EXPERIMENTAL STUDY OF LIQUID NITROGEN COOLING BY VAPOR PUMPING

Yu. A. Kirichenko, S. M. Kozlov,
O. S. Komarevskii, and V. E. Seregin

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Cooling of liquid nitrogen to the thermodynamic triple point is studied experimentally. The effect of mass of the cooler and pumping regime on cryoagent flow rate is considered.

One of the most widespread methods for cooling cryogenic systems is pumping of the cryoagent vapor, upon which liquid evaporation results, accompanied by a reduction in the liquid temperature. System cooling occurs due to loss of a portion of the cooling agent. This method is distinguished by its simplicity and reliability and has found wide use in systems for long-term preservation of cryogenic liquids [1], in sublimation refrigeration accumulators [2], and for production of low temperatures, and slushlike and solidified coolants.

There exist methods for calculating cooling of cryoagents by vapor pumping which consider variability in their thermophysical properties, thermal leakage and heat capacity of the cooling chamber and cryostat liquid [1-4]. In [5] a study was made of the effect of nonequilibrium in the pumping process (superheating of the liquid relative to the current saturation temperature develops), characteristic for cooling of large volumes of cryoagents, for example, in industrial containers for storing cryogenic liquids. The last study mentioned established the existence of two regimes — a volume boiling regime (at high pumping rates) and a surface evaporation regime, with liquid superheats of up to 14 K being recorded in nitrogen before activation of boiling centers. The superheating can be explained by formation of a boundary layer several mm thick near the phase boundary with the gas, across which the main temperature change occurs.

However systematic experimental studies of the pumping process with consideration of heat influx to the cooled zone and heat capacity of the crystat are lacking.

Using the method of vapor pumping to the thermodynamic triple point, the present study will investigate the effect upon coolant loss of the pumping regime, heat leakage, and the presence of the body being cooled within the cryostat. The experiments were performed with a glass cryostat with working volume of $8 \cdot 10^{-3}$ m^3. To study the effect of mass of the cooler construction upon coolant loss a model cooled object was mounted in the lower portion of the working volume, in the form of a copper (type M1) block, composed of 253 vertically oriented tubes 6 mm in diameter with a wall thickness of 1 mm. The block was arranged in the form of a cylinder 105 mm in diameter with a height (corresponding to the tube height) of 80 mm, with a mass of 2.85 kg, and copper volume of $3.2 \cdot 10^{-4}$ m^3 (0.32 liter). Thus a sufficiently massive copper construction with well developed heat liberation surface was obtained. Two germanium resistance thermometers were installed in the upper and lower portions of the block, near its face surfaces. Similar thermometers monitored liquid temperature below the block, near the phase boundary (two thermometers were installed on a float, one in the liquid in the immediate vicinity of the phase boundary, the other at a depth of 10 mm), as well as temperature of the vapor in the region where it was collected into the pump line. A constantin wire heater was placed on the bottom of the working volume, allowing introduction of a thermal flux of up to 110 W into the liquid. The heater was connected to a B5-21 regulated power supply. Vapor was pumped by an NVZ-20 forevacuum pump. The pumping rate was controlled by a system of coarse and fine adjustment valves. At the pump output the quantity of vapor removed was determined by an RG-40 rotation gas counter. The gas temperature

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at the counter input was monitored by a chromel-alumel thermocouple. Pressure in the working volume was determined by a spring-type reference vacuum meter. The temperature sensor leads passed through a hermetic coupling in the cryostat lid to a regulated voltage source and digital voltmeters. Liquid level and filling of the volume were monitored visually. The measurement scale was calibrated in terms of volume, and provided an accuracy of $2 \cdot 10^{-6}$ m^3.

The experiments were performed in the following manner. Initially with the heater switched off and atmospheric pressure above the liquid, parasitic heat influx into the working volume was determined from the rate of nitrogen loss. The average value of this influx comprised 3.7 W. The required amount of liquid was then placed in the working volume and a constant thermal load produced by the heater, which was maintained constant during the course of pumping to an accuracy of ±2%. In the first series of experiments pumping was commenced by disconnecting the working volume from the atmosphere and simultaneously opening a valve to the vacuum line. In following experiments the valves at the input of the vacuum line were opened beforehand and pumping was commenced by starting the pump. That method was used in order to eliminate an abrupt drop in pressure in the chamber upon its connection to the pre-evacuated vacuum line, which had a significant volume, and also in order to be able to preadjust the pump rate at the initial moment by selecting a cross section such that for given heat production by the heater a constant (atmospheric) pressure was maintained in the volume. At intervals during the pumping the following quantities were recorded: vapor, liquid, and copper block temperature, pressure in the working volume and at the input to the gas counter, liquid level, quantity of vapor removed by pump. Attainment of the triple point was determined from the pressure in the working volume, and also monitored using the cryoagent temperature, as well as visually (appearance of solid phase skin on the liquid surface). In various series of experiments the initial liquid level, heater power, and pumping rate were varied. Experiments were also performed with and without the copper block installed.

Experimental data were processed with a computer. To calculate derivatives finite difference approximations of second order accuracy on a nonuniform grid were used, which allowed measurements at arbitrary time intervals, chosen in accordance with the rate of parameter change. The experimental data were compared to results of a theoretical calculation of the process of nitrogen cooling by vapor pumping, performed by numerical integration of the ordinary differential equation

$$\frac{dT}{d\theta} = \left[\frac{\dot{Q}}{\dot{m}} + u - i_V\right] \Big/ \left[\left(\frac{\partial u}{\partial T}\right)_s (1-\theta) + C_m \frac{m_m}{m_0}\right] \qquad (1)$$

with initial condition $T|_{\theta=0} = T_0 = T_s(p)$. Here m_0 is the initial liquid mass, i_V is the specific enthalpy of the vapor, m_m is the mass of the material in thermal contact with the liquid, C_m is its specific heat, $\theta = (m_0 - m)/m_0$ is the relative fraction of liquid pumped off at the current time, $\dot{Q}$ is the thermal influx into the system. As in processing the experimental data, in performing the numerical calculations the special program of [6] was used for calculation of the thermophysical properties of the nitrogen. In addition to the relative mass fraction of the liquid pumped off θ the quantity $\psi = (V_0 - V)/V_0$, the relative volume fraction of the cryoagent removed, was also used to represent the data. The value of ψ was determined visually from the change in liquid level in the glass cryostat.

It is evident from Eq. (1) that there is a characteristic value of the parameter $(\dot{Q}/\dot{m})_1 = (i_V - u)|_{T=T_0}$, at which the thermal influx is expended completely in vapor formation and $dT/d\theta = 0$. Thus, for nitrogen $(\dot{Q}/\dot{m})_1 = 1.988 \cdot 10^5$ J/kg, and the corresponding value of the parameter $(\dot{Q}/V_V)_1 = 9.159 \cdot 10^5$ J/m³, where $\dot{V}_V$ is the volume pumping rate. If pumping is carried out at a constant volume rate ($\dot{Q}/V_V$ = const), then as a result of decrease in density of the vapor being pumped with approach to the triple point, pumping efficiency will decrease. Therefore there exists a second characteristic value of the parameter $(\dot{Q}/V_V)_2 = \rho_V(i_V - u)|_{T=T_{tp}}$ which when exceeded makes it impossible to cool the system to the triple point. For nitrogen $(\dot{Q}/V_V)_2 = 1.44 \cdot 10^5$ J/m³, while the ratio $(\dot{Q}/\dot{V}_V)_1/\dot{Q}/\dot{V}_V)_2 = 6.37$. The dimensionless parameters $(\dot{Q}/\dot{m}) = (\dot{Q}/\dot{m})/(\dot{Q}/\dot{m})_1$, $(\dot{Q}/\dot{V}_V)_1 = (\dot{Q}/\dot{V}_V)/(\dot{Q}/\dot{V}_V)_1$ and $(\dot{Q}/\dot{V}_V)_2 = (\dot{Q}/\dot{V}_V)/\dot{Q}/\dot{V}_V)_2$ were used to represent the experimental results.

During the course of the experiments the pumping rate was varied over a wide range: for cooling of the system from T_s at atmospheric pressure to T_{tp} from 1 to 150 min were required. Values of the parameters $(\dot{Q}/m)_1$ and $(\dot{O}/\dot{V}_V)_2$ varied from 0.002 to 1.5 and 0.007 to

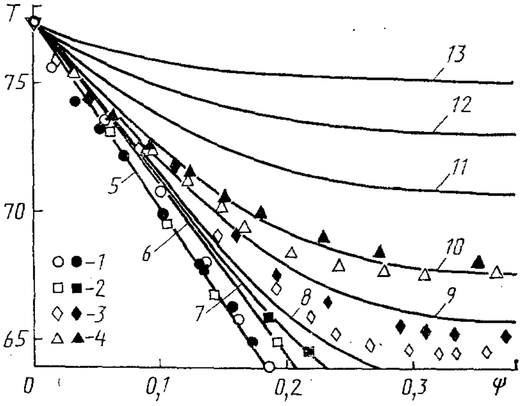

Fig. 1. Nitrogen cooling curves for vapor pumping: 1) Q = 3.7 W; 2) 34.4 W; 3) 65.1 W; 4) 103.8 W. Light symbols, H_0 = 0.214 m, dark, 0.186 m. Mean values of parameter$(\dot{Q}^{\sim}/\dot{V}_V)_2$ for indicated H_0 values: 1) 0.08 and 0.115; 2) 0.56 and 0.79; 3) 1.06 and 1.41; 4) 1.70 and 2.03. Solid lines, calculations with Eq. (1) assuming constant ratio $(\dot{Q}/\tilde{\dot{V}}_V)_2$ during entire pumping process: 5) $(\dot{Q}^{\sim}/\dot{V}_V)_2$ = 0.1; 6) 0.5; 7) 0.75; 8) 1; 9) 1.5; 10) 2; 11) 3; 12) 4; 13) 5, t, K

7.6 respectively. The pumping rate, calculated for vapor parameters at the pump input, ranged from $2\cdot10^{-4}$ to $2.5\cdot10^{-2}$ m^3/sec. We note that over the entire range of thermal influxes and pump rates the liquid state was close to saturation at the current pressure. The mean superheating of the liquid volume at $\dot{V}_V = (4{-}20)\cdot10^{-4}$ m^3/sec did not exceed 0.4 K, and at the maximum pump rates of about $2.5\cdot10^{-2}$ m^3/sec reached 1.3 K. However, it should be noted that the measured superheat values could be affected by the order of reading the sensors during experiment.

The mean temperature differential of the phase boundary surface relative to the current liquid temperature in the volume varied over the range 0.1-0.4 K. Thus, the surface evaporation regime was not realized in the experiments even at the slowest pump rates.

The relatively low superheating of the liquid surface and the absence of a clearly expressed boundary layer on the surface in the experiments can be explained by the fact that in practically all the experiments the active heater was located on the bottom of the working volume, upon which active bubble boiling occurred, insuring intense mixing of the liquid over the entire volume.

Figure 1 shows a comparison of two series of experiments on pumping for identical sets of heat liberation and different initial liquid levels H_0 and theoretical calculations assuming constant volume pumping rate during the entire process. It should be noted that the flowmeter used in the study had a certain critical minimum measurable flow value, equal to $2\cdot10^{-5}$ m^3/sec, below which the value of $\dot{V}_V$ could not be recorded. At $2\cdot10^{-5}$ m^3/sec $< \dot{V}_V < 2\cdot10^{-4}$ m^3/sec the flowmeter indicated artificially low flow rate values. At high flow rates the values obtained with the flowmeter and by decrease in level agreed satisfactorily. Nevertheless for uniformity in processing the $\dot{V}_V$ were calculated from the rate of decrease in liquid level, recorded visually. It is true that this introduced another source of error — increase in liquid level upon volume boiling to the presence of a certain volume of vapor within the liquid. As a result, in the initial pumping period slightly reduced values of relative volume flow rate are obtained (see Fig. 1). Another cause of divergence of the experimental results from theoretical calculations performed with the assumption $(\dot{Q}^{\sim}/\dot{V}_V)_2$ = const, is nonconstancy of this parameter during the course of pumping. In the majority of experiments the value of $(\dot{Q}/\dot{m})$ decreased with passage of time, while the parameter $(\dot{Q}/\dot{V}_V)$ might either decrease of increase. Most typical was a decrease in $(\dot{Q}/^{\sim}\dot{V}_V)_2$ calculated from cold vapor saturation parameters by a factor of 2-5 times during the course of pumping.

In those experiments in which the value of the parameter $(\dot{Q}^{\sim}/\dot{V}_V)_2$ changed insignificantly during the course of pumping, good agreement was obtained between experiment and

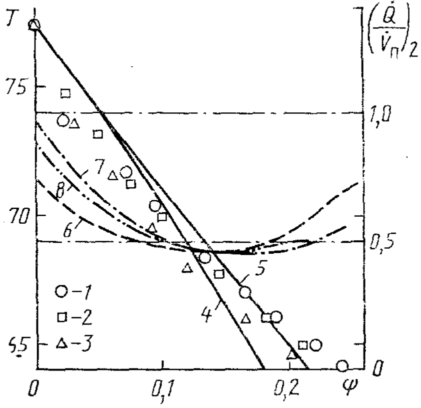

Fig. 2. Nitrogen cooling curves for experiments with close to constant value of parameter $(\dot{\tilde{Q}}/\dot{V}_V)_2$, constant power input Q = 34.2 W and various initial levels: 1) H_0 = 0.3 m and $(\dot{\tilde{Q}}/\dot{V}_V)_2$ = 0.61; 2) 0.216 m and 0.7; 3) 0.157 m and 0.54. Calculations: 4) $(\dot{\tilde{Q}}/\dot{V}_V)_2$ = 0; 5) 0.57 (mean experimental value). Dashed lines show change in parameter $(\dot{\tilde{Q}}/\dot{V}_V)_2$ during pumping: 6, 7, 8, for experiments 1, 2, 3 respectively. T, K.

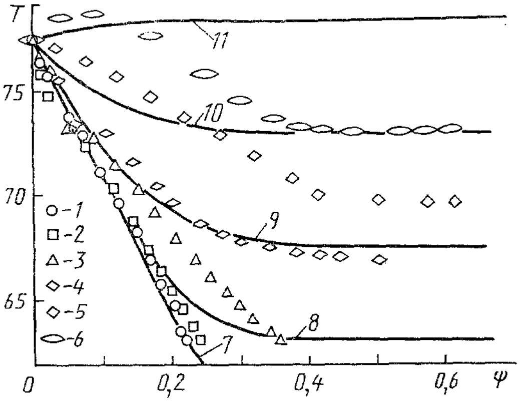

Fig. 3. Cooling curves for experiments with copper mass installed in working volume: 1) $\dot{Q}$ = 6.74 W and $(\dot{\tilde{Q}}/\dot{V}_V)_2$ = 0.43; 2) 10.27 and 0.56; 3) 18.78 and 1.15; 4) 34.15 and 1.94; 5) 64.83 and 3.3; 6) 104.3 W and 3.98. Curves, calculation with Eq. (1): 7) $(\dot{\tilde{Q}}/\dot{V}_V)_2$ = 0.5; 8) 1; 9) 2; 10) 4; 11) 7.

theory. Thus, Fig. 2 shows results of a series of experiments with pumping at constant valve opening, fixed power output from the heater, and various initial liquid levels. It is evident that the relative volume loss of cooling agent for cooling to the triple point is practically independent of H_0 and agrees with the calculated value for $(\dot{\tilde{Q}}/\dot{V}_V)_2$ = 0.57, corresponding to the mean experimental value of this parameter.

Figure 3 shows system cooling curves with the copper mass installed at an initial liquid level of H_0 = 0.216 m and various heater powers selected so that the parameter $(\dot{\tilde{Q}}/\dot{V}_V)_2$ varied from a low value, such that the presence of heat liberation had a weak effect on the cooling curve, up to values exceeding unity, and even the critical value, which comprises 6.17 for nitrogen (when this value is exceeded the derivative (dT/dψ) changes sign). Curves of the change in $(\dot{\tilde{Q}}/\dot{V}_V)_2$ during pumping shown in the same figure are quite typical – there is a decrease with time in this parameter by an average factor of two times. The limiting temperature to which it is possible to cool the system at $(\dot{\tilde{Q}}/\dot{V}_V)_2 > 1$ is determined by the value of $(\dot{\tilde{Q}}/\dot{V}_V)_2$ at large ψ. The figure caption presents mean values over the entire cooling period.

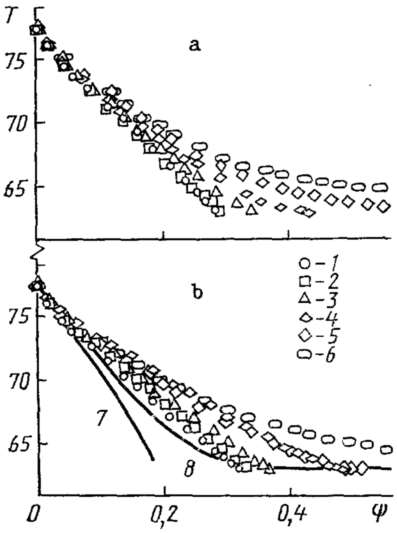

Fig. 4. Comparison of results of two series of pumping experiments with identical heater power levels and pump rates: a) without copper mass; b) with mass installed in working volume: 1) $\dot{Q}$ = 13.8 W, a, $(\widetilde{\dot{Q}/\dot{V}_V})_2 = 0.84$, b, $(\widetilde{\dot{Q}/\dot{V}_V})_2 = 0.91$; 2) 15.8, a, 0.91, b, 0.97; 3) 17.8, a, 0.98, b, 1.06; 4) 19.8, a, 1.16, b, 1.4; 5) 21.8, a, 1.27, b, 1.32; 6) 23.8, a, 1.53, b, 1.55; 7) calculatin with $\Gamma = 0$, $\widetilde{\dot{Q}/\dot{V}_V})_2 = 0$; 8) calculation with $\Gamma = 0.19$, $(\widetilde{\dot{Q}/\dot{V}_V})_2 = 1$

It follows from analysis of the solutions obtained for Eq. (1) that the limiting temperature to which it is possible to cool liquid nitrogen by pumping on the vapor depends on the value of the parameter $(\widetilde{\dot{Q}/\dot{V}_V})_2$ and can be determined from the expression

$$T_{\min} = 63{,}15 + [10{,}2105\,((\widetilde{\dot{Q}/\dot{V}_V})_2 - 1)^{0{,}44052} - 5{,}5398\,((\widetilde{\dot{Q}/\dot{V}_V})_2 - 1)^{0{,}16650}] \qquad (2)$$

with a mean square uncertainty of 0.17%, maximum relative uncertainty of 0.3%, and absolute uncertainty not exceeding 0.25 K. This dependence of minimum temperature on the parameter $(\widetilde{\dot{Q}/\dot{V}_V})_2$ as $\psi \to 1$ is shown in Fig. 3. Equation (2) is applicable over the range $(\widetilde{\dot{Q}/\dot{V}_V})_2 =$ 1-15.

In the series of experiments illustrated in Fig. 3 and subsequent ones the retardation of copper block temperature relative to liquid temperature was studied. In the series described and other experiments at moderate cooling rates, at which the duration of the experiment was 30-90 min, the mean temperature difference between block and liquid comprised only 0.08 K. At the maximum pumping rates of $2.5 \cdot 10^{-2}$ m^3/sec (experiment lasting 1-2 min) a difference of 0.3-0.45 K was reached, i.e., still sufficiently low, which validates the assumption made in the calculations.

Preliminary calculations revealed that the heat capacity of the copper mass cooled with the liquid only has an effect at $(\widetilde{\dot{Q}/\dot{V}_V})_2$ values close to unity for nitrogen. Figure 4 shows results of two series of experiments, in which identical quantities of liquid nitrogen were placed in the working volume (without copper mass, $H_0 = 0.187$ m, with mass $H_0 = 0.216$ m), with evacuation rate at the pump maintained practically constant: $V_p = (3.9{-}4.9) \cdot 10^{-4}$ m^3/sec, and heater power varied such that the mean value of the parameter $(\widetilde{\dot{Q}/\dot{V}_V})_2$ varied about unity. It was determined by calculations that by nitrogen vapor pumping massive copper objects can be cooled from the atmospheric pressure boiling point down to the triple point with $m_m/m_0 \simeq 80$, which corresponds to a ratio of copper volume to initial liquid volume $\Gamma = V_m/V_0 \approx 7$. The experiments of the present study achieved a maximum Γ value of only 0.19, since a well developed heat liberation surface was required, and it was necessary that the entire block be submerged beneath the liquid at the end of pumping. Aside from the experimental data, Fig. 4 also shows calculated curves for the regime parameter range studied. It is evident that on the whole the agreement is satisfactory, although because of change in the parameter $(\dot{\tilde{Q}}/\dot{\tilde{V}}_V)_2$ during pumping direct comparison of the experimental results with and without the copper mass is impossible. Thus, we have performed an experimental study of the process of cooling by cryoagent vapor pumping. The results confirm the validity of the calculation method proposed previously [7]. An analytical expression has been obtained for determination of the minimum attainable temperature as a function of the parameters characterizing the cooling process.

NOTATION

C, specific heat; i, specific enthalpy; m, mass; $\dot{m}$, mass pumping rate; p, pressure; $\dot{Q}$, heat influx; T, temperature; V, volume; $\dot{V}$, volume pumping rate; u, specific internal energy; $\Gamma = V_m/V_0$, geometric parameter; $\theta = (m_0 - m)/m_0$, relative mass fraction of pumped liquid; $\phi = (V_0 - V)/V_0$, relative volume fraction of pumped liquid; ρ, density. Subscripts: 1, parameters corresponding to first characteristic value of ratio $(\dot{Q}/\dot{V}_V)$; 2, parameters corresponding to second characteristic value of ratio $(\dot{Q}/\dot{V}_V)$; 0, initial values; ~, dimensionless parameters; p, parameters at pump; v, vapor; m, value on submerged mass surface; tp, triple point; s, saturation.

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FACTORS AFFECTING THE RELIABILITY OF PRECISION MEASUREMENT OF LIQUID HELIUM TEMPERATURE IN A DYNAMIC REGULATION AND STABILIZATION MODE

A. G. Demishev, V. Z. Suplin,
V. F. Khirnyi, A. F. Ryazantsev,
and I. Yu. Nemish

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A study has been made of anomalies detected during precision measurements of liquid helium temperature, using secondary thermometers in a dynamic mode. An explanation is suggested for the physical nature of these anomalies.

The ever growing interest in precision measurement and stabilization of the temperature of experimental equipment in liquid helium has made it essential to study the heat exchange process and the dynamics of the temperature of liquid helium under variable conditions. The studies were carried out in a KG-100 cryostat [1] as applied to the problem of ensuring that the temperature of an experimental device immersed in the liquid helium remains stable (with an accuracy of the order of 10^{-3} K) for a long time. The heat release of the device during active operation was of the order of 0.2 W (constant thermal background) with a fluctuation

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