A Portable Dilution Refrigerator

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Abstract—Refrigerating units of two dilution cryostats manufactured in the form of inserts for a commercial portable nitrogen-free cryostat for liquid helium are described. The ^{3}He circulation in both apparatuses is achieved by its evacuation via its condensation on a wall at a temperature of 0.35–0.5 K, which is cooled by the sorption-based evacuation of ^{3}He from a separate bath. One of the apparatuses operates periodically. A temperature below 0.1 K is maintained in it for several hours, after which the sorption pump needs to be regenerated for ~0.5 h. The other apparatus includes an additional bath with liquid ^{3}He that maintains its circulation during the sorption-pump regeneration thanks to the large specific heat of this liquid. The minimum temperature reached in experiments was 0.035 K. The refrigerators operate at a circulation rate of ~2–10 μmol/s and consume 1–2 l/day of liquid helium for maintaining their operation. The refrigerators are designed to cool photodetectors or samples in experiments that do not require a high refrigerating capacity.

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 photodetectors and particle detectors [1–3]. A specific feature of these devices is that no appreciable power must be dissipated in them. For example, if we deal with the detection of soft X-ray photons with a resolution of a few electronvolts, then, at a time constant of low-temperature detectors of ~10–100 μs, the measured radiation flux must be at a level of units to several tens of femtowatts. The heat flux to the detector caused by all other sources must then be strictly limited in order that the statistical noise be no higher than the detector's energy resolution. 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 [2, 3]. 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 $T < 0.1$ K, the thermal conductivity of materials is usually low and the thermal resistance of the sample–cooler contact is high.

Dilution refrigerators produced by a number of companies (e.g., Oxford Instruments) are widely used to obtain temperatures ranging from several units to several tens of millikelvins [4]. However, to make them more versatile and reach temperatures that are as low as possible, these apparatuses are designed for a higher capacity that, at a temperature sufficiently low for the detector operation, is as high as tens or even hundreds of microwatts, which is obviously too high. This excess obviously requires additional expenses and leads to a complicated design, larger dimensions, and a considerably increased amount of consumed liquid helium.

As is known, the cooling effect in a dilution refrigerator is achieved owing to the dilution of ^{3}He in ^{4}He [5]. The capacity of such a refrigerator is proportional to the ^{3}He circulation rate. In classical designs (see [5]), the range of this rate is 0.01–1 mmol/s, and it is not clear if the circulation rate can be reduced considerably without losing operating stability. At the same time, as was shown in [6, 7], when ^{3}He is evacuated from the still by its condensation on a cold wall and the cold condensate drains down to the dilution chamber [8], 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 [7]. This paper describes the design of a refrigerating unit we developed in which this principle is implemented.

DESIGN OF THE REFRIGERATOR

Two versions of the refrigerator design are depicted in the schematic diagram shown in Fig. 1. One of them (the right drawing) maintains a low temperature during the regeneration of sorption pumps. The other, with a somewhat simpler design (Fig. 1, the left drawing), operates in a periodical mode. Both apparatuses contain the following functional units located in a common vacuum volume.

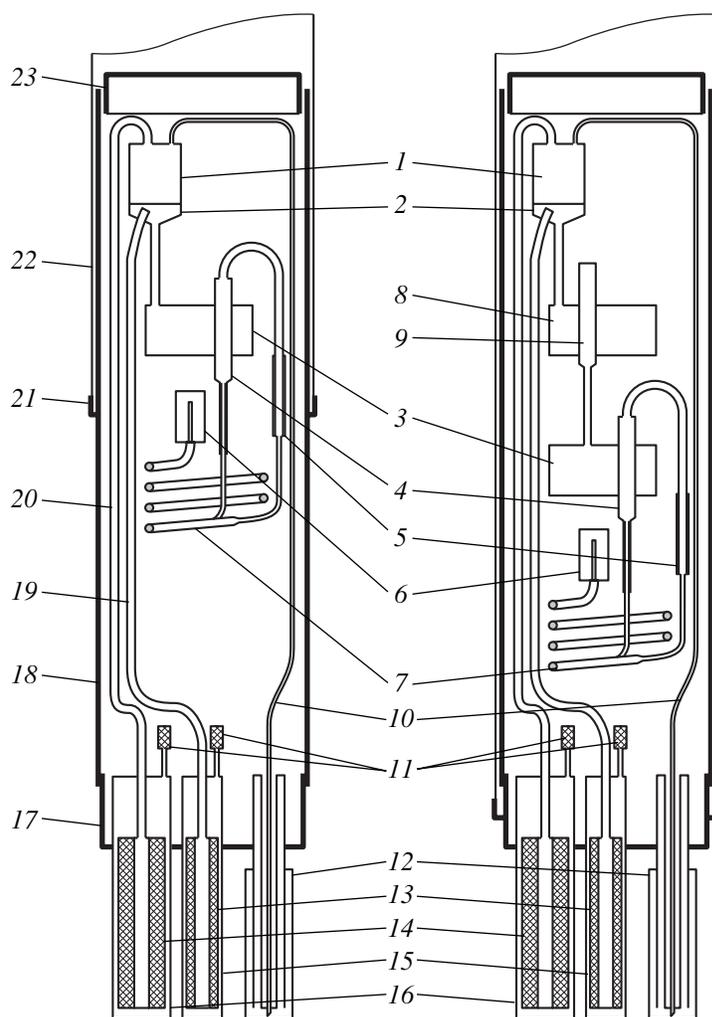(i) A ^{4}He loop incorporating chamber *1* with $T \sim 1$ K (1-K chamber) and ^{3}He condensation volume *2* that

Fig. 1. Schematic drawing of the refrigerating units of dilution refrigerators: (*1*) ^{4}He bath; (*2, 9*) ^{3}He condensers; (*3, 8*) ^{3}He baths; (*4*) condenser for mixture vapors; (*5*) still; (*6*) mixer; (*7*) heat exchanger; (*10*) capillary for pouring ^{4}He; (*11*) thermal valves; (*12*) ^{4}He condenser; (*13*) ^{3}He sorber; (*14*) ^{4}He sorber; (*15, 16*) copper housings with ^{3}He and ^{4}He sorbers, respectively; (*17*) oxygen-free copper block; (*18*) copper shield; (*19, 20*) tubes for evacuating ^{3}He and ^{4}He, respectively; (*21*) copper sleeve; (*22*) shell, stainless-steel tube; and (*23*) cap of the refrigerating unit.

compose a common unit, ampoule *12* for ^{4}He condensation, capillary *10* for transferring ^{4}He, and sorption pump *14* that evacuates helium vapors through stainless-steel tube *20* with a ~5-cm-long copper insert. The lower part of this insert is soldered through the cap of housing *16*. One of thermal valves *11* serves to control the heat exchange between sorber *14* and its container, housing *16*.

(ii) The main ^{3}He loop containing ^{3}He bath *3* and condenser *4* of ^{3}He–^{4}He mixture vapors. Bath *3* in the left version in Fig. 1 is cooled by the direct evacuation of liquid-^{3}He vapors through tube *19*, which is similar to tube *20*, by sorption pump *13* equipped with thermal valve *11*. In the right design in Fig. 1, bath *3* is cooled with the help of an intermediate ^{3}He bath (*8*) and condenser *9*. To establish a heat exchange between gas flows during the ^{3}He condensation, three short copper

sleeves (not shown in Fig. 1) were soldered on each of tubes *19* and *20* uniformly over their lengths and connected in pairs with heat conductors.

(iii) Dilution loop containing mixer *6*, heat exchanger *7*, still *5*, and condenser *4* mentioned earlier and installed in bath *3*.

The entire low-temperature section is enveloped by cylindrical annealed oxygen-free copper shield *18* with cap *23*. Using a low-melting InSn solder, the shield is soldered to block *17* made of oxygen-free copper and supporting housings *15* and *16* with sorbers *13* and *14*, respectively, and ampoule *12*. Stainless-steel tube *22* with copper sleeve *21* soldered to it with the ПСР45 solder is put on the shield. Tube *22* is soldered to block *17* (Fig. 1, the right drawing) or shield *18* (Fig. 1, the left drawing) through sleeve *21* with the InSn solder. The right and left apparatuses operate jointly with an

Airliquid portable cryostat (France) with a 50-mm-diameter throat and a Kriogenmash cryostat (Russia) with a 58-mm-diameter throat, respectively. The corresponding dimensions of tube *22* for the right and left refrigerators are $\varnothing 46 \times 0.4$ and $\varnothing 56 \times 0.3$ mm. In order to avoid a loss in the stability of the tube walls, brass rigidity rings are soldered over it.

During operation, cap *23* is positioned in the bottom part of the portable-cryostat throat where the temperature is no higher than 50–70 K in the absence of the insert. The cap carries a copper tube that connects the refrigerator's lower part, shown in Fig. 1, to the upper part situated above the refrigerator flange (description of the system of heat conductors and shields and the upper part is beyond the scope of this paper). Consequently, a considerable heat flux of 50 mW arrives at the cap. An additional contribution is provided by the capillaries through which the gases are supplied (not shown in the figure). Therefore, to efficiently transfer heat to the cap in the right design (Fig. 1), the cap is soldered to shield *18* with the InSn solder. Unfortunately, this significantly complicates the assembling and dismantling procedures that are inevitable in the development period. The large diameter of the outer tube in the left apparatus (Fig. 1) allowed the diameter of shield *18* to be increased and heat-conducting copper rods (not shown in Fig. 1) linking cap *23* to block *17* to be placed inside it. With allowance for the fact that the flange of tube *22* is vacuum tightened in the room-temperature region using a dismountable seal with a rubber ring, shifting upwards the zone where sleeve *21* is soldered to the shield allows the accomplishment of only one soldering operation during assembling and dismantling—soldering of shield *18* to block *17*.

Let us describe the main units of the refrigerator.

The ^{4}He sorption pump is a cylindrical container filled with a sorber. The container is made from an 18-mm-diameter stainless-steel tube with 0.2-mm-thick walls. Two bottoms are welded into it: one of them is blind, and a 4-mm-diameter stainless-steel tube with 0.2-mm-thick walls is welded into the other for evacuation. A stainless-steel tube–separator with ≈ 100 1-mm-diameter holes drilled over its entire surface is set up inside the sorber. The space between the outer tube and the separator is filled with activated charcoal before the blind bottom is welded. The volume occupied by the charcoal is 30 cm^3, which is sufficient for sorption of ~0.15 mol of ^{4}He.

A heater made of a 0.1-mm-diameter constantan wire is wound over the entire container length and glued solid with БФ2 adhesive. The total resistance of the heater is 2.3 kΩ. One terminal is soldered to the container's bottom and the second to the metallic coating on a cylindrical isolator made of alundum ceramics, placed on the evacuation tube, and soldered to the container's top. A feeding constantan wire is soldered to the site to which the heater's terminal is also soldered. The constantan and copper wires of a differential ther-

mocouple are soldered to a metallized area, which is insulated from both the feeding wire and the housing.

An oxygen-free-copper tube 50 mm long that is intended to cool the gas flow from the sorber during the gas desorption is soldered with the ПСР solder to the free end of the evacuation tube.

A container with charcoal is installed in the sorber's cylindrical copper housing *16* so that the bottom part of the copper spacer is soldered through the housing's cover, and there is an annular gap of ~0.8 mm between the container and the housing. The wires run through a vacuum-tight adapter mounted on the same cover, which also carries a thermal valve and the tube for evacuation and filling the space inside the housing. The bottom is soldered on the opposite side of the housing.

The thermal valve consists of a ~2-cm-long copper tube capped at one end; its other end is soldered to a cupronickel tube ~3–4 cm long with dimensions of $\varnothing 2 \times 0.2$ mm. The latter is soldered to the cover of the sorber's housing. The copper tube is filled with several grains of activated charcoal, and a heating resistor with a nominal value of several tens of kiloohms is soldered to its outside.

Heat-exchanging ^{3}He at a pressure of a few torrs is enclosed inside housing *16*. This gas is fully sorbed by the charcoal contained in the thermal valve at liquid-helium temperature. When the valve is heated to ~15–20 K and a power of ~1–2 mW is supplied to the heater, the gas is desorbed and ensures good thermal contact between the container with the charcoal and the housing cooled with liquid helium.

Housing *16* is inserted into block *17* and soldered to it (see Fig. 1), so that three-quarters of its length protrudes outward from the vacuum volume. Hence, during its operation, the housing is directly immersed into liquid helium, which ensures its reliable cooling as long as the liquid remains in the portable dewar. This is important, since, when the sorber is being regenerated, a power of ~0.5 W is dissipated in it, and when the system is switched to the evacuation mode, about 100 J are withdrawn in a time of ~1 min. If there were no direct contact between the sorber's housing and liquid helium, parasitic thermal effects impairing the refrigerator operation would then arise. In a refrigerator prototype briefly described in [7], in which the heat released in the sorbers was removed using a copper heat conductor, it was observed that, at the moment any sorber was switched from the regeneration to the evacuation mode, the temperature of the other sorber's housing increased jumpwise by several degrees. This resulted in a desorption of the heat-exchanging helium from its internal wall, and heat was transferred via this gas to the sorber itself, which was also heated. If liquid helium was evacuated during this period, its temperature increased jumpwise by several tenths of a degree.

The ^{3}He sorption pump for the refrigerator shown in the left part of Fig. 1 is identical to the ^{4}He pump; for the "right" refrigerator, the only difference is the

dimensions: the container filled with charcoal is made from a 12-mm-diameter stainless-steel tube with 0.2-mm-thick walls and contains approximately 0.05 mol of ^{3}He.

Condenser *12* (Fig. 1) is assembled of three copper-tube segments inserted one into another. This is done to improve the heat exchange and accelerate the ^{4}He condensation, which is especially essential when the condenser is filled with liquid. The volume of the condenser ($\approx$4–5 cm^3) is chosen so that, at the end of the regeneration process, it is filled almost completely. This reduces the parasitic volume occupied by a dense gas at a pressure close to atmospheric pressure, and the greater part of ^{4}He charged into the apparatus condenses. A capillary running from the condenser's bottom serves to transfer liquid helium from the condenser to bath *1*.

^{4}**He bath** *1* is made of copper and has an internal volume of 4 cm^3 equal to the volume of ^{4}He helium remaining in the liquid phase after the liquid is transferred. Under bath *1*, there is an adjacent cavity into which ^{3}He condenses during the regeneration of sorber *13*. ^{3}He drains down into the ^{3}He bath (*8* and *3* in Fig. 1 for the right and left drawings, respectively) through a stainless-steel tube ($\varnothing 3 \times 0.1$ mm). ^{3}He is evacuated by the cold sorber through the same tube, the condenser, and tube *19*.

^{3}**He baths** *3* and *8* are made of copper, and, to improve the heat exchange between helium and the bath, ribs 0.5 mm thick separated by 1-mm-wide gaps were cut in their bodies using a spark tool. The internal volumes of both baths for the right apparatus are ~4 cm^3, and 0.1 mol of ^{3}He can be condensed in each bath. For the left apparatus, the volume of this bath has a doubled volume in order to increase the store of the liquid and thus increase the time of maintaining the low temperature.

A copper tube—a condenser of ^{3}He vapors—is soldered into bath *8* (Fig. 1, the right drawing). The tube's interior surface is large enough (~20 cm^2) for the vapor overheating relative to the wall due to the Kapitza resistance at $T \approx 0.4$ K and a heat flux of 0.1 mW to be no higher than 0.01–0.02 K. Condensing ^{3}He drains down into bath *3* along a thin-walled stainless-steel tube ($\varnothing 4 \times 0.2$ mm) 10 cm long. Hence, at $T_8 <$ (the indices correspond to the baths' positions in Fig. 1, the right design), an efficient heat exchange exists between baths *3* and *8*. At $T_8 > T_3$, a heat exchange occurring only through the material of the stainless-steel tube and the gas column inside it is several orders of magnitude lower. Therefore, during the regeneration of sorber *13*, the temperature of bath *3* filled with liquid ^{3}He, the specific heat of which is considerable, changes rather slowly. This allows the ^{3}He circulation to be maintained in the mixture loop during the regeneration of sorber *13*, if the speed of the latter process is high enough.

Dilution loop. A copper tube—condenser of ^{3}He vapors—is soldered into both bath *3* (for the both designs) and bath *8*. The gas arriving from the still is condensed in this tube. The condensate drains down, passes through the internal capillary of the heat exchanger (stainless steel of cupronickel, 0.5–0.7 mm, and the wall 0.1–0.2 mm thick), and enters the upper part of the mixer, where the light fraction enriched with ^{3}He is located. Subsequently, after traversing the stratification boundary, ^{3}He is diluted with the heavy fraction enriched with ^{4}He and diffuses into the still through the heat-exchanger's outer capillary (stainless steel or cupronickel ~20 cm long, $\varnothing$1.6–1.4 mm, and the wall 0.2 mm thick) and the capillary connecting the heat exchanger to the still. Being evaporated in the still, the gas returns to the condenser, and the cycle closes.

Forced cooling device. It is obvious that the normal operation of the refrigerator requires that all of its main loops be properly thermally insulated from one another and from the walls having liquid-helium temperature. This is achieved by using sufficiently long thin-walled stainless-steel tubes simultaneously operating as gas lines and fastening elements. However, this leads to a very slow initial refrigerator cooling process.

The first stage of cooling to liquid-nitrogen temperature requires several hours (this usually occurs at night) after the bottom part of the refrigerator is immersed into a dewar with liquid nitrogen. At this stage, the heat transfer through radiation and heat conduction via the residual gas is quite efficient. Air is pumped out of the refrigerator's vacuum jacket through a narrow tube, since it is unnecessary to achieve a high vacuum at room temperature, thus resulting in an appreciable amount of gas in the jacket.

When the apparatus is moved from nitrogen to a helium dewar, the vacuum improves drastically thanks to the freezing of all gases except for helium. The latter is sorbed on cold walls; moreover, its amount is small. Therefore, heat transfer through the gas is precluded. The heat transfer through radiation depends on the temperature as T^4 and also becomes inefficient. At the same time, the specific heat of massive refrigerator components manufactured from copper is still rather high at nitrogen temperature. Experience has shown that such units as the ^{3}He bath and the mixer are cooled for more than two days. Therefore, operating the refrigerator requires that the initial cooling be intensified. A mechanical thermal valve serves for this purpose, which is based on a stainless-steel bellows element with bottoms welded at its ends. One bottom has a hole with a soldered tube. The gas supplied along the capillary from the ^{3}He sorber volume enters through this tube into the bellows. The second bottom is blind, and a copper support, to which a flexible heat conductor made from an annealed copper foil, is screwed into it. The bellows element is fixed in a housing, a stainless-steel tube, in which there are windows for introducing copper-wire heat conductors soldered to the parts to be cooled. A nut, into which a copper screw with a flat end is screwed, is fixed at the tube's end opposite to the site

of the bellows attachment. The screw and the support are connected to the liquid-helium bath through copper heat conductors.

Free ends of heat conductors from elements are inserted into the gap of ~3–5 mm between the screw's end and the support so that these ends form a stack of contacts. Gaps of ~0.5–1 mm remain between both the contacts and the heat conductors and the valve elements. Therefore, as long as the pressure in the bellows is low, no heat flux is transferred to the ^{3}He baths and the mixer. To create a thermal contact, the ^{3}He sorber is heated to 50–70 K. The helium contained in it is desorbed, and a pressure of several atmospheres (estimated as ~5–10 atm) develops in the system. The bellows element elongates, and the stack of contacts is clamped between the screw and nut, thus ensuring the efficient heat transfer. The heat flux through the contacts depends substantially on the state of their surfaces. For example, the cooling times for ordinary stripped copper contacts, indium plated contacts, and contacts plated with chemically deposited gold are ~5–6, ~1–3, and ~0.5–1 h, respectively.

Note that it is inexpedient to use ^{4}He for controlling the thermal valve, since the valve itself always has a low temperature. For this purpose, the pressure in the bellows cannot exceed atmospheric pressure. This complicates the adjustment of gaps in the valve, since the bellows stroke decreases, and reduces the heat conduction through the contacts, since it is proportional to the pressing force. There is an additional substantial factor: during the refrigerator operation, the pressure in the ^{4}He loop periodically rises slightly above the atmospheric level; therefore, the helium that fills the ^{4}He sorber cannot be used for controlling the valve. On the contrary, after the refrigerator operating mode establishes under periodic ^{3}He desorption cycles, the gas pressure remains low due the condensation on the wall being in contact with the 1-K chamber. This guarantees against unsanctioned valve operation.

TEMPERATURE MEASUREMENTS AND DATA COLLECTION

To monitor and control the refrigerator operation, the temperatures of at least seven (or six) points must be known. These are the temperatures of two sorbers, the 1-K ^{4}He chamber, two (or one) ^{3}He baths, the still, and the mixer. At the adjustment stage, it is also desirable to measure the temperatures at other points—sorbers' thermal valves, the copper shield, flows of the desorbed helium in front of the inlet to the corresponding condenser, etc. Hence, it is necessary to have many thermometers combined in a computer data collection system, without which online control is virtually impossible. However, the fact that it is unnecessary to know these temperatures with a high absolute accuracy simplifies the development of the measurement system.

The temperatures varied in the range ~4–100 K (the sorbers and thermal valves) are measured by copper–constantan differential thermocouples for determining the thermal emf relative the copper blocks of the sorbers' housings—the "cold ground." These thermometers have low sensitivities in the range 4–10 K, where the measurement accuracy was no higher than several degrees. However, since the desorption goes at high temperatures when the differential thermal emf exceeds 10 μV, the use of the thermocouples is justified.

PH1-12 packageless 1-kΩ resistors with resistive films based on ruthenium oxide and deposited on a ceramic substrate were used to measure temperatures below 4 K down to tens of millikelvins. These thermometers have a low sensitivity at $T > 5$ K, where their readings can be used only for qualitative monitoring of the preliminary-cooling processes. In the range $T = 10$–100 K, it is more convenient to use the differential thermal emf measured at a zero current through resistance thermometers. Its value differs slightly from the thermal emf of the copper–constantan thermocouple, since stainless-steel tubes are also included in the circuit, but this difference converted into temperatures leads to values underestimated by only several degrees. Note that this is quite convenient, since it becomes possible to monitor the operation of the bellows thermal valve by a rapid rise in the observed temperature of appropriate units upon appearance of a contact.

The resistors were taken from a common batch. Two of them were calibrated at the Institute for Physical Problems by measuring the magnetic susceptibility of cerium–magnesium nitrate in the range from 4.2 to 0.03 K. One resistor was calibrated at PTB, Berlin. The calibration characteristics normalized to resistance values of the resistors at room temperature coincided with the characteristic averaged over these three thermometers to an accuracy no worse than 1 mK at 0.05 K. Subsequently, we used this averaged characteristic, and the resistance of each individual resistor used in temperature measurements was normalized to its resistance at room temperature.

The measurement currents in the thermometers were supplied from a common voltage source—a 12-bit DAC with a maximum output voltage of 10 V. Resistors with rated values of 10 MΩ are connected in series with the source. The resistors were soldered directly to the pins of a metal–glass vacuum-tight connector on the vacuum side. This protected the ballast resistors against possible changes in the measurement currents caused by moisture condensation on their surfaces. To reduce the number of wires running from the connector installed on the refrigerator's warm cap to the cold zone and thus to improve the reliability of operation, each resistor (except for the one installed on the mixer) was linked to the connector by only one PH1-12 0.08-mm-diameter constantan wire, with a resistance of ~100 Ω. Therefore, the total voltage drop across the resistor and the connecting wire relative to the "cold ground" was

measured. It was verified that the resistance of the wires was virtually temperature-independent, and its known value was subtracted from the total resistance measured. The resistor's second contact area was soldered to the grounded pin of a cermet thermal isolator (see below).

High-frequency oscillations of technogenic origin and from the controlling computer penetrate to a thermometer and overheat it, which is especially noticeable when measuring the lowest temperatures. Conventional shielding measures were insufficient. For example, we could not register a temperature of the mixer below 0.15 K, although it obviously continued to decrease after this value was reached. This could be judged by the temperature of the still that continued to increase owing to the decreasing concentration of ^{3}He contained in it, as the mixture's temperature was falling. Including a resistor of ~10 kΩ, which was placed inside the apparatus, into the signal-detection circuit by connecting it in series to the thermometer improved the situation. Its resistance is much lower than the input impedance of the measuring device, and its connection has no effect on the measurement accuracy, but a parasitic signal penetrating via this channel is attenuated noticeably. In this way, we managed to record temperatures of 0.05–0.1 K. In order to suppress the thermometer overheating to a higher degree, it was placed directly in the liquid inside the mixer, and a temperature of 0.035 K was obtained in this configuration. The actual temperature in the mixer was probably even lower, but this result is also quite good in the light of the problems we face.

Another factor causing thermometers to overheat is a heat flux through wires. This effect must always be taken into consideration, if the wires are in vacuum and an efficient heat exchange is absent. Estimates show that a constantan wire 0.08 mm in diameter and 50 cm in length conducts 20–30 µW from room temperature to a level of 4 K. This is a small value from the standpoint of the thermal load on liquid helium in a portable dewar, constitutes an appreciable fraction of the heat input to the ^{3}He baths, and is absolutely unacceptable for the mixer and thermometers. For example, if a wire running from the vacuum-tight connector is soldered directly to the thermometer on the 1-K chamber, then, at $T = 4$ K, it will be overheated by 2–3 K because of the low thermal conductivity of the resistor's substrate.

In order to remove the effect of a heat flux through the wires, we used either integral ceramic packages (from K581 РУ4A logic memory circuits) or those cut into parts with metal pins baked into ceramics. To do this, a metal cover and the microcircuit itself were removed. If the remaining package with 16 pins was used as a whole, a copper-foil heat conductor, which was brought into contact with cold parts, was then soldered to the gold-plated metallized area, to which the cover was soldered. Two wires soldered to each pin ran from the warm and cold zones. All of the wires running from the warm connector were soldered to the pins of the unbroken package. To fasten the thermometers, the package was broken into pieces containing two or three pins. A resistor was soldered between two pins, a conducting wire was soldered to one of them, and the second (as well as the free pins, if they existed) was soldered to the part the temperature of which was measured.

In order to assess the effect of this system, we measured the overheating of the pin to which heat was conveyed from an electrical heater. It was established that the temperature dependence of the thermal conductivity does not contradict the T law typical of many materials in the 1-K region [9]. The numerical values of the thermal resistance between neighboring pins at $T = 1$ and 0.4 K are ~10 and ~2 µW/K, respectively. To additionally reduce the risk of overheating, one more element of thermal insulation was soldered to the element to which a thermometer was attached. The "warm" wire was first soldered to the pin of the additional element, and a wire 10–20 cm long was laid from this pin to the thermometer. The wire running to the mixer has an intermediate thermal contact with the ^{3}He bath.

To amplify the signals from temperature sensors, match these signals to the range of the measuring-ADC input signal, and suppress in-phase interference, differential amplifiers based on AD620 precision operational amplifiers (Analog Devices) are used. Apart from these amplifiers, the interface unit includes switches controlled by logic signals and intended to power the heaters of the sorbers and thermal valves from a source of 25–40 V and the still. The switches also set the measurement current through the resistance thermometers. The currents through the still's heater and thermometers are supplied from two ADCs.

Signals are measured and digitized by an NI-6014 ADC/DAC computer board (National Instruments, United States). This board ensures measurements of eight signals in the differential mode. The data collection and the control over the refrigerator operation were executed using a program developed for this purpose under the control of the Windows operating system.

OPERATION OF THE REFRIGERATOR

Let us follow the steps of the process of reaching a low temperature, beginning with the ^{4}He desorption and condensation, after all of the units were cooled to $T = 4$–5 K. In the initial state, all of the voltages powering the sorbers' heaters, the thermal valves, and the still are turned off. There is no heat-exchange helium inside the cavities of the assembly of sorbers, which are properly thermally insulated from the liquid helium in the portable dewar. The sorbers contain approximately 0.15 and 0.05 (in the right apparatus in Fig. 1) or 0.15 (in the left apparatus in Fig. 1) mol of ^{4}He and ^{3}He. Since the gas pressure is low, the bellows-based switch is open. The amount of ^{3}He condensed in the lower ^{3}He bath (bath *3*, the right drawing in Fig. 1) is 0.1 mol, while 0.04 mol of a 25–30% solution of ^{3}He in ^{4}He is con-
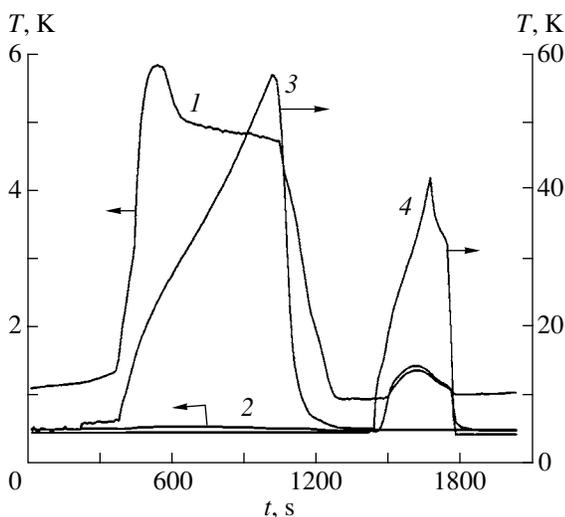
Fig. 2. Time dependences of the (*1*) ^{4}He bath, (*2*) ^{3}He bath, (*3*) ^{4}He sorber, and (*4*) ^{3}He sorber temperatures during the ^{3}He liquefaction. The power delivered to the ^{4}He and ^{3}He sorbers at the stages of their heating is 0.8 and ~0.3 W, respectively. Their temperatures fall abruptly upon the operation of the thermal valves, when a power of 2–4 mW is delivered to them.

Fig. 3. Sorber's temperature T and relative volume V of liquid helium remaining in the bath after its evacuation to $T = 1$ K as functions of the amount of heat delivered to the ^{4}He sorber. The volume filled with activated charcoal is 30 cm^3; the sorber is filled with 0.14 mol of ^{4}He.

tained in the mixer. The procedure of filling the system with the gases requires that the ^{3}He and ^{4}He desorption–evacuation process, which is described below, be repeated several times, but we will not consider this question in detail.

After the heater of the ^{4}He sorber is turned on, its temperature increases and helium desorption is initiated (Fig. 2). The gas that passes through the lower (copper) section of the evacuation tube is cooled from the sorber's temperature to ~5 K and passes to the condenser through the ^{4}He bath. At the initial stage, as long as the gas pressure is below atmospheric pressure, the bath temperature increases to a somewhat higher value owing to the adiabatic gas compression. However, after the pressure reaches the atmospheric value, the gas begins to condense and the pressure stabilizes. The bath temperature slightly diminishes and acquires the temperature of the gas flow. The heating of the sorber stops when its temperature reaches 50–55 K. At the power of 0.6–0.8 W typically delivered to the sorber, the desorption process lasts ~10 min (see Fig. 2). After that, the heater of the corresponding thermal valve is turned on, and, thanks to the appearance of a heat-exchange gas, the sorber is rapidly cooled and the gas pressure in it drops. Liquid helium is poured from the condenser to the bath; this corresponds to a kink in the time dependence of the ^{4}He-bath temperature at ~4.2 K. (If the sorber is cooled more slowly, a step arises in this dependence at ~4.2 K.) Evacuation of helium vapors then begins, and the temperature decreases to ~0.9–1 K.

Because the gas pressure is rather high during the ^{4}He desorption, an appreciable amount of helium
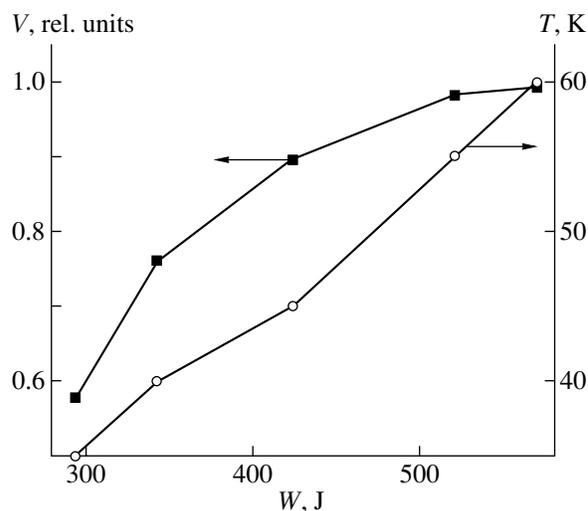
remains in the gas phase. During liquid helium transfer, part of liquid in the condenser also evaporates and fills it with a gas. By the end of this operation, 65–70% of the entire ^{4}He turns out to be in the bath (this was measured from the amount of helium removed from the refrigerator into a preliminarily evacuated vessel with the warm sorber). There is also a fraction of the liquid that evaporates upon cooling, so that no more than 50% of the stored ^{4}He (i.e., ~0.07–0.08 mol) is used for the ^{3}He condensation. This amount of stored ^{4}He is in principle sufficient for condensation and cooling to 1–2 K of a somewhat larger amount of ^{3}He, provided that there is an ideal heat exchange between the tubes through which the gas flows with allowance for a much lower heat of evaporation for ^{3}He compared to that for ^{4}He. However, if the process goes not too slowly, only 0.05 mol of ^{3}He can be actually condensed. Therefore, the ^{3}He-sorber volume in the right apparatus in Fig. 1 is 2.5 times smaller than the ^{4}He-sorber volume. In the left apparatus in Fig. 1, in order to extend the duration of low-temperature experiments, the both sorbers have equal volumes; however, to condense the total amount of ^{3}He stored in the sorber, its condensation process has to be repeated two or three times.

The ^{4}He desorption processes are mostly responsible for the helium losses from a portable dewar vessel during the operation of the refrigerator. Therefore, it is necessary to know the temperature to which the sorber must be heated, so as, on the one hand, to achieve as complete utilization of the helium stored in it as possible and, on the other hand, to stop heating in due time and not to evaporate helium from the dewar uselessly. For this purpose, we measured the dependences of the relative amount of liquid ^{4}He remaining in bath *1*
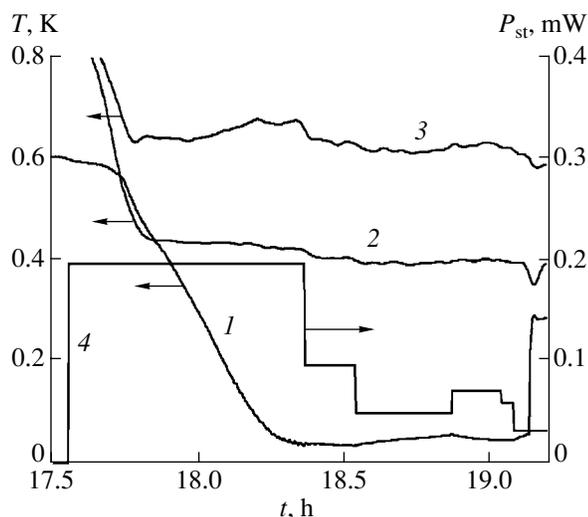
Fig. 4. Time dependences of the (*1*) mixer, (*2*) ^{3}He bath, and (*3*) still temperatures and (*4*) the power delivered to the still for the refrigerator constructed according to the left drawing in Fig. 1.

Fig. 5. Time dependences of the (*1*) mixer, (*2*) ^{3}He bath, and (*3*) still temperatures for the refrigerator constructed according to the left drawing in Fig. 1. The power delivered to the still, $P_{st} = 100$ μW. Within a time interval of ~100–200 s, $P_{st} = 200$ μW, which leads to an appreciable increase in the temperature of the still, a slight increase in the ^{3}He-bath temperature, and more rapid cooling of the mixer than at a lower still-heating power. The initial amount of ^{3}He condensed in the ^{3}He bath is 0.14 mol. The temperature level in the mixer is determined by the parasitic heat input (~2 μW) delivered to it.

(Fig. 3) after its cooling to 1 K and the temperature to which the sorber was heated on the amount of heat delivered to the sorber. The relative time for which a low temperature of the bath remained unchanged at a constant heat flux delivered to it was taken as the measure of the amount of helium. As is seen in Fig. 3, it is practically senseless to heat the sorber above 55 K: in this case, >98% of the maximum amount of ^{4}He is gathered in the bath, and the desorption itself consumes ~70% of the delivered heat. The rest of the heat is expended on the heating of the sorber itself.

Similar measurements were also performed for ^{3}He. It turned out that, in this case, it is sufficient to heat the sorber to 40–45 K. The total amount of heat expended on a single desorption of both gases is 600 J, which corresponds to a consumption of 0.2 l of liquid helium. Hence, at a power $P_{st} = 50$ μW delivered to the still and continuous operation of the refrigerator, the total amount of ^{3}He that must be liquefied is 0.2 mol; this requires three or four ^{4}He regeneration procedures. Thus, maintaining the circulation regime requires the consumption of only 1 l of liquid helium per day.

The ^{3}He condensation proceeds similarly to the ^{4}He condensation process but consumes a halved sorber heating power (see Fig. 2). Thanks to the low vapor density at the condensation temperature, almost the entire ^{3}He store is liquefied.

After the heater of the ^{3}He sorber is turned off and the corresponding thermal valve is switched on, bath *3* (Fig. 1, to the left) (or bath *8* together with bath *3* (Fig. 1, to the right)) is cooled to 0.35–0.4 K owing to the evacuation of vapors (Figs. 4–6). Since the initial temperature of liquid ^{3}He is quite low (~1 K), several percent of its initial quantity is expended on its cooling. The remaining liquid evaporates, thereby ensuring the
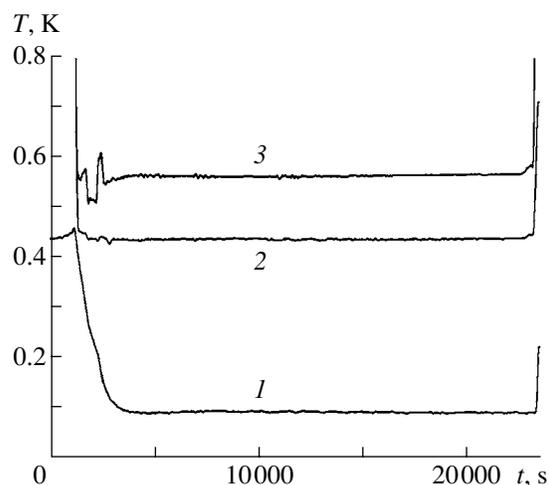
operation of a dilution cycle. Since the total heat of evaporation of 0.05 mol is ~1.2 J, the duration of the work until a repeated regeneration is needed is mainly determined by the power delivered to the still.

Figure 4 demonstrates the operation in the circulation mode. Our experience has shown that the still's heater must be turned on when the mixer's temperature reaches ~0.6 K. If we wait for a moment when the mixer's temperature approaches the temperature of the ^{3}He bath due to the heat exchange, a stratification of the mixture may additionally occur in the heat exchanger's capillaries. In this case, a cooling effect cannot usually be achieved. As can be seen in Fig. 4, a minimum temperature of 0.035 K at a power $P_{st} = 200$ μW delivered to the still can be reached in a time slightly longer than 0.5 h after the process was started. Despite the quite small dimensions of the heat exchanger, the reached temperature is fairly low and obviously meets the requirements for the operation of detectors. The refrigerator also operated reliably at a power $P_{st} = 50$ μW, but the temperature increased to 0.05 K, which, in this particular case, was associated with the presence of a small gas leak from the volume of the ^{3}He thermal valve into the refrigerator's vacuum jacket. This leak was found by a helium leak detector upon turning on the valve's heater. In accordance with the thermodynamics of the operation of the dilution refrigerator, this additional heat input can be assessed as ~0.4 μW.

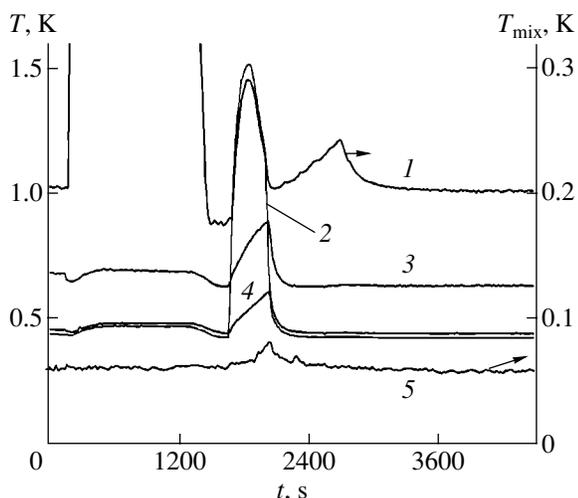Figure 5 demonstrates the operation of the left refrigerator in Fig. 1 for almost 6 h at a comparatively

Fig. 6. Time dependences of temperatures T for: (*1*) ^{4}He bath, (*2*) ^{3}He bath *8* (Fig. 1, right drawing), (*3*) still, (*4*) ^{3}He bath *3* (Fig. 1, right drawing), and (*5*) mixer. A slight increase in the ^{3}He-bath and still temperatures in the time interval of ~200–1500 s is associated with an increase in the ^{4}He-bath temperature during the ^{4}He regeneration, primarily due to an increase in the viscous resistance of the tube between the ^{3}He bath and the condenser. The power delivered to the still and the ^{3}He and ^{4}He sorbers during their heating is 100 μW and 0.3 and 0.8 W, respectively.

Fig. 7. Mixer cooling rate as a function of the ^{3}He circulation rate at a mixer temperature of 0.25 K. The apparatus contains approximately 0.02 mol of ^{3}He and 0.03 mol of ^{4}He.

high power delivered to the still. In this experiment, the ^{3}He sorber was filled with 0.14 mol of the gas, which condensed in two stages.

Figure 6 shows the possibility of maintaining a low temperature in the mixer during ^{3}He regeneration in the right refrigerator in Fig. 1. The mixer's temperature rises only slightly at the end of the regeneration when the still's temperature reaches ~0.8 K, and a perceptible amount of ^{4}He appears in the mixture vapors (i.e., the ^{3}He circulation actually decreases).

In the conventional dilution refrigerator design, a diaphragm between the still and the evacuation tube plays an important role. It restricts the transfer of super-fluid ^{4}He over the film from the cold zone to the warmer zone. In our case, the still is the warmest place, and the thermomechanical effect does not induce the helium transfer over the film. There is another force related to the osmotic pressure that, in our case, far exceeds the thermomechanical effect and leads to the transfer of ^{4}He from the still to the condenser. It is, however, obvious that, in this way, only an amount of ^{4}He can arrive at the still that corresponds to its equilibrium concentration in liquid ^{3}He at the condenser temperature. This is ~2–3%, which corresponds to a ^{4}He flow at a level of 0.2 μmol/s even at a maximum circulation rate of 10 μmol/s. This flow can be attained by limiting the gas transfer with a diaphragm that has a hole <0.2 mm in diameter but introduces an unacceptably high resistance to the gas flow. Hence, in our case, there is no need for a diaphragm.
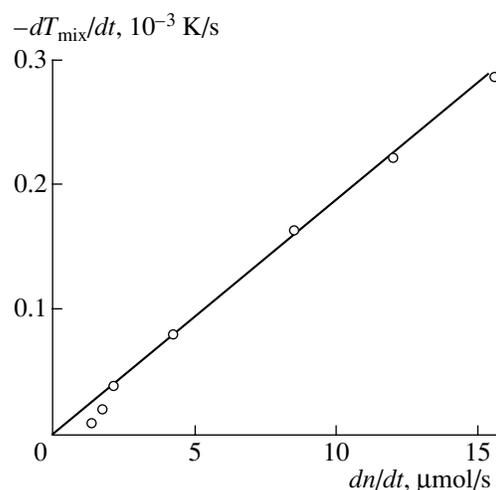
This is confirmed by the experimental results. Figure 7 shows the mixer cooling rate as a function of the ^{3}He circulation rate primarily determined by the power delivered to the still with small corrections for the dependence of the mixture's heat of evaporation on both the temperature and the fraction of ^{4}He in vapors [10]. In this experiment, the evacuation tube with an internal diameter of 3 mm had no diaphragm. The mixer cooling rate was measured at $T = 0.25$ K, when the heat fluxes to it play a comparatively insignificant role, and the main process is the heat removal from the liquid with a high specific heat (~0.02 mol of ^{3}He and ~0.03 mol of ^{4}He). As is seen, the cooling rate is proportional to the ^{3}He circulation rate at its value >2 μmol/s. If the ^{4}He flow from the still to the condenser were determined by the diameter of the evacuation tube, it would be ~3 μmol/s [11]. This is five times smaller than the ^{3}He flow during a circulation rate of 16 μmol/s (a power of 0.4 mW) and exceeds this flow by 50% at 2 μmol/s (a power of 0.05 mW). This would have affected radically the cycle productivity. However, this does not occur. An abrupt decrease in the cooling rate to a value below <2 μmol/s during circulation is probably caused by a flow of superfluid helium over the film in heat-exchanger's capillaries; this may cause the formation of regions of different phases in the mixture in the capillaries, which disturbs the operation of the refrigerator. Note that, when a 1-mm-diameter diaphragm was installed in the evacuation tube, a qualitatively similar picture was observed (see Fig. 4 where an abrupt increase in the mixer's temperature is observed at a still power of 0.03 mW).

As was mentioned above, after the refrigerator is preliminarily cooled and filled with the working gases, it is sufficient to expend ~1 l of liquid helium per day to
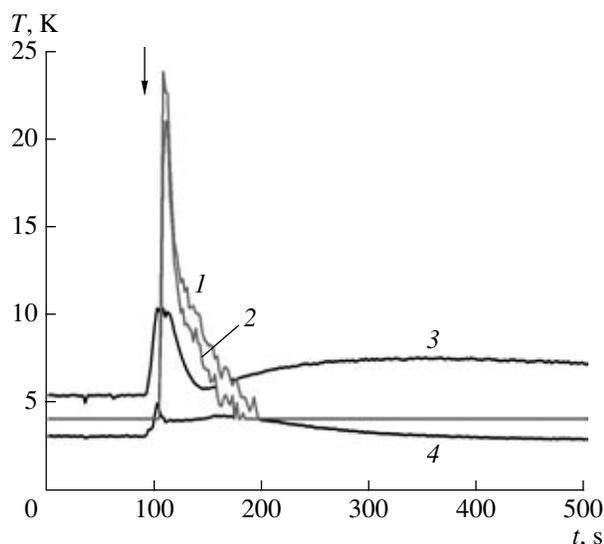
Fig. 8. Changes in the temperature of different units of the refrigerator upon its transfer from the exhausted dewar to another one filled with liquid helium. The moments the transfer was started are indicated with arrows. The moment the immersion of the refrigerator into the liquid began corresponds to the maximum sorber temperature: (*1*) ^{4}He sorber, (*2*) ^{3}He sorber, (*3*) ^{4}He bath, and (*4*) mixer.

maintain the operation of the refrigerator itself. There are, however, other heat-inputting sources, and liquid helium is actually preserved in a 30-l dewar for about a week. After that, the efficiency of ^{4}He liquefaction during regeneration sharply drops and further operation becomes impossible. However, if necessary, the already cooled apparatus can be moved to another dewar with liquid helium. This operation can be done within several tens of seconds (Fig. 8). In this case, judging by the temperature of sorbers, which, in this time interval, are heated by 20–30 K, the outer shield holds the same low temperature and the losses in liquid helium in this transfer procedure are some fraction of 1 l. This is one order of magnitude smaller than for the first immersion into liquid helium of the insert cooled to liquid-nitrogen temperature. A few minutes later, the refrigerator is again ready to operate. Note that, after the liquid in the dewar is exhausted, a quite low temperature is maintained inside it for a long time owing to the high specific heat of the cold gas filling the vessel. For example, a workday elapsed between the moment the absence of liquid helium in the dewar was revealed and the moment when the insert was carried over to the helium-filled dewar (Fig. 8). In this period, the temperature of the empty bath for ^{4}He, which reaches the temperature of the copper shield rather quickly, increased by only ~6 K.

CONCLUSIONS

The refrigerating units developed demonstrated stable operation with a quite modest amount of consumed liquid helium. The units are compact and reach the operating mode within a short time. Only low-power electrical signals are used to control their operation. The refrigerators have no mechanical cooled gas valves (as in [4]), which potentially increases their reliability and allows their computer-controlled operation.

The modest amounts of the working gases required for their operation are an important advantage of these apparatuses. Thanks to the small dimensions of the refrigerator's parts, these are generally quite strong if their walls are reasonably thick, and, after the refrigerator is filled with gases, the latter can be locked inside it. According to tests performed, the sorbers withstood a pressure of ~150 atm without any signs of failure. Since they can be filled under normal conditions with a gas volume 100 times larger than their own volume, the pressure developed by the gas upon its heating to room temperature will not exceed 100 atm. The refrigerator-filling mixture was enclosed in the dilution loop, and this did not cause a failure in the warmed apparatus. A hazardously high pressure can develop only in bath *3* of the right design in Fig. 1. If the apparatus is equipped with a container volume of ~0.1 l, the pressure in this loop can be reduced to an acceptable level. Small additional volumes can also be used as containers for other gases in order to overcome psychological discomfort and to increase the margin of safety. The apparatus can thus be fully autonomous.

1. Wollmann, D.A., Irvine, K.D., Hilton, G.C., *et al.*, *J. Microsc. (Oxford)*, 1997, vol. 188, no. 3, p. 196.

2. Frank, M., Hiller, L.J., Legrand, J.B., *et al.*, *Rev. Sci. Instrum.*, 1998, vol. 69, no. 1, p. 25.

3. Akimov, D.Yu., *Prib. Tekh. Eksp.*, 2001, no. 5, p. 6 [*Instrum. Exp. Tech.* (Engl. Transl.), 2001, no. 5, p. 575].

4. www.oxinstr.com.

5. Pobell, F., *Matter and Methods at Low Temperatures*, Berlin: Springer, 1996.

6. Edel'man, V.S., *Prib. Tekh. Eksp.*, 2002, no. 3, p. 139 [*Instrum. Exp. Tech.* (Engl. Transl.), 2002, no. 3, p. 420].

7. Edelman, V.S., *Physica B* (Amsterdam), 2003, vols. 329-333, p. 1574.

8. Edel'man, V.S., *Prib. Tekh. Eksp.*, 1972, no. 4, p. 229.

9. Olson, J.R., *Cryogenics*, 1993, vol. 33, no. 7, p. 729.

10. Radebaugh, R., *NBS Technical Note*, 1967, no. 362.

11. Keller, W.F., *Helium-3 and Helium-4*, New York: Plenum, 1969.