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EXPANSION OF BRUKER VERTEX 70v FTIR SPECTROMETER CAPABILITIES



To expand the capabilities of VERTEX 70v Bruker spectrometer (Germany), a vacuumized L-type module attachment to the spectrometer has been designed and manufactured. The proposed configuration enables to mount and to adjust optical mirrors with a diameter of up to 50 mm and to couple the attachment to cryostat with temperature control within the range from 2 to 330 K.

The research has been carried out within the framework of instrument-making program of the NAS of Ukraine, grant # P- 2/16-40.

Keywords: spectrometer, module attachment, and cryostat.

Infrared (IR) Bruker Vertex 70v Fourier Spectrometer (Germany) is a high-resolution research class spectrometer. Due to the option of extending the spectral range with the help of additional configurations, the Bruker Vertex 70V infrared spectrometer enables measurements from terahertz to near-infrared ($30\text{--}12500\text{ cm}^{-1}$) spectral bands.

The mentioned infrared spectrometer is based on patented Bruker interferometer with rock solid mirrors. It contains two retro-reflex cubic angular mirrors mounted on an inverted pendulum mechanism, the periodic oscillation of which around the equilibrium position changes the path difference of interferometer arms. Because of this design, the slope and mechanical displacement of interferometer mirrors are limited, which ensures a high resolution and stability of the device as compared with the Michelson interferometers.

The data from the spectrometer detector are taken off and digitized using a two-channel 24-bit delta-sigma ADC (ADC DigiTect™). Special OPUS software helps to control the spectrometer and to record the interrogated Fourier transformation.

Vertex 70v spectrometer model provides the ability to make measurements in vacuum, which enables to almost completely eliminate the atmospheric disturbances (caused by water vapor or CO_2) in the resulting spectra. It should be noted that the pumping of spectrometer is more efficient than its blowing with inert gases or use of desiccants. The configuration enables to pump out both the whole device and the sample holder or the optical bench separately. Pressure sensors are provided for monitoring pressure in the sample holder and in the optical bench. Five ports for input infrared radiation and two ports for output significantly extend the spectrometer functionality. Through these ports, additional optical components or accessories can be connected to the spectrometer.

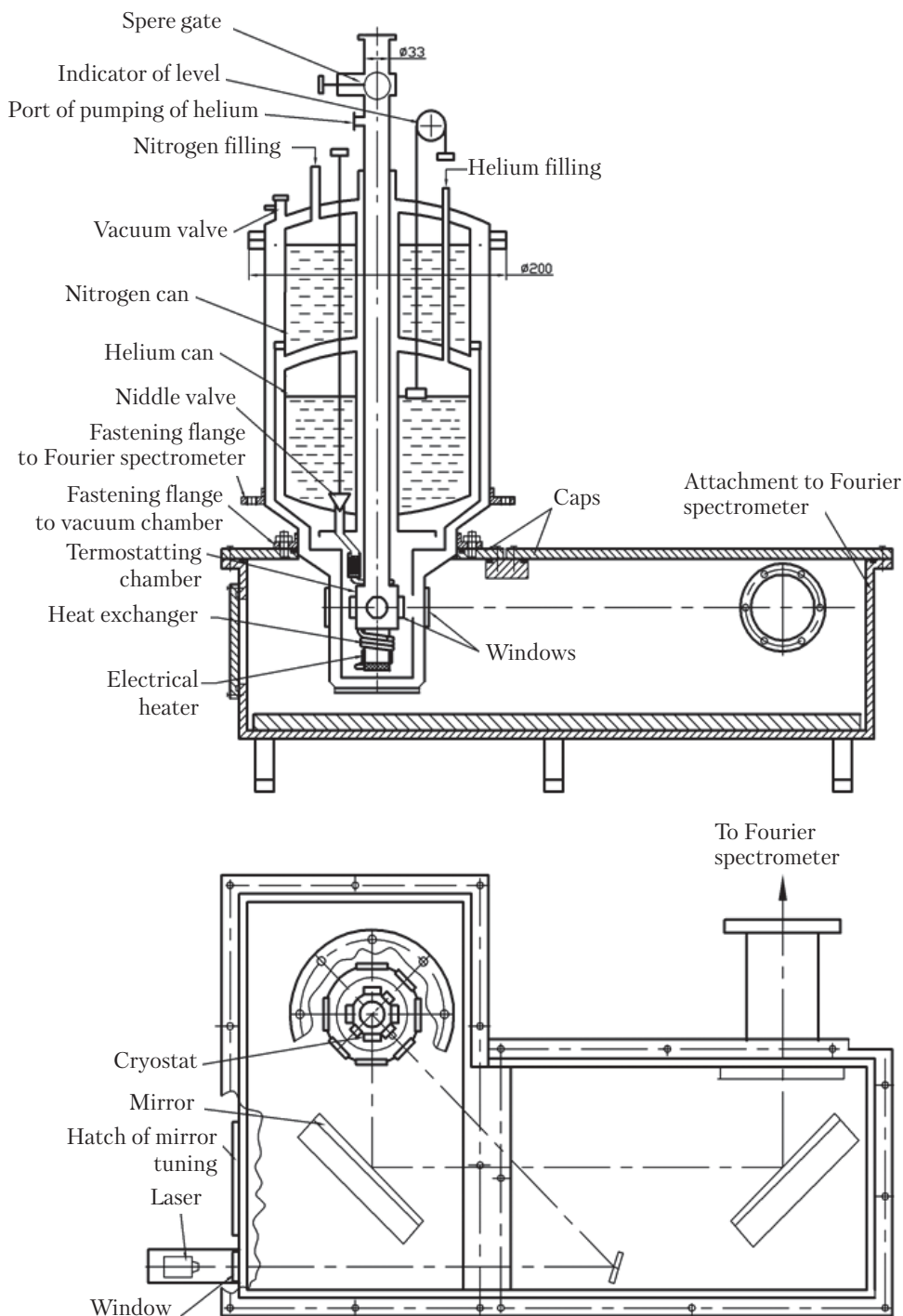


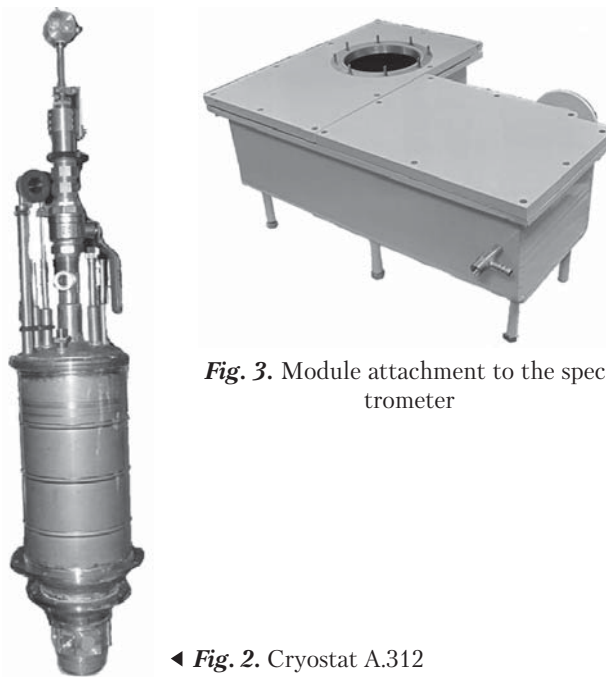
Fig. 1. Layout of module attachment with embedded cryostat

For low-temperature measurements the spectrometer can be equipped with *Optistat* CF cryostat (*Oxford Instruments*, UK) with contact cooling and blowing with refrigerant vapors. This cryostat enables to adjust the sample temperature in the range from 3.8 to 300 K with an accuracy of 0.1 K. Its disadvantage is sample vibration during the measurements while pumping refrigerant vapor, whereas the continuous flow cryostats manufactured at the Institute of Physics Ukraine do not have this shortcoming.

The research is aimed at expanding the functionality of the Bruker Vertex 70v Infrared Bright Spectrometer, in particular, at providing spectral measurements of optical radiation, transmission, absorption, and reflection in the wavelength range of 0.8–330 μm and at eliminating the aforementioned disadvantage for low-temperature studies. In order to achieve this goal, a vacuum unit for the given spectrometer model has been designed and manufactured for recording the optical radiation spectra; also, a multi-purpose temperature-controlled cryostat of continuous flow type adapted to this spectrometer, with an operating temperature range from 2 to 330 K and special sample holders has been designed and manufactured.

Today, both European [1, 2], and Russian manufacturers [3] offer attachments to expand the capabilities of IR spectrometers. Unfortunately, there are no such proposals from Ukrainian manufacturers, since the domestic manufacturer of spectrometers, OJSC *Selmi* (Sumy), has shut down. For this reason, domestic researchers have to buy these attachments abroad, which requires considerable funds. Therefore, one of the tasks was to show the possibility of developing and manufacturing domestic prefabricated attachments for the Bruker Vertex 70v Infrared Fourier Spectrometer.

The developed vacuum module attachment (Fig. 1) enables to measure the optical radiation spectra in the range of 0.8–330 μm . It is equipped with a vacuum flange for commuting with spectrometer, with the flange having a variable opti-



◀ Fig. 2. Cryostat A.312

Fig. 3. Module attachment to the spectrometer

cal window. Due to this the user can select a window with optimal optical parameters for the studied part of the spectrum.

It should be noted that the technical parameters of designed attachment match the best foreign counterparts.

The essential difference of the designed vacuum module, as compared with foreign counterparts, is its connectability to the device vacuum system, which eliminates the need to use a window on the connecting flange. This enables to extend the measurement range to the far IR (terahertz). In addition, the device vacuuming effectively eliminates the atmospheric factor (CO_2 and water vapor) influence on the spectral measurements.

The integrated multi-purpose helium cryostat embedded into the vacuum module greatly expands the Bruker Vertex 70v capabilities as it enables to perform low-temperature optical measurements in the temperature range 2–330 K. In addition, it can be mounted in the standard cuvette compartment of the spectrometer to measure the absorption, transmission, and reflection

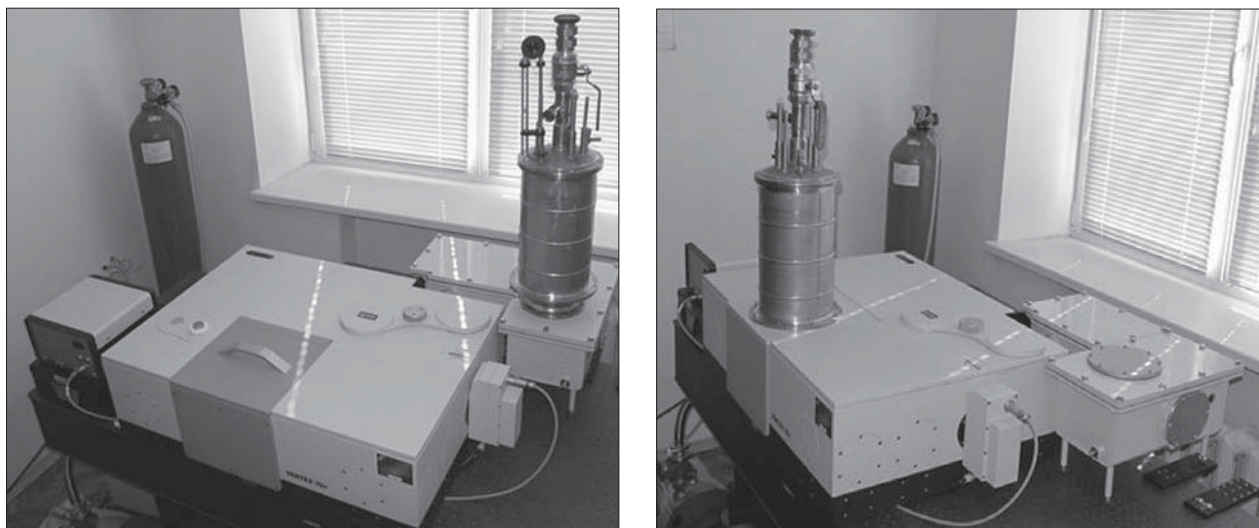


Fig. 4. Module attachment with embedded cryostat and spectrometer mounted in various configurations

optical spectra. The helium cryostat design makes it possible to use it both in the device and in the vacuum photoluminescent module. Due to the original solution [3, 4], both with liquid helium and liquid nitrogen can be used as main cryogenic agent.

In addition to optical measurements, the cryostat designed enables combined magnetic and electro-optical measurements. For implementing the magneto-optical and electro-optical research, it is equipped with specialized manipulators.

The manipulator for electro-optical studies makes it possible to work with samples in the standard Plcc 20 pins case (plastic leadless chip carrier), as well as with flat samples of arbitrary shape having up to 15 mm in diameter.

The magneto-optical manipulator enables to use neodymium magnets with a diameter of 20 mm to create radial and axial magnetic field with an induction of up to 1 T.

The cryosystem technical parameters are given in Table 1.

Technical Parameters of UTREX Cryosystem

Temperature range, K	1.6–4.2–330 77–330
Cryogenic agents	Liquid helium, liquid nitrogen

Liquid helium consumption:	
a) while cooling the cryostat, cm ³ , at most	≤500
b) for keeping a temperature of 1.6 K, cm ³ /hour, at most	≤150
c) for keeping a temperature of 4.2 K, cm ³ /hour, at most	≤250
Continuous work time at 1.6 K without cryogenic agent refilling, hours, at least	≥6
Continuous work time (with a capacity of up to 0.1 W fed from outside) at 4.2 K, hours	8
Liquid nitrogen tank volume, cm ³	1800
Liquid helium tank volume, cm ³	3000
Cryostat charge tube diameter, mm	33
Thermostat chamber dimensions, mm	Ø33 × 60
Cryostat external dimensions, mm	Ø226 × 875
Cryostat tail diameter, mm	Ø90
Distance from the bottom to the horizontal optical axis of cryostat windows, mm	61
Optical entries:	
a) two ZnSe (0–180°) windows:	
clear aperture of cold windows, mm	16
external windows with these positions, mm	37
b) two KRS-5 (90–270°) windows:	
clear aperture of cold windows, mm	16
of external windows with these positions, mm	37
c) three KU-1 (0–45–135–225°) fused quartz windows:	
clear aperture of cold windows, mm	9
clear aperture of external windows with these positions, mm	22

Weight:	
a) Cryostat, kg	11.2
b) Manipulator №1, kg	1.13
c) Manipulator №2, kg	0.7
Electric heater parameters:	
a) Electric heater mounted on the outer wall of thermostat chamber:	
Resistance, ohm	100
Material	nickel-chromium, Ø0.14 mm; L = 1.5 m
	nickel-chromium, Ø0.14 mm; L = 0.2 m
b) electric heaters mounted on the manipulators in the place of sample location:	
Resistance, ohm	21
Material	nickel-chromium, Ø0.14 mm; L = 0.3 m
Voltage on heaters connected in series, V	0–40
Working pressure of burst discs of safety valves, Pa	5–7×10 ⁴

REFERENCES

1. *Specifications of laboratory infrared Fourier spectrometers*. URL: <https://www.bruker.com/ru/products/infrared-near-infrared-and-raman-spectroscopy/ft-ir-routine-spectrometers/tensor/technical-details.html> (Last accessed: 04.04.2017).
2. *Attachments for IR Fourier spectrometers*. URL: <http://www.intertech-corp.ru/aboutproduct.asp?gr=-15&subgr=33&prid=134>; <http://okb-spectr.ru/products/es/modern/> (Last accessed: 04.04.2017).
3. *Ukrainian Patent on an invention No. 87503*. Zharkov I.P., Ivaschenko O.M., Safronov V.V., Pogrebniak S.V. A method and device for adjusting of temperature. Bul. No. 14 from 27.07.2009. IPC G05D 23/30, F25B9/00, G05D23/19, published. 27.07.09.
4. Russian Federation patent on an invention No. 2366998. Zharkov I.P., Ivaschenko O.M., Safronov V.V., Pogrebniak S.V. Method of adjusting and stabilizing of temperature and device for his realization Bull. No. 25 from 10.09.2009, IPC G05D 25/30.

Recieved 12.04.17

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РОЗШИРЕННЯ ФУНКЦІОНАЛЬНИХ
МОЖЛИВОСТЕЙ ІНФРАЧЕРВОНОГО
ФУР'Є-СПЕКТРОМЕТРА BRUKER VERTEX 70v

З метою розширення функціональних можливостей інфрачервоного Фур'є-спектрометра Vertex 70v виробництва «Bruker» (Німеччина) розроблено конструкцію та виготовлено вакуумований Г-подібний модуль-приставку до нього. Запропонована модель дає можливість встановлювати і юстиувати оптичні дзеркала діаметром до 50 мм та приєднувати до неї терморегульований в діапазоні 2-330 К кріостат.

Роботу виконано в рамках програми наукового приладобудування НАН України, грант П-2/16-40.

Ключові слова: Фур'є-спектрометр, модуль-приставка, кріостат.

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РАСШИРЕНИЕ ФУНКЦИОНАЛЬНЫХ
ВОЗМОЖНОСТЕЙ ИНФРАКРАСНОГО
ФУРЬЕ-СПЕКТРОМЕТРА BRUKER VERTEX 70v

С целью расширения функциональных возможностей инфракрасного Фурье-спектрометра VERTEX 70v производства фирмы «Брукер» (Германия) разработано конструкцию и изготовлено вакуумированный Г-образный модуль-приставку к нему. Предлагаемая модель дает возможность устанавливать и юстировать оптические зеркала диаметром до 50 мм и присоединять к ней терморегулируемый в диапазоне 2–330 К криостат.

Работа выполнена в рамках программы научного приборостроения НАН Украины, грант П-2/16-40.

Ключевые слова: спектрометр, модуль-приставка, криостат.