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COOLING OF CRYOAGENTS BY THE PUMPING-OUT OF VAPOR

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The cooling of cryogenic liquids by the pumping-out of vapor is considered theoretically, taking account of the variability of the thermodynamic properties, the heat fluxes to the cryostatting zone, and the specific heat of the vessel walls.

Cooling of cryogenic liquids by the pumping-out of vapor is widely used in outfitting sublimational cold-storage units (SCSU) [1], obtaining slushlike cryoagents [2], and simply for reduction in temperature level. In the evaporation of liquid to replace the vapor which has been removed, the remaining mass of liquid is cooled, on account of the latent heat of vaporization, and also as a result of the work done by the vapor on emission from the container.

In designing cooling systems in which pumping-out of vapor is employed, as well as the maintenance posts of SCSU [3], it is necessary to have information on the cryoagent losses on cooling to a specified temperature. An approximate method of calculating the cooling was outlined in [1], under the following assumptions: that the process is equilibrium (the liquid and vapor temperatures are equal to the saturation temperature at the given pressure); the latent heat of vaporization is constant; the heat fluxes to the system are negligibly small in comparison with its heat content; consumption of cryoagent in cooling the cryostat may be neglected; the mass rate of pumping-out is constant. In [4], the cooling of helium on pumping out its vapor was calculated, taking account of the variability of its properties and the external heat fluxes. However, constant mass rate of pumping-out and negligibly small specific heat of the cryostat was assumed. In [5], the cooling of nitrogen, oxygen, neon, and para-hydrogen by pumping-out was calculated, under assumptions analogous to those in [1]. Simple approximations were used for the dependence of the specific heat and the latent heat of vaporization on the temperature, which means that it is possible to obtain simple analytical computational relations.

The present work is devoted to theoretical investigation of the cooling of widely used cryogenic liquids - hydrogen, oxygen, nitrogen, argon, and methane - by the method of evacuating the vapor space, taking account of the variability of their properties, the heat fluxes in the cryostatted zone, and the specific heat of the cryostat walls.

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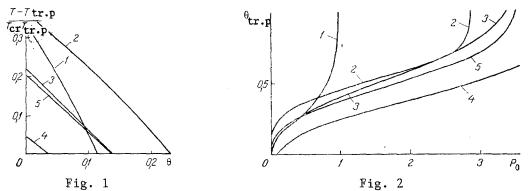


Fig. 1. Cooling curves of cryoagents in the case of no heat fluxes and negligibly small specific heat of the cryostat: 1)  $H_2$ ; 2)  $O_2$ ; 3)  $N_2$ ; 4)  $Ar_2$ ; 5)  $CH_4$ .

Fig. 2. Dependence of the relative losses of cryoagents in cooling to the triple point on the initial pressure  $P_0$ , MPa, in the case of no heat fluxes and negligibly small specific heat of the cryostat; 1-5) as in Fig. 1.

Consider a vessel of mass  $M_w$  partially filled with cryoagent (initial mass  $m_0$ ) in equilibrium with its vapor at an initial pressure  $P_0$  and temperature  $T_0 = T_s(P_0)$ . At time  $\tau_0$ , pumping-out of vapor begins. The following assumptions are made: 1) the temperature of the liquid and vapor at each time are equal to the saturation temperature corresponding to the given pressure; 2) the vapor mass is negligibly small in comparison with the liquid mass (this is true down to very small liquid levels, since the ratio  $\rho_V/\rho_L$  decreases on approaching the triple point); 3) the heat flux to the vessel is constant, and is equal to Q; this is valid, since the temperature difference between the surrounding medium and the cryoagent varies very little in the course of pumping-out; 4) vapor leaves the cryostat at the liquid temperature, with negligible kinetic energy; this is the strongest of the assumptions made, and may be violated in practice, but the heating of the vapor along its path until it leaves the cryostat depends on many factors, such as the magnitude of the heat fluxes, the rate of pumping-out, and the geometry of the system; in addition, the part of the cryostat for which this assumption holds may always be mentally isolated (by moving the upper boundary of the system sufficiently close to the phase interface); 5) the cryostat temperature is equal to the liquid and vapor temperature.

The heat-balance equation for the system takes the form

$$\frac{d}{d\tau}(mu_{\rm L}) + M_{\rm W}C_{\rm W}\frac{dT}{d\tau} = \dot{Q} - \dot{m}_{\rm V}^{\prime}.$$
(1)

Introducing the dimensionless variable  $\theta = \int_{\tau_0}^{t} \dot{m}/m_0 d\tau = (m_0 - m)/m_0$ , the relative mass fraction of liquid pumped out at the given time, Eq. (1) takes the form

$$\frac{d}{d\theta} \left[ (1-\theta) u_{\rm L} \right] + \frac{M_{\rm W}}{m_0} C_{\rm W} \frac{dT}{d\theta} = \frac{Q}{m} - i_{\rm V}, \tag{2}$$

and hence

$$(1-\theta)\frac{du_{\rm L}}{d\theta} + \frac{M_{\rm w}}{m_0}C_{\rm w}\frac{dT}{d\theta} = \frac{\dot{Q}}{\dot{m}} + u_{\rm L} - i_{\rm V}$$

Since saturation is assumed at each instant, it follows that

$$\frac{du_{\mathrm{L}}}{d\theta} = \left(\frac{du_{\mathrm{L}}}{\partial T}\right)_{\mathrm{s}} \frac{dT}{d\theta}$$

Finally, the ordinary differential equation

$$\frac{dT}{d\theta} = \left[\frac{\dot{Q}}{m} + u_{\rm L} - i_{\rm V}\right] / \left[\left(\frac{\partial u_{\rm L}}{\partial T}\right)_{s}(1-\theta) + C_{\rm W}\frac{M_{\rm W}}{m_{\rm 0}}\right]$$
(3)

Material	<sup>T</sup> tr.p <sup>K</sup>	Ptr.p <sup>, bar</sup>	PV tr.p, kg/m <sup>3</sup>	py  <sub>Tb</sub> , kg/m <sup>3</sup>
H <sub>2</sub> O <sub>2</sub> N <sub>2</sub> Ar CH <sub>4</sub>	13,8 54,36 63,15 83,80 90,68	0,070431 0,001490 0,124640 0,689078 0,117435	0,1239 0,0034 0,666 4,05 0,2496	1,338 4,473 4,607 5,767 1,813
Material	$\left(\frac{\dot{Q}}{\dot{m}}\right)_1 \cdot 10^{-5},  \mathbf{J/kg}$	$\left(\frac{Q}{V}\right)_{1} \cdot 10^{-5}, J/$	$\operatorname{m}^{3} \left  \left( \frac{\dot{Q}}{\dot{V}} \right)_{2}, J/n \right $	$\mathbf{n^3} \left  \left( \frac{\dot{Q}}{\dot{V}} \right)_2 \right  \left( \frac{\dot{Q}}{\dot{V}} \right)_1$
H <sub>2</sub> O <sub>2</sub> N <sub>2</sub> Ar CH <sub>4</sub>	4,469 2,125 1,988 1,621 5,110	5,98 9,505 9,159 9,348 9,264	$5,62 \cdot 10^4 \\ 8,55 \cdot 10^2 \\ 1,44 \cdot 10^5 \\ 6,73 \cdot 10^5 \\ 1,362 \cdot 10^6 $	0,0938 0,00087 0,157 0,724 0,147

TABLE 1. Characteristic Parameter Values Determining the Course of Pumping-Out

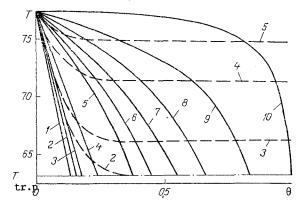


Fig. 3. Theoretical cooling curves of nitrogen T, K, at  $P_0 = 1.013$  bar, as a function of the rate of pumping-out: 1)  $\tilde{Q} = 0$ ; 2) 0.157; 3) 0.25; 4) 0.5; 5) 0.75; 6) 0.8; 7) 0.85; 8) 0.9; 9) 0.95; 10) 1; complex curves) constant mass rate of pumping-out ( $\dot{Q}/\dot{m} = const$ ); dashed curves) constant volume rate ( $\dot{Q}/\dot{V} = const$ ).

with the initial condition

$$T|_{\theta=0} = T_0 = T_s(P_0), \tag{4}$$

describing the temperature variation in the course of pumping-out, is obtained. It is nonlinear, since  $u_L$ ,  $i_V$ ,  $(\partial u_L/\partial T)_s$ , and  $C_w$  depend on the temperature and may only be solved with sufficient accuracy by numerical methods.

The problem in Eqs. (3)-(4) is solved using a procedure which realizes a combination of Adams methods [6]. In calculating the thermophysical properties of the cryoagents, the program of [7] is used. The dependence  $C_W(T)$  for three typical structural materials - copper M1, steel 12Kh18N10T, and aluminum alloy AMg5 - is specified using splines according to the data of [8].

Integration is taken either to  $\theta$  = 1 or to the satisfaction of the condition T = T<sub>tr.p</sub>.

Cooling curves of five cryoagents with the pumping-out of vapor at  $P_0 = 1.013$  bar, Q = 0, and  $M_W = 0$  are shown in Fig. 1, from which it is evident that 11-13% of the initial mass must evaporate for the cooling of hydrogen, nitrogen, and methane from the boiling point to the triple point, around 23% for oxygen, and 3.5% for argon, the boiling point of which exceeds  $T_{tr.p}$  by no more than 3.5 K.

The dependence of the cryoagent consumption in cooling to the triple point in the absence of heat fluxes and with negligibly small specific heat of the cryostat on the initial pressure is shown in Fig. 2, from which it is evident that all the given cryogenic liquids, except for hydrogen, may be cooled by the method of vapor pumping-out to the triple-point temperature  $T_{tr.p}$  at initial pressures exceeding 1 MPa, consuming less than 50% of the initial mass here.

It is evident from Eq. (3) that, for each cryoagent, there is a characteristic value of the parameter  $\dot{Q}/\dot{m}$  at which the heat influx completely compensates the energy consumption in vapor formation and cooling does not occur:  $(\dot{Q}/\dot{m})_1 = (iV - uL)_{T=T_0}$ . Values of  $(\dot{Q}/\dot{m})_1$  and correspondingly  $(\dot{Q}/\dot{V})_1$  at  $P_0 = 0.1013$  MPa are shown in Table 1. Note that, for all the given cryoagents except hydrogen, the mean value  $(\dot{Q}/\dot{V})_1 = 9.32 \cdot 10^5 \text{ J/m}^3$  may be used with an error of no more than 2%. In the

TABLE 2. Maximum Value of  $M/m_0$  at Which Cooling of the System from  $P_0 = 0.1013$  MPa to the Triple Point is Possible with  $\dot{Q} = 0$ 

	Wall material			
Material	copper M1	steel 12Kh18N10T	alloy AMg5	
H <sub>2</sub> O <sub>2</sub> N <sub>2</sub> Ar CH <sub>4</sub>	$ \begin{vmatrix} > 300 \\ \sim 35 \\ \sim 80 \\ \sim 250 \\ \sim 92 \end{vmatrix} $	$ \begin{array}{c c} > 300 \\ \sim 32 \\ \sim 90 \\ \sim 250 \\ \sim 90 \end{array} $	>300 ~17 ~40 ~120 ~48	

pumped decreases in the course of the process, which leads to reduction in its effectiveness. As result, there is a second characteristic value of the parameter:  $(\dot{Q}/\dot{V})_2 = (i_V - i_V)_2$  $u_L)_{T=T_{tr.p}}$ , above wh ich the system cannot be cooled to the triple point. Cooling curves of nitrogen from P<sub>0</sub> = 0.1013 MPa with different values of the parameter  $\dot{Q} = (\dot{Q}/\dot{V})/(\dot{Q}/\dot{V})_1$ are shown in Fig. 3 for the cases of constant mass and volume rate of pumping-out. The value Q = 0.157 corresponds to  $(\dot{Q}/\dot{V})_2$  for nitrogen. If it is possible to cool the liquid to the triple-point temperature at any  $\hat{Q} < 1$  with a constant mass cooling rate, conceivably with large mass consumption, taking account of the decrease in density of the pumped-out vapor leads to cooling curves of qualitatively different form. In the case of a constant volume rate of pumping-out, for  $(\dot{Q}/\dot{V}) > (\dot{Q}/\dot{V})_2$ , the condition dT/d $\theta$  = 0 is satisfied at a certain temperature, and further pumping-out at the same volume rate or with the same heat influx is useless, since it does not lead to further reduction in temperature. As follows from the structure of Eq. (3), taking account of the consumption of cryoagent in the cooling of cryostat has no influence on the value of  $(\dot{Q}/\dot{V})_2$ . As noted in [9, 10], disequilibrium of the evaporation and vapor heating in the pumping-out line may lead to additional losses in cooling efficiency.

For parametric investigation of the cooling of cryoagents by the pumping-out of vapor, taking account of the specific heat of the cryostat, the basic parameters of the given process are determined, together with their possible range of variation. Since  $(\dot{Q}/\dot{V})_1$  and  $(\dot{Q}/\dot{V})_2$  are sufficiently large and they may scarcely be attained in the theoretical operating conditions of SCSU maintenance posts, the scale of  $\dot{Q}/\dot{V}$  is taken to be  $(\dot{Q}/\dot{V})_2$ , its limiting value at which cooling of the system to  $T_{tr.p}$  is still possible; in addition,  $(\dot{Q}/\dot{V})_2$  is fixed for the given cryoagent, and does not depend on  $P_0$ . The dimensionless parameter  $\tilde{Q} = (\dot{Q}/\dot{V})/(\dot{Q}/\dot{V})_2$  is now determined. The characteristic rates of pumping-out of the vacuum pumps employed lie in the range  $\dot{V} = 10^{-3} - 10^{-1} \text{ m}^3$ /sec. The range of variation in heat influx may be estimated as  $\dot{Q} \simeq 0.1$ -10 W. Correspondingly, the characteristic range of variation in  $(\dot{Q}/\dot{V})_2$ , the following values may be chosen for parametric investigation:  $\tilde{Q} = 0.1, 0.3, 0.9$ .

Another parameter of the given process is the ratio of the mass of the structure in thermal contact with the liquid to the initial mass of liquid introduced in the system  $M/m_0$ . Since the specific heat of the structural materials in the temperature range which is of interest here is approximately an order of magnitude less than the specific heat of cryoagents along the saturation line  $C_s$ , it must be expected that the influence of this parameter is sufficiently weak. For quantitative estimation of this factor, calculations are made for the case  $\dot{Q} = 0$ ,  $P_0 = 0.1013$  MPa; in the course of the calculations, the maximum values of  $M/m_0$  for which cooling of the system to  $T_{tr.p}$  is possible are calculated (Table 2). As is evident from the data in Table 2, not only the cryoagent itself but also sufficiently massive structures may be cooled by the method of pumping out vapor. Note also that, since the values of  $C_w$  and  $\rho_w$  are close for copper and stainless steel, one of these materials may be expected in both cases.

The investigation may be undertaken in the most general form if  $M/m_0$  is expressed as  $M/m_0 = \Gamma \rho_W/\rho_{L0}$ , where  $\Gamma$  is the ratio of the volume of the structure in thermal contact with the cryoagent to the initial volume of liquid. This parameter characterizes the structural features of the system. The characteristic range of variation in  $\Gamma$  is estimated for the example of a spherical cryostat (this form is widely used in practice, since it allows the heat flux to the cryostatted zone to be minimized). It is readily evident that  $\Gamma = 3\delta/r$  for a sphere. The ratio  $\rho_W/\rho_{L0}$  for the given cryoagents and structural materials varies in the range 1-130.

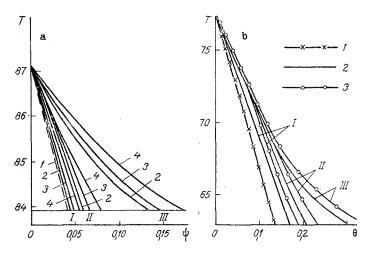


Fig. 4. Influence of the heat fluxes and specific heat of the cryostat on the cryoagent loss in cooling the system by the pumping-out fluid  $\psi$  for argon when  $\dot{Q} = 0$ ,  $\Gamma = 0$  (1),  $10^{-2}$  (2),  $10^{-1}$  (3), 0.5 (4);  $\tilde{Q} = 0.1$  (I), 0.5 (II), 0.9 (III); b) dependence of T, K, on the relative mass fraction of fluid pumped out  $\theta$  for nitrogen:  $\dot{Q} = 0$ ,  $\Gamma = 0$  (1); alloy AMg5,  $\Gamma = 0.5$  (2); steel 12Kh18N10T,  $\Gamma = 0.5$  (3);  $\tilde{Q} = 0.1$ (I), 0.5 (II), 0.9 (III).

Taking the characteristic cryostat dimension  $r = (5-25) \cdot 10^{-2}$  m and wall thickness  $\delta = (0.2-2) \cdot 10^{-3}$  m, the range of variation  $\Gamma = 2 \cdot 10^{-3} - 10^{-1}$  is obtained and, taking account of the presence of reinforcing ribs, the coolant pipeline, and other structural elements in the cryostat, this range must be expanded to  $2 \cdot 10^{-3} - 5 \cdot 10^{-1}$ . Thus, the ratio M/m<sub>0</sub> for real SCSU structures may lie in the range 0.002-65. Of course, for specific combinations of structural materials and cryoagents, this range may be considerably narrowed. Comparison with existing literature data confirms the correctness of the estimates made. Thus, for the KT-7 Soviet SCSU filled with colid nitrogen,  $\Gamma = 0.036$ , M/m<sub>0</sub> = 0.118 [11], while in a US two-component SCSU in a volume with solid argon  $\Gamma = 0.027$ , M/m<sub>0</sub> = 0.124, and with solid CO<sub>2</sub>,  $\Gamma = 0.028$ , M/m<sub>0</sub> = 0.141 [12].

The influence of  $\Gamma$  in the case where argon is the cryoagent is illustrated in Fig. 4a, from which it is clear that the cryoagent consumption in cooling the system to  $T_{tr.p}$  at large  $\tilde{Q}$  may exceed the consumption for the idealized case ( $\dot{Q} = 0$ ,  $\Gamma = 0$ ) more than fourfold; even in the case of moderate heat fluxes ( $\tilde{Q} = 0.1$ ), pronounced increase in the consumption associated with cooling of the cryostat walls made from alloy AMg5 is observed. In the case where a steel or copper cryostat is used, this increase in cryoagent consumption is even more considerable.

A similar picture is seen for methane. This is associated with the higher temperature level for these cryoagents, in which the specific heat of the structural materials is considerable.

Calculations for the case when hydrogen is used as the cryoagent shows that, with increase in Q to 0.9, the liquid consumption on cooling to  $T_{tr.p}$  may be increased by a factor of approximately 1.5. The influence of the parameter  $\Gamma$  and the wall material is slight, however, because of the small specific heat of the materials at hydrogen temperatures.

Analogous calculations for oxygen show that increase in  $\Gamma$  to 0.1 and in  $\tilde{Q}$  to 0.9 has practically no influence on the liquid consumption in cooling the system; this is evidently explained by the low absolute value of  $(\dot{Q}/\dot{V})_2$  for this cryoagent. Note, however, that for oxygen it is quite probable that  $\dot{Q}/\dot{V}$  exceeds the critical value  $(\dot{Q}/\dot{V})_2$ .

In the case of nitrogen, with the maximum chosen values of  $\tilde{Q}$  and  $\Gamma$ , increase in the cryoagent consumption to more than double is possible.

The influence of the material from which the cryostat is made is shown in Fig. 4b for the example of nitrogen. With identical  $\tilde{Q}$  and  $\Gamma$ , the consumption of cryoagent in cooling a steel cryostat is higher than in the case of a cryostat made from aluminum-magnesium alloy,

although the specific heat of AMg5 is higher. This is explained in that the actual cryoagent consumption at fixed  $\Gamma$  is determined by the volume specific heat of the cryostat material, which is higher in steel because of its higher density. In addition, in choosing the material, the higher strength characteristics of steel must be taken into account; this allows structures with lower  $\Gamma$  to be used.

## NOTATION

C, specific heat; i, specific enthalpy; m, liquid mass; ṁ, mass rate of pumping-out; M, cryostat mass; P, pressure; Q̇, heat flux; Q̃ =  $(\dot{Q}/\dot{V})/(\dot{Q}/\dot{V})_1$ , Q̃ =  $(\dot{Q}/\dot{V})/(\dot{Q}/\dot{V})_2$ , dimensionless parameters; r, radius; T, temperature;  $\tau$ , time; V, volume; V̇, volume rate of pumping-out; u, specific internal energy;  $\Gamma = V_W/V_{L^0}$ , geometric parameter;  $\theta = (m_0 - m)/m_0$ , relative mass fraction of pumped-out liquid;  $\psi = (V_0 - V)/V_0$ , relative volume fraction of pumped-out liquid;  $\delta$ , wall thickness of cryostat;  $\rho$ , density. Indices: V, vapor; L, liquid; 0, initial conditions; tr.p, triple point; s, saturation; w, wall; cr, critical point.

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## RAISING THE ACCURACY OF MEASURING THE DIELECTRIC

## PERMITTIVITY OF FLUIDS

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UDC 621.317.33

Dielectric permittivity measurements are performed for certain nonpolar liquid dielectrics with a relative error of 0.05% by using a perfected phase method.

The dielectric permittivity is one of the most characteristic macroscopic dielectric parameters and its knowledge is necessary practically everywhere that such substances and materials are utilized. Exact knowledge of the dielectric permittivity in application to liquid substances permits reliable information to be obtained about the nature of thermal motion of molecules, about the molecular structure (mutual molecule orientation, dynamics

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