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COMPLEX OF CRYOGENIC APPARATUS FOR FOURIER-TRANSFORM INFRARED *BRUKER VERTEX 70V* SPECTROMETER

Introduction. *Infrared Fourier-transform Bruker spectrometer of Vertex 70v type is known to be a high-resolution research device that has a rather high cost. The device manufacturer provides the possibility of expansion of its functional possibilities due to cooperation with western partners, with the use of additional attachments to the spectrometer and continuous flow cryostats for temperature dependent measurements.*

Problem Statement. *The imported attachments are standard-type, not customized and quite expensive. Therefore, the objective of this research is to expand functional capabilities of the spectrometer using Ukraine-made pieces.*

Purpose. *To design the configuration and to create a cryogenic apparatus for expanding the functional capabilities of Bruker Vertex 70v infrared Fourier-transform spectrometer (Germany).*

Results. *The cryogenic apparatus consisting of the cryosystem with temperature controlled within 2.2—330 K based on a helium continuous flow cryostat with a set of special manipulators and the cryosystem with temperature controlled within 80—500 K based on a combined nitrogen cryostat with vacuum refrigerant duct of continuous flow type and integrated reservoir of liquid cryoagent has been created. The developed complex of cryogenic equipment is fully compatible with Bruker Vertex 70v infrared Fourier spectrometer and enables temperature-dependent measurements of infrared transmission, reflection, and photoluminescence spectra.*

Conclusions. *The designed cryosystems are as good as the best world analogs in terms of technical characteristics and have an essential advantage over them in terms of cryoagent consumption rate.*

Keywords: Fourier-transform spectrometer, temperature-controlled helium, and nitrogen cryosystems.

Bruker Vertex 70v infrared Fourier-transform spectrometer is a high-resolution and very expensive research device. Therefore, the task of expanding its capabilities by efforts of Ukrainian researchers is rather relevant. The device manufacturers, in cooperation with their western partners, envisaged the possibility of expansion of its capabilities with the use of additional attachments to the spectrometer and by equipping it with continuous flow cryostats (*OptistatCF* (*Oxford Instruments*, United Kingdom) [1] or *ST-100-FTIR* (*Janis*, USA) [2], in the case of liquid helium or *OptistatDN* [3] and *VNF-100* [4] of the same manufacturers, respectively, in the case of liquid nitrogen) for cooling the samples with cryoagents. If the sample is placed in vacuum, *OptistatDN-V* [5] and *VPF-100* are proposed to be used [6]. In all these cryostat systems, temperature control is based on the principle of pumping a cryoagent through the thermostated chamber of the cryostat and heating it (the cryoagent) while pumping. Pumping generates vibrations of the test sample while measuring and is a significant disadvantage for spectral measurements.

As previously reported [7], in order to expand the capabilities of *Vertex 70v* infrared Fourier-transform spectrometer manufactured by *Bruker* (Germany), a vacuum *L*-shaped module attachable to the spectrometer has been designed and manufactured. This module enables mounting and adjusting optical mirrors with a diameter of up to 50 mm and attaching a flow helium cryostat with the possibility of temperature control within the range of 2.2—330 K. This cryostat has no above mentioned disadvantages. Also, there have been manufactured special manipulators [8] for the module to make magneto- and electro-optical studies at a low temperature. However, there are many tasks that require temperatures in the range from liquid nitrogen temperature and above room temperature. Such measurements can be useful, for example, for studying temperature dependence of bandgap energy of semiconductor materials, phonon anharmonicity, structural and phase transitions in inorganic and organic compounds, temperature-dependent interactions between complex polymers, and kinetics of chemical reactions. Unfortunately, the previously designed cryostat did not provide such an opportunity. For this reason, it was necessary to create an auxiliary flow cryostat with temperature control within the range from 80 to 500 K, in which the sample is placed in the vacuum chamber of the spectrometer. In this case, the sample is protected from vibrations and loss of desired optical signal due to its reflection and absorption by the optical windows of the cryostat.

Foreign manufacturers of flow cryostats use an external vessel as a source of cryoagent and flexible metallic sleeves through which vibrations are transmitted to the cryostat for pumping the cryoagent through the cryostat and through the pump. Proceeding from this and taking into consideration researches [9, 10], it was decided to use a reservoir integrated into the cryostat as source of cryoagent, from where the cryogenic agent can be pumped through the thermostat chamber under pressure. The functional diagram

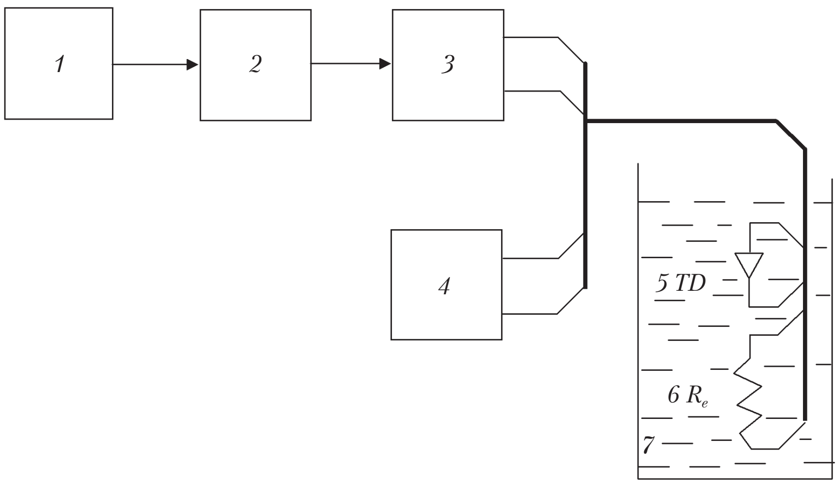

Fig. 1. Flowchart of the pressure-keeping system: *1* — pressure sensor; *2* — generator discriminator; *3* — amplifier; *4* — protective comparator; *5* — thermal diode (protective sensor of liquid nitrogen level); *6* — evaporator resistance (R_e); *7* — cryostat tank with liquid cryoagent

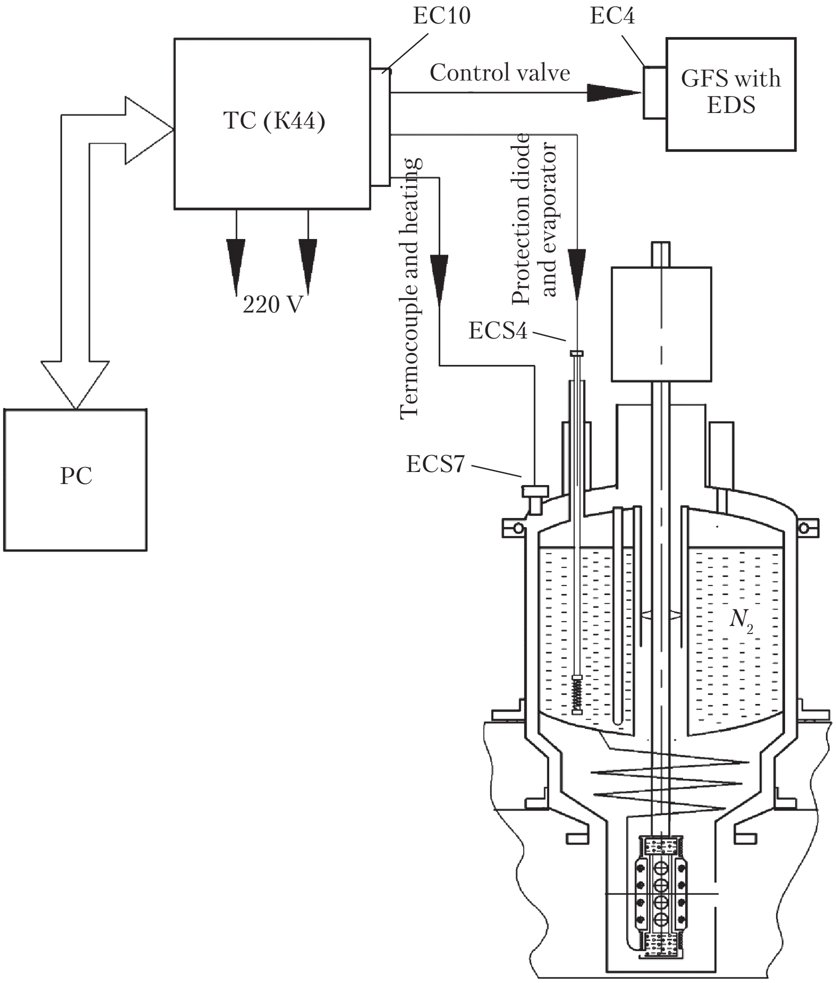

Fig. 2. Flowchart of the flow cryostat system with integrated tank for cryoagent: EC10; EC4; ECS 4; and ECS7 are electric connectors; TC (K44) is temperature controller; GFC is gas flow controller with built-in electrodynamic valve (EDC); PC is personal computer

of the system for creating and keeping the excessive pressure is shown in Fig. 1.

The pressure keeping system includes an inductive pressure sensor, a generator discriminator, a power amplifier, an evaporator, a protective comparator, and a liquid cryoagent level sensor. The inductive pressure sensor contains an inductor with a ferrite core. Varying pressure of the gaseous cryoagent makes the ferrite core move, and, consequently, changes the coil inductance, which, in turn, causes varying frequency of the generator and changing output voltage of the frequency discriminator supplied to the input of the power amplifier. Its load is the evaporator (R_c) mounted on an inserted piece located in the internal reservoir with a cryogenic cryostat fluid. The protective comparator contains a diode protective sensor (PS) mounted on the inserted piece above the evaporator. The circuit controls the level of cryogenic fluid in the cryostat reservoir and switches off the evaporator when the cryoagent level is lower than the permissible one.

Fig. 2 shows a functional diagram of the cryosystem, and Fig. 3 features the structure of nitro-

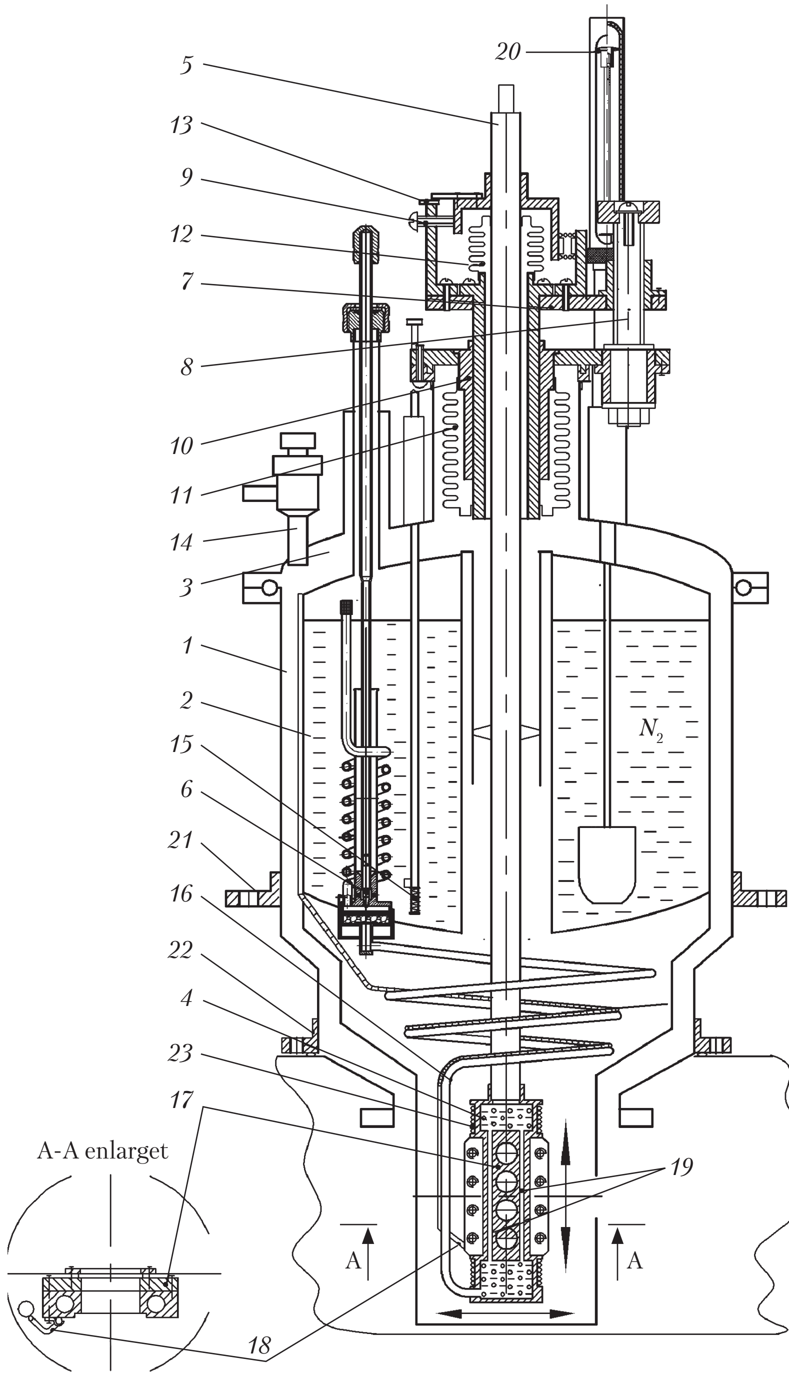

Fig. 3. Scheme of nitrogen cryostat with extended rage of temperature control and built-in vacuum cooling conduit and sample transfer mechanism: *1* — outer case of the cryostat; *2* — nitrogen tank; *3* — cryostat cover; *4* — heat exchanger; *5* — tube for suspending the sample holder; *6* — locking device; *7* — vertical transfer mechanism; *8* — screw; *9* — horizontal transfer mechanism; *10* — guide rod; *11* — bellows; *12* — bellows; *13* — horizontal displacement reference scale; *14* — vacuum valve; *15* — evaporator; *16* — nitrogen feed tube; *17* — sample holder; *18* — thermocouple; *19* — nitrogen transit channels; *20* — level indicator; *21* — flange for fastening to spectrometer; *22* — flange for fastening to attached module; *23* — heater

gen cryostat with extended range of temperature control, which has a built-in vacuum cooler and a mechanism for moving the samples.

When using the cryostat as a stand-alone device, its vacuum space is evacuated by a vacuum pump through the vacuum valve. While working with a cryostat with attached module or with a spectrophotometer, the cryostat and the attachment, or the cryostat and the spectrophotometer have common vacuum space that is evacuated

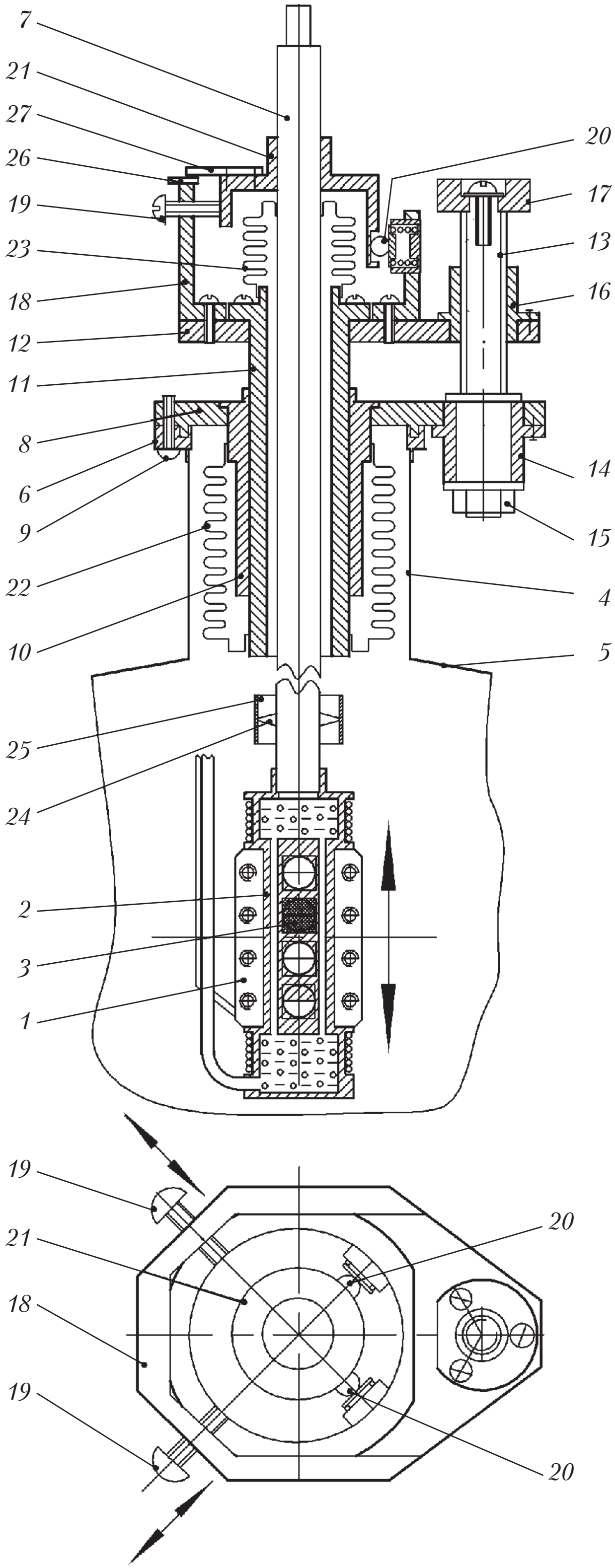

Fig. 4. Detailed scheme of the transfer mechanism built in the nitrogen cryostat: *1* — heat exchanger; *2* — pad; *3* — tested samples; *4* — tube for fastening the transfer mechanism; *5* — cover; *6* — mounting flange; *7* — tube connecting the transfer mechanism with the heat exchanger; *8* — base flange; *9* — screws; *10* — guide rod; *11*— slider; *12* — upper flange; *13* — screw; *14* — plug; *15* — nut; *16* — threaded sleeve; *17* — handle; *18* — cup; *19* — angular tilt screws; *20* — spring stops; *21* — sleeve; *22* — bellows; *23* —bellows; *24* — spacer; *25* — tube; *26* — horizontal displacement reference scales (mm); *27* — arrow indicator of displacement

through the vacuum port of the attachment or the spectrometer.

The cryostat consists of a sectional body, inside of which there is placed a nitrogen tank with a suspended screen surrounding the heat exchanger with a pad. The nitrogen tank is suspended on the cover of the body using three thin-walled tubes made of low thermal conductivity material. The suspension tubes of the nitrogen tank are required for:

- introducing a needle valve into the tank;
- placing a vertical tube of a floating pointer of liquid nitrogen level;
- filling the tank with liquid nitrogen
- mounting an evaporator.

In the cavity of the nitrogen tank there are placed a float, a cryogenic pump, and a nitrogen feed unit. When the needle valve is closed, gaseous nitrogen comes from the tank through the tube, while if the valve is open liquid nitrogen inflows from the tank. The liquid nitrogen level in the tank is visually monitored with the help of the pointer placed inside the glass ampoule.

The heat exchanger with the pad is suspended on the tube fixed in the upper part of the cryostat. It is moved by the transfer mechanism together with the heat exchanger and the pad, both vertically and horizontally. The structure of the transfer mechanism is shown in Fig. 4.

The transfer mechanism is designed to move the heat exchanger (*1*) with the pad (*2*) for mounting samples to be tested (3) both vertically (up-down) and horizontally (see the top view in Fig. 4). The mechanism is fixed at the upper part of the cryostat on the tube (*4*) located in the center of the cover (*5*) that has a mounting flange (*6*). The heat exchanger (1) with the pad (*2*) is located in the tail part of the cryostat and is connected to the transfer mechanism through the tube (*7*) fixed at the upper part of the mechanism.

The transfer mechanism consists of the base flange (*8*) fixed on the flange (*6*) through seals on the screws (*9*). On the flange (*6*), there is mounted the guide rod (*10*) with the vertically moving slider (*11*) that is connected to the upper flange (*12*) and moves together with it with the help of the screw (*13*). The screw is fixed on the flange (*8*) and can rotate by means of the plug (*14*) and the nut (*15*). While rotating, it moves vertically the threaded sleeve (*16*) fixed on the flange (*12*). The screw (*13*) rotates by means of the handle (*17*) and, while rotating, moves vertically the cup (*18*) containing the screws (*19*) and spring stops (*20*) together with the flange (*12*) on which the cup is fastened. Through these screws and stops, the sleeve (*21*) moves with the tube (*7*) fixed therein. Thus, while the handle (*17*) is rotating, the tube (*7*) together with the heat exchanger and the pad moves up or down. The bellows (*22*) are used for sealing the gap between the tube (*10*) and the slider (*11*) from the vacuum space of the cryostat, whereas the bellows (*23*) enable the movement of the tube (*7*), provided vacuum is maintained in the vacuum space of the cryostat.

In addition to moving vertically, the transfer mechanism enables angular tilts of the heat exchanger with the pad relative to the vertical axis of the cryostat in two vertical planes. As a result, the heat exchanger with the pad moves horizontally in two planes.

The angular tilts of the tube (*7*) are realized by the two screws (*19*) that move its upper part through the sleeve (*21*) relative to the spacer (*24*) fixed on the tube (*7*) and tilting with it in the tube (*25*) fastened on the inner side surface of the nitrogen tank of the cryostat. The part of the tube (*7*), which is above the spacer tilts to one side while the part of the tube below the spacer leans in the opposite direction. To change the tilt of the tube to the diametrically opposite one, the screws (*19*) are rotated to in the contrariwise with respect to the initial direction. The spring stops (*20*) mounted against the screws (*19*) bump into the sleeve (*21*) on the opposite side. These tilts have an angle less than 1° and can be considered linear displacement (instead of arc one) of the tube (*7*) with the heat exchanger and the pad. To measure the horizontal displacements of the heat exchanger with the pad on the cup (*18*), two scale bars (*26*) are mounted at an angle 90° along the displacement directions. On the sleeve (*21*), there are the two pointers (*27*) that measure the linear displacements of the heat exchanger with the pad relative to the scale bars (*26*).

Within the range of 80—300 K, temperature of the sample fixed to the heat exchanger with the pad is controlled by varying the rate of feed of the cryoagent into the heat exchanger and by heating the heat exchanger by electric heater. The flow of cryoagent coming from the nitrogen tank to the heat exchanger channels through the feeding tube and the tube on which the heat exchanger is suspended to the gas flow controller (GFC) is controlled by a temperature controller that sends a control signal through the electrical connectors EC10 and EC4 to the electrodynamic valve (EDV) mounted in the GFC at the outlet through which the cryogenic gas stream escapes from the cryostat. The heating of the heat exchanger is controlled by a temperature controller (TC) that supplies voltage to the electric heater.

The program for varying the temperature is given by a personal computer connected to the TC. To measure the temperature, on the pad with the sample, there is mounted a thermocouple connected to the TC via ECS 7 connector on the cryostat.

To increase the pressure of nitrogen vapor, in the tank of the cryostat, there is installed an evaporator that affects the rate of flow of liquid nitrogen into the heat exchanger. To protect the evaporator from overheating, if the level of liquid nitrogen in the tank falls below the upper part of the evaporator, there is a protective diode that is activated when the level of liquid nitrogen drops below the permissible one and triggers a signal to the TC via ECS4 connector to switch off the evaporator. Within the range of 300—500 K, temperature on the pad with the sample is controlled by TC that supplies voltage only to the electric heater on the heat exchanger.

The cryostat enables pumping nitrogen vapor to achieve low temperatures (up to 65 K). In this case, it is possible to extinguish vibrations of the sample by putting a mechanical isolation between the pump and the cryostat in the form of a dense reinforced rubber corrugated pipe located at the outlet of the cryostat to which flexible corrugated sleeve is tightly connected. The other end of this sleeve is tightly connected to a reinforced corrugated pipe tightly mounted on the evacuating junction pipe of the cryostat. In addition, the pump is mounted on the floor through a thick rubber porous plate to reduce vibrations such as a running pump.

The technical characteristics of the transfer mechanism are as follows.

- 4 holes for mounting the samples;
- the possibility of up and down shift by ± 11 mm; and
- the possibility of horizontal shift in two planes for ± 3 mm.

The technical characteristics of the cryosystem are given in Table.

Technical parameters of the Cryosystem

Range of temperature control, K	65 ÷ 80 ÷ 500
Cryoagents	Liquid nitrogen
Liquid nitrogen consumption:	
while cooling down the cryostat, cm³, at most	500
while keeping a temperature of 80K, cm³/hour, at most	150
while keeping a temperature of 65 K, см³/hour, at most	250
Continuous operation at a temperature of 65 K without refill with cryoagents, hours, at least	4
Continuous operation (under supply of a power of at most 0.1 W from outside) at a temperature of 80 K, hours	8
Volume of the chamber with liquid nitrogen, cm³	2500
Dimension of the heat exchanger, mm	Ø 36 × 90
External dimension of the cryostat, mm	Ø 224 × 604
Weight of cryostat, kg	6
Parameters of the electric heater mounted on the heat exchanger:	
Resistance, ohm	70
Material	Nickel-chromium, Ø 0.14 mm $L = 2$ m

Thus, to expand the capabilities of *Vertex 70v* infrared spectrometer manufactured by *Bruker* (Germany), a flow cryostat with temperature control within 80—500 K and with the sample located in the spectrometer vacuum chamber has been developed. The designed cryostat enables to avoid problems associated with the sample vibration and the loss of desired optical signal due to its reflection and absorption by optical windows of the cryostat, and has as good capabilities as its best foreign analogs.

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КОМПЛЕКС КРІОГЕННОЇ АПАРАТУРИ ДЛЯ ІНФРАЧЕРВОНОГО ФУР'Є-СПЕКТРОМЕТРА *BRUKER VERTEX 70v*

Вступ. Відомо, що інфрачервоний Фур'є-спектрометр *Bruker Vertex 70v* є приладом дослідницького класу з високою роздільною здатністю, проте є високовартісним. Виробниками приладу, завдяки кооперації із західними партнерами, передбачено збільшення його функціональних можливостей за рахунок розширення спектрального діапазону за допомогою додаткових приставок до спектрометра та оснащення кріостатами продувного типу для температурнозалежних вимірювань.

Проблематика. Додаткова імпортна апаратура виготовляється стандартною, не враховуючи особливості кожного експерименту і, в свою чергу, має високу ціну. Тому завдання розширення функціональних можливостей спектрометра вітчизняними засобами замість імпортованих є актуальним.

Мета. Розробка конструкції та виготовлення комплексу кріогенної апаратури для розширення функціональних можливостей інфрачервоного Фур'є-спектрометра *Vertex 70v* виробництва *Bruker* (Німеччина).

Результати. Створено комплекс кріогенної апаратури у складі терморегульованої у діапазоні 2,2—330 К кріосистеми на базі гелієвого кріостата рідинно-проточного типу з набором спеціалізованих маніпуляторів, а також терморегульованої у діапазоні 80—500 К кріосистеми на базі комбінованого азотного кріостата з вакуумним холопроводом проточного типу та інтегрованим резервуаром рідкого кріоагента. Розроблений комплекс кріогенної апаратуру є повністю адаптованим для роботи з інфрачервоним Фур'є-спектрометром *Bruker Vertex 70v* та забезпечує можливість проведення температурно-залежних вимірювань спектрів інфрачервоного пропускання, відбивання та фотолюмінесценції.

Висновки. Характеристики розроблених кріосистем за фунціональними показникам не поступаються кращим західним аналогам, а за витратами кріоагента мають суттєві преваги.

Ключові слова: Фур'є-спектрометр, терморегульовані гелієва, азотна кріосистеми.

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КОМПЛЕКС КРИОГЕННОЙ АППАРАТУРЫ ДЛЯ ИНФРАКРАСНОГО ФУРЬЕ-СПЕКТРОМЕТРА *BRUKER VERTEX* 70*v*

Введение. Известно, что инфракрасный Фурье-спектрометр *Bruker Vertex* 70*v* является прибором исследовательского класса с высокой разрешающей способностью, но является дорогостоящим. Изготовители прибора, благодаря кооперации с западными партнерами, предусматривали расширение его функциональных способностей при помощи дополнительных приставок к спектрометру и оснащения криостатами продувного типа.

Проблематика. Дополнительная импортная аппаратура изготавливается стандартной, не учитывая особенностией каждого эксперимента и, в свою очередь, имеет высокую цену. Поэтому задача расширения функциональных возможностей спектрометра отечественными средствами вместо импортных является актуальной.

Цель. Разработка конструкции и изготовление комплекса криогенной аппаратуры для расширения функциональных возможностей инфракрасного Фурье-спектрометра *Vertex* 70*v* производства *Bruker* (Германия).

Результаты. Создан комплекс криогенной аппаратуры в составе терморегулируемой в диапазоне 2,2—330 К криосистемы на базе гелиевого криостата жидкостно-проточного типа с набором специализированных манипуляторов, а также терморегулируемой в диапазоне 80—500 К криосистемы на базе комбинированного азотного криостата с вакуумным хладопроводом проточного типа и интегрированным резервуаром жидкого криоагента. Разработанный комплекс криогенной аппаратуры является полностью адаптированным для работы с инфракрасным Фурье-спектрометром *Bruker Vertex* 70*v* и обеспечивает возможность проведения температурно-зависимых измерений спектров инфракрасного пропускания, отражения и фотолюминесценции.

Выводы. Характеристики разработанных криосистем не уступают лучшим мировым аналогам по функциональным показателям, а по затратам криоагента имеют существенные преимущества.

Ключевые слова: Фурье-спектрометр, терморегулированные гелиевая, азотная криосистемы.