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OPTIMIZATION OF THE PROCESS OF OBTAINING LOW TEMPERATURES IN UNIVERSAL LIQUID-FLOW CRYOSTATS

In order to quickly and economically achieve low temperatures, the process of obtaining them in home universal liquid-flow cryostats using liquid helium and nitrogen is considered. To control the level of a cryoagent, an original capacitive system for measuring the level of different liquid cryoagents has been designed, which has no analogs in the world. It has been experimentally shown that the most optimal and economical way to obtain low temperatures is to pump cryoagent vapors directly from the working chamber of the cryostat.

Keywords: cryostat, liquid helium, liquid nitrogen, capacitive level gauge.

1. Introduction

Liquefied gases are usually used to obtain and maintain low temperatures. In a Dewar vessel containing liquefied gas that evaporates at the atmospheric pressure, the constant temperature of normal boiling of the refrigerant is well maintained. The most commonly used refrigerants are liquid nitrogen and liquid helium.

Previously used liquefied hydrogen and oxygen are now rarely used due to the increased explosiveness of vapors. Nitrogen and helium are practically inert, and the danger is only a sharp expansion in the transition from the liquid to gaseous state.

By reducing the pressure over the free surface of the liquid, we can get a temperature below the normal boiling point of this liquid. For example, pumping a nitrogen vapor, we can achieve a temperature down to the triple point temperature equal to 63 K, pump-

ing a hydrogen vapor (above the solid phase) can give a temperature of 10 K, and pumping a helium vapor allows one to get (under favorable conditions of the experiment) a temperature of about 0.7 K. To conduct a research at low temperatures, experimenters use specialized devices to obtain and maintain these temperatures – cryostats.

The leading manufacturers of the cryogenic equipment are the following companies [1–5]: Oxford Instruments Ltd. (UK), “Cryo Industries of America Inc.” (USA), Lake Shore (USA), JANIS Research Company, Inc. (USA), Cryomagnetics corp (USA), and RTI Company (Russian Federation).

2. Main Part

Most of the above manufacturers prefer flow cryostats (so-called “continuous flow gas cryostat”), where the heat exchange gas is forced to flow through the working chamber of the cryostat, and the required sample temperature is created by an electric heater wound on the outside of the working chamber or heat exchanger.

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The advantage is that such cryostats are simple in design and relatively cheap. Their significant disadvantages are large consumption of a cryogen and a low stability of maintaining the temperature at the given level. Such cryostats can work with both helium and nitrogen as a working cryogen. For the operation of such a cryostat, it must be provided with a source of a cryogen (Dewar vessel), flexible overflow siphons such as “Bellows in the bellows”, a pump for pumping the cryogen, rotameter, *etc.*

Another scheme of operation of a thermocontrolled cryostat, built on the so-called “Swenson’s method” [6]. It assumes that the cryostat has a built-in tank with liquid helium. From it, liquid helium forcibly enters the heat exchanger, where it evaporates and, after heating to the required temperature, is fed into the working chamber with the test sample.

The temperature is regulated by a heater mounted on the outer surface of the heat exchanger and a valve. This method is schematically explained in Fig. 1. In the vacuum cavity of the cryostat, a nitrogen tank, a thermostat chamber with the object of study, and

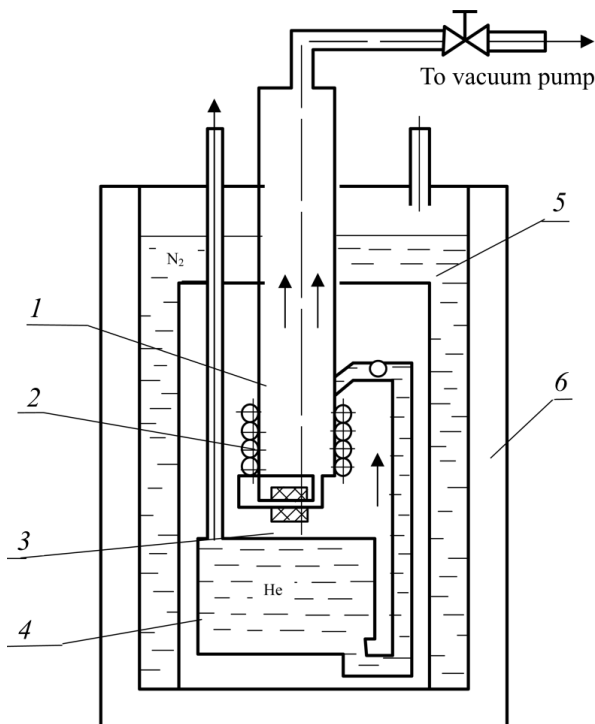


Fig. 1. Scheme of the Swenson method: heat exchanger (1), chamber of thermostating (2), sample (3), helium can (4), nitrogen can (5), vacuum cavity (6)

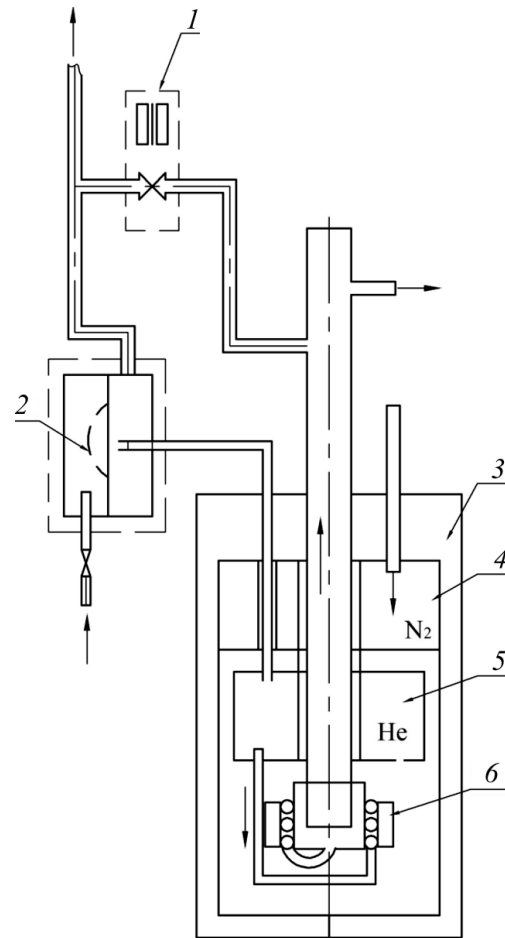


Fig. 2. Scheme of the method of temperature control on a.s. USSR No. 436334: backpressure valve (1), manostat (2), vacuum cavity (3), nitrogen can (4), helium can (5), chamber of thermostating (6)

a tank with liquid helium are placed. The regulation and stabilization of the temperature are achieved by supplying liquid helium from the tank through a tube firstly into the heat exchanger in contact with the walls of the chamber and then into the volume of the chamber.

At the Institute of Physics of NASU, a method of temperature regulation and stabilization was proposed and developed [7]. It is based, like Swenson’s method, on the heat exchange between the object of study, located in the thermostat, and flows through this chamber with a cryocarryer. The essence of this method is explained in Fig. 2.

In contrast to the Swenson method, the supply of a cryoagent from a tank with the liquid or gaseous

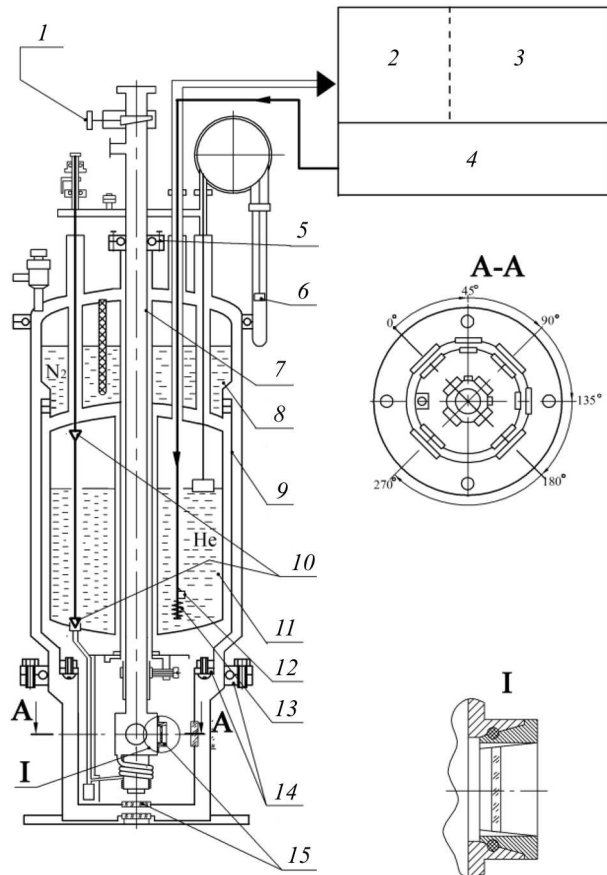


Fig. 3. Design of a universal liquid-flow cryostat: sluice (1), pressure sensor (2), gas flow regulator (3), temperature controller (4), flange of column-in change (5), level indicator (6), column-in (7), nitrogen can (8), nitrogen screen (9), double valve cryoagent supply (10), helium can (11), level sensor (12), evaporator (13), flange of tail changing (14), changeable windows (15)

cryoagent to the thermostat is due to gas support. This occurs in a closed space of the tank filled with liquid helium, which is in the boiling state. The set constant pressure in the tank is provided by the manostat established on the line that removes helium vapors from the tank. This allows one to obtain stable conditions for the passage of the cryoagent through the heat exchanger and more finely control the mass of the cryoagent passed through the temperature chamber by means of a valve mounted on the line of discharge of the spent cryoagent from the temperature chamber.

This valve regulates the supply of a cryoagent to the temperature chamber.

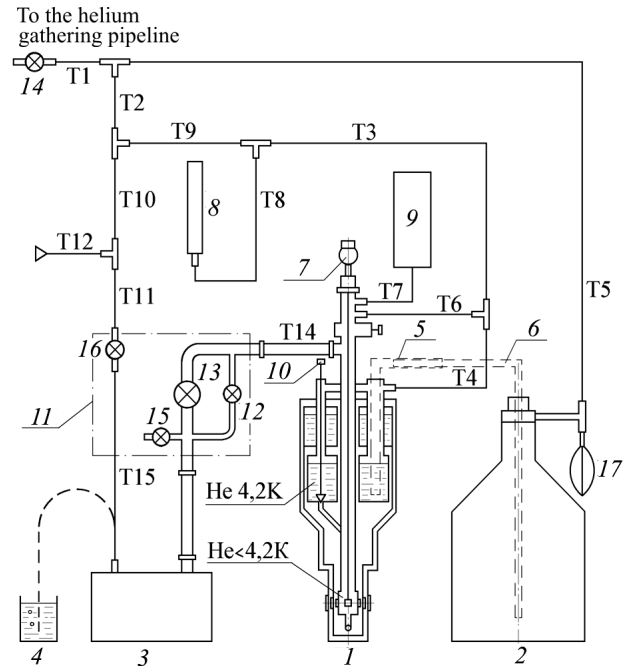


Fig. 4. Scheme of the stand for tests of the cryostat at temperatures below 4.2 K: cryostat (1), vessel with liquid helium (2), vacuum pump (3), vessel with water (4), overflow siphon (5, 6), manipulator (7), water manometer (8), vacuum meter (9), valve of liquid helium supply (10), pumping post (11), Needle valve (cross-section diameter – 3 mm) (12), bellows valve (cross-section diameter – 10 mm) (13), valves (14, 15, 16), rubber ball (17), T1–T15 – rubber tubes

When using liquid cryostats in the temperature control mode in the range 4.2–300 K, the flow of a cryogen in comparison with the flow decreases by 1–2 orders of magnitude and is of the order of 0.1 liter/h, and the stability of temperature maintenance is within 0.1 K and better. Comparative data on the consumption of a cryogen in the mode of reaching low temperatures below 4.2 K are absent. Therefore, we consider the process of achievement of low temperatures on the example of a standard universal optical cryostat of the domestic production [8–12] in more details.

The design of such cryostat is schematically presented in Fig. 3, and the scheme of the test bench is given in Fig. 4.

As mentioned above, by reducing the pressure above the free surface of the cryoagent, it is possible to obtain a temperature below the normal boiling point of the cryoagent itself. As can be seen from the design of the cryostat, there are several

ways to reduce the pressure above the surface of the cryogen:

1. Carry out pumping of cryoagent vapors directly from the tank filled with a cryoagent and, by opening the cryoagent supply valve to the working chamber of the cryostat shaft, to supply the cooled liquid to the test sample.

2. Simultaneous pumping of cryoagent vapors directly from the cryoagent tank and from the cryostat shaft with the cryoagent supply valve open to the cryostat shaft.

3. Pump the cryoagent vapors directly from the working chamber of the cryostat filled with a cryoagent to the level of the location of the object of study with the closed supply valve of the cryoagent to the working chamber of the cryostat.

To maintain a stable temperature of the test object, it is necessary to keep the necessary stable level of a liquid cryoagent in relation to the test object in the working chamber of the cryostat. The known methods of monitoring the level of a liquid cryoagent were developed by various manufacturers (“Cryomagnetics corp” (USA), “RTI Company” (Russian Federation), *etc.*). The level of liquid helium is controlled by the phenomenon of superconductivity, and the level of liquid nitrogen is controlled by capacitive coaxial level sensors, from which two main modifications are distinguished: continuous and segmented ones [14].

To perform the task of measuring the level of liquid cryoagents, a capacitive level meter was created from a coaxial three-tube capacitor with an increased sensitive surface area, the design of which is shown in Fig. 5, and its structural and functional diagram in Fig. 6. Tubes 1, 2, and 3 of different diameters are concentrically installed in each other. Tubes 1 and 3 are soldered to each other by wires 5. Threads 6 are wound along the entire length of tube 2. Threads are wound in turns around the inner and outer surfaces of the pipe walls and fixed with glue.

The level meter works as follows. Let the generated frequency be f_0 . When the capacitor is filled with liquid helium, its capacity increases, and the generated frequency decreases. Changing the oscillation frequency leads to a change in the output voltage of the frequency detector supplied to the DC amplifier and then through the switch to the indicator. The indicator device mounted in the temperature regulator has a scale of 0–100%.

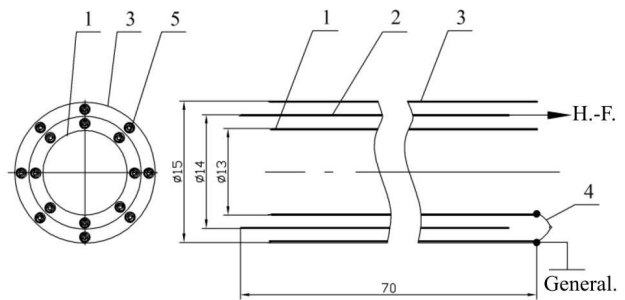


Fig. 5. Design of the level sensor of the segmented type: metal tubes (1, 2, 3), wires (4), threads (5)

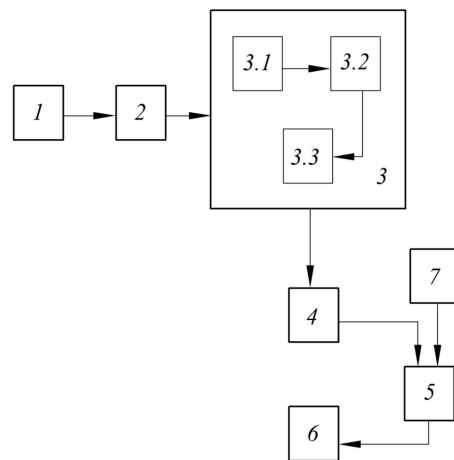


Fig. 6. Block diagram of a coaxial level gauge of the segmented type: capacity level gauge (1), RF generator (2), path of processing of frequency-modulated signals as a part (3): the amplifier-limiter (3.1), the frequency detector (3.2), the preliminary amplifier (3.3), DC amplifier (4), switch (5), indicator (6), PC (7)

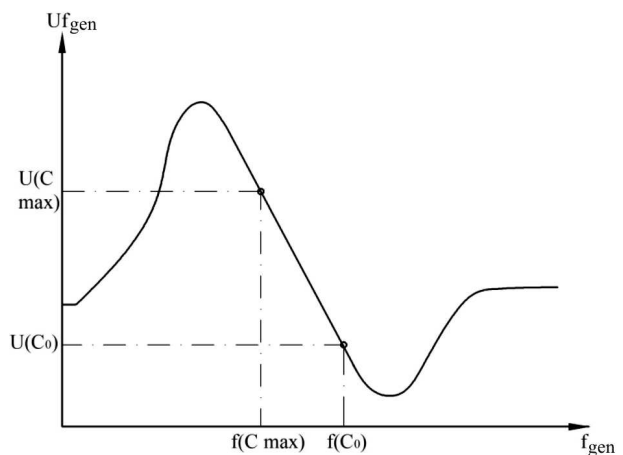


Fig. 7. Amplitude-frequency characteristic of a frequency detector

To record small changes in the frequency ($2\div 3\%$) of RF signals when measuring the level of liquid helium, a resonant measurement method was used, which, in contrast to that used in other developments [13], is more sensitive and accurate [15]. This method consists in connecting the measured capacitor to the LC circuit of a high-frequency sinusoidal voltage generator. The essence of the method is that a change in the frequency of the RF generator is registered by a resonance that coincides in frequency with the measuring circuit. The resonance method allows one to obtain a measurement error up to 0.1%. One of the types of the resonance method is the principle of frequency detection. In Fig. 7, the amplitude-frequency characteristic of such detector is shown. The bandwidth of the frequency detector and its settings are selected so that, in the frequency band from $f(C_0)$ to $f(C_{\max})$, the amplitude of the output signal is a linear function of the frequency.

The capacity of the measured tubular condenser filled with a liquid cryoagent varies from C_0 (gas phase) to C_{\max} (liquid phase). When the generator frequency changes from $f(C_0)$ to $f(C_{\max})$, the output voltage of the frequency detector changes from

$U(C_0)$ to $U(C_{\max})$. The implementation of a similar measurement method is shown in Fig. 6.

The tubular measuring capacitor 1 is connected to the LC circuit of high-frequency generator 2, the frequency of which changes, when it is filled with a cryogenic liquid, because the dielectric constant of the liquid and gaseous phases are different. Next, the RF signal is fed to the processing path of frequency-modulated signals 3, which contains amplifier-limiter 3.1, frequency detector 3.2 and pre-amplifier 3.3. From the output of the pre-amplifier, the voltage is fed to the scaling DC amplifier (DC) 4, and then fed through a computer-controlled switch to the indicator. The gain of the PPP and the dynamic range of the output voltage are selected so that this device allows one to measure the levels of both liquid helium and liquid nitrogen by changing the inclusion factor of the level indicator using a switch. This allows one to use one capacitive sensor to measure different liquids without changing its design.

The results of studies of the consumption of the cryogen in different modes of pumping are shown in Tables 1 and 2.

3. Conclusions

As can be seen from the above results, the most effective way is to pump the cryogen from the working chamber of the cryostat while maintaining the level of a cryogen at a stable level, as it allows one to quickly get, when using liquid helium, a low temperature (1.4 K) for 0.5 h at the liquid helium consumption not more than 0.8 c/h, and, when using liquid nitrogen, 65 K for 0.5 h at a consumption of liquid nitrogen not more than 0.9 liter/h.

1. <http://wwwoxfordinstruments.com>.
2. <http://www.janis.com>.
3. <http://www.cryoindustries.com/products.html>.
4. <https://www.cryomagnetics.com>.
5. <http://www.cryo.ru/>.
6. C.A., Swenson, R.N. Stahl. An efficient cryostat for producing temperatures between 4 and 80 K. *Rev. Sci. Instrum.* **25** (6), 608 (1954).
7. V.S. Medvedev, V.M. Ermakov, P.V. Vodolazskii *et al.* *Thermocomtrolled Cryostat Unit*. A.c. SSSR No. 436334, MKI G 05d 23/30, G 05d 16/06, publ. 15.07.1974. Byul. Izobr. SSSR No. 26, 27.02.1975 (un Russian).
8. I.P. Zharkov, V.M. Ermakov, V.V. Safronov, A.G. Tchmul. The cryosystems UTRECS for the spectral analysis: New methods and approaches. *Vestn. KhGU, Ser. Biofiz. Vestn.* **3** (434), 125 (1999).

Table 1. Consumption of liquid helium in different pumping modes

Pumping mode	Reached minimal temperature, K	Time of reaching of minimal temperature, min	Cryoagent consumption, ltr/hour
From can and column-in	1.5	40	0.5
From column-in	1.4	30	0.25

Table 2. Consumption of liquid nitrogen in different pumping modes

Pumping mode	Reached minimal temperature, K	Time of reaching of minimal temperature, min	Cryoagent consumption, ltr/hour
From can and column-in	59.36	21	0.33
From column-in	49.5	15	0.2

9. I.P. Zharkov, V.M. Ermakov, V.V. Safronov, A.G. Chmul'. Universal thermocontrolled cryostat system. In: *Proceedings of the First Ukrainian Sci. Conference on Physics of Semiconductors UNKN-1* (Odessa State Univ., 2002) (in Russian).
10. I.P. Zharkov, Yu.I. Zhirko, A.N. Ivashchenko, V.V. Safronov, A.I. Solonetskii. Extension of functional potentialities of thermocontrolled cryostats of the flow fluid type. *Prikl. Fizika* No. 3, 110 (2011).
11. I.P. Zharkov, O.M. Ivashchenko, V.V. Safronov, S.V. Pogrebnyak. *Method and Unit for the Temperature Control*. Patent of Ukraine No. 87503, Byul. No. 14, 27.07.2009, MPK G05D 23/30, F25B9/00, G05D23/19, publ. 27.07.2009 (in Ukrainian).
12. http://cryo.ru/index.php?option=com_content&task=section&id=5&Itemid=56.
13. https://sibcontrols.com/ru/chto_takoje_jemkostnyj_urov-njemjer.
14. A.M. Meerson. *Radiomeasurung Texhnique* (Energiya, 1978)(in Russian) [ISBN: 7700243925]. Received 15.07.21

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ОПТИМІЗАЦІЯ ПРОЦЕСУ ОТРИМАННЯ НИЗЬКИХ ТЕМПЕРАТУР В УНІВЕРСАЛЬНИХ РІДИННО-ПРОТІЧНИХ КРІОСТАТАХ

З метою швидкого і економного досягнення низьких температур розглянуто процес їх отримання у вітчизняних універсальних рідинно-протічних кріостатах з використанням рідкого гелію та азоту. Для контролю рівня кріоагента розроблено оригінальну ємнісну систему вимірювання рівня різних типів рідкого кріоагента, яка не має аналогів у світі. Експериментально показано, що найоптимальнішим і найекономічнішим шляхом отримання низьких температур є відкачка парів кріоагента безпосередньо з робочої камери кріостата.

Ключові слова: кріостат, рідкий гелій, рідкий азот, ємнісний рівнемір.