

# LIQUID He<sub>3</sub>, LIQUID MIXTURES OF He<sub>3</sub> AND He<sub>4</sub>, AND DILUTION REFRIGERATION

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## LIQUID He<sup>3</sup>, LIQUID MIXTURES OF He<sup>3</sup> AND He<sup>4</sup>, AND DILUTION REFRIGERATION (\*)

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**Abstract.** — Basic properties of pure liquid He<sup>3</sup> and of dilute solutions of He<sup>3</sup> in superfluid He<sup>4</sup> are reviewed as they relate to the design and operation of dilution refrigerators. Brief discussions are given both of the intrinsic limitations of the dilution refrigerator and of instabilities which can degrade performance. Finally some characteristics of a new generation of practical dilution refrigerators are displayed and discussed.

**I. Basic Properties of Liquid He<sup>3</sup> and of Dilute Mixtures of He<sup>3</sup> and He<sup>4</sup> as They Relate to the Dilution Refrigerator.** — We have already discussed elsewhere [1] the essence of this section, so we will confine ourselves to a few remarks in review. Pure, or concentrated, liquid He<sup>3</sup> can be regarded as a normal Fermi liquid with sufficient accuracy for the present purposes. That is, we can ignore the very interesting departures [2] at low temperatures of the specific heat from a linear dependence on  $T$ , of the thermal conductivity from a  $T^{-1}$  law, and of the viscosity from a  $T^{-2}$  law. Dilute solutions of He<sup>3</sup> in superfluid He<sup>4</sup> have thermal properties very similar to those of the ideal Fermi-Dirac gas. Transport phenomena in the dilute solutions at temperatures below 10 to 20 m °K are also rather like those in a dilute Fermi gas. They may be deduced from an effective interaction [3] dependent on momentum transfer in a mutual collision of quasiparticles and attractive for low values of momentum transfer. Above about 10 m °K the contribution of He<sup>4</sup> phonons to the thermal conduction in dilute solutions is already important. By 100 m °K He<sup>4</sup> phonons, scattered by He<sup>3</sup> quasiparticles and by boundaries, carry essentially all the heat [4]. There are no measurements of capillary viscosity at low temperatures in dilute solutions. At low temperatures the capillary viscosity has been estimated by Roach [5]. Above 100 m °K we can use the effective viscosity measured by Webeler and Allen [6] to estimate viscosity as a function of temperature and concentration.

In addition to the properties individually of concentrated He<sup>3</sup> and dilute solutions, the solubility of He<sup>3</sup> in He<sup>4</sup>, and vice versa, is of great importance. A number of measurements have been made, particularly by Edwards and collaborators, [7] and these have been reviewed in reference 2. The principal qualitative

point is that at  $T = 0$  a single He<sup>3</sup> atom is more strongly bound to liquid He<sup>4</sup> than it is to liquid He<sup>3</sup>, so that even at  $T = 0$  He<sup>3</sup> will dissolve in He<sup>4</sup>. The solubility is limited, however, by the Fermion nature of He<sup>3</sup> quasiparticles in superfluid He<sup>4</sup> which requires additional He<sup>3</sup> atoms to enter the liquid in higher translational energy states. Similarly, a single He<sup>4</sup> atom is also more strongly bound to liquid He<sup>4</sup> than to liquid He<sup>3</sup> so that as  $T$  approaches zero the concentration of He<sup>4</sup> in He<sup>3</sup> tends rapidly to zero. Certainly below 100 m °K an accurate picture of a mixture of He<sup>3</sup> and He<sup>4</sup> is that, assuming excess He<sup>3</sup>, essentially pure He<sup>3</sup> will float over a dilute solution containing near  $T = 0$  a He<sup>3</sup> molar concentration of about 6.5 %. At higher temperatures the increase in solubility of He<sup>3</sup> in superfluid is proportional to  $T^2$ .

In the most common type of dilution refrigerator, the refrigeration is produced by dilution of He<sup>3</sup> by the superfluid in the mixing chamber. The refrigeration rate at constant temperature is of the form  $\dot{n}_3 T \Delta s$ , or explicitly,

$$\dot{Q} = \dot{n}_3 (10^3 \text{ K}^{-1} RT^2) f(T). \quad (1)$$

where  $\dot{n}_3$  is the molar rate of solution of He<sup>3</sup>,  $R$  is the gas constant, and  $f(T)$  is a function of temperature which is one in the limit of low temperatures, but which decreases as the temperature increases.

The superfluid properties of He<sup>4</sup> are of great importance to the dilution refrigerator. For the superfluid in equilibrium it is required that  $\mu_4$ , the partial thermodynamic potential for He<sup>4</sup>, be uniform. If this requirement is not met then the superfluid will accelerate, at least until a critical velocity is reached, until  $\mu_4$  is uniform. There are three contributions to  $\mu_4$ , aside from a constant term. These arise from mechanical pressure  $P$ , He<sup>3</sup> impurities dissolved in the superfluid (leading to an *osmotic pressure*  $P_{\text{osmotic}}$  which is a function of He<sup>3</sup> concentration  $x$  and temperature  $T$ )

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and  $\text{He}^4$  excitations (leading to *fountain pressure*  $P_{\text{fountain}}$ ). It is customary to write

$$\mu_4 = \mu_{4,0} + v_4(P - P_{\text{osmotic}} - P_{\text{fountain}}), \quad (2)$$

where  $v_4$  is the partial atomic volume for  $\text{He}^4$  and  $\mu_{4,0}$  is a constant. In the temperature range of interest here the term in  $P_{\text{fountain}}$  is negligible.

Equation (2) has several applications for the dilute solution part of a dilution refrigerator. The first is that the increasing temperature as one proceeds from dilution refrigerator mixing chamber to higher temperature still, or whatever, requires that  $\text{He}^3$  concentration  $x$  decreases to keep  $\mu_4$  constant. It is especially to be noted that in the superfluid in equilibrium  $x$  decreases with increasing  $T$  while the solubility of  $\text{He}^3$  in  $\text{He}^4$  increases with increasing  $T$ . Secondly, motion of  $\text{He}^3$  in the superfluid is caused by true osmotic pressure gradients which are in turn resisted by viscosity in the  $\text{He}^3$  quasiparticle gas to obtain a steady state.

Constancy of  $\mu_4$  in the superfluid requires a definite variation of  $x$  with  $T$  and thus affects importantly the ability of the solution to absorb heat. There is a sort of *osmotic enthalpy*, valid for the dilute solution and depending on the temperature of the mixing chamber, which can be used in heat exchange and similar computations.

We now wish to present the above results in a reasonably compact form. For numerical computations we have used work cited above and in addition the

tables computed by Radebaugh [8]. The numbers given below have a rather uncertain accuracy, but they will serve for qualitative understanding and semiquantitative calculations. Table I gives estimates of the limiting properties of concentrated  $\text{He}^3$  (c) and dilute

TABLE I

*Some limiting properties of  $\text{He}^3$  and saturated solutions of  $\text{He}^3$  in superfluid  $\text{He}^4$  near  $T = 0$ . Here  $\eta$  is viscosity,  $\kappa$  is thermal conductivity,  $u_3$  is internal energy at zero pressure,  $h_a$  is osmotic enthalpy, and  $\Delta s_{\text{eff}}$  is the increase [in entropy of a  $\text{He}^3$  atom on passing from concentrated  $\text{He}^3$  to dilute solution.*

$$\begin{aligned} \eta_c T^2 &= 2 \times 10^{-6} \text{ dyne s K}^2/\text{cm}^2 \\ \kappa_c T &= 33 \text{ erg/s cm} \\ \eta_d T^2 &= 5 \times 10^{-7} \text{ dyne s K}^2/\text{cm}^2 \\ \kappa_d T &= 30 \text{ erg/s cm} \\ u_{3c}/k &= 1.5 \text{ K}^{-1} T^2 \\ h_a/k &= 6.5 \text{ K}^{-1} T^2 \\ T \Delta s_{\text{eff.}}/k &= 10 \text{ K}^{-1} T^2 \\ x &= 0.064(1 + 10.8 \text{ K}^{-2} T^2) \end{aligned}$$

solutions (d) near  $T = 0$ . These are most useful in estimating the behavior of the coldest parts of the refrigerator, and in particular the mixing chamber and contiguous parts. The properties of the dilute solution which is in osmotic equilibrium ( $\mu_4$  constant) with the saturated solution in the mixing chamber are also

TABLE II

*Dependence on  $T$ , for two fixed mixing chamber temperatures, of several physical quantities in a dilute solution in osmotic equilibrium. Here  $C_V$  is effective osmotic heat capacity per unit volume;  $B = d \ln \rho/dT$ , where  $\rho$  is mass density,  $\kappa$  is thermal conductivity and  $\eta$  is viscosity*

$T$ °K	$C_V$ $10^5 \text{ erg/cm}^3 \text{ K}^0$	$B$ $\text{K}^{-1}$	$\kappa$ $10^3 \text{ erg/s cm K}^0$	$\eta$ $10^{-4} \text{ dyne s/cm}^2$
$T_{\text{mixing chamber}} = 10 \text{ m } ^\circ\text{K}$				
.022 5	.56	.055	2.5	10.
.032 5	.82	.079	2.6	4.5
.042 5	1.07	.096	2.8	2.5
.055	1.34	.116	3.3	1.4
.075	1.75	.128	4.2	.70
.095	1.99	.127	5.5	.42
.15	2.03	.098	9.6	.29
.225	1.65	.048	14.5	.26
.325	1.23	.024	18.	.25
.425	.96	.015	23.	.24
.525	.78	.010	28.	.25
$T_{\text{mixing chamber}} = 50 \text{ m } ^\circ\text{K}$				
.055	1.47	.125	2.8	1.6
.075	1.87	.141	3.4	.80
.095	2.11	.138	4.6	.45
.15	2.25	.106	8.0	.30
.225	1.85	.055	12.5	.27
.325	1.39	.028	16.0	.26
.425	1.08	.016	20.	.25
.525	.89	.011	24.	.25

important, in regard particularly to its ability to absorb heat and to the possibility of a convective instability. Some properties relevant to convection are given in Table II for two mixing chamber temperatures. The figures are estimates only. We quote in figure 1 from

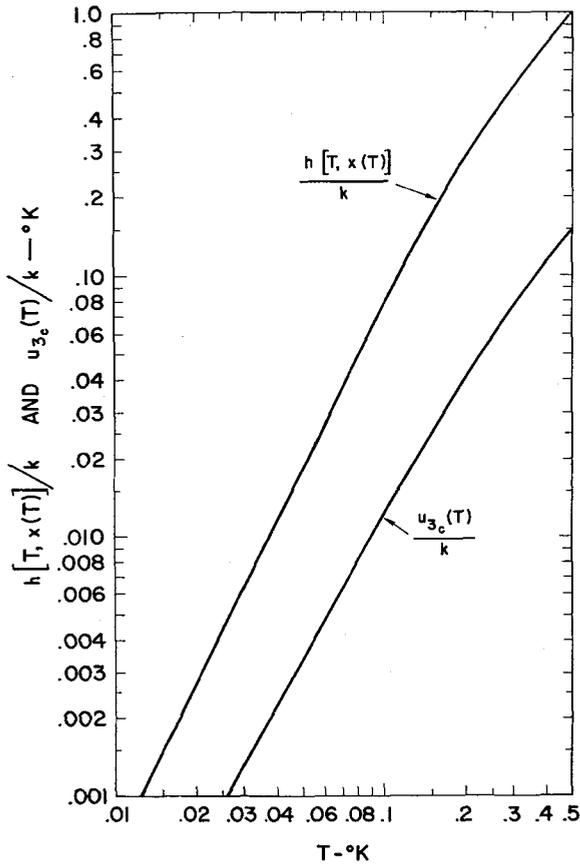


FIG. 1. — Internal energy per atom of concentrated He<sup>3</sup> and osmotic enthalpy per He<sup>3</sup> atom of a dilute solution in equilibrium with saturated solution at  $T = 0$ . Both energies are expressed in degrees by division by Boltzmann's constant.

reference 1 the values calculated there for the internal energy of concentrated He<sup>3</sup> at zero pressure,  $u_{3c}$ , and for the osmotic enthalpy,  $h$ , of a dilute solution in equilibrium with saturated solution at  $T = 0$ . These are useful in discussing heat exchangers. In conjunction with the values of  $T\Delta s$  given in figure 2 and taken from reference 1, the values of  $u_{3c}$  given in figure 1 can be used to relate the temperature of the mixing chamber,  $T_{MC}$ , the temperature,  $T_i$ , of concentrated He<sup>3</sup> entering the mixing chamber from the exchanger, and the rate  $\dot{Q}_{ext}$  at which heat is added externally. The equation is

$$\dot{Q}_{ext} + \dot{n}_3 R[u_{3c}(T_i) - u_{3c}(T_{MC})] = \dot{n}_3 RT \Delta s(T_{MC}). \quad (3)$$

In the case where  $\dot{Q}_{ext}$  is zero the calculated relation between  $T_i$  and  $T_{MC}$  is given in figure 3. Finally, in figure 4, we give a curve from reference 1 showing how the He<sup>3</sup> concentration varies with  $T$  both for a saturated solution (right branch) and for a solution in

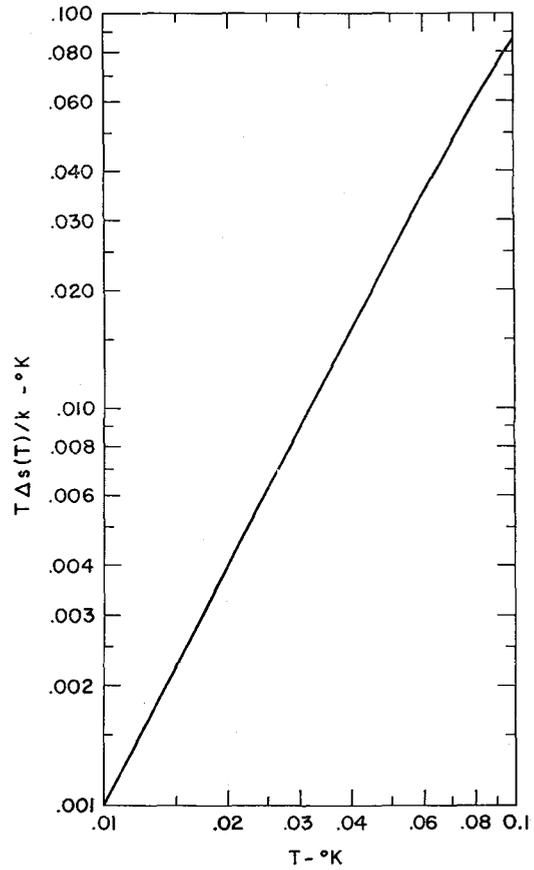


FIG. 2. — Refrigeration capability (expressed in degrees) of a single cycle dilution refrigerator.

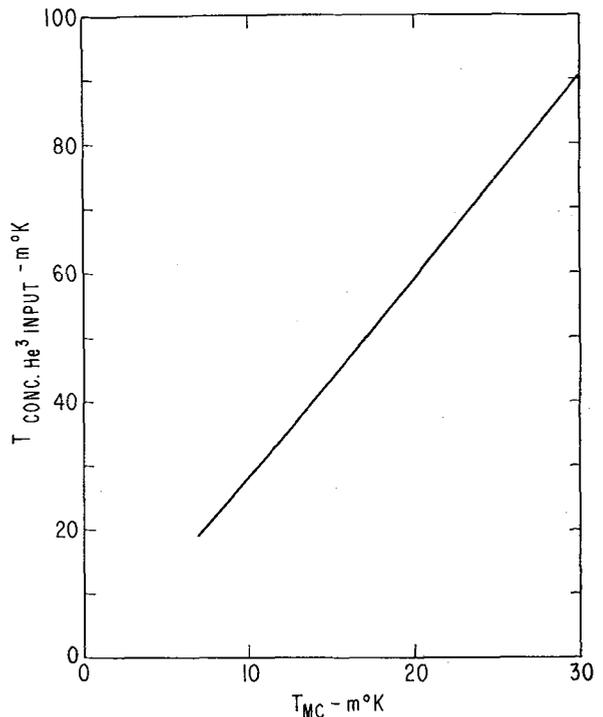


FIG. 3. — Dependence of mixing chamber temperature,  $T_{MC}$ , on the temperature of the concentrated He<sup>3</sup> input to the mixing chamber under conditions of no external heat load.

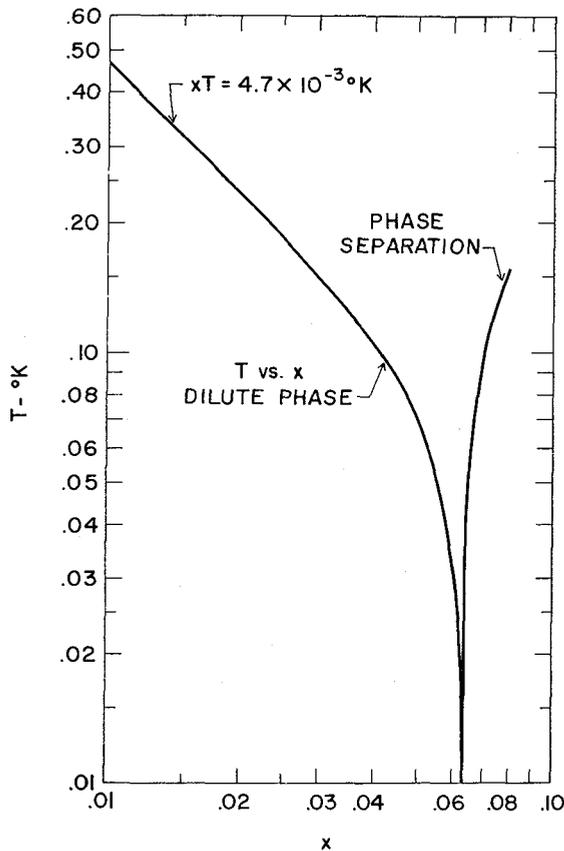


FIG. 4. — Computed values for the dilute solution branch of the phase separation curve near  $T = 0$  and also for the variation of  $x$  with  $T$  in the dilute solution with the mixing chamber at  $T = 0$ .

osmotic equilibrium with saturated solution at  $T = 0$  (left branch). This figure is helpful in attempting to understand possible effects of superfluid on the concentrated side of a dilution refrigerator.

**II. Intrinsic Limitations.** — Details of the factors which limit the low temperature which may be attained with a dilution refrigerator have been discussed in reference 1 and by Varoquaux [9]. No experiments on the limitations of the superleak operated refrigerator have been performed, to our knowledge, but the limiting behavior of a device in which concentrated  $\text{He}^2$  dissolves in  $\text{He}^4$  has been studied qualitatively [10, 11]. The nature of the limitations may be understood at once from simple reasoning based on the properties of Fermi liquids. Consider the simplest case, in which the mixing chamber has no concentrated  $\text{He}^3$  input as such but communicates with the outside only by a tube containing dilute solution. Imagine, further, that there is no extraneous heat leak to the mixing chamber. Now suppose that we are extracting  $\text{He}^3$  from the mixing chamber at some rate via superfluid helium and remember that at very low temperatures not only the viscosity but also the thermal conductivity of the liquid is high. Owing to viscosity ( $T^{-2}$ ) the  $\text{He}^3$  is heated up, by an amount proportional to  $T^{-1}$  for a

given heat dissipation since the osmotic heat capacity varies as  $T$ . A temperature gradient is thereby generated, proportional to the rate of removal of  $\text{He}^3$ , which allows the frictionally generated heat to be conducted ( $T^{-1}$ ) back into the mixing chamber. The ability of the mixing chamber to absorb heat is proportional to the  $\text{He}^3$  flow rate and to  $T^2$ . The result then is that, independent of flow rate, the low temperature which can be reached will be the solution of an equation which depends on  $T^6$ :  $T^2$  from refrigeration capability,  $T^2$  from viscosity,  $T$  from thermal conductivity, and  $T$  from osmotic heat capacity. According to our quantitative estimate [1] the intrinsic temperature

$$T_{\text{MCintrinsic}} = 4 \left( \frac{1 \text{ mm}}{d} \right)^{1/3} \text{ m } ^\circ\text{K}, \quad (4)$$

where  $d$  is the diameter of the tube by which  $\text{He}^3$  is extracted. This temperature is rather close to values which are observed experimentally.

**III. Instabilities.** — Mixing chamber temperatures for the continuously operating dilution refrigerator result from a dynamic equilibrium between heat introduced both by external means and by incoming warm  $\text{He}^3$  and the refrigeration produced when  $\text{He}^3$  dissolves in superfluid. If the equilibrium is disturbed in some way then the mixing chamber temperature will change. In our experience it is frequently the case that, at the lowest temperature, say, the mixing chamber temperature will be quite stable, usually better than a few tenths of a percent over periods of many hours. However, under some conditions and in some refrigerators temperature fluctuations are more violent and can take place on a time scale of seconds or minutes. The performance of the refrigerator is usually degraded when they are present. The problem is important, but complicated. In this section we will identify some of the sources of the instabilities and discuss their characteristics.

The possibility for gravitational convection in the dilute solution was suggested by H. London and discussed by Hall, Ford, and Thompson [12]. Owing to the requirement for osmotic equilibrium the weight density of dilute solution increases as  $T$  increases. Since it is usual and convenient in cryostat design to have the «hot» end up, the resultant configuration will be unstable against convective mixing whenever there is a temperature gradient. To avoid convection it is possible to arrange the parts of a refrigerator so that «hot» is down, but this may be awkward and inconvenient. We have in the past advocated an arrangement of «U» bends in interconnecting tubes so that the helium in part of each tube is gravitationally stable. This will prevent thermal shorts through convective mixing but may not eliminate more subtle effects of a convective eddy connected with interruption of the osmotic pressure gradient driving the  $\text{He}^3$  flow.

If we argue essentially on dimensional grounds then we expect that if a convective instability is to be damped then we should have

$$\frac{C_V BT}{\kappa\eta} \rho_0 g(\cos \theta) a^4 \text{grad ln } T \gtrsim 1. \quad (5)$$

where  $C_V$  is the osmotic heat capacity per unit volume ;  $B = d \ln \rho / dT$ ,  $\rho$  being density

$$\left[ B \simeq -0.53 \left( \frac{\partial x}{\partial T} \right)_{\mu_4} \right];$$

$\rho_0 g$  is the weight density of He<sup>4</sup> ;  $\theta$  is the angle of the tube with the vertical ;  $a$  is a characteristic length perpendicular to the tube axis and to the plane of the eddy ; and the gradient is taken along the tube. Using Table II we have computed the quantity  $C_V BT \rho_0 g / \kappa\eta$  for mixing chamber temperatures of 10 m°K and 50 m°K. We were rather surprised at the results, which shows that the possibility for convection is greatest at temperatures near 0.1 °K and much less great above 0.5 °K and below 50 m°K. There is not a large effect of mixing chamber temperature. It thus appears desirable to be cautious about large tube dimensions down to rather low temperatures.

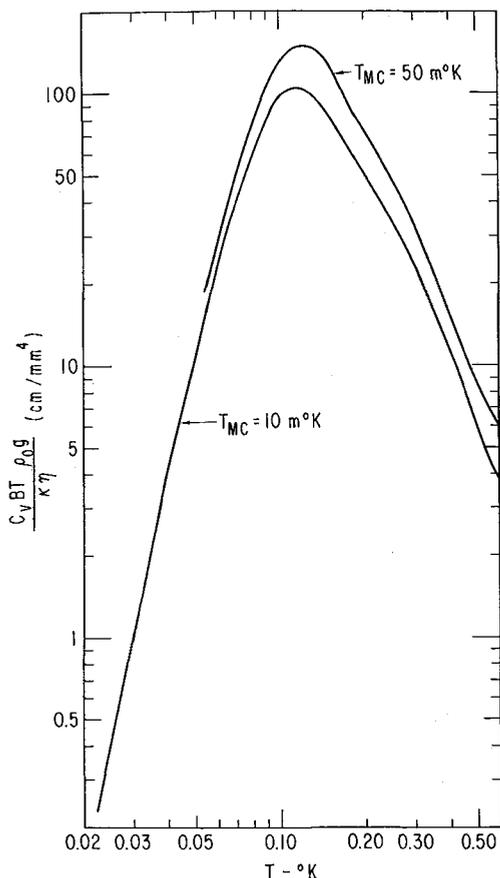


FIG. 5. — Plot of a quantity important for convection (see text) as function of temperature for two mixing chamber temperatures. Owing to a calculational error, all values on the graph are 1.77 times too small.

Another type of instability can take place on the concentrated side of the exchanger at temperatures above 0.4 to 0.5 °K. If the flow impedance factor is too low for concentrated He<sup>3</sup> it is possible for the absolute pressure at, say, the still or below it to fall below the thermodynamic equilibrium vapor pressure. In this case a bubble of vapor can form in an interconnecting tube or in the still exchanger, forcing He<sup>3</sup> temporarily downward, the driving energy coming from heat exchange with the still. It might be possible for a condition to be maintained continuously with vapor in the tube between still and exchanger and with a resultant high heat load of condensation on the exchanger. But if the pressure required to drive vapor through the tube at the steady state molar flow rate is greater than the vapor pressure, then liquid will form again in the still exchanger and interconnecting tube. The whole cycle will then be repeated. It is easy to avoid the above problem by means of a secondary flow restricting impedance which can be inserted very conveniently in a continuous counterflow exchanger of concentric tube type.

A third possible instability can result from superfluid on the concentrated He<sup>3</sup> side of the refrigerator. Superfluid gets into the concentrated side either by being circulated in the main helium stream or by creeping under bulk He<sup>3</sup> [13] up the walls of tubes interconnecting the mixing chamber with the exchangers. The latter effect can be observed readily when a dilution refrigerator operating continuously is made to operate in the *single cycle* mode by closing the valve through which He<sup>3</sup> is recirculated. Circulated He<sup>4</sup> tends to degrade the performance of a dilution refrigerator as a result of the heat load put on the exchanger by the heat of separation which must be absorbed as phase separation occurs. This effect is not a large one below 0.1 °K, however. Another possible effect which can lead to a mechanical instability results directly from properties of the superfluid, is most likely to occur when *step* type heat exchangers are used (temperature changes in steps rather than continuously), and may be understood with the help of figure 4 and eq. (2). Suppose that a plug of superfluid has been formed and is being swept through a step exchanger by the pressure of He<sup>3</sup>. The superfluid is presumably saturated with He<sup>3</sup> at a concentration given by the right branch of figure 4. Now when the superfluid reaches the outlet of the step exchanger it can be pushed as a body through the tube interconnecting it with the next step. At some instant the superfluid will come in contact with concentrated He<sup>3</sup> at a lower temperature and hence will attempt to reduce He<sup>3</sup> concentration. Reference to figure 4, however, shows that whereas the condition for saturation ( $\mu_3$  equal in concentrated and dilute phases) requires  $x$  to decrease, the condition for osmotic equilibrium at zero pressure requires  $x$  to increase. There results a gradient in  $\mu_4$  which makes the superfluid accelerate toward the higher  $T$  step in order

to build up a mechanical pressure gradient large enough to offset the change in  $x$ . These pressures are substantial, especially at higher temperatures, and we can expect some sort of violent effect in the device unless the action of response to the gradient in  $\mu_4$  tends to break up the superfluid. That the effect can be substantial may be seen by inspection of Table III, which

TABLE III

The maximum transient pressure across a non-accelerating column of superfluid connecting saturated solution at a low temperature  $T_L$  and a higher temperature  $T_H$ . Pressures are given in torr, temperature in  $^{\circ}\text{K}$ .

$T_L/T_H$	.05	.07	.10	.15	.20	.30	.40	.50	.60
.03	1	3	6	16	26	58	102	160	231
.05	—	2	5	13	25	57	101	159	230
.07	—	—	4	12	23	55	99	157	228
.10	—	—	—	8	20	52	96	154	225
.15	—	—	—	—	11	44	88	146	217
.20	—	—	—	—	—	32	76	134	205
.30	—	—	—	—	—	—	44	102	173

was computed using Tables in Radebaugh's [8] report. Below 0.1  $^{\circ}\text{K}$  the effect will not be particularly large, though it still may be harmful to some extent. In this connection we note that the best protection, aside from reducing the amount of  $\text{He}^4$  circulated, is to use a continuous counterflow exchanger of concentric tube type between still and step exchangers. The concentrated  $\text{He}^3$  should flow in the central tube, which should be of such small dimensions that phase separating  $\text{He}^4$  tends to form in a fine mist and be swept along in the main helium stream without forming larger units of superfluid.

**IV. Practical Devices.** — By now a large number of successfully operating dilution refrigerators have been built. We cannot within the scope of this paper review all the progress which has been made. What we will do is discuss some of the characteristics of a new dilution refrigerator built and operated at the University of California at San Diego by the author, together with R. E. Rapp and R. T. Johnson. Full details of the work will be published elsewhere.

Our new refrigerator combines a number of desirable features into a relatively simple and compact device which has a high performance. The components of the device are a still of the  $\text{He}^4$  film flow suppressing type [14], a continuous counterflow heat exchanger of the concentric tube type and containing the secondary impedance within the hottest part of the central tube, a set of sintered copper foil type step exchangers of compact design and placement, and a mixing chamber of design suitable to the experiment at hand.

In some preliminary experiments to test different types of continuous exchangers we used an arrangement illustrated schematically in figure 6. The film

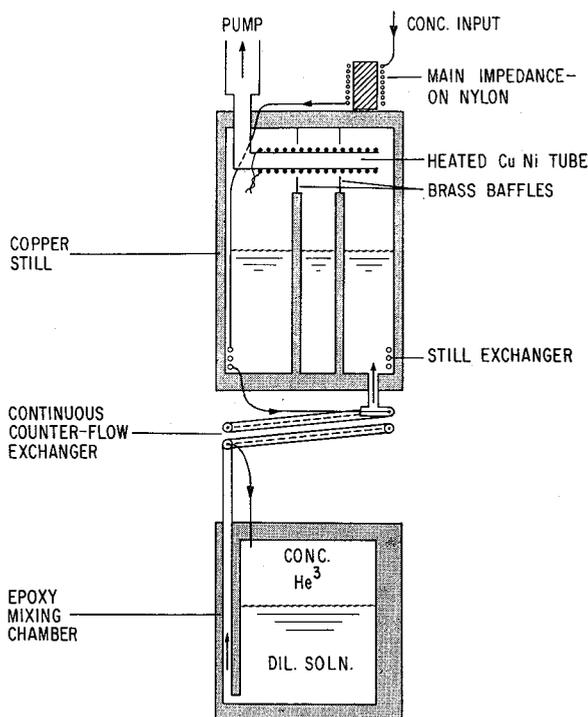


FIG. 6. — Schematic diagram of a simplified dilution refrigerator with  $\text{He}^4$  film suppressing still and concentric tube heat exchanger.

flow suppressing characteristics of the still, which was pumped through a heated 2.4 mm diameter  $\times$  30 mm long Cupro-nickel tube, can be found by examining figure 7. Critical still power for film destruction is

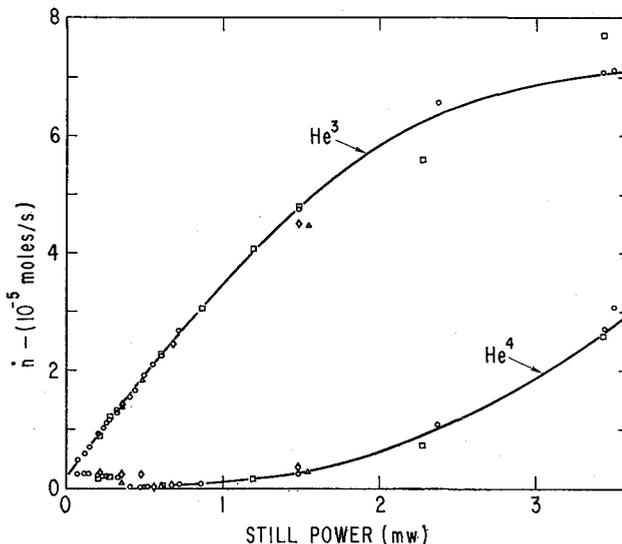


FIG. 7. — Dependence of  $\text{He}^3$  and  $\text{He}^4$  molar flow rates on still power showing the film suppression effect.

around 0.4 mW for this size of pumping tube. We find critical power roughly proportional to tube diameter from other experiments. For the present heated pumping tube the useful maximum He<sup>3</sup> flow rate is 6 to 7 × 10<sup>-5</sup> moles/s. Higher throughput would require a larger heated pumping tube and a concomitantly higher heater power.

We performed a large number of experiments on continuous exchangers. The results can be summarized by remarking that our best results were obtained with the smallest diameter tubes that we tried, the outer tube being 1.04 mm I. D. and the inner tube being 0.25 mm I. D. and 0.41 mm O. D. Better results were obtained with an exchanger using tubes of these dimensions for a length of 1.5 meter than for a length of 1 meter. The lowest M. C. temperature achieved with this configuration was 22 m °K and corresponded to a He<sup>3</sup> flow rate of 10<sup>-5</sup> moles/s. The effect of the additional circulated He<sup>4</sup> on going below critical still power was appreciable, but not profound. Referring to figure 3, for a mixing chamber temperature of 22 m °K the incoming He<sup>3</sup> will be at 65 m °K, so this simple continuous exchanger is rather effective. Attempts to increase surface area for heat exchange by increasing the size of tube met with degraded performance. Further, in the larger tube or plate geometries the mixing chamber temperature rapidly rose once the still power was set below critical.

Although a simple continuous exchanger is inadequate for achieving very low temperatures, particularly at high He<sup>3</sup> molar flow rates, it is entirely suitable for achieving equilibrium temperatures of 0.1 °K or higher and with high refrigeration capacity. This is illustrated in figure 8. Near 0.1 °K the refrigeration capacity can be deduced with sufficient accuracy just from figure 2, the heat input from circulated He<sup>3</sup> being small relative to the external heat input.

To achieve lower temperatures it is necessary to have more adequate heat exchange, as can be arranged with step exchangers. However, we should continue to use the continuous exchanger between still and step exchangers to inhibit instabilities and reduce the total helium required. Such an arrangement was used by Elmholm et al. [15].

For high He<sup>3</sup> throughput some modification needs to be made of the step exchangers that we used in reference 1. The flow impedance factor  $Z (\equiv \Delta p / \eta \dot{V})$ , where  $\Delta p$  is the pressure drop needed to produce volume flow rate  $\dot{V}$  of fluid with viscosity  $\eta$ ) must be reduced from that common amongst our sintered copper powder steps [1] while maintaining the high surface area available with the powder. Since the problem which the steps are to solve occurs at low temperatures we use the low temperature approximation to find formulas suitable for computations. We will assume that the principal thermal resistance is the Kapitza thermal boundary resistance from helium to copper. This will presumably always estimate the resistance even in cases [16] where the He<sup>3</sup> resistance

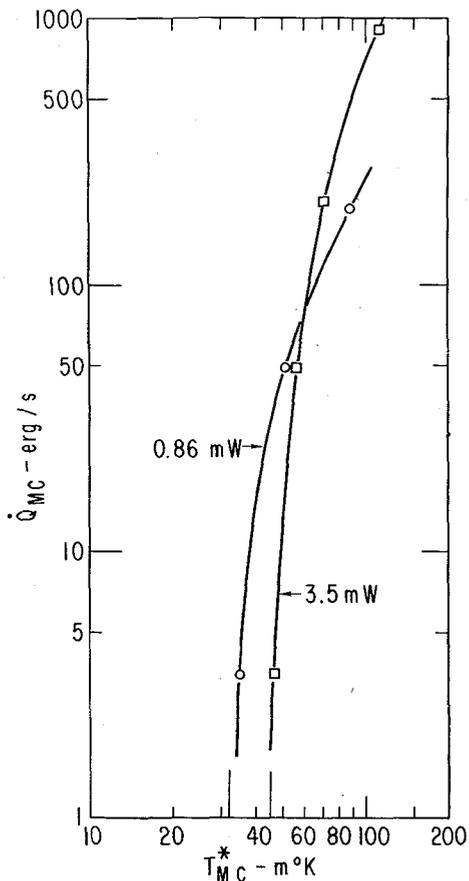


FIG. 8. — Dependence of mixing chamber temperature on external power input to mixing chamber for two still powers. The heat exchanger is a simple concentric tube device one meter long and otherwise described in the text.

is important. Two quantities are of interest. These are  $(\tau/t)$ , the ratio of the thermal relaxation time to the time helium is in an exchanger; and  $(\Delta T/T)$ , the fractional increase in temperature of helium in an exchanger (or whatever), due to viscous heating. For optimum conditions  $(\tau/t)$  should be about one and  $(\Delta T/T) \ll 1$ . Approximate formulas for these quantities are, for concentrated (c) and dilute (d) phases,

$$\left(\frac{\tau}{t}\right)_d = 3 \times 10^3 \left(\frac{\dot{n}_3}{10^{-4} \text{ moles/s}}\right) \times \left(\frac{20 \text{ m } ^\circ\text{K}}{T}\right)^2 \left(\frac{1 \text{ cm}^2}{A}\right), \quad (6a)$$

and

$$\left(\frac{\tau}{t}\right)_c \approx \frac{1}{4} \left(\frac{\tau}{t}\right)_d, \quad (6b)$$

for the same parameters;

$$\left(\frac{\Delta T}{T}\right)_d = 5 \left(\frac{\dot{n}_3}{10^{-4} \text{ moles/s}}\right) \left(\frac{Z}{10^8 \text{ cm}^{-3}}\right) \times \left(\frac{20 \text{ m } ^\circ\text{K}}{T}\right)^4, \quad (7a)$$

and

$$\left(\frac{\Delta T}{T}\right)_c \approx \frac{1}{8} \left(\frac{\Delta T}{T}\right)_d, \quad (7b)$$

for the same parameters.

Examination of figure 3 shows that for mixing chamber temperatures in the range of 8 m °K the incoming helium must be cooled to a temperature near 20 m °K. Hence an impedance factor of order  $10^{-2}$  ( $10^8 \text{ cm}^{-3}$ ) will suffice ; see eq. (7). This can readily be obtained in a copper foil type of heat exchanger such as that used so successfully by Neganov and coworkers [17]. Other types of foil exchangers have been developed by Roubeau and Varoquaux [18]. The greatest problem is surface area, as may be seen from eq. (6). Since it is best not to exceed a certain size of step convenient to one's general mechanical layout, it seems clear that for achieving the lowest temperatures a large number of low impedance steps in series will be required.

The geometry of our copper foil exchangers is shown in figure 9. These are made from a double roll

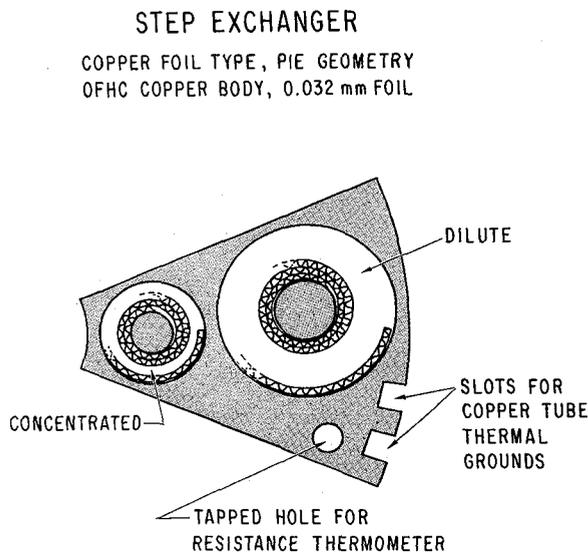


FIG. 9. — Cross-sectional diagram of a typical sintered copper foil type heat exchanger.

of copper foil, one of which is corrugated. The assembly is sintered in hydrogen at 700 °C for one hour. A typical dilute section has a surface area of  $10^3 \text{ cm}^2$ , a helium volume of  $1.7 \text{ cm}^3$ , and an impedance factor of  $0.02$  ( $10^8 \text{ cm}^{-3}$ ) ; while a concentrated section has an area of  $400 \text{ cm}^2$ , a helium volume of  $0.5 \text{ cm}^3$ , and an impedance factor of  $0.1$  ( $10^4 \text{ cm}^{-3}$ ). By using a « pie » geometry we achieve a very compact design and efficient use of space. The general means of assembly of the exchangers to still and mixing chamber is shown in figure 10, where only one step and no interconnections or copper foil are shown. The mechanical support is of *Delrin*. It is thermally grounded at two points, as shown.

The results of detailed experiments using the above device are shown in figure 11, where the no-load mixing

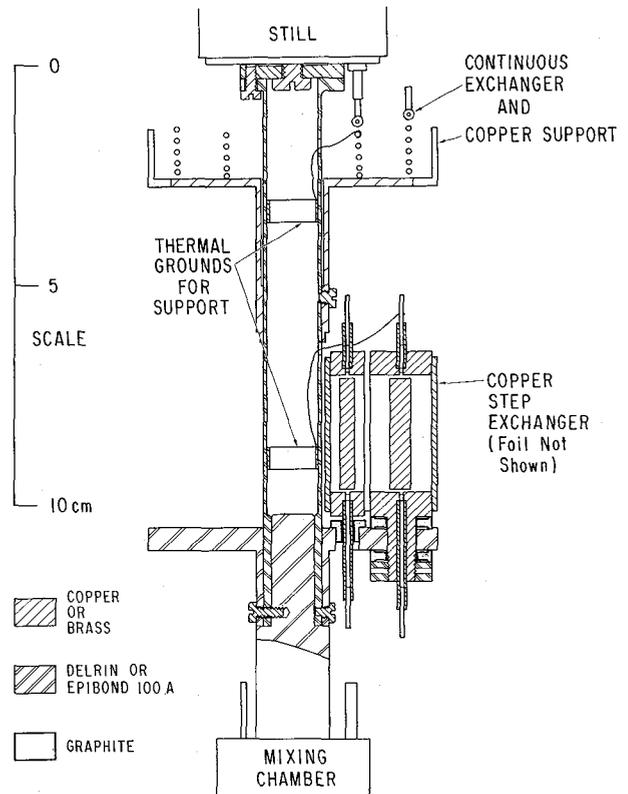


FIG. 10. — Scale drawing of the heat exchanger assembly in the UCSD refrigerator. The central mechanical support and support for the step exchangers are of « Delrin ». No copper foil in the step, only one step, and no interconnections are shown to avoid confusion.

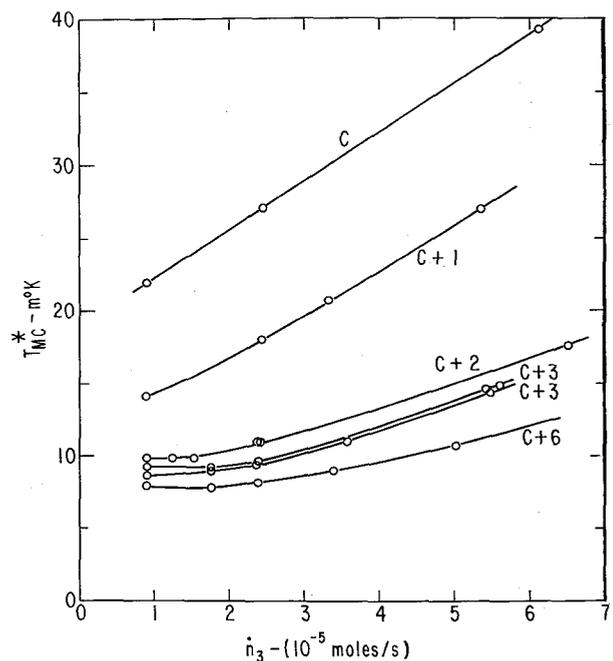


FIG. 11. — Dependence of no-load mixing chamber temperature on He<sup>3</sup> molar flow rate for several heat exchanger configurations. C indicates a continuous exchanger and the number indicates the number of steps.

chamber temperature is represented as a function of the He<sup>3</sup> molar flow rate for various exchanger configurations. In the figure, C refers to the continuous counterflow exchanger, and the number refers to the number of step exchangers used with the continuous exchanger. The two curves labeled C + 3 were obtained for two different mixing chambers. Just the continuous exchanger is quite effective for higher temperatures. It is quite likely that for very heavy heat load applications at higher temperatures a single continuous exchanger will suffice. Addition of a single step exchanger has a very profound effect. Addition of more exchangers brings down the high more than the low flow rate end. It is interesting that to lower the mixing chamber temperature at low flow rates from 10 to 9 m°K required a change from two to three steps, while a further decrease of 1 m°K to 8 m°K required three more for a total of six steps.

Regarding heat absorption capability one may say that at low temperatures the mixing chamber temperature will begin at no load with the temperatures indicated in figure 12 and then increase from there in such a way that the difference in performance for different numbers of steps gradually disappears. This emphasizes the point that no more steps need be added than are required to do the job at hand.

We wish to conclude this section by making some comments on the mixing chamber. It can be designed with great flexibility. It is important to remember that a unique characteristic of a dilution refrigerator is that the cold dilute solution in the mixing chamber can be moved around as required by the experiment. To illustrate this point consider figure 12, which shows a mixing chamber which [19] we are using to make measurements of weakly magnetic phenomena at very low temperatures. In this device the refrigeration is produced in the upper chamber where the phase boundary is caused to be located. It was necessary to reduce interaction between 17 Hz CMN susceptibility measurements and static measurements of, say, nuclear magnetism in copper, using a superconducting device sensitive to  $\varphi_0 = h/2e$ . We therefore physically separated the two measurement functions as

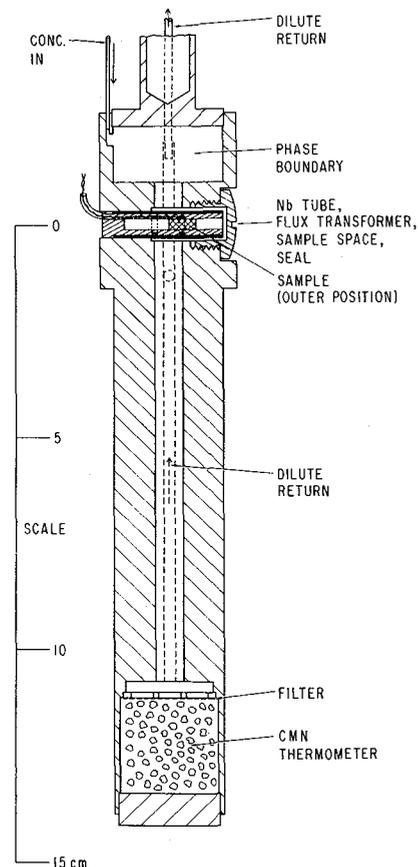


FIG. 12. — Epoxy mixing chamber for a system which allows simultaneous measurement of the 17 Hz susceptibility of CMN and the static magnetization of weakly magnetic materials.

shown and swept the dilute solution first by the weakly magnetic sample, then down a long and relatively small tube, by the CMN, and finally up a small diameter tube in the wall of the epoxy chamber. Another important technical feature of this mixing chamber is the port giving access to the upper measuring system. This screwed joint can be opened to insert samples and then sealed again [20]. Such easy access to the mixing chamber gives great flexibility and should be quite important in future applications of dilution refrigerators.

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- [19] HIRSCHKOFF (E. C.), SYMKO (O. G.), and WHEATLEY, (J. C.), to be published in the *Journal of Low Temp. Phys.*
- [20] The sealing compound was developed by Dr. MOTA (A. C.) and Dr. BLACK (W. C.) and consists of 30 gm of glycerine to 8 gm of « Ivory Flakes ». It is a translucent jelly at room temperature, but is warmed for application.