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A Continuously Operating Dilution Microcryostat

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Abstract—A dilution microcryostat based on the technology of sorption evacuation of ^{3}He and ^{4}He and condensation evacuation of a ^{3}He + ^{4}He mixture in a dilution cycle is described. A low temperature of ~0.1 K during regeneration of sorbers is continuously maintained owing to the high specific heat of the cryostat unit, in which circulating helium condensates. The cryostat is manufactured in the form of an insert in an industrial 35-L portable Dewar flask for liquid helium. A cold sample holder is positioned in the upper part of the device, thus facilitating the access to it. The complete volume of liquid helium suffices for 6–7 days of operation including the consumption of ^{4}He for preliminary cooling of the cryostat from nitrogen temperature. During this period, a low temperature of the sample holder can be maintained with an accuracy of ~1 mK. The device operates under computer control.

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INTRODUCTION

Refrigerator units of two dilution cryostats, which were manufactured in the form of an insert in an industrial portable nitrogen-free cryostat for liquid helium, were described in [1]. In both devices, circulation of ^{3}He is performed by its pumping out by condensation on the wall with a temperature of 0.35–0.5 K, which is cooled by the sorption pumping-out of ^{3}He from a separate bath. One of the cryostats operates in a periodic regime. A temperature below 0.1 K is maintained in it for several hours; after that, the regeneration of the sorption pump, which lasts ~0.5 h, is required. In the other cryostat, an additional bath with liquid ^{3}He is introduced, which, owing to the high specific heat of the liquid, provides maintenance of the circulation during the regeneration of the sorption pump. The cryostats operate at the circulation in a range of ~2–10 μmol/s.

On the basis of the periodically operating unit, a self-contained dilution microcryostat was manufactured [2], in which the sample holder is positioned in the upper part of the device, thus facilitating access to it. The holder is linked to the mixer through a heat conductor made of annealed copper and is surrounded with radiation shields at temperatures of 0.4, 4.2, and ~100 K. Windows that absorb thermal radiation and that transmit measured radiation with low attenuation are mounted in the upper lids of the shields and the warm cap of the instrument. At present, this instrument is used in studies of detectors in the terahertz region [3].

However, there are many tasks that require long-duration maintenance of samples (detectors) at low operating temperatures, e.g., during astronomical observations. Below, we describe a continuously operating autonomous dilution microcryostat, whose operation is based on the idea (proposed in [1]) of using a substance with a high specific heat for maintaining the ^{3}He circulation in a dilution cycle during the regeneration of the sorption pump that evacuates ^{3}He. The design solutions that were presented in [2] were used when developing and manufacturing the cryostat.

DESIGN OF THE MICROCRYOSTAT

The instrument has many common structural features with the microcryostats described in [1, 2], thus allowing its general description without specific details. Its simplified scheme (Fig. 1) shows the arrangement of the main units. The microcryostat contains the following functional units that are placed in the common vacuum volume:

(i) ^{4}He unit that includes a 1-K chamber and a ^{3}He condensation volume, which constitute a common unit, and a sorption pump that evacuates helium vapors. In contrast to the cryostat in [2], the ^{4}He sorber (as well the ^{3}He sorber) is above the ^{4}He bath and both ^{3}He baths. Heat is withdrawn from the sorber to liquid helium in the portable Dewar flask through copper heat conductors. The sorber on which a heater is wound is manufactured from stainless steel and placed in a sealed copper container. The evacuation channel consists of a stainless-steel tube into which a ~5-cm-long copper tube is soldered. The latter passes through the container cover and is soldered to it. In this tube, ^{4}He condenses and trickles down to the 1-K chamber. A thermal valve, during whose heating/cooling the heat-exchanging gas (^{3}He) is desorbed/sorbed, serves for controlling the heat exchange between the sorber and the container walls. This is necessary when changing from the ^{4}He desorption regime to its evacuation.

(ii) The upper ^{3}He unit that contains the ^{3}He sorber and the condenser of ^{3}He vapors, which is soldered into it and serves for the lower ^{3}He bath. The ^{3}He bath is cooled by the evacuation of vapors of liquid ^{3}He by its own sorption pump, which is analogous to the ^{3}He pump. When the pump is regenerated, the bath is filled with ^{3}He, which is liquefied when being in contact with the 1-K chamber.

(iii) The lower ^{3}He unit that contains the second ^{3}He bath, which is under the first bath and is connected through a stainless-steel tube to the condenser of ^{3}He vapors of the upper circuit. Both circuits are sealed with respect to each other. If the temperature of the upper bath is lower than that of the lower bath, a good thermal contact establishes between them; otherwise, the thermal interaction is substantially weakened.

(iv) The dilution unit that contains a dilution bath (mixer), a heat exchanger, a still, and a condenser of vapors of a ^{3}He–^{4}He mixture. The condenser is filled with plates of holmium that has the high specific heat at $T < 1$ K [4]. For a Ho mass of ~50 g, its specific heat at $T = 0.4$ K is ~2 J/K. This allowed us not to use a large amount of ^{3}He for maintaining the working conditions during the regeneration of ^{3}He in the upper circuit, as was proposed in [1], and to restrict ourselves to a rather small amount of ^{3}He, only 0.015 mol in the lower circuit for establishing thermal coupling. The dilution circuit is placed above the sorption pumps, and the condenser included in it is connected to the lower ^{3}He bath via a copper heat conductor. The temperature difference between the upper ^{3}He bath and the condenser at $T = 0.4$ K is approximately 0.02 K/100 μW.

As in the cryostat in [2], the sample holder is placed in the upper part of the instrument and connected to the mixer through a copper heat conductor, which is brazed with a copper–silver solder to the mixer and holder. Owing to the absence of superconductors in the heat-transferring line, the temperature difference is an order of magnitude lower than in [2] and amounts to ~2 mK/μW at $T = 0.1$ K.

As in the device described in [2], the sample holder is surrounded by three heat shields: the shield connected to the still that has a temperature of 0.6–0.7 K and shields with temperatures of 4.2 and ~100 K. An increase in the temperature of the inner shield from 0.4 K (in the cryostat from [2]) to the temperature of the still has virtually no effect on the heat inflow from this shield to the holder, which remains at a negligibly low level. However, this allowed the virtually complete utilization of the heat flux from a level of 4.2 K through fastening elements as an addition to the power that specified the ^{3}He circulation and a decrease in the heat inflow directly to the condenser in the lower ^{3}He bath to 1–2 μW.

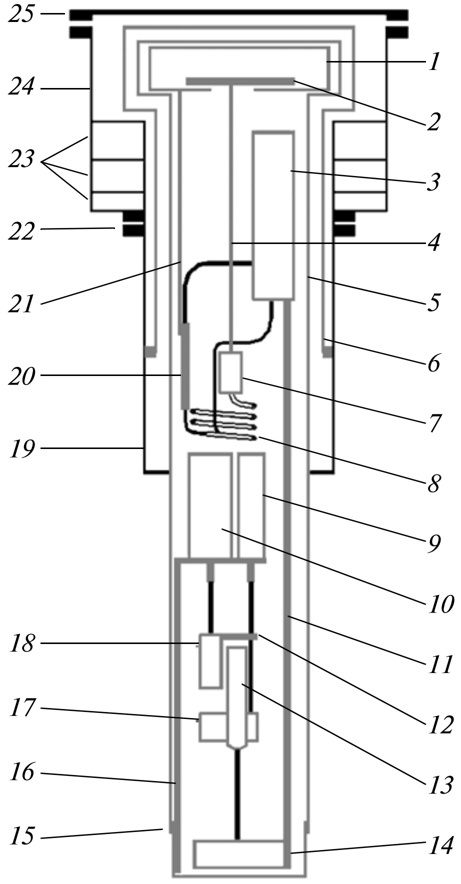

Fig. 1. Simplified diagram of the microcryostat that shows the arrangement of the main units: (*1*, *5*, *6*) 0.6-, 4.2-, and 100-K shields, respectively; (*2*) platform at 0.06–0.1 K; (*3*) holmium-filled condenser of the mixture vapors; (*4*, *11*, *16*, *21*) 0.1-, 0.4-, 4.2-, and 0.6-K copper heat conductors, respectively; (*7*) mixer; (*8*) tubular heat exchanger; (*9*) ^{3}He sorber; (*10*) ^{4}He sorber; (*12*) condenser of ^{3}He arriving at the upper bath; (*13*) ^{3}He condenser of the lower circuit; (*14*) lower ^{3}He bath; (*15*) vacuum-tight soldered spots of shield *5*; (*17*) upper ^{3}He bath; (*18*) ^{4}He bath (1-K chamber); (*19*) outer stainless-steel tube with a 56-mm diameter; (*20*) still; (*22*) flange of the outer tube; (*23*) vessels for collecting the working gases that are stored in them at a pressure of 25–50 atm at room temperature of the instrument; (*24*) housing; and (*25*) detachable cap. Stainless-steel tubes and parts are black-colored; copper parts are gray-colored.

OPERATION OF THE MICROCRYOSTAT

As the instruments described in [1, 2], the microcryostat operates when being immersed into liquid helium, with which a portable cryostat with a 58-mm-diameter passage (produced by Geliimash, Russia) is filled. The full height of this cryostat with the insert is 1 m.

Most units of the instrument are analogous to those of the cryostat described in [2]. Therefore, their behavior in the regime of preliminary cooling with the use of electromagnetic switches, filling with the working gases, their holding during warming of the insert,

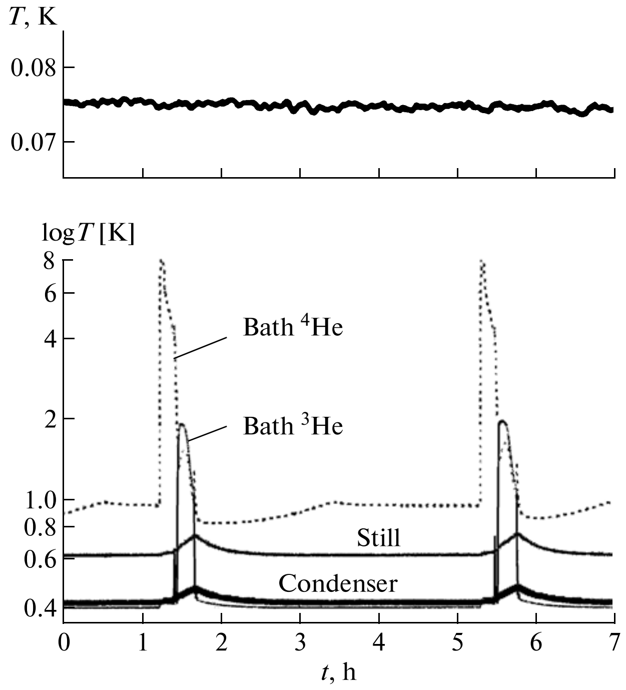

Fig. 2. Time dependences of the temperatures of the mixer (upper curve) and the ^{4}He bath, upper ^{3}He bath, still, and condenser of the mixture vapors (lower curves).

and the first cooling of the mixer to a low temperature are almost identical. However, in order to cool the condenser that has a high specific heat to the working temperature, an additional ^{4}He and ^{3}He regeneration cycle is required. In addition, because of a temperature difference between the upper ^{3}He bath and the condenser, which is due to the power dissipation in the still, at a mixer temperature lower than 0.1 K and a circulation more than 4–5 μmol/s, the still is heated to a temperature above 0.8 K and the cooling efficiency decreases.

The operation of the cooled instrument during the ^{3}He regeneration is demonstrated by the curves shown in Fig. 2. During a computer-controlled experiment, heating of the ^{4}He sorber was turned on twice, at time moments of 1.25 h and 5.25 h, and ^{4}He condensation occurred in the 1-K chamber. During this process, because of an increase in the heat inflow to the upper ^{3}He bath, the latter was slightly heated (by ~0.02 K) and the condenser temperature remained almost constant. Then, the sorber was cooled and the evacuation of ^{4}He vapors began. After the liquid was cooled to a temperature below 2 K, the heater of the ^{3}He sorber was turned on and the upper bath was heated to approximately 2 K. At the end of the desorption process, ^{3}He was cooled to ~1 K; subsequently, the evacuation of ^{3}He was turned on and its temperature returned to the level observed before the beginning of the regeneration process of the sorbers. During the ^{3}He desorption and cooling process, the condenser and still were warmed only by 0.04–0.05 K and ~0.1 K, respectively. In this case, the efficiency of the dilution cycle remained virtually constant and the temperature of the sample holder remained at a constant level with an accuracy to within thermometer noise of ~1 mK (a Lakeshore thermometer was used).

The virtually complete coincidence of the time dependences of the temperatures for both regeneration cycles shows that the instrument can stably operate for a long time at a constant temperature of the sample holder. A successful experiment on maintaining a constant low temperature for 3 days was performed, during which 20 computer-controlled desorption cycles were performed with a period of ~4 h.

The results of measuring the steady-state temperature T_{mix} of the mixer as a function of the power P_{mix} supplied to it from the heater are presented below for two values of the still heating power P_{st} = 75 and 175 μW:

P_{st}, μW	75	75	75	75	175
P_{mix}, μW	0	1	2	4	4
T_{mix}, K	0.075	0.14	0.16	0.21	0.13.

P_{st} was defined as the power dissipated in the heater plus the heat inflow to the still through the fastening elements and the superfluid in the filled capillary. The latter inflow was evaluated from the condenser heating rate during the regeneration at the disabled heater of the still and at approximately equal temperatures of the upper and lower ^{3}He baths. This value was 25 ± 5 μW.

The temperature settling time for T changing from 0.075 to 0.14 ± 0.001 K and a power of 1 μW supplied to the mixer is ~1.5 h. It is obvious that, if a feedback is introduced, this time can be reduced by a factor of several tens by controlling the power supplied to the mixer.

In order to maintain the operation of the dilution cryostat during the regeneration of the ^{4}He and ^{3}He sorbers, powers of 1.08 and 0.3 W are fed to them, respectively. A larger part of this power (70–80%) is expended for the regeneration itself; this fraction of heat is transferred to liquid helium in the cryostat during the next sorption when the corresponding baths are pumped out, and the other part is expended for heating gases and the gases themselves. The total heat supply per day (for a 4-h regeneration period) is 4300 J, thus leading to the evaporation of 1.7 L of liquid helium from the 35-L portable Dewar. In reality, the liquid helium in it is exhausted within 6–7 days; i.e., a much larger amount of liquid helium evaporates per day, approximately 5 L. We believe that when the design is optimized by selecting the height of the contact of the 100-K shield with the outer stainless-steel tube, using a tube with a thinner wall, installing an additional intermediate shield for reducing the radiation load on the shield at helium temperature, etc., the thermal loss can be substantially reduced and the time of continuous operation of the cryostat can be extended. A trivial way to increase the duration of the continuous operation is the use of a portable cryostat with a large volume (of course, in this case, the size and mass of the installation will be larger).

Note that, when the cryostat will be used in astrophysical measurements, it must be placed high above sea level at a low atmospheric pressure. In this case, the heat loss for ^{4}He desorption can be appreciably reduced because, first, the desorption can be finished at a lower temperature and, second, a smaller amount of liquid ^{4}He is required for liquefying the same amount of ^{3}He owing to smaller losses for its self-cooling at a lower start temperature and a lower initial temperature of the liquefied gas.

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