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SPECTRAL APPARATUS WITH A CRYOGENIC, HIGH-THROUGHPUT, MULTIPASS GAS CELL FOR STUDIES OF ABSORPTION OF RADIATION BY GASEOUS MEDIA

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Spectral systems with an MKhK-6 cryogenic, high-throughput, multipass gas cell for studying the absorption spectra of gaseous media with high spectral resolution in the 0.1–6 μm range at pressures of 100 to $5 \cdot 10^6$ Pa and temperatures of 180–300 K are discussed. Their use in measurements of spectral absorption coefficients, temperature dependences of the spectral transmission function, and parameters of spectral absorption lines is examined.

Keywords: *absorption spectrum, spectral absorption coefficient, spectral transmission function, cryogenic multipass gas cell.*

Many applied problems associated with radiative transfer in the atmospheres of planets and with radiative heat transfer require information on the temperature dependences of the spectral transmission functions of optically active gaseous components in electronic and rotational-vibrational bands, rotational-vibrational bands, and pressure induced absorption bands owing to the effect of intermolecular collisions on molecular quadrupole moments [1–3]. Studies in the UV and, especially, in the vacuum ultraviolet require special radiation sources for spectral calibration and calibration of measured spectra, along with vacuum equipment in all the components of measurement systems in order to eliminate atmospheric absorption effects.

In this paper we discuss high-resolution spectral measurement systems for the 0.1–6 μm range with a cryogenic high-throughput multipass gas cell. They supplement previously developed spectral systems [3–6] with a capability for study of gaseous media at lower temperatures and in the vacuum and near ultraviolet, and they can be used to analyze the contents o f industrial atmospheric effluents and process media by optical spectral methods [7, 8].

Two spectral systems with a high-throughput MKhK-6 cell have been developed for experimental studies of molecular absorption by gaseous components of the atmosphere at reduced temperatures. These systems can be used to study the spectral transmission function and the parameters of absorption lines at temperatures of 180–300 K. The operating principle of these systems is based on models of UV, visible, and near IR absorption by atmospheric gases under different propagation conditions in the actual atmosphere. The multipass cell makes it possible to model different temperatures, the total and partial pressures of gaseous components of the atmosphere, and the concentration of absorbing and line-broadening gases.

Figure 1 shows the optical configuration of the multipass cell during operation with SDL-1 and SDL-2 spectrometers. Radiation from a source is projected through the input window 1 by spherical mirrors 2 and 3 in the illumination section onto the plane of the input stop of the multipass part of the cell. After the radiation has passed repeatedly between the front mirror 5 and the back mirrors 4 and 4′ located a distance of R_{mir} = 0.5 m from the front mirror in a White system configuration, it is focused onto the output stop of the MKhK and mirrors 6 and 7 direct it through the output window 1′ onto the input slit of the spectrometer. The input and output stops of the MKhK are interchangeable, with sizes of 6 × 15, 2 × 15, and 1 × 15 mm. The thickness of the absorbing medium can be varied by increasing the optical path with multiple passages of the radiation beam between the three spherical mirrors 5, 4, and 4′, which are adjusted relative to one another by a rotation device. The maximum thickness of the absorbing medium is 20 m. The optical part of the cell is mounted on an optical bench located in a vacuum chamber. The optical bench is made of invar, which has a low coefficient of thermal expansion.

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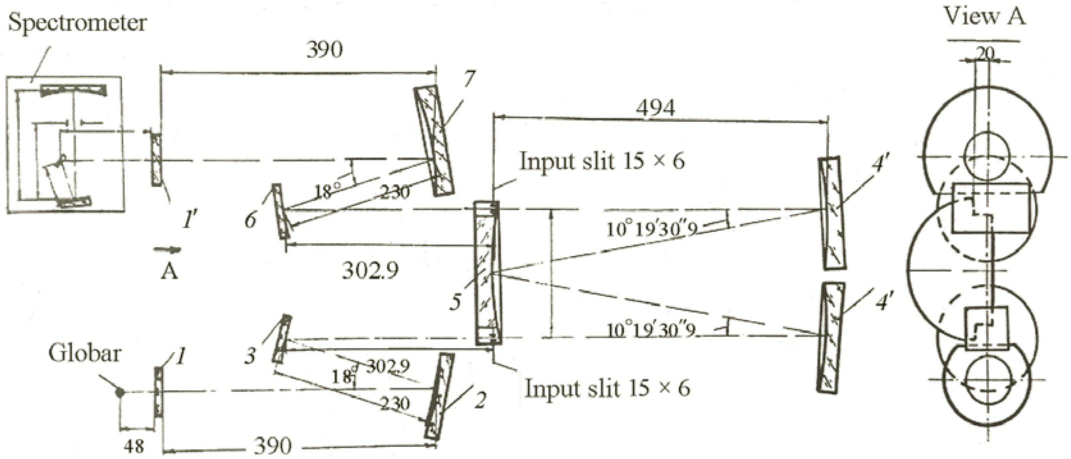

Fig. 1. Optical configuration of the MKhK-6 multipass cell.

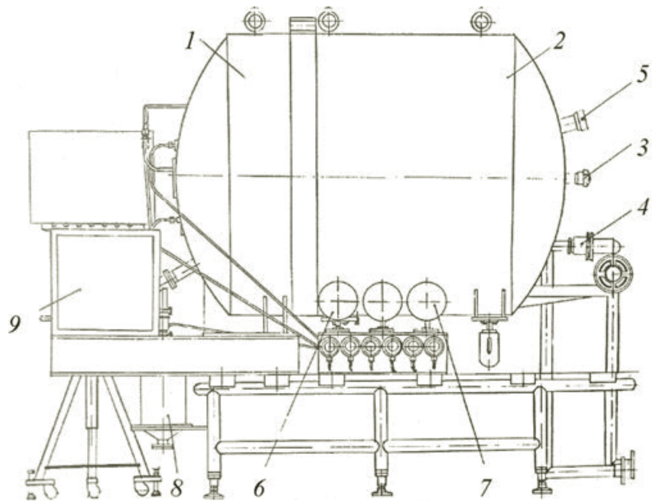

Fig. 2. Overall view of the cryogenic system with the MKhK-6.

Figure 2 is an overall view of the system. The vacuum chamber consists of two parts, 1 and 2. The front part 1 is fixed to a mounting table and the rear part 2 can be moved along rails, which permits free access to the optical part of the cell. The two parts of the cell are connected with bolts and a vacuum seal is provided by aluminum gaskets. A protective valve 4, mechanisms 3 for steering the back mirrors, and a viewing window 5 are mounted at the end of part 2 of the cell. The state of the material in the cell is monitored by a pointer vacuum gauge 6, a manometer 7, and a mercury manometer. The working pressure in the cell can be varied from 13 to $5 \cdot 10^5$ Pa. An NVR-16D vacuum pump and a vacuum dose system are used for pumpout and gas feed.

The temperature inside the cell was measured by IS-568 temperature sensors glued onto the optical rail. Their outputs passes through sealed leads to an Shch-34 digital ohmmeter. The working space of the cell was cooled using a refrigerator wrapped around the housing of the MKhK and made of aluminum pipes with protective shields and a system of pipes through which liquid nitrogen flows. The inlet and outlet of the pipe from the cell were sealed with teflon chevron baffles. The liquid nitrogen is delivered from a 900-liter TRZhK-2U transport reservoir through a flexible pipe and shut-off valve. As it passes through the refrigerator pipe, the liquid nitrogen is vaporized and the vapor is released to the atmosphere through a pipe in the upper part of the refrigerator. The cryogenic section is a component of the multipass gas cell and is intended for cooling of the gaseous components of the atmospheres during the spectral measurements. The cryogenic cell is thermostatically controlled with the aid of a foam-plastic thermal insulator. The optical part of the cell with the spectral instrumentation is installed through double protective windows separated by an air gap. In order to maintain a constant vacuum in the inlet and outlet windows during operation, a fore-tank 8 is mounted in the system to prevent condensation on the protective windows.

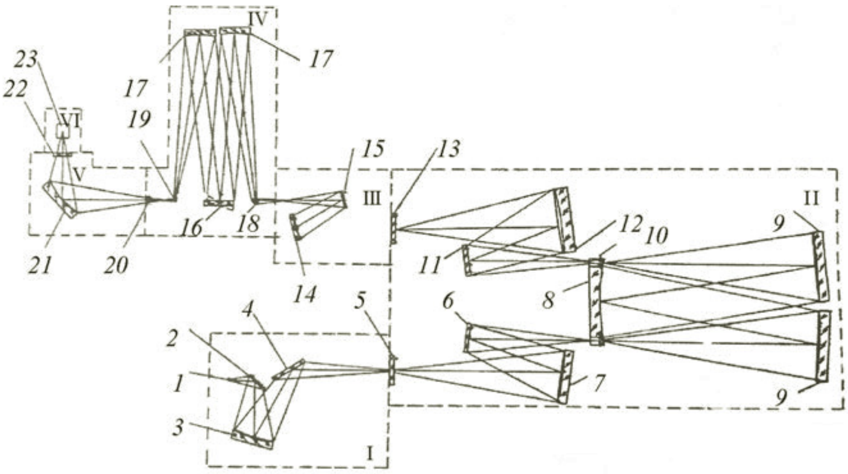

Fig. 3. Optical configuration of the spectral system with the cryogenic multipass gas cell attached to a modernized IKM-31 monochromator.

Depending on the spectral region being studied, interchangeable inlet and outlet protective windows of quartz, BaF_2, CaF_2, LiF, or MgF_2 are used. The spectral systems with MKhK-6 multpass cell are high-throughput systems and operate with two different spectrometers.

One cryogenic cell is matched to high-throughput SDL-1 or SDL-2 spectrometers which serve for work in the 0.2–6 μm range. The SDL-1 is a double spectrometer. The focal distance of the parabolic mirror objectives is 500 mm and the relative aperture is 1:3. Four pairs of interchangeable diffraction gratings (1200, 600, 300, and 100 lines/mm) are used as dispersion elements in the working range (0.2–6 μm). The gratings all operate in first order. The parameters of the interchangeable gratings were chosen so as to ensure that at any point in the working range of a spectrum ≥40–50% of the energy is reflected at the maximum of the radiation source.

An FEU-39A (FEU-100) and an FEU-62 photomultiplier were installed by turns to cover a spectral range of 0.2–1.2 μm. Vacuum optical matching attachments were developed to match the optical part of the MKhK-6 with the spectrometer. A chart recorder 9 is used to produce a visible image of the recorded spectra.

The second spectral system with an MKhK-6 multipass cryogenic cell is intended for work in the 0.11–1 μm range. Figure 3 shows the optical configuration of the cryostat attached to a modernized IKM-731 monochromator. The illuminator I consists of a radiation source 1 and three mirrors 2, 3, and 4 which are used to focus an image of the source onto the focal plane of the spherical mirror 7 of the receiver part of the cryogenic cell. Then the radiation that passes through the high-throughput multipass cell II and the optical attachment III is focused onto the plane of the input slit of the IKM-731 monochromator IV. The input slit of the monochromator is in the focus of a rotatable mirror 18 and a spherical mirror 17; the mirrors are used to direct the radiation onto the diffraction grating 16. After diffraction the beam of parallel rays is collected by the spherical mirror 17 and directed by a rotatable mirror 19 onto the output slit of the monochromator, which is located in the focal plane of the output spherical mirror 17. In the receiver chamber V a toroidal mirror 21 focuses the light onto the detector area of the photomultiplier 23 in module VI. Vacuum optical attachments are used to match the cryogenic multipass gas cell to the IKM-731 monochromator. The vacuum optical attachments, illuminators, and monochromator are pumped down by a 2NVR-5DM vacuum pump and a TsVA1-2 vacuum system. The vacuum was measured by VDG-1 and VIT-2 gas-discharge strain manometers with thermocouple and manometer lamps. The spherical mirrors 8 and 9 ensure multiple passage of the radiation in the MKhK-6; 6, 7, and 11, 12 are mirrors for input and output matching of the optical attachments to the MKhK-6; 14 and 15 are the mirrors on the optical attachment for coupling to the IKM-731 monochromator; 5, 13, 20, and 22 are interchangeable inlet and outlet protective windows for modules I–IV of the system; and 10 is the output stop of the multipass cell.

Spectral calibration of the measured absorption spectra is based on the hydrogen emission lines from VMF-25P and LD2-D arc lamps with barium fluoride windows in the 120–170 nm range and on mercury and helium emission lines generated by a DRGS-12 arc lamp in the 0.2–0.4 μm range. The absorption spectra in the visible and IR can be calibrated based on the position of the absorption lines of SO_2, NO_2, CO, HCl, NO, and water vapor. Figure 4 is an example of a UV absorption spectrum of SO_2 recorded by means of the spectral measurement system with the cryogenic multipass cell and an SDL-1 spectrometer.

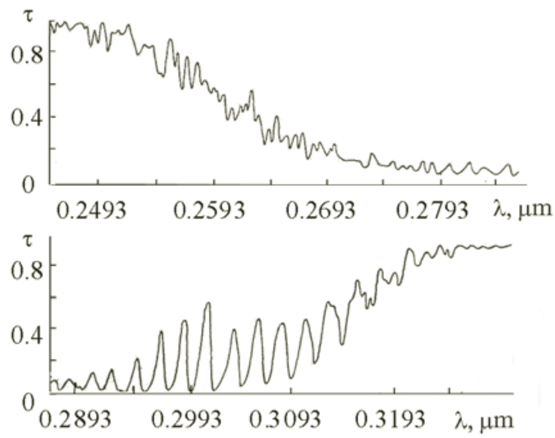

Fig. 4. UV absorption of SO_2 at wavelengths of 0.245–0.329 mm; the partial pressure of SO_2 is P_{SO_2} = 50 Pa and the optical path length is L = 200 cm.

In sum, high-resolution spectral measurement systems with an MKhK-6 high-throughput cryogenic multipass gas cell together with SDL-1 and SDL-2 spectrometers and a modernized IKM-731 monochromator have been developed, built, and tested. These measurement systems provide a high spectral resolution Δ = 0.05–0.1 cm^{-1} for studying the absorption spectra of gaseous media in the 0.1–6 μm at temperatures of 180–300 K and pressures of 0.001–50 atm. These systems have full metrological support for the measured characteristics.

REFERENCES

1. K. Ya. Kondrat′ev and N. I. Moskalenko, *Thermal Radiation of Planets* [in Russian], Gidrometeoizdat, Leningrad (1977), pp. 62–84.
2. K. Ya. Kondrat′ev and N. I. Moskalenko, *Key Problems in Research on Planets in the Solar System* (*Greenhouse Effect in Planetary Atmospheres*) [in Russian], VINITI, Moscow (1983), pp. 3–156.
3. N. I. Moskalenko, S. O. Mirumyants, N. F. Liktev, and R. Sh. Misbakhov, *Equilibrium and Nonequilibrium Radiative Processes: High-Temperature Media, Radiative Heat Transfer* [in Russian], Izd-vo KGEU, Kazan (2014), pp. 67–102.
4. N. I. Moskalenko, S. A. Sementsov, R. S. Sadykov, S. O. Mirumyants, and L. S. Semenov, *Zh. Prikl. Spektrosk*., **32,** No. 2, 377–381 (1980).
5. N. I. Moskalenko, Yu. A. Il′in, and G. V. Kayumova, *Zh. Prikl. Spektrosk*., **56** (1), 122–127 (1992) [*J. Appl. Spectrosc*., **56** (1), 93–97 (1992)].
6. N. I. Moskalenko and Ya. S. Safiullina, *Izv. Vyssh. Uchebn. Zaved., Problemy Énergetiki*, Nos. 11–12, pp. 22–32 (2009).
7. N. I. Moskalenko and Ya. S. Safiullina, *Al′ternativnaya Énergetika Ékologiya*, No. 6, pp. 89–98 (2009).
8. N. I. Moskalenko, Ya. S. Safiullina, M. S. Sadykova, and N. F. Loktev, *Al′ternativnaya Énergetika Ékologiya*, No. 2, pp. 43–54 (2010).
9. L. P. Shishatskaya, *Opt.-Mekh. Prom-t′*, No. 9, pp. 54–59 (1984).
10. V. M. Tait and R. V. Shatskaya, *Atlas of the Multiline Spectrum of Molecular Hydrogen* [in Russian], Izd. AN ÉSSR, Tallin (1981), pp. 3–79.