceptibility were measured at eight to ten different temperatures for $1.3 < T < 4.2$ K. The average of at least four readings was obtained at each temperature. The helium vapour pressure was maintained by a manostat in the pumping line and values of temperature were obtained from published tables of vapour pressure vs. temperature (26).

At the end of the calibration run, the inner dewar was refilled with liquid helium, the water-cooled electromagnet was rolled into place and the cooling salt was magnetized in a field of 18 kG. The remaining helium exchange gas was pumped out of the chamber when the heat of magnetization had dissipated, and, at a pressure of $\sim 10^{-5}$ Torr, the salt was slowly demagnetized.

3.3 Results

The susceptibility of the CMN was found to vary as $1/T$ in the liquid helium region as expected, and, from extrapolation of a least-squares' fit of the data points, temperatures below 1.3 K were obtained. The resistance also varied with reciprocal temperature in the calibration range, but below 1 K it did not follow a simple relationship. Various expressions have been proposed to express the

resistance as a function of temperature (27), some with secondary correction curves, but none was considered accurate enough over an extended range. It was therefore decided to obtain the temperature graphically over the immediate range of interest on a linear scale. Fig. 3 shows the overall calibration of R1 and Fig. 4 is the curve for $85 < T < 120$ mK. Values for R2 are similar. Data for the curves were obtained from nine different calibration runs, the first seven were taken between 6 December 1969 and 30 March 1970; the last two were obtained in November 1971. Below 0.13 K the early values of R1 differed by $\sim 5\%$ from those of November 1971 probably because the resistor had been heated very slightly to re-attach a lead. The reduction in resistance of the later values is consistent with resistor heat treatment (28). At 0.1 K the repeatability of the November data is about 2% and above 0.2 K, the reproducibility of all data is better than 2% in good agreement with Harrison's (25) estimate.

3.4 Magnetoresistance

Low temperature sensors are usually affected to some extent by the presence of a magnetic field. The effect may tend to increase or decrease the reading according to the material employed. In some cases, the change may be a complicated function of the field and temperature. For carbon composition resistors the effect depends distinctly upon the method of manufacture.

The magnetoresistance of Allen Bradley resistors is positive for $T < 4$ K. Above this temperature some researchers (29, 30, 31, 32)

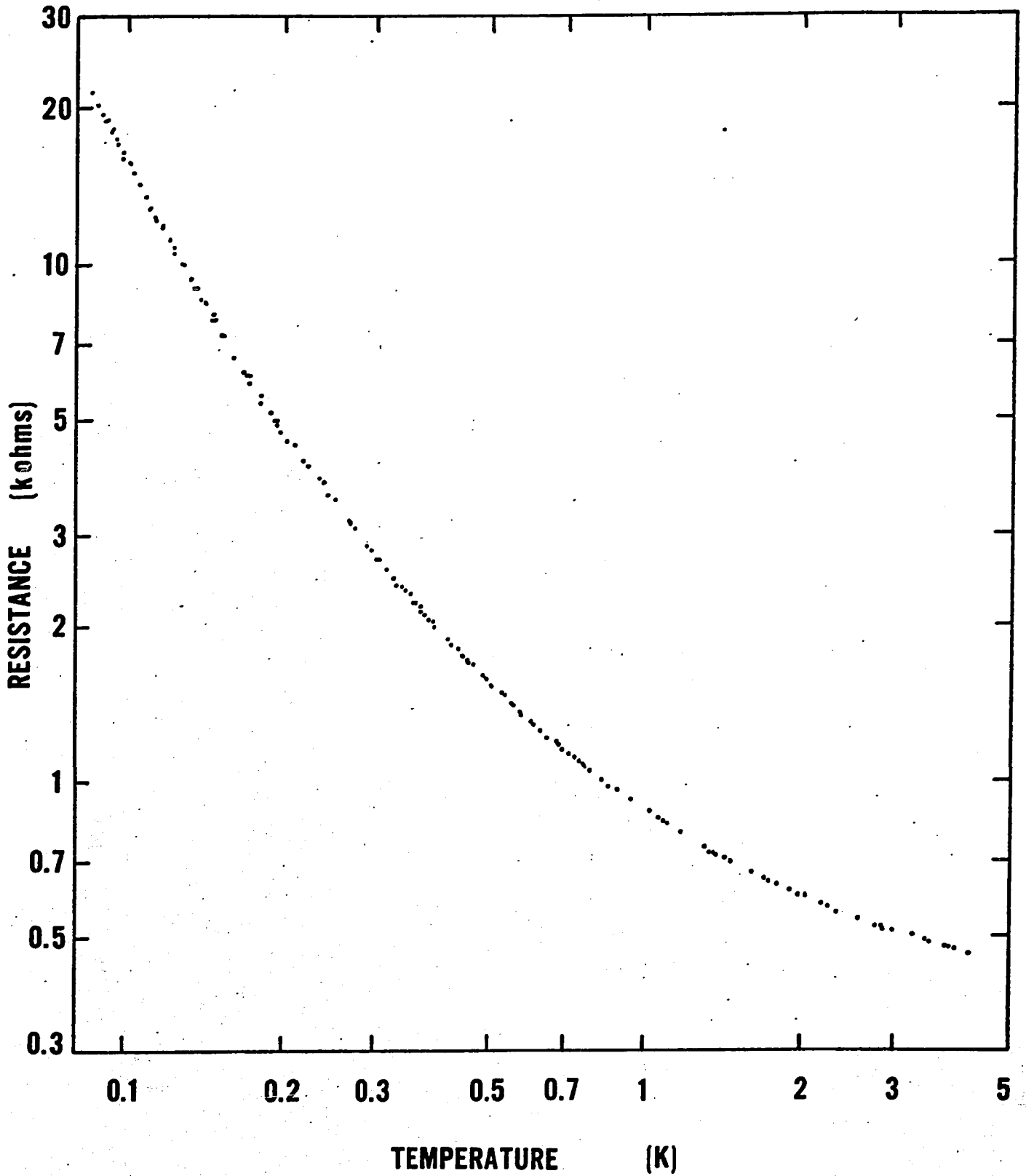


Fig. 3. Calibration curve of R1

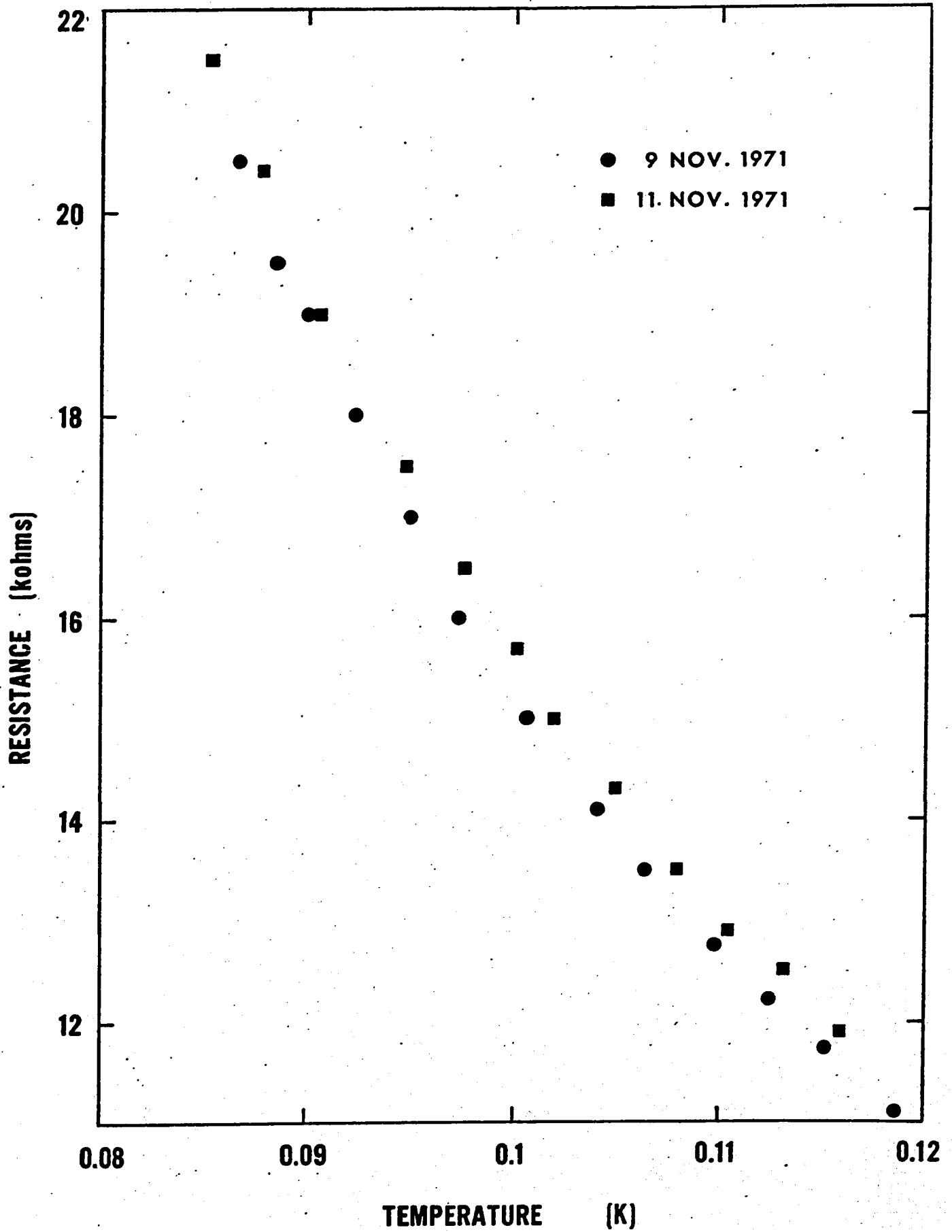


Fig. 4. Calibration curve of R1 (detail)

observed a positive effect whereas others (33, 34) claim negative shifts. The discrepancy in results suggests that a possible third factor may be involved such as the power dissipated in the resistor during measurement. In any case, this brand of resistor is not suitable for very low temperature measurement because the resistance varies too rapidly with temperature. The magnetoresistance is independent of the direction of the field.

For Speer resistors, Black et al. (17) found a negative effect with slope decreasing after 4 kG. The total change in resistance at 10 kG and 0.3 K was about 5%; at 0.5 K, it was 2.6%, and at 1.1 K, 1.3%. The effect is thus approximately inversely proportional to temperature. No difference in magnetoresistance was noted among the seven resistors tested ranging from 51 to 500 Ω and 1/10 to 1 W.

A smaller though similar variation was found by Gordy and Fritzsche (31) for 470 Ω 1/2 W resistors. The effect was inversely proportional to temperature, and at 0.35 K levelled to 2% at 9 kG where it remained constant to 13 kG, the maximum field used.

At 1 K, Edelstein and Mess (18) found a total shift in resistance for a single 220 Ω 1/2 W resistor of -0.9% at 9 kG, with no further change to 12 kG, in reasonable agreement with Black et al. No evidence of a levelling off is seen at lower temperature though for fields up to 7 kG. At 0.113 K the change is 5.6% and is still rising. No dependence on the direction of the field was observed. The temperature variation appears to be more rapid than linear below

0.23 K, but scatter in the points makes a reasonable estimate difficult.

Saito et al. (35) provide the only high field data published on Speer resistors, however, the lowest temperature used was 1.5 K for four 470 Ω 1/2 W samples. Three 220 Ω 1/2 W resistors were examined at 4.2 K. In both cases the effect was initially negative, levelling at about 1% between 20 and 30 kG at 4.2 K, changing sign at about 35 kG and rising to + 3 to 4% at 75 kG. At 1.5 K the cross-over occurs at 37 kG for two samples and at 52 kG and 64 kG for the others. At 75 kG $\Delta R/R = +2.5, 1.8, 0.9$ and 0.4% respectively. The scatter in the data makes it impossible to determine the temperature variation.

There is general agreement on a negative magnetoresistive effect at ~ 1 K amounting to about 1% near 10 kG where it remains approximately constant for several kG. The uncertainty in the measurements at low temperature and low field make extrapolation to higher fields rather hazardous. The same can be said for extrapolation to low temperature from the high field, high temperature data.

Despite the lack of solid information, it is interesting to note that at 1.5 K, the crossover point for two of Saito's resistors occurs at higher fields than at 4.2 K. At these fields the slope of the curves is shallow and suggests that at low temperature the crossover may occur at even higher fields. One is thus led

to believe that any observable effect at very low temperature and high magnetic field will be small. In all likelihood, it will probably be masked by the 2% uncertainty in calibration.

CHAPTER IV

THE SENSITIVITY OF NUCLEAR EMULSION AT LOW TEMPERATURE

4.1 Introduction

Nuclear emulsion is a particular type of photographic emulsion used to record the passage of charged particles. It is thicker than ordinary photographic emulsion, contains a greater concentration of silver bromide and is finer grained. It is available with or without glass backing, in several thicknesses (50 - 600 μ) and in various formulations for different applications.

Layered blocks of pellicles may be built up to provide an extended three-dimensional detector of particles. These stacks are particularly useful in beam work and ionospheric flights.

Some nuclear emulsion is available in gel form, which, when warmed may be poured into any desired shape and thickness. A further advantage of emulsion gel is that previously-recorded cosmic ray tracks are broken up in the warming and pouring process thus clearing the pellicle of an annoying background of spurious long tracks. Emulsion can not be shielded from cosmic rays because of their very high energies, and the pellicles act as a continuous detector as soon as poured. When bought preformed emulsion usually

contains a few tracks which increase in number with time. There is therefore a limited useful age of emulsion beyond which the background is considered too great.

Besides serving as a detector of ionizing particles, nuclear emulsion can also yield information on the nature of the particle and its energy. The tracks of α -particles and protons above the minimum of ionization, for example, are much denser than those of electrons, and measurement of the range in the emulsion allows an estimate of the particle's energy to be made from the stopping power of the emulsion.

Neutrons may be indirectly detected by their interaction with the hydrogen in the gelatine (proton recoil) or by loading the medium with suitable target nuclei such as boron or lithium (36). In this latter case, the reaction products identify the original particle.

The disadvantages of nuclear emulsion are the delay involved in processing and the tediousness of searching for the tracks under a microscope; however, in certain very-low-temperature nuclear orientation experiments, the use of emulsion is unrivalled.

4.2 Processing of Nuclear Emulsion

Nuclear emulsion is handled in the darkroom in much the same way as in tray processing of common photographic film, except that the temperature of the different baths is not the same and the times involved are somewhat longer.

Thick pellicles may be processed unmounted, but they are usually supported on filter paper or lintless cloth while in the solutions. After processing they are mounted on specially-prepared glass plates. Thin pellicles are mounted before processing to reduce handling difficulties and track distortion. They may be developed in the usual way in ID-19 developer diluted with an equal volume of water for 5 - 15 minutes depending upon the emulsion used. A water presoak is sometimes used to swell the emulsion so that the developer will penetrate more rapidly. Emulsion thicker than 100 μ , however, requires special presoaking in cold ($\sim 5^{\circ}\text{C}$) developer to allow the inactivated solution to penetrate to the interior without over-developing the surface. Isothermal development at 10°C may be used, although best results are obtained with Brussels' amidol developer (37) and a warm ($24 - 25^{\circ}\text{C}$) development stage lasting about one hour. The same process is often used with thin emulsion when maximum discrimination is needed.

After development, the pellicles are transferred to an acetic acid stop bath at 5°C and then fixed in plain hypo, at $\sim 10^{\circ}\text{C}$, for about 50% longer than the time required to clear the emulsion. The temperature of the stop bath is kept low to arrest the development process quickly and the fixer is kept cool to prevent unnecessary emulsion swelling. The time spent in these baths is longer for glass-mounted emulsion (plates) than pellicles because there is only one side exposed to the solution; the time also increases as the square of the emulsion thickness. Barkas (38) suggests $80 z^2$ minutes for fixing where z is the thickness in units of 100 μ , and $40 z^2$

minutes for washing. To reduce gelatine losses the wash water temperature is kept to 12 - 15°C.

The washed emulsions are dried by successive immersion in ethyl alcohol solutions of increasing concentration, such as 20, 40 and 60%, and containing about 5% glycerine. The alcohol causes less distortion than air drying and also inhibits the growth of organisms in the gelatine. The glycerine acts as a plasticiser and helps to control the emulsion shrinkage caused by silver loss in the fixer. The time spent in each solution is equivalent to the penetration time, except for the last bath where the pellicles remain until they have returned to their original linear dimensions. The final thickness is usually about half the unprocessed thickness (shrinkage factor = 2).

Since the thickness of the dried emulsion depends upon the humidity of the air, the plates are usually stored at constant temperature ($\sim 20^{\circ}\text{C}$) and relative humidity ($\sim 55\%$).

4.3 Low Temperature Sensitivity

The latent image formed in photographic emulsion does not remain stable but is subject to a certain degree of fading. This deterioration increases with temperature, time, particle energy and the presence of oxygen. It decreases with loading by materials of high pH and is lower in emulsion with large grain (39). At low temperature the actual formation of the latent image may be interfered with.

According to Gurney and Mott (40), the latent image is formed in a two-step process:

- (i) Photons are absorbed by the silver halide causing the release of electrons which become trapped at sensitivity centres.
- (ii) Positively-charged silver ions migrate to negative traps where silver particles form.

At very low temperature, ionic mobility is essentially zero and the second step can not be completed. It is only as the emulsion warms that the latent image can form. Any factor causing destruction of the traps or neutralization of the ions, including electron-positive hole recombination, results in a loss in sensitivity.

Emulsion which performs well at room temperature does not necessarily give good results at low temperature, although very sensitive emulsion with large grain might be expected to do so. A comparative study by Avan and Dubois (41) of Ilford G5, K5, K0 and C2 emulsion exposed to α -particles showed that only G5 and K5 were usable at 77 K and only G5 retained an appreciable sensitivity at 4.2 K. K0 and C2 have low sensitivity at room temperature whereas K5 (mean grain diameter 0.2μ) and G5 (mean grain diameter 0.27μ) both have good sensitivity. Similar results were obtained by Debeauvais-Wack (42) with C2 and G5 at 77 K. Estimates of the relative sensitivity of K5 and G5 are summarized in Table I. From these results it is evident that G5 is more suitable than K5 for use at 4.2 K.

Since many nuclear orientation methods require temperatures below 4 K, the relative sensitivity of G5 emulsion was measured at

0.1 K, a temperature which can be readily achieved and maintained by adiabatic demagnetization of chromium-potassium sulphate. A preliminary estimate was also obtained at 4.2 K. To determine any possible effect of temperature cycling on the latent image, pellicles that had been irradiated at room temperature were cooled to 0.1 K and then rewarmed.

TABLE I

COMPARISON OF THE SENSITIVITY OF
ILFORD K5 AND G5 NUCLEAR EMULSION AT LOW TEMPERATURE

Emulsion	Temperature (K)	Relative Sensitivity (%) or Observations	Reference
K5	77	41 - 59	43
		70	44
		Rayons α presque continus	41
K5	20	29	43
	4.2	Traces se perdent dans le voile sensibilité faible aux rayons α	44
			41
G5	77	Rayons α presque continus	41
		α en grains séparés	42
		75	45
	20	70	43
G5	4.2	Rayons α à structure lacunaire	41
		>75	45

4.4 Experimental

(a) Measurements at 4.2 K

A preliminary experiment was made to establish qualitatively the sensitivity of Ilford G5 emulsion at 4.2 K. Pellicles measuring 2.5 cm x 7.5 cm x 150 μ thick were poured on clean microscope slides following the manufacturer's instructions. A small beaker containing the strips of gel was warmed to 45°C in a water bath and as soon as the gel had melted, it was poured down a 3 mm glass stirring rod onto the plate. Use of the rod helps to control the pouring and filling of the corners of the glass slide. A surprising quantity of emulsion can be poured onto the plate before the hydrostatic pressure exceeds the surface tension. By placing the plates on a large (level) piece of glass it is possible to salvage emulsion lost from "breaks" when they occur. The amount of emulsion that can be poured without using waxed retaining walls depends upon the temperature of the liquid. Thicker layers may be obtained with cooler emulsion, but the possibility of lumps occurring increases with decreasing temperature.

The emulsion was removed from the glass by first cutting around the edge with a scalpel and then lifting one corner. The resultant pellicles are about 2 mm undersize, however tearing due to edge adhesion is eliminated.

One pellicle and one plate were wrapped together in black paper and then sealed in polyethylene. The package was placed between two small blocks of styrofoam to keep it vertical and the ensemble was placed at the bottom of a double dewar. Liquid nitrogen was

poured into the outer dewar and when the temperature of the inner dewar had stabilized an hour later, as indicated by the resistance of an Allen Bradley 220 Ω $\frac{1}{2}$ W resistor, the inner dewar was filled with liquid helium.

The dewar system was then placed in a water tank containing a two Curie Pu-Be neutron source to irradiate the emulsion. The water protects personnel by serving as a moderator for the high-energy neutrons, assuring that most of them are effectively thermalized by the time they leave the tank. Two layers of 1/8 inch lead sheet surrounded the dewars to attenuate the γ -radiation emanating from the source and produced in the water during the neutron slowing down process. The emulsion was exposed for 5 hours at a distance of 8 cm from the centre of the source.

Two control emulsions poured at the same time as those exposed at low temperature were given the same irradiation at 22°C in empty dewars.

The test and control emulsions were processed according to the following schedule:

Water presoak	5°C	1 hour
Developer presoak (D-19 diluted 1:6)	5°	30 min.
Develop	20°	30 min.
Stop (0.5% acetic acid)	5°	20 min.
Fix	8°	4 hours
Fix	8-16°	30 min.
Wash	17°	1 hour
Dry (5% glycerine, 95% ethyl alcohol)	21°	20 min.

The pellicles were then mounted on glass plates and allowed to dry completely in air at 21°C and 55% relative humidity.

In this preliminary experiment, Kodak D-19 prepared developer was used for convenience. The utmost in sensitivity and discrimination is not obtained with this product, but for qualitative measurements its use was considered acceptable.

(b) Measurements at 0.1 K

In the second experiment, Ilford G5 emulsion was irradiated at 0.1 K in the arrangement shown in Fig. 5. The same cryostat as was used in calibrating the resistance thermometers was employed.

To cool the emulsion, a salt pill containing a 57 g slurry of chromium potassium sulphate in glycerine was prepared. Mixed in it were several hundred short pieces of No. 40 copper wire about 5 mm long to improve the thermal conductivity. The mixture was sealed with epoxy resin in a stainless steel tube 2.5 cm in diameter and 9.3 cm long supported by three taut nylon threads. The temperature of the pill was monitored by the previously calibrated thermometer R2 attached to a piece of coil foil extending into the container from above. Two additional pieces of foil 2.5 cm wide entered the cooling salt from below.

Two prepared pellicles measuring 2.5 cm x 7.5 cm x 400 μ thick were sealed in separate black vinyl envelopes (not shown in the diagram) and placed vertically between the lower foils. Mounted on another piece of coil foil placed between the pellicles, but thermally isolated from the outer foils, was the thermometer R1. All

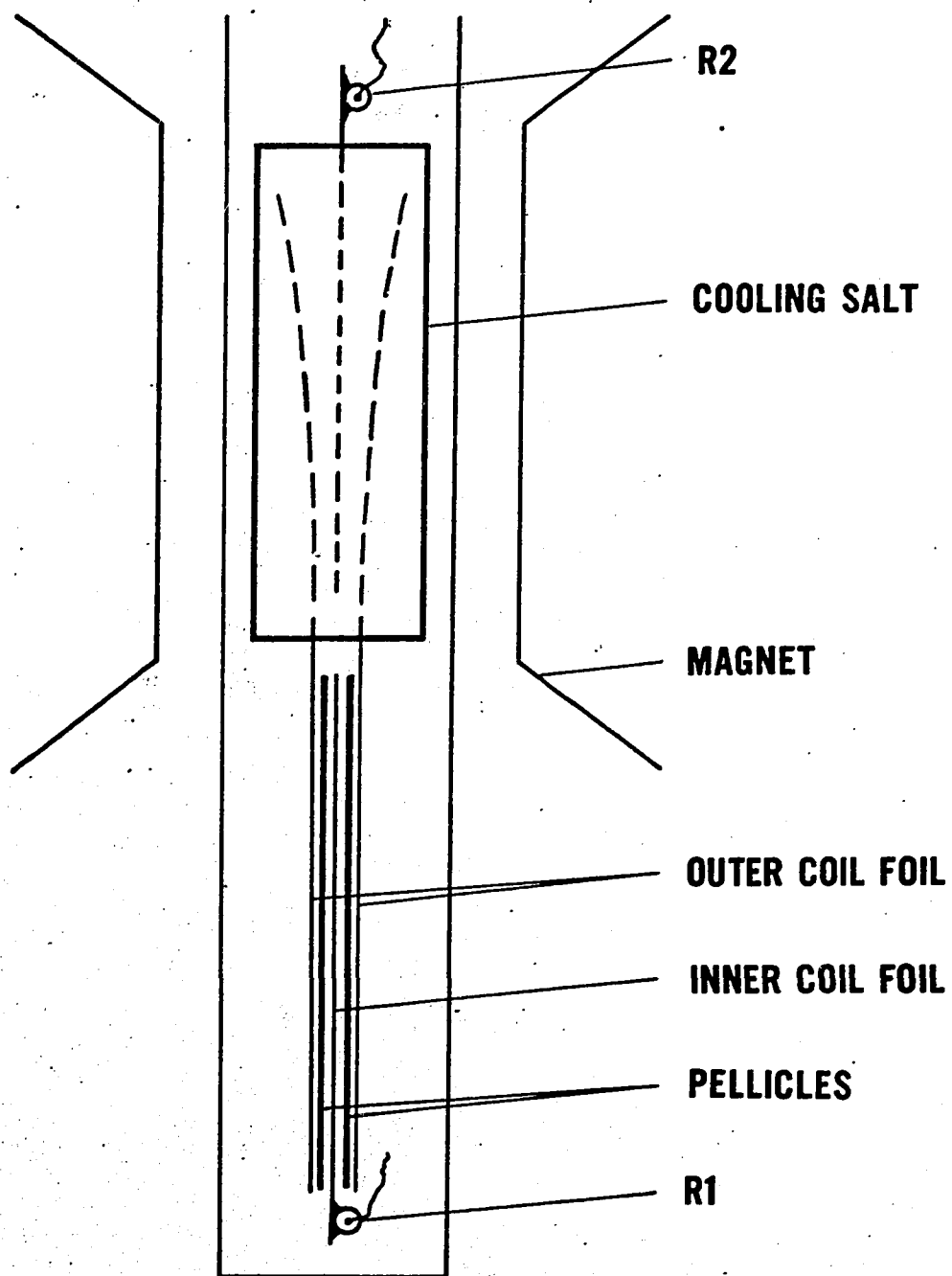


Fig. 5. Simplified diagram of the cryostat

contacting surfaces were thinly smeared with glycerine to improve the thermal contact. Glycerine is used instead of grease because it is water soluble and will not harm the emulsion if the envelope should rupture during the experiment. The pellicles and coil foils were kept in intimate contact by two pieces of 1/8 inch bakelite 2.5 cm wide and 7.5 cm long placed on the outside of the outer foils and tied tightly with masking tape.

The pellicles were cooled to 77 K in about 2 hours by pouring liquid nitrogen in the outer dewar of the cryostat and to 4.2 K in another hour by filling the inner dewar with liquid helium. Pumping on the liquid helium further reduced the temperature to 1.3 K at which point the cooling salt was magnetized to 18 kG. Five minutes later the residual thermal exchange gas was removed from the chamber, and, at a pressure of 10^{-5} Torr the salt was slowly demagnetized. Final demagnetization was achieved by careful, slow withdrawal of the magnet from the cryostat.

The pellicles were irradiated along the short axis with neutrons from the 2 Curie Pu-Be source at a distance of 9 cm for 125 minutes. During this time, the emulsion cooled from 0.148 K to 0.135 K. (The experiment was terminated at this point because of a sudden rise in temperature caused by insufficient liquid helium in the dewar.) The temperature of the pellicles was measured on the same bridge and with the same power dissipation as was employed in the resistor calibration. The shielding around the source amounted to 8 mm Pb. Control emulsions were given the same exposure at 25°C with empty dewars around the cryostat.

After the low temperature irradiation, the pellicles were allowed to warm to 0°C in about 2 hours and were placed in a refrigerator. The control pellicles were then irradiated and all were developed without delay in Brussels' amidol developer. A stock solution of this developer was prepared earlier without amidol which oxidizes rapidly. To prepare a working solution, 0.9 g amidol was added to 200 ml of stock. In processing the emulsions, no cold water presoak was given to avoid possible fading of the latent image. The following procedure was followed:

Developer presoak	4°C	1½ h
Warm-up period	4-24°	½ h
Develop	24°	½ h
Stop	4°	1½ h
Fix	6°	30 h
Wash	12-16°	10¼ h
Dry 20% alcohol + 5% glycerine	10°	1 h
40% alcohol + 5% glycerine	10°	1 h
60% alcohol + 5% glycerine	10°	6 ¾ h

The pellicles were then mounted on prepared glass and allowed to dry as before. In the warm development stage, the pellicles were blotted dry and placed in a sodium sulphite solution (18 g/l) to prevent oxidation of amidol in the surface of the emulsion.

(c) Effect of Temperature Cycling

Two wrapped 400 µ pellicles were mounted in the cryostat and irradiated as before for 2 hours at room temperature. Immediately after the irradiation, one pellicle (the room temperature control)

was removed and placed in the refrigerator and an unexposed, replacement pellicle was installed to retain the original geometry for reasons of thermometry. The outer dewar was then filled with liquid nitrogen, and 1½ hours later, liquid helium was poured into the inner dewar. Forty minutes was required to complete the transfer and fifteen minutes of pumping reduced the temperature from 4.2 K to 1.3 K. The pellicles remained at 1.3 K for 45 minutes during magnetization of the salt and evacuation of the chamber. Demagnetization quickly reduced the emulsion temperature to 0.11 K, and 0.10 K an hour later as cooling slowed. At the end of the second hour, the temperature had reached 92 mK, but the salt was warming rapidly and 2½ hours after demagnetization with the emulsion at 0.117 K, exchange gas was readmitted. The cryostat was allowed to warm overnight with liquid nitrogen remaining in the dewar for about 10 hours.

The pellicles were still well below 0°C when they were removed 13 hours after pumping on the helium bath was halted. They were processed and mounted as for the cold-irradiated emulsions.

4.5 Results and Discussion

(a) Measurements at 4.2 K

The pellicle irradiated at 4.2 K and the controls were observed under a binocular microscope employing oil immersion

objectives. No measurements were made on the cooled plate as the emulsion appeared to shred in the developer and floated away—probably because of the difference in thermal expansion between glass and gelatine.

It was immediately obvious that the emulsion exposed at low temperature still retained a good fraction of its room temperature sensitivity by the profusion of electron tracks originating from the incompletely-attenuated γ -radiation. The tracks, however, were not long enough to obtain reasonable measurements. Proton-recoil tracks longer than 200 μ and lying in a plane parallel to within a few degrees of the surface were selected for study.

The rate of energy loss of a particular particle at the end of its track is approximately the same for all initial energies and is proportional to the number of silver bromide grains ionized and reduced to silver in the developer. When a loss in sensitivity or fading occurs some of these ionized grains are not fully reduced and gaps appear in the track. The relative sensitivity of an emulsion is thus determined by comparing the number of grains or the total gap length per unit distance over the same residual range with that of the control.

For heavily ionizing particles, such as protons, the grain density at the end of the track may be too great to count reliably. It is then not unreasonable to begin counting at a fixed but short distance from the end of the track, especially if very accurate

estimates are not required. In the present measurements, grains were counted over the next-to-the-last 100 μ of track.

For the low temperature pellicle, the average number of grains per 100 μ for 25 tracks was 54, and for the room temperature control, it was 95, giving a relative sensitivity of about 60%.

This figure is considered to underestimate the potential of the emulsion because D-19 developer was used instead of Brussels' amidol which is known to give better results [see (b) below]. The experiment was conducted to ascertain that the emulsion was indeed sufficiently sensitive at liquid helium temperature to warrant cooling to 0.1 K.

(b) Measurements at 0.1 K

The grain density at the end of the proton tracks in emulsion irradiated at room temperature and developed in Brussels' amidol developer proved to be too high to obtain realistic measurements. The gap length was therefore measured, and, from an estimate of the average grain diameter, the number of grains per unit distance was inferred. This method was considered more accurate than attempting to measure actual grain density at a fixed distance from the end of the track as in (a) above.

The last 73.3 μ of track was analysed corresponding to a single field of view of the microscope.

The average diameter of grains in the room temperature plate, obtained from 105 measurements, was found to be $0.56 \pm 0.01 \mu$, and the average gap length, from measurements on 70 tracks, was

$10.3 \pm 0.3 \mu$ leaving $73.3 - 10.3 = 63 \mu$ of equivalent continuous track. The number of grains per 73.3μ is $\frac{63}{0.56} = 113$.

In the low temperature plate, the average number of grains counted per 73.3μ for 115 tracks was 83.6 ± 0.9 , yielding a relative sensitivity for G5 emulsion at 0.1 K of $74 \pm 2\%$. This result is in excellent agreement with the work of Waniek (45) at 4.2 K and Sedrine and Weill (43) at 20 K, and has already appeared in abbreviated form (46).

The high grain count in the low temperature plate (120/100 μ) confirms the superiority of the warm-stage Brussels' developer compared to D-19.

(c) Effect of Temperature Cycling

The pellicle irradiated at room temperature and cooled to 0.1 K was compared visually with its uncooled control without quantitatively analysing the tracks. Ten fields of view were observed.

There was no noticeable reduction in the density of proton and electron tracks (number of tracks per field of view examined throughout the emulsion thickness) nor in the grain density of the proton tracks.

It would appear that cooling the emulsion from room temperature to 0.1 K and rewarming it has no appreciable effect on the preformed latent image. This temperature stability and the low time-related fading reported by Barron and Wolfendale (47)

suggest that the retained high sensitivity at 0.1 K should hold well into the mK range.

It may be concluded that Ilford G5 emulsion is suitable as a detector for ionizing particles at very low temperature and should prove useful in certain nuclear orientation experiments taking place below 1 K.

CHAPTER V

COOLING OF EMULSION IN A MAGNETIC FIELD

5.1 Introduction

The brute force method of orientation is applicable in principle to all nuclei with spin and magnetic moment different from zero (5). The basic simplicity of the method is attractive in some ways although the small degree of orientation obtained by readily available temperatures and magnetic fields limits its usefulness. Despite this drawback, Dabbs et al. (48), for example, were able to show that the angular momentum of the compound state in ^{115}In for 1.458 eV neutrons is $J = I + \frac{1}{2} = 5$. They cooled their indium target to 40 mK in a 16.4 kG field to obtain a polarization of 2.1%. Marshak (49) also used the method in measurements of neutron transmission through polarized ^6Li nuclei. A survey of nuclei that have been polarized by the brute force method is given by Shirley (50).

Methods of orientation which depend upon low temperature to populate the lowest hyperfine levels can only be used with samples with short spin-lattice relaxation times (T_1) in order that the nuclei may be cooled by the lattice in a reasonable period. In

arrangements employing adiabatic demagnetization cooling, the maximum value of T_1 that may be tolerated depends upon the exposure time needed and the rate of warming of the assembly, and may be as high as several minutes. This figure would have to be reduced considerably if the energy absorbed by the target is high enough to warm it. Substances with short T_1 are principally the metals.

Nuclear emulsion may be loaded with metals in the form of wires (51) and powders (52). The wires are usually wound on a former and the emulsion poured around them. They offer the advantage of delimiting the scanning area during analysis and of more efficient cooling since they can be connected directly to the heat sink. In the case of powders, a sandwich arrangement consisting of poured emulsion, sprayed fine powder and second poured layer is probably better than a non-uniform dispersion of the material throughout the emulsion. Distortion of tracks around the metal, which is occasioned by the loss of silver during processing and the consequent shrinkage of the emulsion, may be reduced by refilling the voids with resin dissolved in alcohol. When interactions with thermal neutrons are to be recorded, ^{10}B ($\sigma = 3800$ Barns) and ^6Li ($\sigma = 910$ B) are useful target nuclei. At warmer temperatures, both have been used together in the form of lithium borate infused into the preformed emulsion (36).

In the arrangement to be described, pellicles were cooled to 0.1 K in the presence of a 70 kG magnetic field. If the emulsion had been loaded with ^{10}B ($\mu = 1.8 \beta_N$, $I = 3$), the expected polarization would have been (cf. section 2.2):

$$f \approx \frac{1}{3} \left(\frac{I + 1}{I} \right) \frac{\mu H}{kT} = 2.1\%.$$

Under the same conditions, the polarization of ${}^6\text{Li}$ ($\mu = 0.822 \beta_N$, $I = 1$) would have been 1.5%.

5.2 Experimental

(a) Apparatus

The experimental arrangement used to cool nuclear emulsion to 0.1 K in a high magnetic field is shown schematically in Fig. 6. The dewars are omitted for clarity.

A new cryostat was constructed for this last phase of the project as the one used previously had insufficient room to accommodate the superconducting solenoids. Stainless steel dewars were used with overall height of 1.52 m and inside diameter 13.7 cm. The cryostat was attached to a vertically movable stage carrying the diffusion pump, vacuum gauge and the pumping line to the experimental chamber attached below (see Fig. 7). It was supported by ball bushings travelling along two vertical steel bars and was actuated by a hand-operated, worm-driven pinion riding on a vertical rack. The stage was counterbalanced with removable lead weights and had enough lift to allow easy removal of the dewars. Flexible lines connected the water supply and external forepump to the diffusion pump, and a removable, 3.8 cm diameter, rubber link joined the helium bath to the main helium pumping and recovery system. Copper baffles were

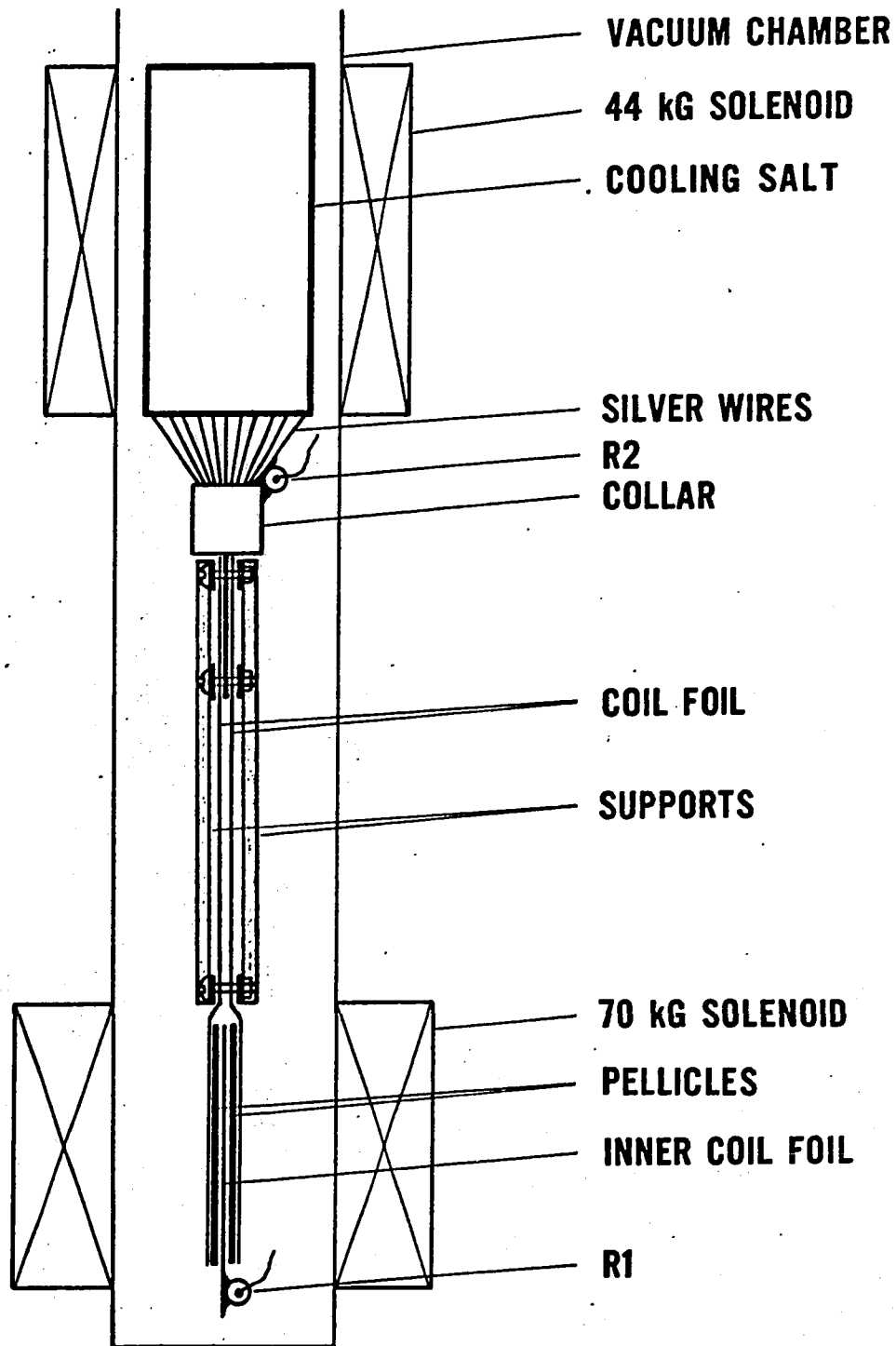


Fig. 6. Arrangement for cooling emulsion in a magnetic field

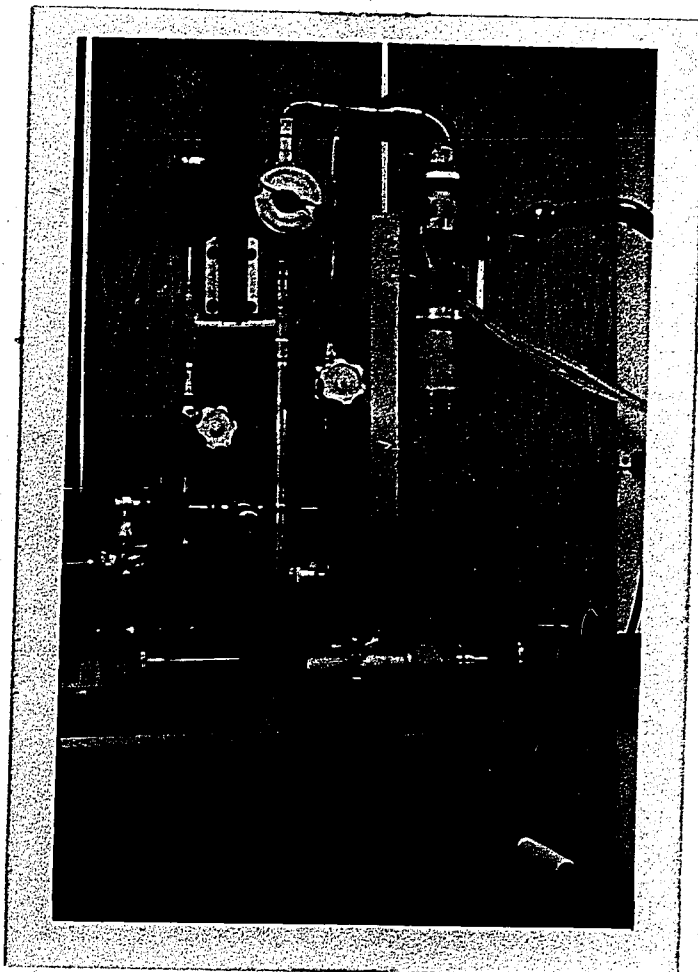


Fig. 7. The movable stage of the new cryostat

- | | |
|------------------------|------------------|
| B. Baffles | L Removable link |
| C Chamber pumping line | P Diffusion pump |
| D Diodes | S Shunt |
| G Vacuum gauge head | |

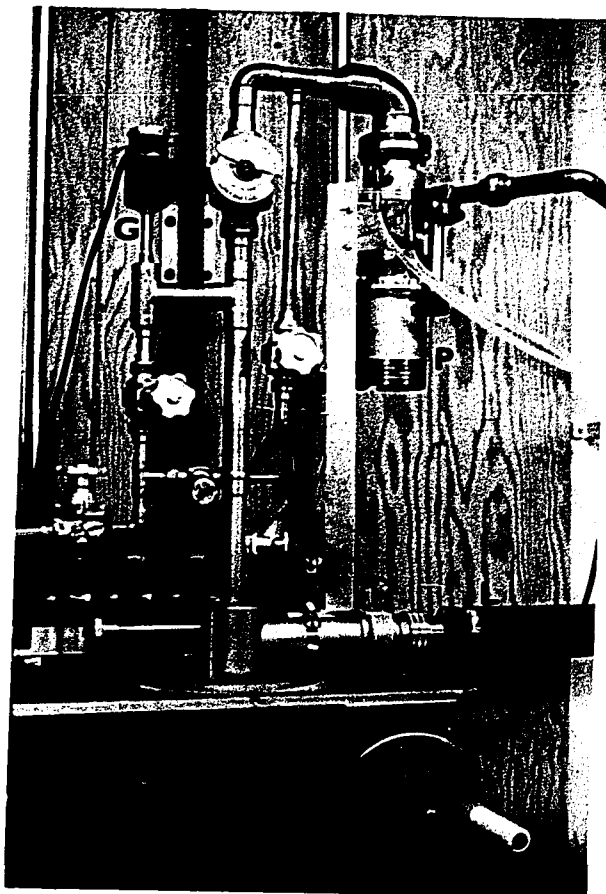


Fig. 7. The movable stage of the new cryostat

B Baffles	L Removable link
C Chamber pumping line	P Diffusion pump
D Diodes	S Shunt
G Vacuum gauge head	

mounted on the chamber pumping line to improve cooling by the evaporating helium during pumping from 4.2 to 1.3 K. Level-sensing resistors were positioned near the top of the pumping line and at the top of the 70 kG solenoid.

The magnetic field for the emulsions was provided by a Ferranti-Packard model 7001-7012C 70 kG superconducting solenoid, and for the cooling salt by an F-P model 8000 44 kG solenoid. They were supported by three thin wall stainless steel tubes anchored to the movable stage. A Harrison model 6260 A, 10 V, 100 A, power supply energized the solenoids and was protected from them (in the case of their becoming normal while energized) by two pairs of International Rectifier 70H5 rectifiers connected back-to-back as shown in Fig. 8:

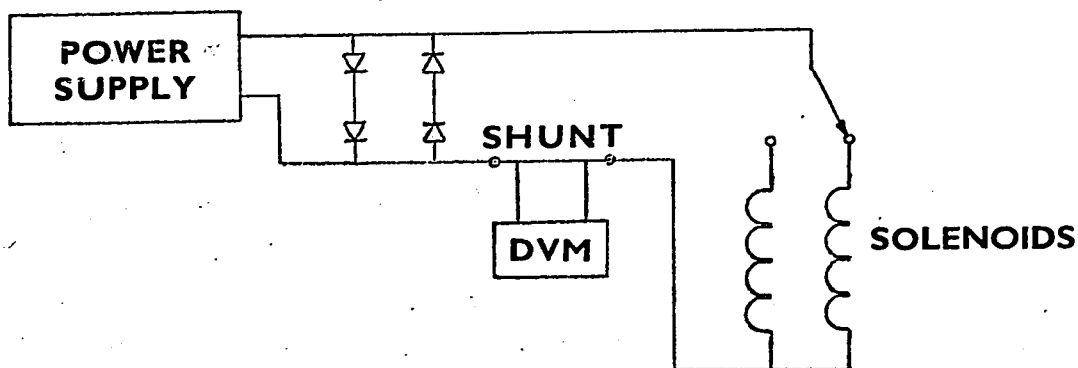


Fig. 8. Wiring Diagram for the Solenoids

The current was measured by a Hewlett-Packard model 3439 A digital voltmeter connected to a 100 A, 50 mV shunt.

A salt pill consisting of 48 g of chromium potassium alum crystallized around ~ 700 silver wires was used to cool the pellicles. The silver wire bundle was made by winding a continuous 0.024 cm diameter wire on a flat bakelite mandrel. The wire was then cemented to a rectangular piece of fibreglass screening attached to one side of the mandrel before the winding. When dry, the wires were cut across the turns to give a single strip of parallel wires supported by the fibreglass. This strip was rolled, together with a thin piece of perforated plastic sheet of 3 mm square mesh, to form a cylinder 2 cm in diameter and 11.5 cm long. The tail of wires which extended beyond the screening and plastic mesh was coaxed into a copper collar and silver soldered to it. Attached to the collar were a thin wall copper tube for thermometer R2 and a copper plate to support the coil foils.

For good thermal contact, the cooling salt was crystallized directly onto the wires. To achieve this, the wire bundle was immersed vertically in a saturated solution of chromium potassium sulphate and the wires were cooled by attaching one leg of a U-shaped strap made of 3 mm x 2.5 cm copper bar to the collar and placing the other leg in a 5 l dewar of liquid nitrogen. An adjustable heater maintained the temperature of the solution above $\sim 16^\circ\text{C}$ below which excessive ice formed on the collar.

The completed salt pill was smeared with Apiezon N grease and hung in the cryostat with nylon threads (not shown in the diagram). Two coil foils, 2.5 cm wide and 34 cm long, were screwed to

the collar plate and were supported by a pair of side braces made of two 3 mm diameter copper rods 27.3 cm long. The pellicles, inside vinyl envelopes, were placed between the ends of the foils below the supports and the thermometer R1 assembly was inserted between the envelopes as in section 4.4 (b). Contacting surfaces were smeared with glycerine.

(b) Procedure

The cooling down procedure with the new cryostat is basically the same as with the one used in the 0.1 K emulsion sensitivity tests and calibration of the resistance thermometers. The main difference lies in the longer times needed to cool the increased mass. Overnight (≈ 12 h) cooling with liquid nitrogen in the outer dewar is required to reduce the temperature to 77 K and slightly more than an hour and 18 - 20 l of liquid helium are needed to cool to 4.2 K. The amount of helium used depends upon the rate of transfer; if it is too rapid, the cooling capacity of the cold helium gas is not fully utilized; if it is too slow, liquid helium is wasted in keeping the idle cryostat cool for longer than is necessary. Towards the end of the transfer, the lower solenoid was energized so that the helium evaporated by Joule heating of the leads could be replaced. The current was applied slowly (3.5 A/min.) to prevent the solenoid from becoming unexpectedly normal, and at 49 A (70 kG), the persistent current switch was closed. Power was then removed, the transfer completed and the bath pumped to 1.3 K.

The upper solenoid magnetising the cooling salt was ener-

gized to 40 A (26 kG) and the chamber pumped out. This solenoid is capable of producing a 44 kG field, however the current required (68 A) necessitates the use of heavy leads and consequent high heat input. Since very high fields were not needed and to preserve helium, it was decided to operate at 40 A and use smaller leads.

The salt was slowly demagnetized when the chamber pressure reached 10^{-5} Torr.

At the end of the run, the lower solenoid was reconnected, the current raised to 49 A, the persistent current switch opened and the solenoid slowly de-energized.

5.3 Results and Discussion

The temperature of the pellicles and cooling salt as a function of time after demagnetization is shown in Fig. 9.

The pellicles attained their lowest temperature of 88 mK an hour after demagnetization and they remained below 0.1 K for 91 minutes. The usable irradiation temperature of $97 \text{ mK} \pm 10\%$ lasted for 106 minutes. If longer times are needed, the demagnetization process may be repeated as often as desired, however, the inner dewar must be refilled with liquid helium before each run.

The temperature of the cooling salt is seen to reach a minimum of about 38 mK 10 minutes after demagnetization and then rises as the pellicles continue to cool. When the temperature difference diminishes to 35 mK the pellicles also begin to warm,

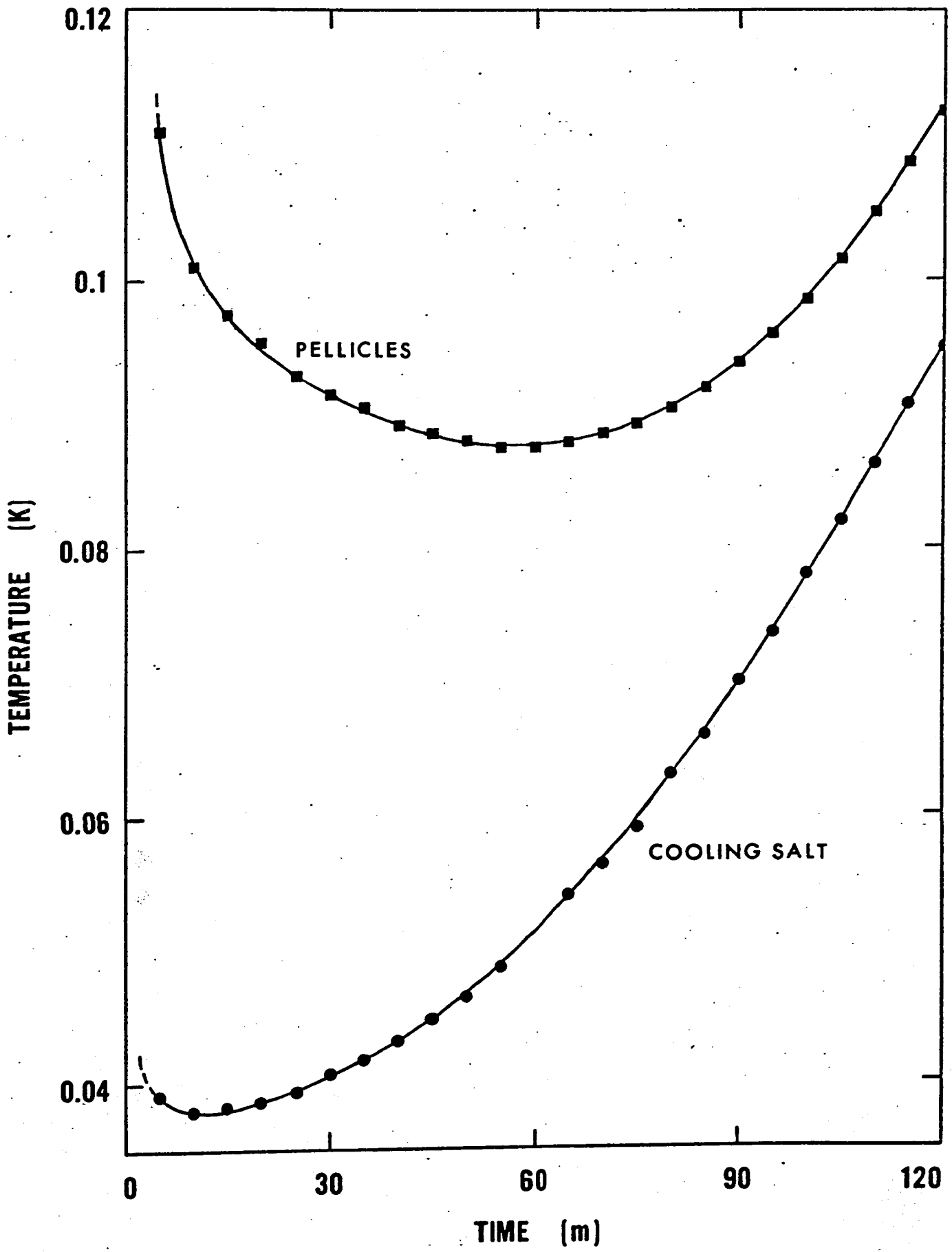


Fig. 9. Temperature curve of pellicles and cooling salt

and both approach the temperature of the pumped helium bath.

It is possible that the salt temperature at minimum is lower than indicated by R2 because of inadequate thermal contact of the resistor. During calibration, the establishing of thermal equilibrium between salt and resistor was evidenced by an abrupt change in slope of the temperature-time curve. (Resistance as a function of magnetic temperature, T^* , was actually measured, but R is also a function of T, and T^* was a function of time during the warming phase.) In the present case, demagnetization lasted 30 minutes and it is believed that the thermometer R2 closely followed the salt temperature. If the cooling of the salt is underestimated, the difference is expected to be slight and is not evident in the warming part of the curve.

The characteristics of R2 could only be obtained to 80 mK during calibration; temperatures below 80 mK were obtained by extrapolating the calibration curve according to published figures (17). The accuracy at 38 mK is considered to be within 15%.

With the H/T_i ratio used in this experiment (20 kG/K), the salt pill should have cooled to about 10 mK (14); however, the fringing field of the lower solenoid, estimated at 230 G at the centre of the salt, and 340 G at the nearest end, prevented complete demagnetization. This residual field may be partially cancelled by connecting the solenoids in opposition and leaving a small current flowing in the upper solenoid.

In spite of the influence of the polarizing solenoid on the

cooling salt, it was still possible to cool the emulsions to 0.1 K for 1 3/4 hours. In the emulsion sensitivity tests, more than adequate statistics were obtained with a two hour irradiation time using a 2 Curie source. It would seem that a single demagnetization is sufficient for interactions involving neutrons from the Pu-Be source. If thermal neutrons are to be used, the increased source-to-emulsion distance required by the paraffin moderator may impose a long irradiation time and the need for more than one demagnetization operation.

5.4 Suggestions for Improvement

The considerations mentioned above should not be thought of as limiting or state-of-the-art, but rather as a point of departure for more refined techniques. The experimental arrangement that was used can be improved in several ways so that the pellicles may be cooled to a lower temperature and for a longer period of time. The difference in temperature between pellicles and salt, for example, may be reduced by installing around the pellicles a thermal shield connected to the salt. Radiation absorbed from the nearest warm surface would then be considerably reduced.

A second improvement would involve the use of thermal switches to provide contact between salt and bath during precooling. Helium is an excellent thermal exchange gas, but it is very difficult to remove from a system at very low temperature. If its

use could be eliminated, a major contribution to the heat flux to the salt and pellicles would also be removed, warm up times would be longer, and the temperature difference between salt and pellicles would be reduced.

Thirdly, a magnetic shield placed above the polarizing solenoid would allow more nearly complete demagnetization of the cooling salt and consequent lower temperature.

A further refinement would incorporate a second paramagnetic salt pill as heat sink for the first. They would be connected by heat switches and demagnetized in sequence. The initial temperature of the main salt would thus be lowered, increasing the ratio H/T_i . Another heat shield could be attached to the second salt which would protect the first (colder) salt and the inner shield.

Keyston et al. (53) describe a system employing some of these improvements which maintains a sample at 20 mK for several hours. At this temperature, 20% polarization of ^{10}B may be obtained in a field of 135 kG. This degree of polarization may be of greater interest for some interactions than may be obtained with the arrangement used and might justify the added complexity of the system.

CHAPTER VI

CONCLUSION

The object of this feasibility study was to show that nuclear emulsion could be cooled to 0.1 K in the presence of a high magnetic field for a period of time long enough to obtain a reasonable irradiation of the emulsion with a convenient source. These conditions may be considered to be the minimum requirements for polarization, by the brute force method, of nuclei imbedded in emulsion.

It has been shown that 400 μ pellicles measuring 2.5 cm x 6.5 cm may be cooled to 88 mK while in a field of 70 kG in the experimental arrangement described. During an experimental run, the temperature of the pellicles remained at 97 mK $\pm 10\%$ for 1 3/4 hours.

If steps are taken to cancel the effect of the fringing field of the polarizing solenoid on the cooling salt, and to reduce the heat leak into the active area of the cryostat, the emulsion will probably cool to a lower temperature and remain there for a longer time. Higher polarizations will thus be possible.

It has also been shown that Ilford G5 nuclear emulsion retains 74 $\pm 2\%$ of its room temperature sensitivity when exposed

at 0.1 K and that temperature cycling has no noticeable effect on the latent image.

It may be concluded that Ilford G5 emulsion is suitable as a detector of ionizing particles resulting from nuclear events taking place at very low temperature and that the nuclei of substances with short spin-lattice relaxation times imbedded in it may be polarized by the brute force method.

REFERENCES

1. Daniels, J.M. and Goldemberg, J. Rep. Prog. Phys 25, 1 (1962)
2. Jeffries, C.D. Ann. Rev. Nucl. Sci. 14, 101 (1964)
3. Wenckebach, W. et al. Physica 50, 289 (1970)
4. Ambler, E. et al. Phil. Mag. 44, 216 (1953)
5. Daniels, J.M. Oriented Nuclei, Polarized Targets and Beams (Academic Press, New York, 1965)
6. Blin-Stoyle, R.J. et al. Prog. Nucl. Phys. 3, 63 (1953)
7. Rose, M.E. Phys. Rev. 75, 213 (1949)
8. Stolovy, A. Phys. Rev. 118, 211 (1960)
9. Wheatley, J.C. Am. J. Phys. 36, 181 (1968)
10. Wheatley, J.C. et al. Physics 4, 1 (1968)
11. de Klerk, D. Handbuch der Physik (S. Flügge, ed.) 15, 38 (Springer, Berlin, 1956)
12. Ancsin, J. and Lamarche, J.L.G. Rev. Sci. Inst. 38, 368 (1967)
13. Baierlein, R. and Bertman, R. Am. J. Phys. 37, 101 (1969)
14. Thoulouze, D. J. Physique 31, C3-119 (1970)
15. Antcliffe, G.A. et al. Rev. Sci. Inst. 39, 254 (1968)
16. Symko, O.G. J. Low Temp. Phys. 1, 451 (1969)
17. Black, W.C. et al. Rev. Sci. Inst. 35, 587 (1964)
18. Edelstein, A.S. and Mess, K.W. Physica 31, 1707 (1965)
19. Rosenbaum, R. Rev. Sci. Inst. 41, 37 (1970)

20. Hudson, R.P. and Kaeser, R.S. *Physics* 3, 95 (1967)
21. Hudson, R.P. *Cryogenics* 9, 76 (1969)
22. Daniels, J.M. and Kürti, N. *Proc. Roy. Soc.* A221, 243 (1954)
23. Yee, R. and Zimmerman, G.O. *Cryogenics* 10, 136 (1970)
24. Hsu, Y. and Lamarche, J.L.G. *J. Low Temp. Phys.* 1, 489 (1969)
25. Harrison, J.P. *Rev. Sci. Inst.* 39, 145 (1968)
26. van Dijk, H. *et al.* The 1958 He⁴ Scale of Temperature, NBS Monograph 10 (1960)
27. Hetzler, M.C. and Walton, D. *Rev. Sci. Inst.* 39, 1656 (1968)
28. Johnson, W.C. and Anderson, A.C. *Rev. Sci. Inst.* 42, 1296 (1971)
29. Neuringer, L.J. and Shapira, Y. *Rev. Sci. Inst.* 40, 1314 (1969)
30. Belanger, B.C. *Rev. Sci. Inst.* 40, 1082 (1969)
31. Gordy, L. and Fritzsche, H. *J. Appl. Phys.* 41, 3546 (1970)
32. Decker, D.L. and Laquer, H.L. *Cryogenics* 9, 461 (1969)
33. Hudson, W.R. *Rev. Sci. Inst.* 39, 253 (1968)
34. Kollias, N. *et al.* *Cryogenics* 11, 61 (1971)
35. Saito, S. *et al.* *Suppl. Bull. Int. Inst. Refrig. Annexe 2*, 41 (1970)
36. Kaplan, N. and Yagoda, H. *Rev. Sci. Inst.* 23, 155 (1952)
37. Dilworth, C. *et al.* *Fundamental Mechanisms of Photographic Sensitivity*, (Academic Press, New York, 1951).
38. Barkas, W.H. *Nuclear Research Emulsions, vol. I* (Academic Press, New York, 1963)
39. Rotblat, J. *Prog. Nucl. Phys.* (O.R. Frish, ed.) vol. 1 (Pergamon, London, 1952)
40. Gurney, R.W. and Mott, N.F. *Proc. Roy. Soc.* A164, 151 (1938)

41. Avan, L. and Dubois, C. Comptes Rendus 251, 1280 (1960)
42. Debeauvais-Wack, M. Nuovo Cimento 10, 1590 (1953);
Comptes Rendus 251, 76 (1960)
43. CERN Emulsion Group. Fifth Int. Conf. Nucl. Photogr. CERN,
Geneva, 1964
44. Sedrine, T.B. and Weill, R. Fifth Int. Conf. Nucl. Photogr.
CERN Geneva, 1964
45. Waniek, R.W. Bull. Am. Phys. Soc. 1, 219 (1956)
46. Johnson, A.W. et al. Can. J. Phys. 49, 2524 (1971)
47. Barron, W.C. and Wolfendale, A.W. Brit. J. Appl. Phys. 8,
297 (1957)
48. Dabbs, J.W.T. et al. Phys. Rev. 98, 1512 (1955)
49. Marshak, H. Bull. Am. Phys. Soc. II 7, 305 (1962)
50. Shirley, D.A. Ann. Rev. Nucl. Sci. 16, 89 (1966)
51. Danysz, M. and Yekutieli, G. Phil Mag. 42, 1185 (1951)
52. Filion, A. M.Sc. Thesis, University of Ottawa, 1969
53. Keyston, J. et al. Cryogenics, 8, 295 (1968)