An autonomous dilution micro refrigerator

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Abstract In the dilution refrigerator \(^3\)He circulates due to its condensation in a vessel with the temperature of 0.3-0.4K. The latter is cooled by the pumping of \(^3\)He from another bath by means of a sorption pump. A temperature <0.1 K is maintained for >12 hours. The sample holder is placed in an upper part of the refrigerator and is connected with a mixer by a copper heat conductor. They are surrounded by screens at temperatures 0.4, 4.2 and \(\approx 100\)K. The low temperature part is tied to the 0.4 K screen and centered by a polymer threads. A heat flux from the 0.4 K screen to the mixer is less than 0.1 µW. Inner volumes are filled with 0.2 mol of \(^4\)He, 0.1 mol of \(^3\)He and 0.05 mol of a mixture 40%\(^3\)He+60%\(^4\)He respectively. These gases remain all time inside the apparatus. The refrigerator is working when inserted in a 35 l transport cryostat with a liquid helium and operate during 5-6 days. The refrigerator is designed to cool down low temperature detectors or samples in experiments that do not require a high refrigerating capacity.

1. Introduction
In recent years, an increasing interest in reaching temperatures below 0.1 K has been evoked by the necessity of solving applied problems, such as cooling of highly sensitive photo detectors and particle detectors. A specific feature of these devices is that no appreciable power must be dissipated in them. Hence, the refrigerator must ensure a capacity compensating for only the parasitic heat flux delivered to the low-temperature cell through fastening elements, vibrations, etc., which can be reduced to fractions of 1 µW. In many scientific measurements, it is prohibited to supply a significant power to a sample (object) under study; otherwise, it will be overheated, since, at T<0.1K, the thermal conductivity of materials is low and the thermal resistance of the sample-cooler contact is high [1].

The cooling effect in a dilution refrigerator is achieved due to the dilution of \(^3\)He in \(^4\)He. The capacity of such a refrigerator is proportional to the \(^3\)He circulation rate. In classical designs (see [1]), the range of this rate is 0.01-1 mmol/s. As was shown in [2], when \(^3\)He is pumped out from the still by its condensation on a cold wall and the cold condensate drains down to the dilution chamber, stable operation is ensured at a circulation rate at a level of 1 µmol/s. This creates prerequisites for developing an autonomous compact and economic dilution refrigerator in the form of an insert into a portable Dewar helium flask [3]. This paper describes the design of the refrigerator in which this principle is implemented.

2. Design of the refrigerator
The refrigerator design is shown in the schematic drawing on figure 1, its photography – on figure 2. The refrigerator contains the following functional units located in a vacuum volume.

(i) A \(^4\)He loop incorporating a camera filled with \(^4\)He (1-K chamber) and \(^3\)He condensation volume, that compose a common unit, ampoule for \(^3\)He condensation, capillary for transferring liquid \(^4\)He, and sorption pump that evacuates helium vapors through stainless-steel tube with a 5cm-long copper
insert. The lower part of this insert is soldered through the cap of sorber’s container. The thermal valves serve to control the pressure of a heat exchange gas between the sorber and its container.

**Figure 1 (left figure).** Simplified schematic drawing of the refrigerator. 1 – cover sealed by In; 2 – stainless steel body; 3 – sample holder; 4, 19 – copper heat conductors; 5 – 100K screen; 6 – 4.2K screen; 7 – 0.4K screen; 8 - condenser for mixture vapors; 9 - tubes for evacuating \(^{3}\text{He}\) and \(^{4}\text{He}\); 10 - mixer; 11 - still; 12 - heat exchanger; 13, 18 - thermal valves filled with an activated charcoal. 14- copper inserts; 15 – \(^{4}\text{He}\) (left) and \(^{3}\text{He}\) (right) sorbers; 16 - copper container with \(^{3}\text{He}\) and \(^{4}\text{He}\) sorbers; 17 - \(^{4}\text{He}\) condenser; 20 – copper screen; 21 - capillary for pouring \(^{4}\text{He}\); 22 – \(^{3}\text{He}\) baths; 23 – \(^{3}\text{He}\) condenser; 24 - \(^{4}\text{He}\) bath; 25 – outer stainless steel tube; 26 – In sealed flange; 27 – containers for working gases.

**Figure 2 (right figure).** Photography of the refrigerator.
(ii) The $^3$He loop containing $^3$He bath and condenser of $^3$He-$^4$He mixture vapors. Bath is cooled by the evacuation of liquid-$^3$He vapors by sorption pump.

(iii) Dilution loop containing mixer, heat exchanger, still, and condenser, mentioned earlier and installed in a $^3$He bath.

The entire low-temperature section is surrounded by cylindrical copper screen. Using a low-melting InSn solder, the screen is soldered to container with sorbers. The bottom end of a stainless-steel tube is attached to copper screen; to the upper end of a tube a flange is welded. In order to avoid instability of a thin wall stainless tube, brass rigidity rings are soldered over it. They are clearly seen on figure 2.

The samples holder is placed on the top of the device. It is connected with the mixer by annealed copper heat conductor. The holder and the heat conductor are surrounded by 3 screens: a 0.4K screen attached to $^3$He bath; a 4.2K screen attached by means of a heat conducting rods with sorbers container; a 100K screen, cooled by vapors of liquid helium in which the refrigerator is inserted. The low temperature part is tied to the 0.4K screen and centered by a polymer thread “Armos” (produced by “Tverhimvolokno”, Russia). A heat flux from the 0.4K screen to the mixer is less than 0.1µW. Analogously the 0.4 K screen is centered relatively of the 4.2K screen (a heat flux about 5µW) and the 100K screen is centered relatively of its surrounding at the room temperature (a heat flux 3-5mW).

The refrigerator is working when inserted in a 35l transport cryostat with liquid helium and it operates during 5-6 days.

3. Operation of the refrigerator

After cooling of inner parts of the refrigerator down to liquid helium temperature it is possible to start (and repeat till all liquid helium in transport dewar evaporates) dilution refrigeration. In the initial state, all of the voltages powering the still, the sorbers' heaters, and the thermal valves are turned off. $^3$He and $^4$He are contained in corresponding sorbers, mixture – in a mixer and partly in a still and condenser. Sorbers are well thermally isolated from the surrounding liquid helium in the portable dewar. After the heater of the $^4$He sorber is turned on, its temperature increases and helium desorption is initiated. The gas flows to the condenser where it condensed. After the heating of the sorber stops and the heater of the corresponding thermal valve is turned on, the sorber is rapidly cooled and the gas pressure in it drops. Liquid helium is poured from the condenser to the bath, out pumping of helium vapors begins, and the temperature decreases to ~1 K. The $^3$He condensation proceeds similarly to the $^4$He condensation process. After the heater of the $^3$He sorber is turned off and the corresponding thermal valve is switched on, $^3$He bath is cooled to 0.35–0.4 K due to the out pumping of vapors.

To produce $^3$He circulation a power is fed to the still, see figure 3. Then $^3$He evaporates from the mixture in the still, condenses in the condenser, flows down through the heat exchanger to the mixer and returns to the still. Initially power fed to the still must be of the order of 0.2 – 0.3 mW to accelerate the mixer cooling. After the mixer temperature reached the 0.1K the power can lowered to ~0.04 mW. Then the temperature remains <0.1K till all $^3$He evaporates from the $^3$He bath, see figure 3.

![Figure 3](image-url)
The effect of the heating of a sample holder can be seen on figure 4. The mixer temperature slightly increases and temperature difference appears between sample holder and mixer. The difference at temperatures >0.5K is rather small and corresponds to the linearly changed heat conductivity for normal metal, figure 5, with $\alpha = 20\text{W/cm*K}$ at 4K. But at lower temperatures the difference rapidly increases evidently due to a thin layer of a superconducting solder between mixer and heat conductor. By using more elaborated technology this shortcoming can be avoided.

Figure 4. Time dependences of the mixer $T_{\text{mix}}$ and sample holder $T_{\text{top}}$ temperatures at different power $P_{\text{top}}$ delivered to the sample holder.

Figure 5. The dependence of reduced temperatures difference on mixer temperature. The straight line corresponds to the heat conduction of a normal metal.

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