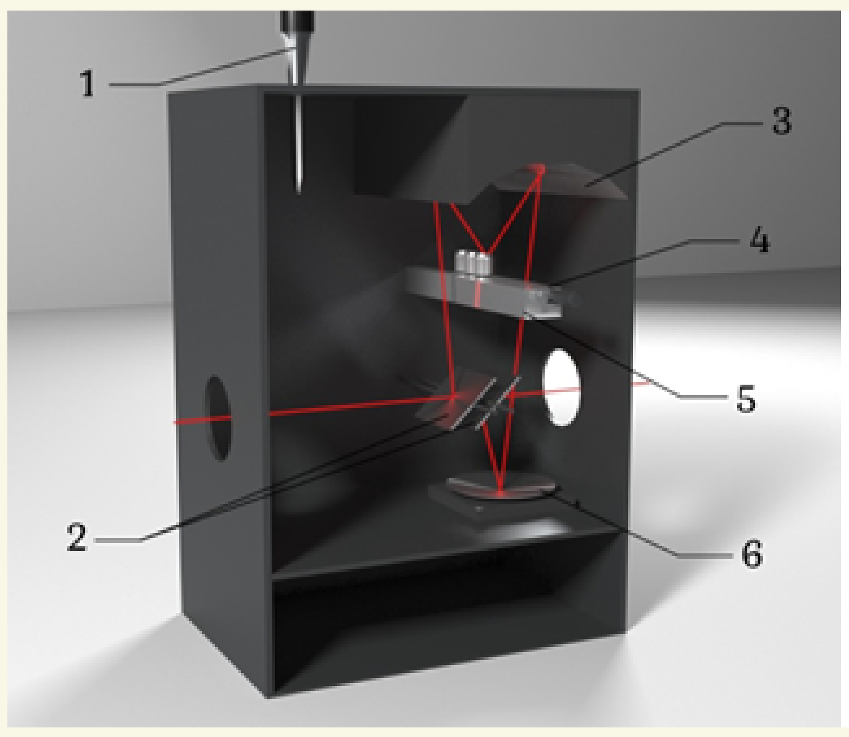
Development of low-temperature cell for IR Fourier-spectroscopy of hydrocarbon materials

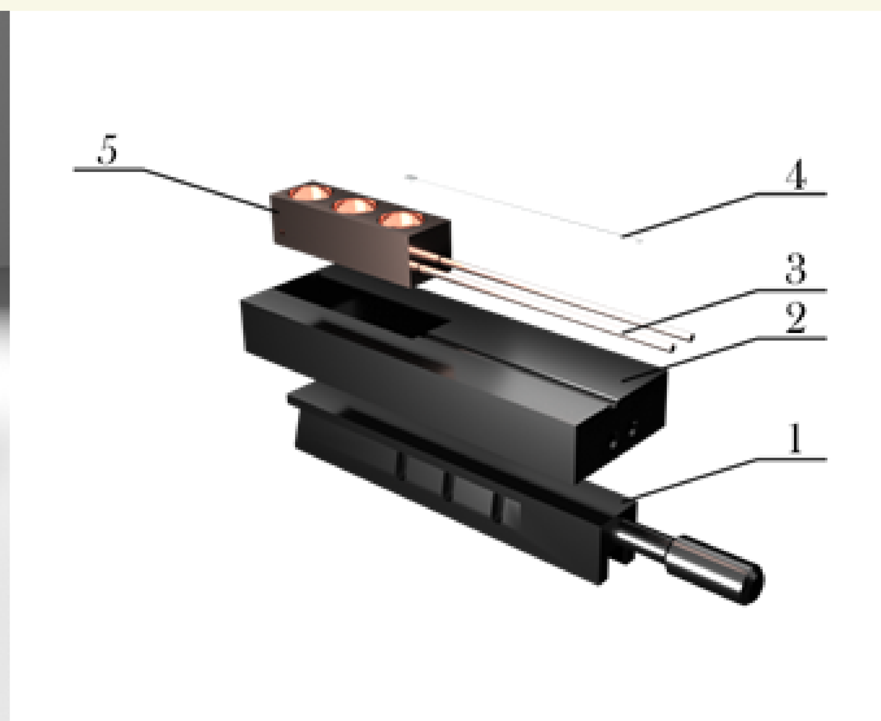
Abstract

Since most IR spectroscopy analysis of hydrocarbons is performed in high vacuum, we are confident that our setup will be effective in generating new low-temperature spectroscopy analysis results if experiments are performed at atmospheric pressure. This work aimed to show the proof of measurement technique efficiency and functionality of the developed low-temperature measuring cell with cryogenic capillary system [1]. In order to confirm them we carried out two experiments with studying ethanol at low temperature and provided their comparison. The first one was conducted using our specially designed low-temperature measuring cell integrated into diffuse reflection attachment of the FSM 2203 Fourier spectrometer and with cryogenic capillary system to achieve low temperature conditions under normal atmospheric pressure.

Methodology

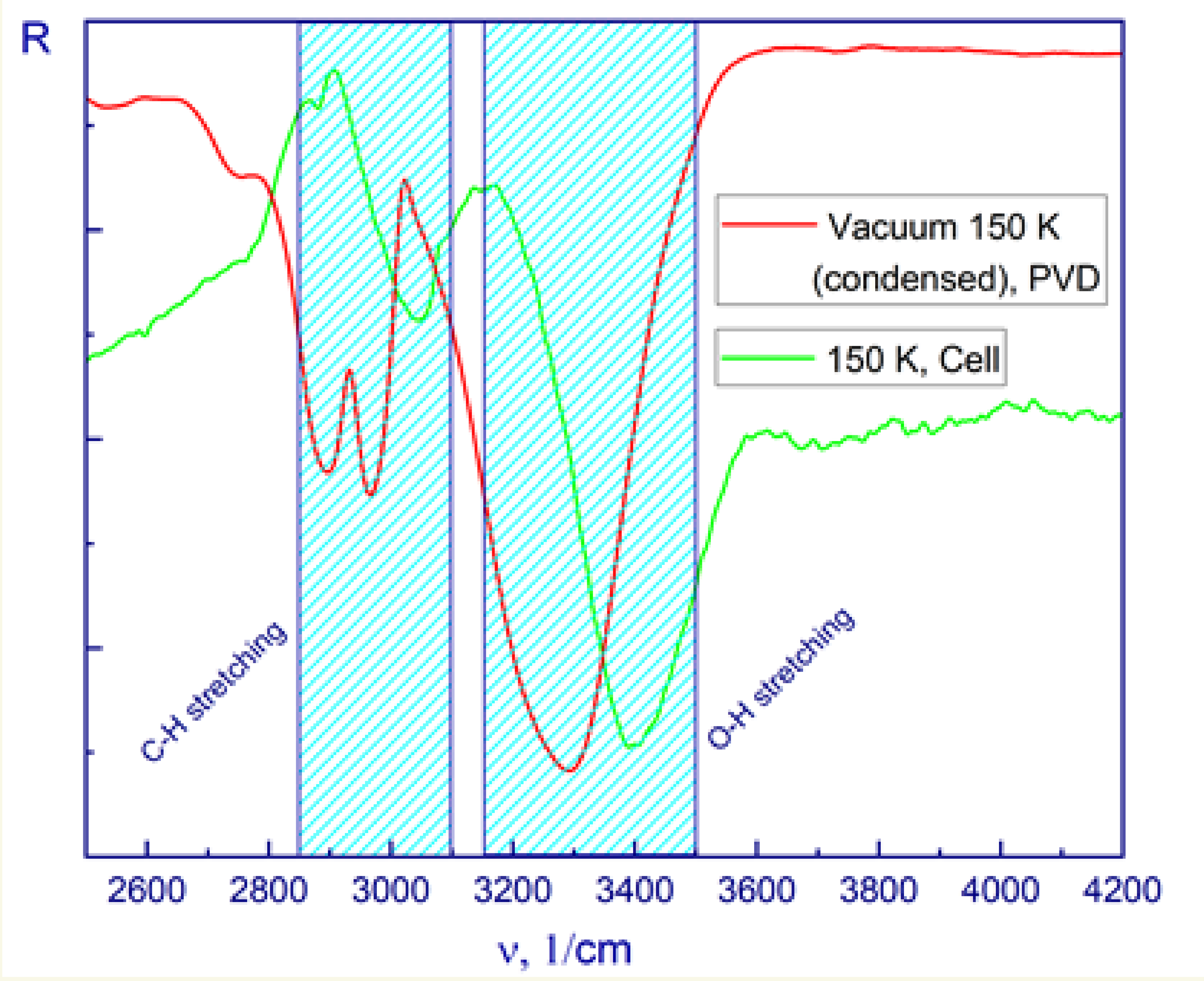
Cooling of the studied samples to a temperature of 80K is carried out due to the continuous circulation of liquid and gaseous nitrogen inside the copper tube of the cryogenic capillary system. Direct contact of the copper tube and copper bar with the cells, as well as the high thermal conductivity of copper, allows for rapid cooling of the substance under study (in our case, ethanol) to the required temperature. Indications of the current temperature of the copper bar are determined using a Lake Shore thermocouple (item 4) installed on the surface of the copper bar. Warming of the samples under study is achieved by stopping the circulation of nitrogen through the cryogenic capillary system and restoring thermodynamic equilibrium between the substance under study and the environment. The circulation of nitrogen through the copper tubes of the cryogenic capillary system is achieved by establishing high pressure by heating in a special Dewar vessel. As a result, nitrogen is “squeezed out” from the vessel into the cryogenic capillary system, with the help of which the samples are cooled. For the sake of the purity of the experiment, a blowing system was also added to the diffuse reflection attachment. Nitrogen gas supplied inside the attachment creates the necessary inert environment and also prevents the formation of frost on the surface of the samples and copper bars. Ethanol produced by SeccoSolv (purity>99.9%, dried, Darmstadt, Germany) was chosen as the test substance.

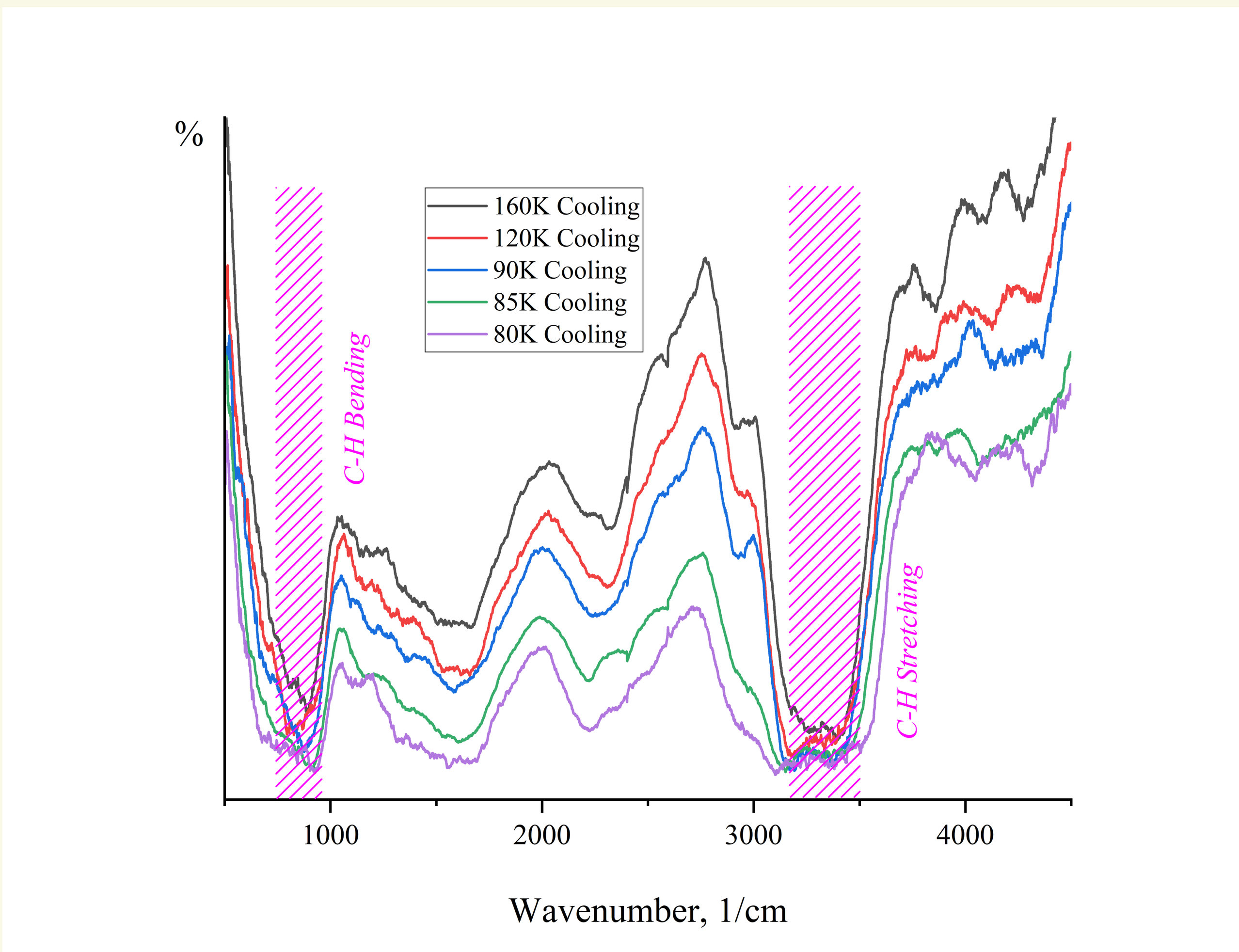




Results

The main result is shown in the figure. As can be seen in the figure, we conducted two types of experiments and now present their comparison and analysis. The first experiment was carried out at normal atmospheric pressure using a developed low-temperature cell with a cryogenic capillary system that provides ethanol cooling, the second in a vacuum using a cryovacuum chamber with the PVD (Physical Vapor Deposition) method. In the experiment with the developed low-temperature cell, we can observe a hypsochromic shift at 150 K relative to the experiment in vacuum. This is due to the method of obtaining the solid sample, since the vacuum sample is obtained by physical vapor deposition, and the atmospheric sample is obtained by cooling the liquid phase of ethanol. Despite the shift, we observe peaks in the absorption bands from 2850 1/cm to 3000 1/cm and from 2950 1/cm to 3100 1/cm, which correspond to the C-H stretching vibrations of ethanol in the PVD and Cell experiments, respectively, from 3150 1 /cm to 3400 1/cm and from 3300 1/cm to 3500 1/cm, which corresponds to the OH stretching vibrations in the PVD and Cell experiments, respectively.





Conclusions

- Has been created the installation for research at low temperatures and normal pressure
- A comparative analysis of two methods for studying the optical properties of ethanol at low temperatures is presented - the PVD method and the developed cell method
- The substance (kerosene) in the solid state was studied in the temperature range 80-160 K and IR Fourier spectra were obtained

References

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[2] Kenbay A.A., Golikov O.Yu., Aldiyarov A.U., Yerezhep D.E. Low-temperature cell for IR Fourier spectrometric investigation of hydrocarbon substances. Scientific and Technical Journal of Information Technologies, Mechanics and Optics, 2023, vol. 23, no. 4, pp. 696–702. doi: 10.17586/2226-1494-2023-23-4-696-702

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