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> LABORATORY TECHNIQUES

A ³He Cryostat Inserted into a Refrigerator with an Impulse Tube

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Abstract—A compact autonomous ³He cryostat inserted into a two-stage refrigerator with an impulse tube is described. The cryostat contains baths filled with ⁴He and ³He and evacuated with cryosorbers to temperatures of ~1 and ~0.35 K, respectively. The low temperature is maintained for 6–8 h at an amount of liquid ³He filling the cryostat of ~0.035 mol. The dimensions of the insert (below the upper flange) are 49 mm (diameter) and 720 mm (length). The insert is introduced into a hermetically sealed tube—well filled with a heat-exchange gas during operation, which promotes the heat removal to levels of 45–50 K (the first stage of the impulse tube) and 3–4 K (the second stage of the impulse tube). The cryostat can be mounted in and extracted from both the warm cryostat and the cryostat cooled to low temperatures.

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INTRODUCTION

In the recent years, the use of so-called dry laboratory refrigerators with an impulse tube (impulse tube refrigerators—ITRs), which allow one not to use large amounts of expensive liquid helium, has become widespread in obtaining low temperatures. The temperatures attained in state-of-the-art ITRs are ~2.5 K or lower, making it possible to install ³He cryogenic units with sorption evacuation. Descriptions of such integrated structures are available on the Internet sites of leading companies producing cryogenic equipment.

However, in some cases, it becomes necessary to rapidly replace samples without stopping the operation of an ITR; to do this, a sample and the cooling units must be placed in a detachable insert. This circumstance is substantial, e.g., in experiments in a strong magnetic field of a superconducting solenoid. Placing a massive solenoid in an ITR leads to an extension of the time required for reaching low temperatures and warming to room temperature to many hours. The experience of using an insert with samples cooled to the temperature of the ITR is described in [1].

Below, we describe an insert with cryosorption evacuation of ³He operating with a two-stage ITR produced by TransMIT (Giessen, BRD) equipped with a hermetic well for placing the insert with a superconducting solenoid. Owing to a heat inflow through current leads, the minimum temperature of the ITR increases to 3.5-4.0 K. At such a temperature, the presence of only a unit with ³He is not quite efficient and it is reasonable to supplement the insert with cryosorption system with ⁴He.

DESIGN OF THE ³He CRYOSTAT

The instrument was designed using the experience obtained during the manufacture of dilution microre-frigerators inserted into a portable vessel filled with liquid helium [2, 3]. Because the description of the basic designs of the main units is available in these papers, here, we restrict ourselves to a simplified dia-gram of the cryostat (Fig. 1). Sorbers of ⁴He and ³He are stainless-steel cylinders filled with activated carbon and placed in sealed copper barrels 9 and 10 without a contact with their walls. The barrels form a part of a block with a copper bottom covering from above the hermetically sealed volume inside housing 16 with a dismountable sealing cone.

Heaters of $\Pi \ni \amalg OK 0.1$ wire are wound over the sorbers and copper–constantan thermocouples are mounted to control the temperature. Thin-wall stainless-steel tubes with copper inserts (not shown in Fig. 1) connect one sorber in the region of the copper block to ⁴He bath *14* and the second sorber, to the ampoule for condensation of ³He *13* and ³He bath *15*. The lower part of the tube running to the ⁴He bath is narrowed to a diameter of ~1 mm for limiting the heat transfer through the superfluid film. The tubes are joined to each other through heat conductors *17* for cooling gaseous ³He with a flow of evaporating ⁴He.

Microsorbers 12 and 18 (copper ampoules with activated carbon equipped with heaters, on thin-wall stainless-steel tubes) control the gas pressure in the barrels of the sorbers: when they are heated, a gas appears in the volume between the corresponding sorber and the barrel and efficient heat exchanges establishes. When the barrels are cooled to the temperature of the copper block, a high vacuum is established

in the barrel volume. Analogous microsorber 19 is open to the hermetically sealed volume inside housing 16. Vacuum in the cavity inside 16 is ensured by sealing cones greased with Apiezon. After the apparatus is assembled, this volume is filled with a small amount of helium evacuated by microsorber 19 during cooling of the low-temperature units.

The entire structure is suspended by thin-wall stainless-steel tubes, inside which there are wires and capillaries for admission of gases. One of them is used to evacuate the volume confined by housing 16. These tubes run through upper flange 1. Between them, there are ballast volumes 3 and 6 storing the gases at a pressure of ~25 atm when the insert is heated to room temperature.

The insert is 49 mm in diameter and 720 mm in height (to flange 1). It is inserted into a hermetically sealed with flange 4 attached to upper flange 5 of the ITR. The tube is equipped with copper rings 7 and 11 related to the first and second stages of the ITR. The temperatures of the rings in the operating mode are 45-50 (ring 7) and 3.5-5.0 K (ring 11). Centering split spring bronze rings—one upwardly and the other downwardly—are installed between the tube's copper rings and the corresponding elements of the insert. The upper rings determine the heat exchange in the zone of 45 K. Owing to a bellows joint 2 mounted between the flanges, the assembly of the sorbers is pressed to ring 11 when the pressure in the tube drops below atmospheric pressure.

OPERATION OF THE CRYOSTAT

The ITR and the insert's parts are cooled to $T \sim 4 \text{ K}$ within 8-10 h. To cool the insert, helium is admitted into the tube-well to a pressure of the same order as the atmospheric pressure at the warm refrigerator. If there are no gases inside the insert, the latter is filled with them owing to sorption in cold sorbers. The amounts of the admitted gases are 0.8-11 (³He) and $1.5-2.0 \, l(^{4}\text{He})$. Then, heating of sorbers with a power of $\sim 0.5 - 1.0$ W is turned on. When the sorbers reach temperatures guaranteeing virtually complete desorption of the gases, heating is turned off and sorbers are slowly cooled owing to a heat flux along the stainlesssteel tubes between the sorbers and the copper block. When the sorbers are heated, a flow of heated gases appears, causing an increase in the temperatures of the copper block with the sorbers, thermal switches, and the ³He and ⁴He baths (Figs. 2b and 2c). However, this heating is insignificant and, ~40 min after this heating began, the ³He and ⁴He baths are cooled to 4 K and ⁴He condenses. After that, the heating of the ³He and ⁴He sorbers is turned on (the heating power is several milliwatts).

After the thermal switches operate, the temperature of the sorbers sharply decreases (Fig. 2a). The baths are cooled to ~ 1 K, and ³He condenses. Then, after the ³He sorber is cooled, its evacuation is initi-

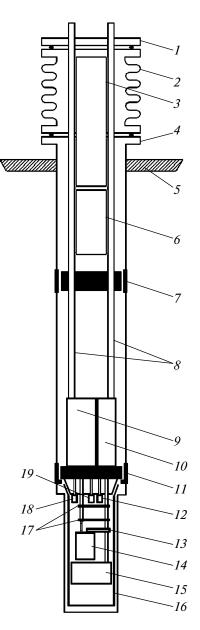


Fig. 1. Simplified diagram of the insert: (1, 4) flanges; (2) bellows; (3, 6) ballast vessels for storing ⁴He and ³He, respectively; (5) upper flange of the ITR; (7) zone of contact with the first stage of the ITR, $T \approx 45$ K; (8) tube for suspension of the lower part of the insert; (9, 10) barrels containing the sorbers; (11) zone of contact with the second stage of the ITR, $T \approx 3.5-5.0$ K; (12, 18, 19) microsorbers of the switches of ³He, ⁴He, and the heat-exchange gas, respectively; (13) copper ampoule for ³He condensation; (14) ⁴He bath; (15) ³He bath; (16) sealing housing with a sealing cone; and (17) copper straps.

ated and the temperature of the ³He bath decreases to $\sim 0.35-0.40$ K (Fig. 2c). Note that, if the ⁴He sorber is rapidly cooled, the temperature of the copper block rises stepwise above 10 K, and the temperature of the switch of the ³He sorber increases to 8 K. Fortunately,

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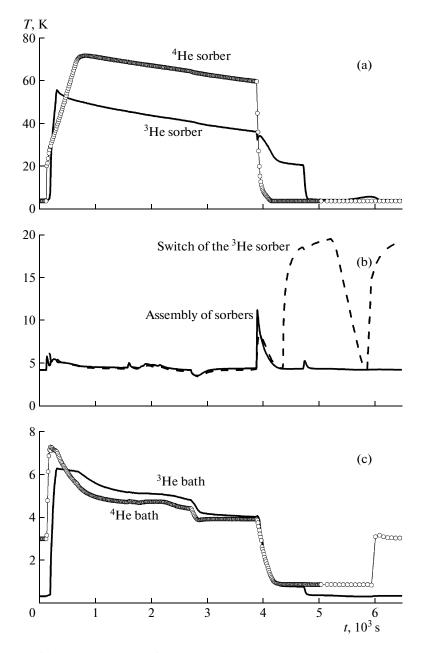


Fig. 2. Time dependences of (a) the temperatures of the sorbers, (b) the copper block that contains the sorbers and the thermal switch of the 3 He sorber, and (c) 3 He and 4 He baths during desorption, condensation of the gases, and cooling by vapor evacuation.

this heating is insufficient for premature operation of this switch. As is seen in Fig. 2b, this switch operates at T = 18 K.

The temperature of the copper block (and the thermal switch) exhibits a curious behavior in the time interval 2700-3000 s—after an abrupt decrease to 3.6 K, it returns to a level of 4.5 K. This is evidently with the specific features of the heat exchange between ring *11* being in good contact with the ITR and the copper block. As the temperature of the ITR, which is always lower than the temperature of the copper block, monotonically decreases, heat-exchange helium may condense in this gap and be kept there owing to the surface tension. As a result, heat exchange increases for a short time. Subsequently, as the ITR is cooled, the liquid recondenses from the gap to the underlying regions of the tube. In this case, a decrease in the density of gaseous helium leads to a decrease in the convection-induced heat exchange and, thanks to a heat inflow from above along the tubes, the block temperature increases. Thus, a decrease in the ITR temperature to a temperature appreciably lower than 4 K has a negative effect on the operation of the insert.

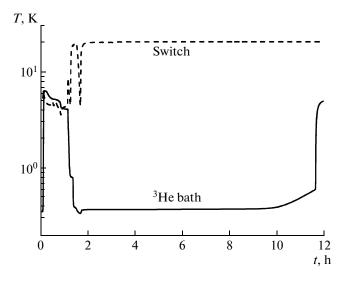


Fig. 3. Time dependences of the 3 He bath and the thermal switch of the 3 He sorber.

The temperature of the ³He bath at a level of 0.38-0.40 is maintained for 8 h (Fig. 3). We assume that the actual temperature is somewhat lower: when heating of the thermal switch is turned off, then, despite a slight increase in the temperature of the ³He sorber (Fig. 2a) and a decrease in the evacuation efficiency, the thermometer readings decrease to 0.34-0.35 K. This can be explained by the removal of the thermometer overheating by the thermal radiation of the heated switch incident on the thermometer. The sorber begins saturating in 8 h, and 10 h after the start of evacuation, the temperature increases to 0.6 K. The liquid in the ³He bath is exhausted. The evaporation heat storage (~1 J) allows us to evaluate that the heat inflow to the ³He bath is $25 \,\mu$ W.

The insert can be taken out from the ITR and inserted into it at low temperatures. In this operation, air must not penetrate into the tube–well; therefore, this procedure is allowed only at an IRT temperature \geq 4.2 K and must be accompanied by blowing of gaseous helium through the open tube, i.e., under conditions simulating the operation with a vessel containing liquid helium.

CONCLUSIONS

The above results show that the described insert is quite operable. Before it being tested, there were suspicions that the effects of overheating relative to the ITR may lead to an appreciable deceleration of processes in the course of desorption, impossibility of reaching a sufficiently low temperature ensuring the efficient condensation of ⁴He, and malfunctions in the operation at the moment of rapid cooling of the sorbers. Estimates of the corresponding heat fluxes owing to the thermal conductivities of the gases were not optimistic. In fact, in the first experiments with a relatively small amount of the heat-exchange gas in the tube, the ³He and ⁴He baths were cooled much more slowly than in the experiment presented in Figs. 2 and 3. We expected that the heat transfer would be intensified owing to convection, and the experiment confirmed these expectations. The condensation stage has actually almost the same duration as in the operation with the dilution microcryostat immersed into a vessel with liquid helium [2, 3]. This means that such a microcryostat can also operate in a "dry" variant without a change in its design, being plunged into the ITR well after the corresponding fitting of the dimensions of the tube-well.

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REFERENCES

- 1. http://cryo.transmit.de
- Herrmann, R., Ofitserov, A.V., Khlyustikov, I.N., and Edel'man, V.S., *Prib. Tekh. Eksp.*, 2005, no. 5, p. 142 [*Instrum. Exp. Techn.* (Engl. Transl), 2005, vol. 5, pp. 693–702].
- 3. Edel'man, V.S., *Prib. Tekh. Eksp.*, 2009, no. 2, p. 159 [*Instrum. Exp. Techn.* (Engl. Transl), 2009, vol. 2, pp. 301–307].