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Simple Nitrogen Sublimation Cryostat for the Temperature Range of 77–45 K

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Abstract—A simple nitrogen sublimation cryostat for operating at 77–45 K was designed, built, and successfully tested. The major part of the apparatus is an SDS-6 commercial dewar vessel 6 L in capacity produced by NPO Geliymash. A temperature of 45 K was achieved after 5 h of forevacuum pumping at 5 L/s.

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An abrupt rise in the cost of energy sources and, as result, in the cost of cooling agents, especially hydrogen and helium, made it virtually impossible for ordinary laboratories to carry out investigations in the temperature region between the nitrogen boiling point (63 K) and the hydrogen boiling point (20 K). Cryogenic apparatuses that do not use expensive and deficient cooling agents would be an acceptable approach in this respect.

As the first step in this direction, we designed and tested quite a simple and low-cost nitrogen sublimation cryostat based on a commercial dewar vessel intended for the generation of enriched parahydrogen. There is an experience [1, 2] of the design and performance of glass sublimation and adsorption cryostats intended for operating at 50–35 K. However, practice showed that glass apparatuses are highly inconvenient because of glass brittleness and maintenance difficulties. Metal apparatuses are evidently necessary.

The development and commercialization of SDS-6 and SDS-35 new-generation nitrogen dewar vessels with low heat losses (less than 1/4 Wt) at NPO Geliymash opens the possibility to create sublimation cryostats operating at 77–45 K with the use of a forepump only with a delivery of at least 5 L/s.

The major part of the sublimation cryostat designed by us (Fig. 1) is a SDS-6 dewar vessel 6 L in capacity with a glass fiber throat $60 \times 0.8 \times 250$ mm in size. The wide throat enables quite a rapid cooling of bulky objects.

Inside the dewar, copper chamber *4* (d = 40 mm) filled with a granulated ortho-para conversion catalyst (iron hydroxide on silica gel) is hanged on holder *3*, which is made of a stainless pipe (20×0.4 mm). German silver capillaries *5* and *6* serve for input and output of converted hydrogen. Vacuum vents *7* and *8* serve to control the gas flow rate and pressure in chamber *4*; the gas pressure is measured with a test vacuum meter *9*.

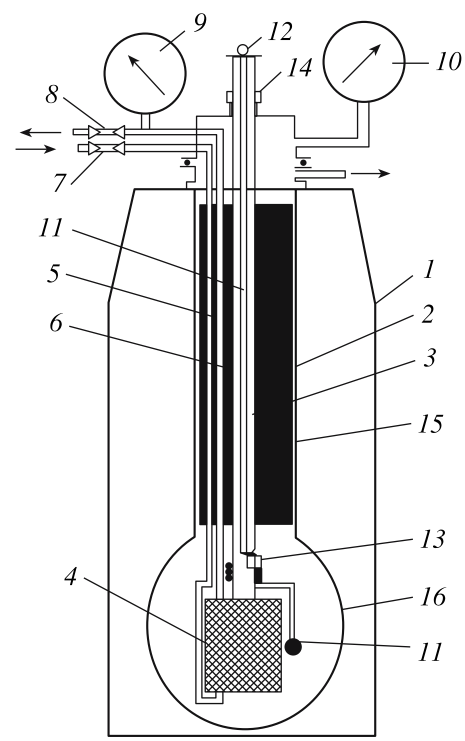

Fig. 1. Sublimation cryostat: (*1*) SDS-6 dewar vessel, (*2*) fiber glass throat, (*3*) reactor holder, (*4*) copper reactor with catalyst, (*5*, *6*) German silver gas inlet/outlet pipes, (*7*, *8*) Du2 vacuum vents, (*9*, *10*) test vacuum meters, (*11*) cold junction of thermocouple, (*12*) sealed joint, (*13*) hole for thermocouple connections, (*14*) mushroom seal, (*15*) heat-insulating glass fiber plug, and (*16*) inner vessel.

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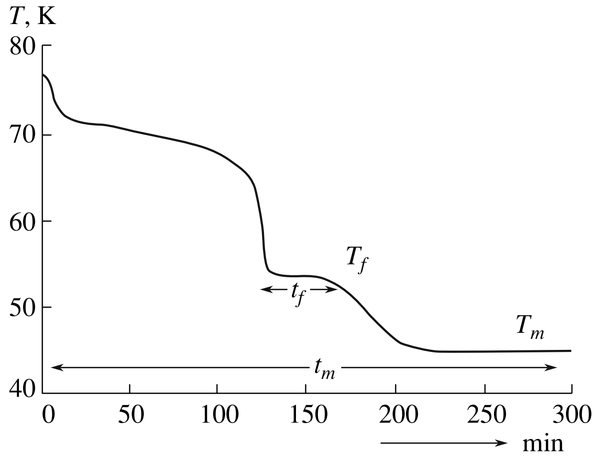

Fig. 2. Reactor cooling dynamics. Notation: t_m—time required for achieving the minimal temperature T_m; t_f—period during which the temperature of the nitrogen triple point (T_f = 63 K) is maintained.

Test vacuum meter *10* is used to control the vapor pressure of liquid nitrogen in the dewar vessel. The temperature in chamber *4* is measured with a copper–constantan thermocouple whose cold junction is mounted to the housing of the chamber. The connections to this junction go from sealed joint *12* inside holder *3*, pass through hole *13* of the holder, and are tightly arranged in it.

Liquid nitrogen vapor is pumped out from inner vessel *16* with a 2NVR-5V forepump through a plastic pipe 20 mm in diameter and 1.5 m long (not shown in Fig. 1). Figure 2 illustrates the cooling dynamics of chamber *4*. The minimal temperature T_m = 45 K is achieved after 5 h. It is noteworthy that this is not the lowest temperature achievable with this dewar. Estimates show that T_m can be reduced to 40 K if using a diffusion pump with a delivery of about 100 L/s and if increasing vacuum pipe diameter to 50 mm.

The cryostat was successfully used to generate enriched parahydrogen. Currently, a system ensuring lower temperatures (to 20 K) is designed on the basis of our cryostat with the use of the Joule–Thompson cycle; this system will generate 99% para-H_2 without using liquid cooling agents at high costs.

The nitrogen sublimation cryostat based on an SDS-6 dewar vessel is an acceptable alternative for investigations at temperatures of 77–40 K without using liquid hydrogen or helium. The use of exchangeable inserts enables one to easily change objects to be investigated.

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