LARGE DILUTION REFRIGERATORS

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We describe the design, construction, and test results of two large dilution refrigerators, capable of circulating 50 and 350 mmol/s of $^3$He, and of absorbing 0.25 and 2.0 W of power in the vicinity of 0.5 K. Both refrigerators are horizontal and have direct horizontal access to the mixing chamber. They feature sintered-copper continuous heat-exchangers, large stills, and thin-wall mylar mixing chambers with useful volumes of 200 and 4000 cm$^3$, respectively. The refrigerators are primarily intended for cooling large polarized targets. A brief review is given of their possible use in other applications, such as stabilization of atomic hydrogen and cooling of intense cold sources of polarized atomic hydrogen, cooling of massive detectors of gravity waves or neutrinos, and cryo-pumping of helium and other light gases.

DESIGN PRINCIPLES

The two dilution refrigerators described in this paper were constructed for cooling large polarized targets. In their design, in addition to heat transfer and flow friction, two important points were stressed: i) rapid cold access to the mixing chamber, and ii) fast evacuation and cool-down of the set-up. Not much attention was paid to reducing the residual heat leaks to the mixing chamber, because the refrigerators were not intended for frozen spin operation at low field.

Figure 1 shows schematically the design of the two dilution refrigerators proper. The rapid-sealing indium joint [1] allows direct horizontal access of the mixing chamber by introducing a special target holder into the access tube. The holder seals the access tube at the height of the still; about 0.7 kN/cm$^2$ force per unit length of wire is required for a superleak-tight joint. The holder closes the access tube with a rubber O-ring at room temperature.

A single stainless-steel envelope contains the still and heat-exchanger, which are built coaxially around the access tube. The mylar extension of this envelope forms the mixing chamber.

The $^3$He is expanded in a cold needle valve before entering the still heat-exchanger. The temperature is 2.0–3.0 K before expansion and a few tenths of a degree lower after. Owing to the rather high $^4$He contamination (15%–40% depending on the temperature of the mixing chamber and on the $^3$He flow), it is likely that a $^4$He-rich liquid phase is formed before expansion, which would explain the cooling that occurs in the expansion to about 0.1 atm pressure.

About half of the boiling heat comes from the recondensing $^3$He in the still heat-exchanger; the other half is supplied by an electric heater. In the smaller refrigerator the heater is made of 0.5 mm Manganin resistance wire, which is wound to a helix of 3 mm inner diameter and laid in horizontal lengths around the access tube. The still heat-exchanger tube is then wound around this heater. In the larger refrigerator the tubular heat-exchanger is wound around the access tube, and the heater on it consists of annuli of thin stainless-steel foils connected electrically in series. In both cases care was taken to ensure good convection and free bubble rise
in the dilute solution. In the calculation of the boiling surfaces we assume the 

correlation \( q/A = 4.0 \times 10^6 \text{ W/m}^2 \text{K} \), which has been deduced from the 
test results of an earlier refrigerator [2] and which has been found to give satis-
factory results in larger ones as well [3]. No significant difference was observed 
in the two different designs. The cold needle valve was found to give a sensitive con-
trol of the \(^3\)He evaporation rate in the still. Degradation of the cooling perfor-
mance became visible, particularly in the lower temperature range, if more than half 
of the evaporation heat in the still came from the still heat-exchanger. This indi-
cates how critical the still heat-exchanger may be in the high-power dilution re-
frigerators.

The main sintered heat-exchanger was constructed of 70/30 CuNi preformed foils, on 
which a layer of 0.7 mm of 325 Mesh (18 \( \mu \text{m} \) surface-to-volume diameter) Cu powder was 
sintered. The sintered halves of the exchanger were bent before being welded to-
gether. The turns of the resulting helix were separated from each other by a plastic 
tube in the smaller refrigerator, and by machined glass-fibre epoxy rings in the 
larger one. In tests without the separating plastic tube, the smaller refrigerator 
showed increased instability in the lowest temperature range; we tend to explain this 
by convectional heat transfer in the dilute stream of the exchanger.

The \(^3\)He trap covering the target in the mixing chamber makes an integral part of the 
target holder. The outlet holes on the \(^4\)He tubes eject the liquid so that concen-
trated droplets are allowed to penetrate through the target before being collected 
into the trap.

The design of the access tube is perhaps the most novel and critical part of the pre-
sent refrigerators. In our polarized targets the beam access to the target is along 
the axis of the refrigerator. The target holder in the access tube has thin beam 
windows of stainless steel on both end-plates; also the thermal radiation screens 
on the five thermal contact rings have each a thin central window. The contact rings 
are spaced by semi-rigid coaxial cables made of beryllium copper. In the larger re-
frigerator these rings are made of titanium, which has a lower thermal dilatation 
than the stainless-steel jacket of the holder. In the smaller refrigerator they are 
made of copper and have expansion screws which allow the rings to be tightened once 
they are inserted into the holder jacket.

The target holder in the access tube may also carry a waveguide(s) for dynamic nuclear 
polarization, and a teflon tube can be mounted in the holder to bring atomic hydrogen 
directly to the mixing chamber.

\(^3\)He CIRCULATION AND CONDENSATION

The following discussion is based on the simplified diagrams of Figs. 2 and 3, which 
show the \(^3\)He and \(^4\)He circuits of the two refrigerators. They have in common that 
there is no pumped \(^4\)He bath condenser near 1 K temperature, but there is a heat ex-
changer between the low-pressure still vapour and the incoming high-pressure \(^3\)He. 
It was foreseen to run the smaller refrigerator using small transportable \(^4\)He dewars. 
This is why an attempt was made to recover the enthalpy of the low-pressure \(^3\)He also 
above 4 K in a special three-flow heat exchanger. The large refrigerator is supplied 
from a cold box with sufficient capacity; here, no attempt was made to recover the 
enthalpy of the low-pressure \(^4\)He above 4 K.

Precooling of the smaller refrigerator is done directly with \(^4\)He using a cold pre-
cooling valve. It is clear that this valve must be superleak-tight, otherwise there 
might be an undesirable increase in the quantity of \(^4\)He in the closed \(^3\)He/\(^4\)He circuit. 
This precooling valve is especially practical when calibrating the NMR signals for 
the measurement of polarization, because pure \(^4\)He makes it possible to obtain a 
stable and uniform temperature in the mixing chamber. In the larger refrigerator the 
precooling is done through a cold \(^3\)He valve. In the former case, precooling from 
300 to 1 K takes about one hour and in the latter about three hours. The larger 
refrigerator is equipped with a \(^4\)He separator and an evaporator whose purpose is to 
make the flow of \(^4\)He in the 300-4 K counter-current exchanger more easy to control.
These were not installed in the smaller refrigerator owing to lack of space. In both cases the $^3$He is pumped with membrane compressors at 0.3-0.5 atm pressure.

Owing to this relatively high pressure (and temperature) of the $^3$He, the condensation pressure of the $^3$He varies from 1.0 to 3.5 atm. The $^3$He pumps of the smaller refrigerator consist of a cascade of five sealed Root's blowers (speed 3000 m$^3$/h) followed by a membrane compressor. The larger refrigerator has a cascade of eight sealed Root's blowers, which have a measured speed of 13'000 m$^3$/h and which can exhaust at 4 atm pressure.

The position of the $^3$He expansion valve before the still heat-exchanger was arrived at by trial and error. The valve was initially placed in the middle of the still heat-exchanger; this gave a significantly worse cooling performance than the present position.

TEST RESULTS

The maximum cooling power and the corresponding optimum helium circulation is given in Fig. 4 for the two refrigerators. The present configurations do not allow lower ultimate temperatures to be reached, which excludes testing the design asymptotic cooling powers [3] below 50 mK. From the data we estimate that the heat leaks to the mixing chambers, in both cases, are about 10 mW, which is believed to come from the access tubes. Thermal radiation from the surrounding 4 K microwave cavity in the large refrigerator contributes about 1 mW; in the smaller one the thermal radiation is insignificant. Single-shot tests indicate that axial conduction in the main heat exchanger is not the cause of the large heat leak.

The minimum continuous helium flow-rate in the large refrigerator is 20 mmol/s, which corresponds to about 0.6 W heat leak through the rather solid support structures connecting the still to the back plate of the refrigerator. In the smaller refrigerator the supporting central tube is much better thermalized by the still vapour; as a result, the $^3$He flow can be reduced to a few millimoles per second.

The rapid direct access to the mixing chamber has operated reliably in numerous test runs. We expect that this loading system will allow quick target material changes in case of radiation damage. In a material test laboratory the smaller refrigerator would allow several cooling cycles, from room temperature to below 0.5 K, in one day, if two or more target holders were used. Because the holder carries the test apparatus or experiment, change-over from one experiment to another would take no more than a few hours.

CONCLUSIONS

The test results show that it is feasible to construct dilution refrigerators with flow rates approaching 1 mol/s. Although at present the flow seems to be limited by the pump performance rather than by any intrinsic property of the helium isotope mixtures, it would be highly desirable to perform measurements of the boiling correlations in the dilute mixtures in the range where the still is usually operated. Another critical parameter is the osmotic pressure in the dilute stream of the main heat-exchanger. At present, better physical insight would be required in order to understand the isotopic mass diffusion, critical velocity, and the couplings between the first, second, and third sound in the dilute stream [4].

The refrigerators were designed for polarized targets in high-energy physics experiments; several successful dynamic polarization tests have been done in the larger one. Other possible applications are experiments which require absorption of a large amount of heat below 0.5 K, e.g. experiments with spin-polarized atomic hydrogen. The last results on spin relaxation and recombination [5] seem to indicate that higher densities could be achieved if a larger recombination heat load could be tolerated before depletion of the mixed state. One could also speculate on the possibi-
ility of making a pulsed source of polarized hydrogen atoms by extracting one of the stored states by means of a microwave pulse, which is strong enough to flip a majority of the spins. The resulting, rather monochromatic, atomic beam could possibly be handled in much the same way as a charged particle beam by applying quadrupole magnets for bending, hexapoles for focusing, and oscillating magnetic fields for accelerating, decelerating, and bunching.

The possible future, directionally sensitive, neutrino detectors [6] require large amounts of nuclear spin-polarized material; it is clear that the present technique should be pushed towards lower temperatures if the "brute force" method of polarization is going to be applied. Large masses tend to have large residual heat leaks, which are best absorbed at intermediate temperatures. The removal of the Zeeman energy of the nuclear spins also requires a substantial cooling power, if reasonable cooling speeds are expected.

The future gravitational-wave antennas may require cooling to lower temperatures than 1 K following improvements in the electronics noise temperature. Although again it is clear that the best way of achieving a low temperature in the bar material is to reduce the residual heat leaks, it is also evident that the refrigerator should allow a fast cooling speed already for a good cycle time in the system debugging.

In the runs of our lower-temperature dilution refrigerators, which do not have a direct access tube, we have systematically examined the behaviour of the residual heat leak to the mixing chambers. We believe that the time-dependent part of the heat leak, which is of order 20 nW/cm² in the beginning, is due to the relaxation of light residual gases in the surroundings of the mixing chamber. This suggests that dilution refrigerators could be used as helium cryopumps. These are usually heavily exposed to thermal radiation, which requires large cooling powers. Although at the moment we do not have applications for such cryopumps other than for the pumping of the isolating vacuum itself, the use of such devices in the fabrication of ultrapure materials, for example, could be envisaged.

REFERENCES

4 de Waele, A.Th.M., private communication; see also Refs. 2 and 3.
Fig. 1: Schematic drawing of the dilution refrigerators with direct horizontal access to the mixing chamber.

1/4 W DILUTION REFRIGERATOR: FLOW DIAGRAM

Fig. 2 Flow diagram of the smaller dilution refrigerator.
Fig. 3 Flow diagram of the large dilution refrigerator

Fig. 4 Cooling power and optimum helium flow in the two refrigerators