Dilution Microcryostat–Insert for Microwave Spectroscopy and Magnetic Resonance

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Abstract—An autonomous dilution microrefrigerator manufactured as an insert into a helium cryostat with a superconducting magnet is described. In this refrigerator, the $^3$He circulation in the circuit filled with a $^3$He–$^4$He mixture (mixer–heat exchanger–still–condenser–heat exchanger–mixer) is achieved due to the condensation of mixture vapors on the walls of the condenser, which is cooled by the $^3$He bath pumped out by a sorption pump, as well as entry of the condensate into the mixer under the action of gravity. An 8-mm-range resonator that contains a sample is connected to the mixer via a heat conductor; the sample is located at the center of a superconducting solenoid with a field of up to 80 kOe. Radiation passes from the generator through a waveguide to the resonator, and the signal that passed through the resonator arrives at the detector. The generator and detector are at room temperature. The device is designed for microwave spectroscopy of magnetic materials at temperatures in the range of ~0.09–3 K. The time within which the temperature is maintained at a level of ~0.1 K is 4–6 h. The results of a test experiment on the antiferromagnetic resonance in a MnCO$_3$ single crystal are presented.

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INTRODUCTION

At present, there is great interest in the development of the temperature range much lower than 1 K. This is due to the prospects for the development and use of low-temperature technologies in the field of ultrasensitive detection of electromagnetic and corpuscular radiations in astronomy and space research, for the needs of quantum electronics, including quantum computers, etc. Currently, refrigerators based on cooling by dilution of $^3$He in $^4$He and providing temperatures of below 0.3 K are produced very sparsely in the world and have a monopolistically high cost, which makes it difficult to access such devices. Commercial devices are often very cumbersome since the manufacturers (Cryogenic, BlueFors, etc.) strive to achieve a high cooling capacity of ~1 mW at 0.1 K. To achieve this, it is necessary to provide the $^3$He circulation rate in the dilution cycle at a level of 1 mmol/s, which requires the use of bulky high-performance vacuum pumps and vacuum lines with a large cross section. In reality, such a high performance is needed in technical fields, such as cooling of multielement radiation detectors or in quantum computers with matrices of thousands and tens of thousands of elements, or when building nuclear demagnetization plants to achieve temperatures lower than 1 mK.

However, due to the very poor heat transfer at low temperatures, it is impossible to dissipate power in samples even of the order of several microwatts, since even a power of the order of several nanowatts often causes unacceptable overheating of a sample relative to the cold platform. Therefore, when conducting scientific research, we can limit ourselves in most cases to the requirement for the performance of the refrigerator at a level of ~1 μW, which is sufficient to obtain a temperature of 0.1 K at a low power dissipated in a sample. This makes it relevant to construct autonomous miniature dilution cryostats, in which $^3$He circulates due to the condensation of helium, which evaporates in a heated still, on a wall cooled by pumping out pure $^3$He with a cryogenic sorption pump to a temperature of 0.4–0.5 K. Liquid $^3$He flows from the condenser through the inner tube in the heat exchanger into the mixer, passes through the dilution boundary into the lower phase with predominance of $^4$He and, via diffusion, returns to the still through the outer tube of the heat exchanger, thereby closing the cycle. As a result, a temperature of ~0.1 K is achieved without using the external evacuation.

Using such a scheme, dilution refrigerators—inserts of periodic and continuous action [1–3] and a dilution microcryostat with cooling by a refrigerator with a pulse tube [4] were created. Devices of this type have been in operation for more than 10 years, and numerous studies of low-temperature detectors based on...
superconducting structures at temperatures in the range of ~0.1–1 K have been performed using them.

In the development of these studies, we have manufactured an autonomous dilution microrefrigerator in the form of an insert into a helium cryostat with a superconducting magnet, which is built according to a scheme similar to that described in [1]. It is designed to conduct research on microwave spectroscopy and electron spin resonance in the frequency range of 25–140 GHz and magnetic fields of up to 8 T at temperatures of dielectric samples down to 0.1 K.

**DESIGN OF THE DEVICE**

The main volume of the used cryostat, which is filled with liquid helium, has the shape of a cylinder with a diameter of 25 cm and a height of 20 cm. A shank with a diameter of 10 cm and a height of 18 cm goes down from it. A solenoid designed for a field of ~80 kOe is placed in the lower part of the shank. The diameter of the solenoid hole is 25 mm, the length of the solenoid is 90 mm, and its diameter is 80 mm. A neck with a diameter of 10 cm and a height of 40 cm goes up from the main volume. A torus-shaped bath for liquid nitrogen is located around the neck, from which a shield goes down that envelopes the helium bath and has a good thermal contact with the neck approximately in the middle of its height. The temperature distribution in the helium volume for the case where the liquid level is at the bottom of the wide part is shown in Fig. 1. Based on these data, the height of the “cold” part of the insert was determined; its photo is shown in Fig. 2.

The insert consists of an inner block and a removable cover. The units of the inner block are attached to its upper flange. On the flange, there are a valve for pumping out the volume of the insert, sealed connectors with wires coming from them, waveguides of the 8-mm range with sealed ends, and valves, which lock volumes of 30, 30, and 15 cm³ with working gases ⁴He, ³He, and a mixture of ~65% ⁴He +35% ³He, respectively, that are located under the flange. These volumes are connected through capillaries to the systems of ⁴He, ³He, and the mixture. The flange of the inner block is jointed to the upper flange of the cover using an indium seal. The flange of the cover with a diameter of 120 mm allows sealing the helium volume of the cryostat with a rubber gasket after placing the insert into it.

A simplified diagram of the cold part of the inner unit is shown in Fig. 3. It contains the following main components.

1. ⁴He (1) and ³He (3) sorbents. They consist of cylindrical stainless-steel capsules with a volume of ~30 cm³ filled with activated charcoal. This amount of coal is sufficient for sorption of ~0.2 mol He. Heaters made of 0.15-mm-diameter manganin wire that were pasted through with BF2 glue are wound over the capsules along their entire length. The total resistance of the heaters is 230 Ω. Thin-walled tubes for evacuating vapors of liquid ⁴He and ³He run down from the sorbents. The capsules are placed in hermetic containers.
that are in thermal contact with the cold inner frame 15. Squeezable copper tubes that are designed to fill the cavities of the containers with heat-exchange helium to a pressure of ~10–20 mbar (at room temperature) and heat switches are soldered into the lids of the containers. The switches consist of ~1-cm-long stainless-steel tubes (Ø 1 × 0.25 mm), on which copper ampoules with a volume of ~1 cm³ with activated carbon are soldered. Heaters—planar resistors of ~10 kΩ—are pasted on the ampoules (the switches and tubes for filling with gases are not shown in Fig. 3). When the ampoules are heated, the absorbed heat-exchange gas is released for controlled cooling of the sorbents.

2. The ⁴He bath (5) with a volume of ~6 cm³. The volume of the bath is sufficient to be filled with 0.2 mol of liquid helium. A capillary for overflow of helium that condenses in volume 18, which is washed with liquid helium in the cryostat, is soldered into the lid of the bath.

3. The ³He bath (7) with a condenser 16 of mixture vapors that arrive from the still 8 through a stainless-steel tube (Ø 4 × 0.2 mm) with a diaphragm 6 with a hole of ~0.5 mm. The bottom of the still is a copper ampoule on whose wall a thermometer is glued. The output of a PTMN wire resistor that serves as the heater of the still is also soldered to the ampoule. The diaphragm reduces the ⁴He flow over a superfluid film into the condenser. There is a copper insert on the ³He pumping tube that is in contact with the ⁴He bath. Copper rings with jumpers are soldered to the ⁴He and ³He pumping tubes for the heat exchange between the streams of evaporating ⁴He and condensing ³He. The ³He bath with a volume of ~8 cm³ is made of copper. To improve the heat exchange between helium and the bath, 0.5-mm-thick ribs with a gap between them of 1 mm and a height of 20 mm are cut on a spark machine at the bottom of the housing.
4. The tubular heat exchanger 9 and mixer 10 with a volume of ~1 cm³. Inside the mixer, columns with a cross section of 0.5×0.5 mm² are cut to the full height to reduce the Kapitza resistance between the walls and helium. A heat conductor 19 consisting of two annealed copper wires with a diameter of 0.7 mm connects the mixer to the resonator 12. The heat exchanger with a length of ~20 cm is made of stainless-steel tubes with dimensions of $\varnothing 1 \times 0.3$ mm ($^3$He flow from the condenser to the mixer) and $\varnothing 3 \times 0.3$ mm ($^3$He diffusion flow from the mixer to the still).

5. The cylindrical resonator with an outer diameter of 16 mm with 0.5-mm-thick copper walls. The resonator has its natural $H_{01n}$-type oscillation frequencies, where $n = 1, 2, 3$ at frequencies of 27, 34, and 43 GHz, respectively, as well as other properly pronounced resonance modes at frequencies of up to 100 GHz. It is enabled for transmitting a microwave signal and attached to two sections of waveguides 4 made of stainless steel with a rectangular cross section of $7.2 \times 2$ mm² with 0.25-mm-thick walls. Such a scheme, in comparison with the registration of a resonator-redirected signal, doubles the parasitic heat inflow to the resonator via waveguides but allows measurements at several frequencies. Waveguides 4 run through the entire insert. In order to reduce the heat flow through them from the room to the resonator, they are connected by height-spaced copper jumpers to the top of the cold frame, to the $^4$He bath, and to the $^3$He bath. The distance from the lower jumper to the resonator is 30 cm. The calculated heat flow to the resonator is approximately 0.5 $\mu$W. To reduce the thermal-radiation flow from the room into the waveguide, friction plugs made of porous Gore-tex material, which scatters IR radiation, are inserted at the very bottom as well as at the points of contacts with thermal jumpers and approximately in the middle between the warm top and the contact with the frame.

6. Cold frame 15. It consists of a base, three bearing copper rods with a diameter of 8 mm, and an upper plate. These parts are brazed with silver solder in a vacuum. A $^4$He condenser (18) is soldered into the hole of the base. In the working position, the upper plate of the frame is located approximately at the level of the lower edge of the upper narrowing of the cryostat. A shield 2 having the form of an inverted 10-cm-high barrel, which protects the sorbent housings from thermal radiation, is pressed against the upper plate. According to Fig. 1, the shield-surrounding stainless-steel tube (cover 14) has a temperature of 70–80 K at most. Thus, the heat gain due to radiation on the inside wall of the shield is quite small. In the lower part, a bronze sleeve with springy blades that touch the walls of the cover is soldered to this shield. This minimizes the radiation flux from the warmer walls of the cover to the cold zone. The same purpose is served by three more flat shields with similar bronze sleeves, one of which is shown in Fig. 3, while the other shields are located above.

7. Capillaries 13 made of stainless steel ($\varnothing 1 \times 0.25$ mm) for supplying working gases.

8. The vacuum cover 14. Its upper part is a stainless-steel tube ($\varnothing 56 \times 0.3$ mm), and the lower part is a copper tube with a copper bottom. There are two holes in the bottom. One of them is for the $^4$He condenser. Vacuum sealing of both the condenser and the
inner volume of the insert is achieved by soldering with low-temperature InSn solder (17). Thus, quite efficient cooling of the supporting frame is achieved at thermal loads of the order of several watts during regeneration of sorbents and their cooling to the operating temperatures. Waveguides and a heat conductor from the mixer to the resonator 12 run into another hole in the bottom of the copper tube. The heat conductor consists of two segments of the annealed copper wire with a diameter of 0.7 mm and a length of ~15 cm. A wire resistor is soldered to the heat conductor. By passing a current through the resistor, the resonator with the mixer can be heated. This part of the device is closed with a copper cover 11 with an indium seal, which allows one to quickly replace test samples without unsoldering the device. The copper cover, as well as the rather massive copper bottoms, reduces the thermal load on the resonator and mixer under possible rapid changes in the magnetic field during measurements.

TEMPERATURE MEASUREMENTS AND DATA ACQUISITION

To monitor and control the operation of the cryostat, it is necessary to know the temperature at eight points. These are the temperatures of the cold frame, two sorbents, the 4He and 3He baths, the still, the mixer, and the resonator. Thus, it is necessary to have many thermometers integrated into a computer data-acquisition system. Without this, operational control is virtually impossible. However, for all points except for the resonator, the temperature values do not need to be known with high accuracy. This simplifies the construction of the measurement system.

Precalibrated copper—constantan differential thermocouples with thermal EMF measurements relative to the copper block of the sorbent housing, “cold earth,” are used to measure the temperatures of the sorbents that vary within 4–40 K. These thermometers are poorly sensitive in the range of 4–10 K, where the measurement accuracy is no better than a few degrees. Since the desorption occurs at high temperatures, when the differential thermal EMF becomes higher than 10 μV, the use of thermocouples was quite justified.

The temperatures below 4 K and down to tens of millikelvin are measured using industrial planar resistors with a nominal value of 1 kΩ with a resistive film element, which is based on ruthenium oxide, pasted to the corresponding blocks with the Stycast 1266 composite. These thermometers, on the contrary, are insensitive at temperatures above 20 K, where their readings can be used only for qualitative control of the precooling processes. Moreover, in the temperature range of liquid nitrogen and below, the used resistors have a minimum resistance. When monitoring the temperatures at the initial stage of cooling with cold helium supplied to the external cryostat, their graphs look as if there is an increase in the temperature, which then decreases with further cooling (Fig. 4). The thermometers were calibrated via comparison with a LakeShore thermometer in the temperature range of 0.1–15 K.

The measuring currents through the thermometers are set from a single voltage source—a 16-bit digital-to-analog converter (DAC) with a maximum output voltage of 10 V—in series with which resistors with a nominal value of ~1 MΩ are connected. There is only one wire from the connector to each resistor (except for those installed on the mixer and resonator) to reduce the number of wires running from the connector, which is installed on the warm lid of the device, to the cold zone. The second manganin potential wire, which is common for this group of resistors, runs from the connector to the “cold ground,” i.e., to the cold frame. The total voltage drop across the resistor and the supply wire relative to the “cold ground” is measured to exclude the thermal EMF. The resistance of the manganin wires is virtually independent of the temperature, and its measured value at room temperature was subtracted from the total measured resistance. A 0.05-mm-diameter copper wire is soldered to the second contact pad of the resistors; its other end is soldered to the housing of the controlled unit.

High-frequency fluctuations of anthropogenic origin and from the control computer, seeping into the thermometers, lead to their overheating, which is especially noticeable when measuring the lowest temperatures. The usual shielding measures were insufficient. Therefore, the wires through which the current is set and the voltage is picked off are connected to the mixer thermometer through planar resistors with a rated value of ~10 kΩ, which are pasted to the 3He bath.

The resistance of the resonator thermometer with a sample is measured according to the four-point scheme without grounding with the passage of signals through four planar 2-kΩ resistances pasted to the waveguide between the resonator and the 3He bath. A ruthenium oxide–based resistor from a batch of such sensors is used as a thermometer, for which a typical calibration was obtained via comparison with an exemplary Lakeshore resistance thermometer.

Another reason for overheating thermometers is the heat flux through the wires. In order to remove heat fluxes through wires, intact or cut-into-pieces ceramic cases with metal legs from Soviet-made K581 RU4A logic memory chips were used, which are baked into ceramics. To do this, the metal cover and the chip itself were removed from the chip. A gold-plated metal frame located under the lid allows soldering the entire body or the body divided into parts to the units of the device. To assess how much such measures remove heat fluxes through the wires, the overheating of the leg, to which heat was supplied from the electric heater, was measured. It was found that the tempera-
ture dependence of the thermal conductivity does not contradict the dependence of $T^2$, which is characteristic of many materials in the region of 1 K. The heat resistance between adjacent legs is approximately $10 \mu W/K$ at 1 K and $\sim 2 \mu W/K$ at 0.4 K [1].

All wires that run from the connectors (0.1-mm-diameter manganin in enamel insulation) are soldered to the legs of the ceramic thermal isolators that are soldered to the copper bodies of the sorbents. Further, 0.06-mm-diameter PESHOM wires run to the thermometers, first to the thermal decoupling elements installed on the controlled units; wires several centimeters long go from them to the thermometers. The wires that run to the mixer and resonator are additionally brought into thermal contact with the $^3$He bath and the waveguide in its middle between the resonator and the $^4$He bath, respectively.

Differential amplifiers based on precision OP177 operational amplifiers (Analog Devices) were used to amplify signals of the temperature sensors, to match them to the dynamic range of the measuring 16-bit analog-to-digital converter (ADC), and to suppress common-mode interference. In addition to amplifiers, the interface unit includes switches controlled by logic signals, which are designed to supply power to the heaters of sorbents and thermal switches from a 24-V source. Three 16-bit DACs are used to power the heaters of the still (a 10-kΩ PTMN resistor) and the mixer (a 150-kΩ PTMN resistor) and to set the measuring current through the resistance thermometers.

The data acquisition and archiving and controlling the operation of the insert is performed under computer control using a program developed for this purpose in the Windows operating system using an NI-6014 ADC/DAC board manufactured by National Instruments. The measurement cycle is organized as follows. The output signals of the operational amplifiers are alternately fed via multiplexers to the input of the 16-bit ADC with a polling frequency of $10^5$ s$^{-1}$. In each channel, the results are averaged over a time of 20 ms, thus making it possible to suppress the network interference at a frequency of 50 Hz. After polling all temperature sensors, the polarity of the current through the thermometers changes with maintenance of its value, and the process recurs. The results are then summed up for thermocouples and are subtracted for resistors, thereby making it possible to eliminate the contribution of the thermal EMF to the results of the resistance measurement.

In accordance with the calibrations, the results are converted into temperatures and displayed on the monitor for on-line control. At the same time, three files are formed for the possibility of subsequent monitoring and analysis, into which the values of the temperatures, measured resistances, voltages, DAC output voltages, and the states of the control logic signals are entered.

An operator can control the operation of the insert using a mouse and the diagram of the insert that is displayed on the monitor. The initial cooling of the $^3$He

![Fig. 4. Change in the temperature of the cold frame $T_{\text{body}}$ and the $^4$He and $^3$He baths ($T_{^4\text{He}}, T_{^3\text{He}}$) during pouring of helium into the cryostat with the magnet. Stop and Start arrows show the moments of stopping and resuming the pouring procedure. The readings of the thermometers correspond to an actual temperature at only $T < \sim 15$ K.](image-url)
bath, mixer, and resonator to a temperature of ~0.5–0.7 K can be performed in an automatic mode. The operation experience shows that it is advisable to control the processes in the manual mode during the initiation of the dilution cycle since, as a rule, it is required during the experiment to stabilize temperatures at intermediate values.

OPERATION OF THE DILUTION CRYOSTAT

The assembled insert is pumped out to a low pressure with the tightness control using a helium leak detector. Subsequently, the heat-exchange gas (approximately 1 cm³ of neon) is supplied to the vacuum volume. The insert is installed in the cryostat, and liquid nitrogen is poured into its nitrogen vessel. Within several hours (from evening to morning), the solenoid and the inner units of the insert cool down to a temperature close to the temperature of liquid nitrogen. After that, cooling to lower temperatures begins when liquid helium is poured into the cryostat. At the same time, one has to make pauses, because the heat-exchange neon freezes and cooling of the internal nodes stops if the helium level in the cryostat approaches its nitrogen vessel. After that, cooling to lower temperatures begins when liquid helium is poured into the cryostat. At the same time, one has to make pauses, because the heat-exchange neon freezes and cooling of the internal nodes stops if the helium level in the cryostat approaches the insert. After the internal components reach a temperature of ~10 K, the cryostat can be completely filled with liquid helium. Its consumption during filling is 15–17 L of liquid, including the expenditure for cooling the solenoid. The poured helium is enough for ~12 h of experimental work.

After cooling the frame to a temperature below 5 K, the desorption/sorption process is activated when the ⁴He and ³He sorbents are heated and cooled (Fig. 5). The sorbent heaters are turned on for heating; after the heaters are turned off, the heat switches are heated for cooling the sorbents. When they are heated, helium is desorbed, and a good heat exchange is established between the sorbents and their containers. As can be seen from Fig. 5, the frame is briefly heated to 8 K. Because of this, as well as due to the large heat consumption for cooling and condensation of ³He and the mixture, the ⁴He desorption/sorption process recurs several times. When the temperature of ³He reaches ~1 K, its sorption evacuation is initiated and the ³He bath cools down below 0.5 K, while the mixer, resonator, and still cool down to 0.5–0.6 K (Figs. 5, 6).

After the mixer is cooled, the still heater is turned on and a stable cooling mode is achieved a few minutes later (Fig. 6). During the experiment, it is possible to slow down the mixer temperature change or stabilize it at a specified level. To do this, the mixer heater is turned on and the power dissipated in it is selected. To increase the total time of the experiment, it is possible to reduce the power supplied to the still during heating of the mixer. The duration of maintaining a low temperature is determined by the fact that approximately 4 J of heat must be supplied to provide the complete evaporation of ³He stored in the bath. Correspondingly, with a power of $P_{\text{still}} = 0.2$ mW, which is supplied to the still from the heater, and taking into account ~50–70 μW of heat supplied through the waveguide and the superfluid film in the capillary to fill the mixer with a mixture of ³He and ⁴He, the duration of the dilution cycle is ~4 h.

Fig. 5. Diagram of the processes during cooling of the ³He bath to a temperature of ~0.5 K.
The lowest temperature of the mixer achieved in our experiment is $T_{\text{mixer}} = 0.1$ K at $P_{\text{still}} = 0.1$ mW and $0.085$ K at $P_{\text{still}} = 0.2$ mW. The dependence of the mixer temperature on its heating power $P_{\text{mixer}}$ is shown in Fig. 7. It corresponds well to the theoretical estimate of the performance of the dilution cycle (solid curve) according to [5], assuming that $\sim 4$ μW of heat is additionally supplied to the mixer. The main source of heat is apparently the insufficient heat exchange between the $^3$He flows from the condenser to the mixer and from the mixer to the still. Unfortunately, the dimensions of the insert do not allow one to significantly (by one order of magnitude or more) increase the heat-exchange area and improve the situation.

The experiments with heating of the mixer allow us to evaluate the Kapitza resistance between the liquid and the mixer walls. When the heater is turned on, the temperature $T_{\text{mixer}}$ increases rapidly, by $\sim 2$ mK at $P_{\text{mixer}} = 4$ μW, after which the liquid in the mixer is heated more slowly (Fig. 8). This is due to the fact that the liquid has a huge heat capacity compared to that of the mixer body. When heating is turned off, a rapid temperature decrease occurs, which is followed by slower cooling due to the mixture circulation. The duration of jumps is approximately 4–6 s and is apparently determined by the heating time of the heater, which is a quite large PTMN resistor, whose wire winding is packed in a plastic with poor thermal conductivity. The time of heating the mixer and resonator by 2 mK is estimated at several tenths of a second. According to the jump values, it is possible to estimate the coefficient $K_{\text{boundary}} \approx 0.7$ W/K$^4$ in the well-known formula that describes the Kapitza resistance:

$$P_{\text{boundary}} = K_{\text{boundary}} (T_1^4 - T_2^4).$$

Temperature variations that are noticeable in the time dependence of the resonator temperature (see Fig. 6) are caused by heating of the resonator with a current through the heater, Foucault currents under changes in the magnetic field $H$, and the absorption of the microwave power in the resonator during experiments. The temperature variations of the mixer are much less pronounced. This is due to the fact that there is a temperature drop along the heat conductor. The power generated by the Foucault currents can be estimated from the mixer heating. It is obviously proportional to the square of the field change rate, which is $P_{dH/dT} \approx 30(dH/dT)^2$ nW/(kOe/min)$^2$ for the used resonator. Similarly, it is possible to estimate the microwave power dissipated in the resonator as $P_{\text{hf}} \sim 2$ nW with a power of 0.5 nW received by the detector, when the signal-to-noise ratio is approximately ten. At this power received by the detector, records of the magnetic-resonance lines were obtained at the lowest temperature of the resonator (0.1 K). They are given in the section “Approbation in the Experiment.” The power
at the input waveguide flange of the insert obviously did not exceed 1 μW.

To estimate the power of room thermal radiation that seeps through the waveguides, an electric stove heated to ≈650 K with a massive blackened heater was brought to the open end of the waveguide. At a resonator temperature of 0.1 K, no change in its temperature was observed under measurement noise of ~0.2 mK. Taking the above results into account, this means that, in this case, the heat flux of the order of 0.2 W is weakened to a level of less than ~0.2 μW. If it is assumed that mainly long-wave radiation can seep into the resonator, whose power for a blackbody at room temperature is half as much as at 650 K, and the degree of blackness of the walls of stainless-steel waveguides is

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**Fig. 7.** Dependence of the mixer temperature on its heating power at \( P_{\text{still}} = 0.2 \text{ mW} \).

**Fig. 8.** Time dependence of the mixer temperature for the heater turned on and off. In the steady-state mode, the heating power is 4 μW.
approximately 0.1, then no more than 10–20 nW of the room’s thermal radiation reaches the resonator under the operating conditions.

APPROBATION IN THE EXPERIMENT

For test magnetic-resonance studies in the temperature range below 0.5 K, single crystals of MnCO$_3$ antiferromagnet were selected, which undergoes an antiferromagnetic phase transition at a Neel temperature of 32.4 K. The antiferromagnetic resonance in this substance has been studied well and is characterized by the fact that its frequency and resonant field strongly depend on temperature, even at a temperature significantly lower than temperature that is appreciably lower than the ordering temperature. This is associated with the interaction of the electron spin subsystem with a subsystem of $^{55}$Mn nuclei, the average magnetic moment of each of them at the temperature $T$ is “paramagnetic,” i.e., it is inversely proportional to the temperature under conditions when the total moment of the nuclear subsystem is far from saturation. As a result, the dependence of the antiferromagnetic-resonance frequency on the magnetic field applied in the light plane of antiferromagnetism and on the temperature is determined by the following ratio:

$$\frac{f}{\gamma} = H^2 + HH_D + 5.8/T + 0.3.$$  (1)

Here, $f$, GHz, is the frequency of the microwave field; $H$, kOe, is the magnetic field; $H_D = 4.4$ kOe is the Dzyaloshinsky field; $T$ is the temperature; and $\gamma = 2.8$ GHz/kOe is the gyromagnetic ratio [6]. The temperature dependence of the resonant field for a frequency of 27 GHz that was obtained using this formula is shown in Fig. 9 as a solid line.

Thus, at a fixed frequency of the microwave generator, the temperature $T$ of the sample can be determined based on the results of measurements of the antiferromagnetic-resonance field $H$. Strictly speaking, this is how the temperature of the nuclear subsystem of the sample is determined, which, as is known, can be overheated relative to the lattice due to a very long relaxation time. At a temperature of 0.1 K, this time may reach several hours [7]. For the lowest temperatures, i.e., significantly lower than 0.1 K, a more precise ratio should be used, in which the polarization of the nuclear subsystem is described not by the Curie paramagnetic law but by the Brillouin function for a nuclear spin of 5/2 (dashed line in Fig. 9). It should be noted that the low temperature of an MnCO$_3$ sample can be measured in this way in a sufficiently strong field (~10 kOe), in which conventional paramagnets turn out to be saturated almost completely and do not show a significant temperature dependence of the intensity of the paramagnetic-resonance signal or magnetic susceptibility in this range.

Figure 10 shows records of the dependences of the microwave power at a frequency of 27 GHz, which is transmitted through the resonator, on the magnetic field that were obtained at different resonator temperatures measured using a resistance thermometer. These curves were obtained under conditions of a low power and a slow passage of the resonance line. The sufficiently low power level was selected experimen-
In Fig. 9, dots show the experimentally obtained values of the resonant field of the antiferromagnetic resonance at different resonator temperatures. The resonant fields correspond well to the theoretical values for temperatures of ≥0.2 K, when the relaxation time of the nuclear subsystem does not exceed tens of minutes. For a resonator temperature of 0.1 K, in accordance with formula (1), we obtain a temperature of the nuclear-spin system of 0.13 K, which is a reasonable value because of the impossibility of thermalizing nuclei during the holding time of ~1 h after cooling the resonator. In this case, the temperature of the sample lattice was probably close to 0.1 K.

Thus, the performed test experiments show that, when loaded into the waveguide path and resonator, the constructed dilution microrefrigerator provides a temperature of the resonator of 0.1 K and a temperature of a dielectric sample down to 0.1 ± 0.01 K in the frequency range of 27–50 GHz under the conditions of a real magnetic-resonance experiment.

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CONFLICT OF INTEREST
The authors declare that they have no conflicts of interest.

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