Fizika Nizkikh Temperatur, 1997, v. 23, Nos. 5/6, c. 655–658

Ultra low temperatures in a helium transport vessel: a dilution refrigerator to achieve 30 mK by continuous adsorption pumping

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Submitted November 18, 1996

A novel design of dilution refrigerator (DR) is described for continuous operation inside a transport Dewar with a neck diameter more than 51 mm. The time required for cooling the mixing chamber from room temperature to 30 mK is 3.5–4.5 h. Warming up to 300 K reguires 0.5–1.0 h The cooling power achieved is 40 μW at 100 mK. The DR is fitted with 6 mm line of sight access down to the mixing chamber. A distinguishing feature of this refrigerator is the absence of a traditional gas handling system, which makes it easy to transport in a small car and quickly commission in a new laboratory within a few hours.

PACS: 07.20.Mc

Many refrigerators have been designed in the last 30 years which use the process of dilution of ^{3}He in ^{4}He to obtain ultra low temperatures. The majority of them use a room temperature ^{3}He circulation system, where one or more vacuum pumps pump ^{3}He from the still and return exhausted ^{3}He gas to condense in the insert.

A modern DR having a reasonable cooling power, is typically supplied with more than 24 wires to the experimental region, often allows sample rotation and can be fitted with coaxial cables for high frequency signal pick up. All this has made it a very popular tool among physicists (and not only physicists) .

It is worth emphasizing that up to now the majority of DR were made as stationary devices. Figure 1 shows an example block diagram of a typical dilution refrigerator from the popular Kelvinox family produced by Oxford Instruments. The main components of this type of standard DR (as shown in Fig. 1) is the refrigerator itself, a special Dewar, a gas handling system mounted in a frame, gas purification system including a cold trap and one or more vacuum pumps. Some of the pumps are required to be hermetically sealed for ^{3}He pumping. The development of the Intelligent Gas Handling System (IGH on Fig. 1) provided with computer control, makes the life of the experimentalist much easier, because to operate the DR one should only fill the Dewar with liquid ^{4}He and «click» on the cooldown button on the computer screen.

A few years ago the problem arose of how to quickly change the sample and cool it down to the millikelvin temperature range. This was solved by the top-loading design of DR [1] and more recently by the development of dilution refrigerators which are insertable directly into liquid helium (see, for example, [2]).

The further development of the low temperature physics of metals, semiconductors and especially mesoscopics has created an interest in a portable dilution refrigerator with a fast sample turnaround time.

This paper describes a novel dilution refrigerator with a 50 mm diameter vacuum jacket, which allows it to be easily installed into the neck of the most common 100 litre helium transport Dewars in Europe and worldwide.

The general view of the DR is shown in Fig. 2. It is easy to see that it has no traditional gas handling system. The only external connections are to the ^{3}He/^{4}He mixture dump and to the 1 K pot pump

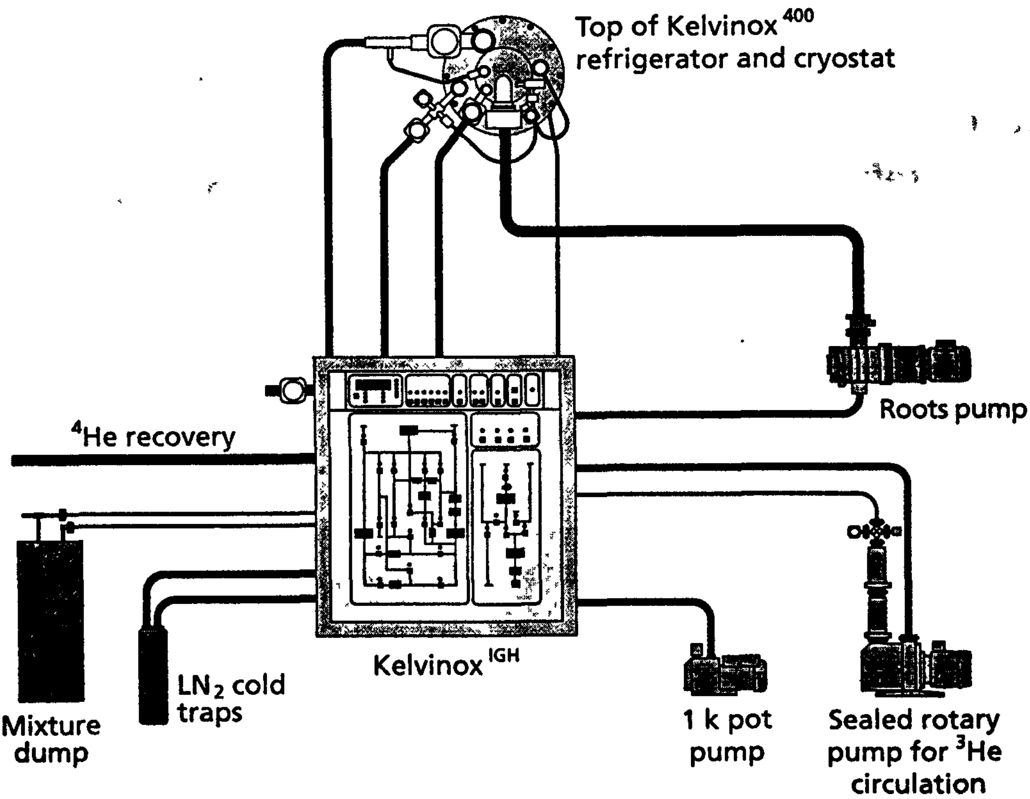

Fig. 1 Block diagram of a typical dilution refrigerator. As an example, the figure shows a Kelvinox 400 DR and its accessories.

by two flexible lines. Only one rotary pump (16 m^3/h) is used to pump the 1 K pot and the sorb heat exchangers.

Special micro-processor controlled electronics have been developed to monitor and control the operation of the refrigerator. Fully automated cool-down is possible, without the need for a computer.

The operation of this DR is based on adsorption pumping by cold activated charcoal, this was fit successfully realized by Boris Naumovich Eselson and his colleagues in the early 1960's for cooling liquid ^{4}He [3] and ^{3}He [4]. It is worth mentioning the growing popularity of this pumping method because of the high pumping speed, purity and absence of vibrations. A number of paper have been published about using this method in the design of dilution refrigerators [5–9].

The DR described here uses a cryogenic circulation cycle with a collector [6],
which was first realized at B. Verkin Institute of Low Temperature Physics and Engineering, Kharkov [7]. Further testing of the cryogenic cycle dilution refrigerator was done in J. Saunders' group at Royal Holloway University of London (Great Britain), this proved the feasibility of this method down to below 10 mK [10]. This success convinced us to design a novel and very compact DR on this basis.

All existing similar DR are fitted with external, gas handling systems (see, for example, [2,7–10]). They also need large electronic systems for automatic control and for stable operation. It was necessary to reconsider a lot of the details of the cryogenic cycle in order to minimize the mass and size of the components, with the aim to arrange all the essential parts of the DR in the cold zone and avoid a standard gas handling system.

The design consists of a vacuum jacket with approximately 47 mm internal diameter, all the elements of the refrigerator are situated inside it, including the 1 K pot with collector, sorbs, still and mixing chamber (see Fig. 2). There is also a 6 mm line of sight access to the mixing chamber for experimental services.

The refrigerator is fitted with a sliding seal to allow the insert to be loaded safely into the Dewar. The total length of the DR from sliding seal down to the bottom of vacuum jacket is 1600 mm but it

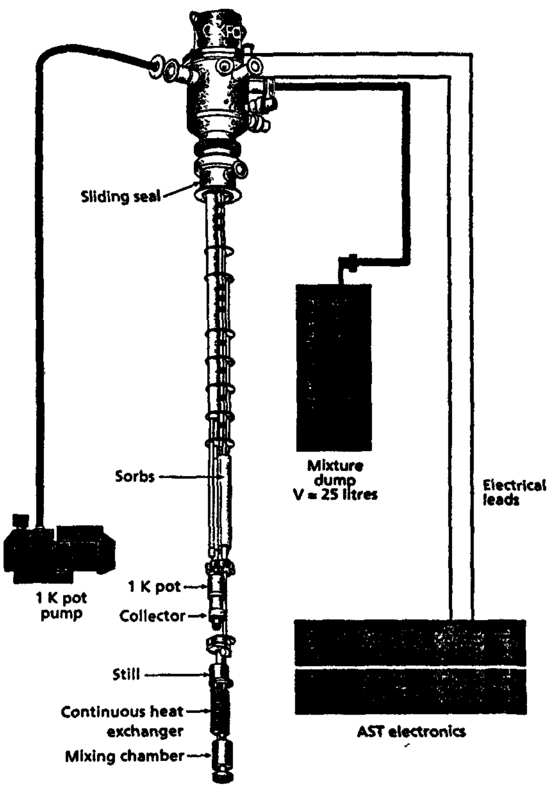

Fig. 2. General view of the new 50 mm dilution refrigerator, for transport Dewar.

can easily be reduced to 1200 mm, which corresponds to the length of a typical transport Dewar.

To achieve more space in the vacuum jacket, the liquid ^{4}He supply to the 1 K pot and sorb heat exchangers enters through a tube at the bottom of the vacuum jacket with a filter and PTFE seal. This design allows us to avoid a standard cold flange with indium seal at 4.2 K and make a simple room temperature clamp connection of the vacuum jacket at the top of the insert.

Development of the cryogenic cycle and the use of electronics for control, helped us to simplify the gas handling system and to eliminate almost all the valves and manometers. As a result, to warm up the refrigerator one only needs to pull the insert out from the Dewar over approximately 30 min. The relief valves ensure that the expanding mixture is safely directed into the storage dump. To prepare the refrigerator for operation after loading it into the Dewar, it is only necessary to open fully two relief valves and close them again when the mixture is sufficiently adsorbed from the dump into insert (by observing a pressure gauge on the mixture storage dump). Refrigerator operation can then be

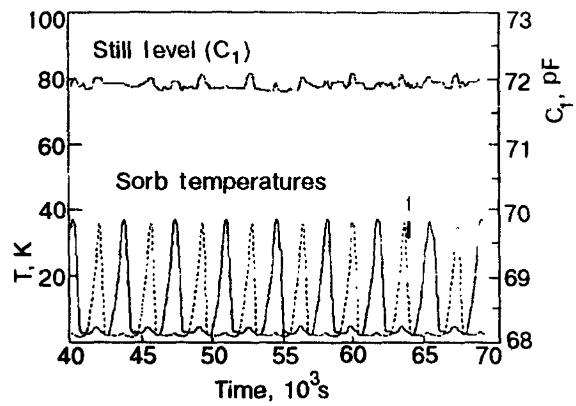

Fig. 3. An example of the diagnostic data recorded with the dilution refrigerator in continuous mode. The cycle time is approximately 30 min. The lower curves show the time evolution of sorb temperatures during the cycles. The upper curve shows the primary capacitance C_1 data of the capacitance level gauge for the level of liquid in the still.

started by simply pressing a button on the electronics box. The electronics can control and monitor all of the key operating systems of the refrigerator.

During operation more than 10 parameters of DR can be recorded simultaneously, such as the temperatures of the sorbs and the liquid levels in the collector and still, using capacitance level gauges. Figure 3 shows typical diagnostic records for 3 parameters of DR — the temperatures of the two sorbs and the level of liquid in the still. This picture has been taken at an early stage of the test. As can be seen the level in still was variable during the cycles, wich was caused by some instability of 1 K pot operation at that time.

One of the remarkable features of the sorbtion pumped DR equipped with a collector is the ability to produce «very long» single shot temperatures down to 10 mK without the use of silver step heat exchangers [10]. Figure 4 shows the capacitance data of a ^{3}He melting curve thermometer, fitted on the mixing chamber base during a cooldown and single shot. On part AB of the curve, one can see ordinary continuous cooling at a flow of approximately 70 μmole/s from 250 mK down to 30 mK, in less than 1 h (B point).

Between points B and C, all the ^{3}He was collected in the mixing chamber, which slightly increased the temperature up to 45 mK. Further cooling to 18 mK was achieved by pumping the still with both sorbs and the flow rate between points D and E was 13 μmole/s. The ^{3}He was all used at point E, when the refrigerator quickly warmed. Experimentally it takes 1 to 1.5 h to return to continuous operation from single shot mode.

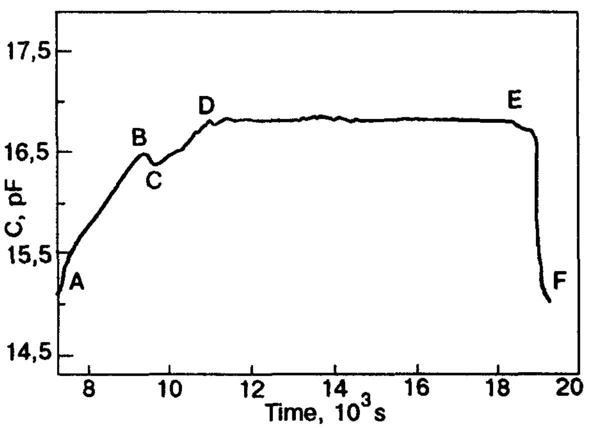

Fig. 4. The primary capacitance C data of the ^{3}He melting curve thermometer, showing the typical cooling of the mixing chamber during single shot operation. The temperature ranges from 250 mK (point A) to 18 mK (line DE).

It is worth noticing that the given result is quite similar to that described earlier in [10], but was achieved with a much smaller mixing chamber and total mixture volume. This can be very useful for quick tests of samples with short measurement times down to below 15 mK. One can estimate the residual heat leak to the mixing chamber from single short results [11]:

$$Q_m = 84 n_3 T_m^2 , \qquad (1)$$

where: Q_m – residual heat leak (W); n_3 – circulation rate (mole/s); T_m – lowest achieved temperature for given circulation rate (K).

The residual heat leak to the mixing chamber of the new DR is about 0.3 μW, from Eq. (1) and the measurements shown in Fig. 4. Because the lowest achievable temperature in single shot mode is a function of the circulation rate, it is clear that the t_h-length of single shots for two different temperatures and ^{3}He flows will be given by ratio

$$(T_1/T_2)^2 = n_3(T_2)/n_3(T_1) = t_h(T_1) t_h(T_2) . \qquad (2)$$

Based on the data from Fig. 4 one can expect to get 14 mK for more then 1 h and 10 mK for about 30 minutes!

During continuous operation, the maximum circulation rate achieved was around 100 μmole/s, with a cooling power of 40 μW at 100 mK. The continuous base temperature was below 30 mK and the still temperature were less than 0.75 K.

The test results of this dilution refrigerator, which has the name «Kelvinox AST Minisorb» allows us to come to these conclusions

– The portable dilution refrigerator which has been developed for operation in a transport Dewar is convenient for a number of experiments, it has a 6 mm line of sight access port down to mixing chamber. An experimental space 30 mm diameter and 80 mm long is provided. 24 constantan wires for electrical measurements are fitted (with a similar quantity for the refrigerator diagnostics) and a few coaxial cables can be fitted for high frequency measurements.

– The base temperature for continuous operation is below 30 mK. Cooling power at 100 mK exceeds 40 μW.

– The use of sorbtion pumping strongly increases the cooling power normally achieved with such a dilution unit, reduces the vibration levels on the insert and also completely eliminates the possibility of blockages.

– The single shot operation demonstrates the ability of the DR to maintain temperatures below 20 mK for a reasonably long time.

– The dilution refrigerator and mixture dump can be easily demounted with help of portable cryopump. As a result this type of dilution refrigerator can be accommodated inside the boot of a small car and transported to any other Cryogenic Laboratory. The commissioning of the refrigerator only takes a few hours, so one can quickly start to do research in a new location.

The authors are very grateful to Alvin Adams and Terry Janaway for their incredible efforts and skills during the construction and testing of this refrigerator.

1. P. H. P. Reinders, M. Springford, P. Hiltton, N. Kerley, and N. Killoran, *Cryogenics* **27**, 689 (1987).
2. J. P. Pekola and J. P. Kalippinen, *Cryogenics* **34**, 843 (1994).
3. B. N. Eselson, B. G. Lazarev, and A. D. Shvets, *Prib. Tekh. Eksp. [Instrum. Exp. Tech. (USSR)]* No. 5, 160 (1961).
4. B. N. Eselson, A. D. Shvets, and N. G. Bereznyak, *Prib. Tekh. Eksp. [Instrum. Exp. Tech. (USSR)]* No. 6, 123 (1961).
5. V. S. Edel'man, *Cryogenics* **12**, 385 (1972).
6. V. A. Mikheev, *Author Certificate of Invention* N1103055, USSR (1984).
7. V. A. Mikheev, V. A. Maidanov, and N. P. Mikhin, *Cryogenics* **24**, 190 (1984).
8. Y. M. Bunkov, D. A. Sergatskov , J. Nyeki, and I. N. Ivoilov *Dilution Refrigerator with Internal Cryogenic* ^{3}*He Cycle*, in: *Quantum Fluids and Solids* (1989), G. G. Ihas and Y. Takano (eds.), AIP Conference Proceeding No.194, AIP New York (1989), p. 397.
9. V. E. Sivokon, V. V. Dotsenko, L. A. Pogorelov, and V. I. Sobolev *Cryogenics* **32**, 207 (1992).
10. V. A Maidanov, N. P. Mikhin, N. F. Omelaenko, E. Ya. Rudavskii, A. S. Rybalko, V. K. Chagovets, V. A. Mikheev, P. Mohandas, B. P. Cowan, and J. Saunders, *Fiz. Nizk. Temp. [Sov. J. Low Temp. Phis.]* **20**, 672 (1994).
11. O. V. Lounasmaa, *Experimental Principles and Methods Below 1 K*, Academic Press, NY (1977).