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Low-temperature cell for IR Fourier spectrometric investigation of hydrocarbon substances

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Abstract

A specialized low-temperature measuring cell with a cryogenic capillary system for infrared spectral analysis of ethanol developed by the authors is presented. The use of the created low-temperature cell is possible for further studies of the low-temperature properties of both pure ethanol and mixtures with its contents, which is currently an urgent task, and the data obtained with its help can be used for ice research. Two methods of ethanol research at low temperature are presented in comparison. In the first method proposed by the authors, a specially developed low-temperature measuring cell based on a diffuse reflection prefix of the Fourier spectrometer FSM 2203 with a cryogenic capillary system is used. This system allows you to achieve the required low-temperature regime at normal atmospheric pressure. The results of the experiment are compared with the traditional method of gas-phase condensation of the test sample under low temperature conditions at the pressure $P = 1.0 \cdot 10^{-5}$ Torr. Infrared spectra of low molecular weight amorphous and crystalline ethanol were obtained at a temperature of 150 K at normal atmospheric pressure and in vacuum. Comparison of experimental results confirmed the operability of the new installation. In the experiments, peaks were observed in the absorption bands from 2850 to 3000 cm^{-1} and from 2950 to 3100 cm^{-1} , corresponding to the valence CH vibrations of ethanol as well as in the absorption bands from 3150 to 3400 cm^{-1} and from 3300 to 3500 cm^{-1} , which corresponds to the valence vibrations of OH. The results of the study showed the prospects of the proposed method and can be useful by researchers in the field of low-temperature spectroscopy at normal pressure.

Keywords

low-temperature cell, cryogenic capillary system, ethanol, IR spectra, diffuse reflection

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Низкотемпературная ячейка для инфракрасных фурье-спектрометрических исследований углеводородных веществ

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Введение. Разработана специализированная низкотемпературная измерительная ячейка с криогенной капиллярной системой для инфракрасного спектрального анализа этанола. Созданная низкотемпературная ячейка может быть применена для исследований низкотемпературных свойств чистого этанола и смесей с его содержанием. Такое использование в настоящее время является актуальной задачей, а получаемые с ее помощью данные могут найти применение для исследования льдов. **Метод.** Выполнено сравнение двух методов исследования этанола при низкой температуре. В первом, предложенном методе применена специально разработанная низкотемпературная измерительная ячейка на базе приставки диффузного отражения Фурье-спектрометра ФСМ 2203 с криогенной капиллярной системой. Использование системы позволило получить требуемый низкотемпературный режим при нормальном атмосферном давлении. Результаты эксперимента сопоставлены с традиционным методом газовой конденсации исследуемого образца в условиях низкой температуры при давлении $P = 1,0 \cdot 10^{-5}$ торр. **Результаты.** Получены инфракрасные спектры низкомолекулярного аморфного и кристаллического этанола при температуре 150 К, нормальном атмосферном давлении и в вакууме. Сравнение экспериментальных результатов подтвердило работоспособность новой установки. В результате экспериментов наблюдались пики в полосах поглощения от 2850 до 3000 см^{-1} и от 2950 до 3100 см^{-1} , соответствующие валентным СН-колебаниям этанола, а также в полосах поглощения от 3150 до 3400 см^{-1} и от 3300 до 3500 см^{-1} , что соответствует валентным колебаниям ОН. **Обсуждение.** Полученные результаты показали перспективность предложенного метода и могут быть полезны исследователями в области низкотемпературной спектроскопии при нормальном давлении.

Ключевые слова

низкотемпературная ячейка, криогенная капиллярная система, этанол, ИК спектры, диффузное отражение

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Introduction

Amorphous ices are the dominant form of water and alcohol in the universe, but they do not naturally form on Earth's lithosphere. Amorphous ices mostly form on interstellar dust, in comets, and many other astrophysical environments including the Saturnian rings. For example, crystalline benzene has been identified in the atmosphere of Titan [1] within the Solar System, while benzene ice has been detected in the direction of a proto-planetary nebula beyond the Solar System [2]. There are plenty of conditions of the amorphous ice formation in nature. Crystalline ice may amorphize under the influence of UV- or ion-irradiation [3]. A significant amount of laboratory researches has been conducted on extraterrestrial ices at low temperatures, primarily focusing on analyzing the infrared (IR) spectra and other characteristics of amorphous molecular solids that consist of one or more components [4]. Unfortunately, this emphasis on amorphous solids has often resulted in the neglect of studying crystalline compounds, despite their presence in various environments within the Solar System.

Among the organic matters that have been found in ices by astronomers were also methanol (CH_3OH) which has received the most detailed and widespread attention [5–8], and solid ethanol ($\text{C}_2\text{H}_5\text{OH}$) which has almost been ignored by astrochemists. Taking into account the importance of ethanol for such areas as energy [9–12], medicine [13, 14], industry [15, 16], study of ethanol in its various states

and conditions is very relevant. Insufficient information about amorphous $\text{C}_2\text{H}_5\text{OH}$, in which phase it is expected in many extraterrestrial environments, makes it impossible to understand and evaluate ethanol-ice by IR astronomers [17].

One of the methods that can help us to get more information about amorphous ethanol is IR spectroscopy that has been invaluable in the study of extraterrestrial ices, both through astronomical observations and laboratory studies. In both cases, the two reference spectroscopic quantities of greatest need are spectral positions and IR intensities, such as band strengths or absorption coefficients for IR peaks. However, although the IR literature contains extensive results on spectral positions, peak assignments, and vibrational modes, quantitative results on intensities for many compounds in the solid state has not been studied in depth [18–21]. The centrality of such reference IR intensities for calculating abundances in astrochemical problems is well illustrated by discussions in both a recent and an older review of icy solids in extraterrestrial environments [22]. IR spectra are crucial in identifying and characterizing extraterrestrial ices, and the quantitative measurements of intensity such as band strengths and absorption coefficients are used to measure abundances. Additionally, optical constants have applications in computational models of solid-phase compositions [23]. IR spectroscopy is the most reliable method for remote identification of molecules and ions in extraterrestrial solids, and it remains possible to not only identify but also

quantify IR detections to obtain molecular abundances by comparing astronomical data to reference measurements from terrestrial laboratories [24]. The IR spectra of ethanol can be used to study the low-temperature chemistry of interstellar clouds and of extraterrestrial objects in our solar system, such as ice-covered moons and trans-Neptunian objects in connection with upcoming NASA and ESA space missions involving IR instrumentation [25].

The present work is aimed at confirming the measurement technique using the developed by us specialized low-temperature cell through conducting fundamental research of ice (amorphous/solid) and liquid ethanol properties in the temperature range of 90–200 K under normal conditions (atmospheric pressure) which are provided by the developed cell.

Methodology

To research the thermophysical properties of ethanol within the framework of this research, we conducted two types of fundamentally different experiments of studying ethanol at low temperature to compare them later. The first one was conducted at low temperatures and normal atmospheric pressure, the second one — at low temperatures in vacuum chamber.

To conduct the first experiment, it was necessary to create such an installation that will allow researching the properties of ethanol in the IR range at low temperatures and at normal pressure. Currently available diffuse reflection attachments for spectrometers do not have the ability to record the spectral characteristics of substances at temperatures different from the ambient temperature. Nevertheless, the forced choice of the diffuse reflection mode of IR spectroscopy is due to the possibility of creating a modified attachment that would allow to record spectra at different temperatures with the simultaneous possibility of cooling the sample.

The basis on which the modernization was carried out within the framework of this research was the IR Fourier spectrometer FSM 2203 of the INFRASPEK company (St. Petersburg, Russia) with a special attachment for diffuse reflection. This IR Fourier spectrometer allows obtaining high-resolution IR spectra for the qualitative and quantitative analysis of the samples under research. Fourier spectrometer FSM 2203 (Fig. 1) operates in the

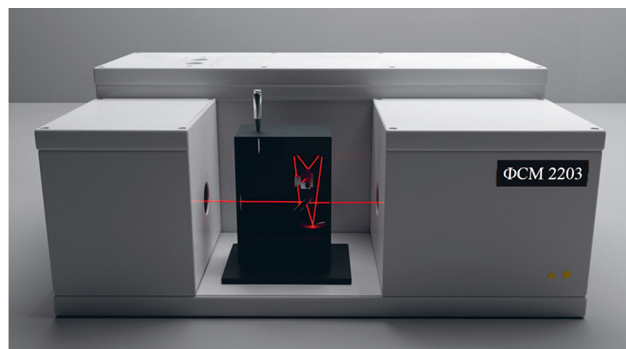


Fig. 1. Infrared Fourier spectrometer "FSM 2203" with a diffuse reflection attachment

Mid-Infrared Region (MIR) with a spectral resolution of 0.1 cm^{-1} in the range of $370\text{--}7800 \text{ cm}^{-1}$.

Since this research implies an experimental research of ethanol thermophysical properties at low temperatures and at normal pressure, the question of creating the possibility of the test sample temperature controlling inside the diffuse reflection attachment arises (Fig. 2), the sample is placed in the cuvette compartment of the FSM 2203 spectrometer. The greatest importance is the possibility of lowering the temperature to 80–90 K. The diffuse reflection method has minimal requirements for preparing a sample of the test material. It is possible to research irregular surfaces or coatings including polymer ones. In addition, it is possible to obtain spectra in a wide range, which gives it an advantage over the classic transmission measurement method.

The main elements of this attachment are the system of mirrors and a retractable sample holder (Fig. 2, pos. 4) in the cells of which the test samples are placed. A micrometer screw (Fig. 2, pos. 1) is used to adjust the vertical position of the sample holder by moving the directive (Fig. 2, pos. 5) in the direction of the vertical axis. The radiation passing through the inlet of the attachment hits the surface of a flat double-sided mirror (Fig. 2, pos. 2), being reflected from it, the beam falls on an elliptical mirror (Fig. 2, pos. 3) which focuses it on the surface of the test sample located in the cell retractable holder. The beam diffusely reflected from the sample surface again hits the elliptical mirror which redirects it to the spherical mirror (Fig. 2, pos. 6), then the beam hits the other side of the two-sided flat mirror exiting into the detector chamber of the FSM 2203 spectrometer. Primary processing of the received IR spectra is carried out in the FSpec software, the analysis is performed using the Origin software.

To be able to lower the test sample temperature inside the cell of the retractable holder of this attachment, a specially developed modification based on the standard

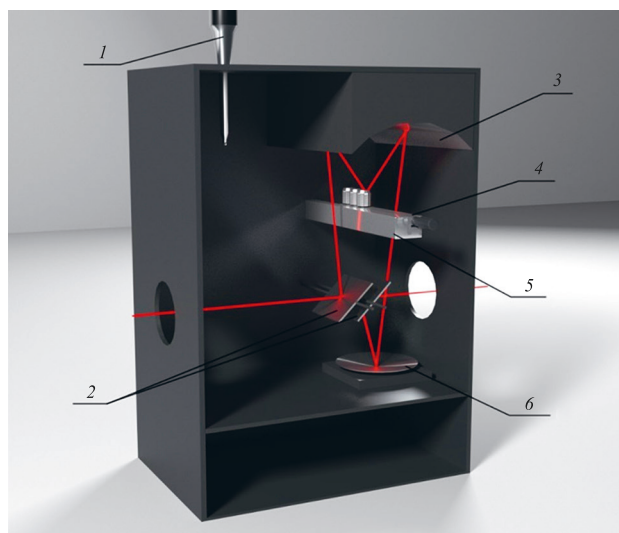


Fig. 2. Schematic representation of the DRA diffuse reflection attachment: 1 — micrometer screw; 2 — double-sided flat mirror; 3 — elliptical mirror; 4 — retractable sample holder; 5 — directive; 6 — spherical mirror

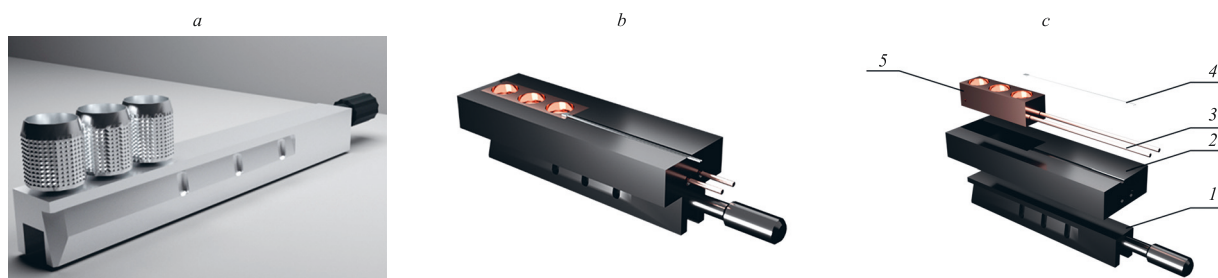


Fig. 3. Three-dimensional model of a retractable holder: standard retractable sample holder (a); modernized specialized retractable sample holder, assembled (b); modernized specialized retractable sample holder, disassembled (c).

1 — holder handle; 2 — heat insulating cover of the holder; 3 — cryogenic capillary system; 4 — temperature sensor; 5 — container

sample holder was used (Fig. 3). The design of the upgraded holder is made up of two main materials: polylactide and copper. The choice of these materials for specific parts of the holder is determined by their thermal conductivity. Thus, the choice of polylactide, which has a thermal conductivity of about 0.11–0.19 W/(m·K), is made with the purpose to prevent the negative effect of the temperature of the external atmosphere on the temperatures of the copper bar and samples placed in the measuring cells. The bar with cylindrical holes for placing the test samples (Fig. 3, pos. 5) is made of copper, a material with high thermal conductivity, to enable rapid and uniform distribution of the temperature supplied through the cryogenic capillary system. The cryogenic capillary system (Fig. 3, pos. 3) is also made of copper for the same reasons.

Cooling of the studied samples to the temperature of 80 K is carried out on account of the continuous circulation of liquid and gaseous nitrogen inside the copper tube of the cryogenic capillary system. The direct contact of the copper tube and copper bar with the cells, as well as the high thermal conductivity of copper, make it possible to achieve rapid cooling of the test substance (in our case, ethanol) to the required temperature. The current temperature of the copper bar is measured using the LakeShore PID controller thermocouple (Fig. 3, pos. 4) mounted on the surface of the copper bar. Heating of the test samples is achieved by stopping nitrogen circulation through the cryogenic capillary system and restoring the thermodynamic equilibrium between the test substance and the environment. The nitrogen circulation through the copper tubes of the cryogenic capillary system is achieved by establishing a high pressure by heating in a special Dewar vessel. As a result, nitrogen vapor is “squeezed out” from the vessel into the cryogenic capillary system with the help of which cooling of the samples is achieved.

For the sake of the experiment purity, a blowing system was also added to the diffuse reflection attachment. Gaseous nitrogen supplied inside the attachment creates the necessary inert environment and also prevents frost formation on the surface of the samples and the copper bar. Ethanol produced by SeccoSolv was chosen as the test substance (purity $\geq 99.9\%$, dried, Darmstadt, Germany).

The second experiment (Physical Vapor Deposition (PVD)) was conducted with using experimental setup for cryovacuum condensation [26] shown in Fig. 4.

A substrate (Fig. 4, pos. 6) is thermally connected to a closed-cycle helium Gifford-McMahon refrigerator and

placed within the vacuum chamber (Fig. 4, pos. 1). The cooling system has two stages, first is capable of cooling the substrate to a temperature of 7.5 K and the second one — cooling to 12 K. A resistor heater is attached to the second stage. The PID temperature controller LakeShore allows for temperature adjustments between 12–200 K [27]. To obtain the condensed ethanol sample, the gas phase was deposited onto the substrate at a pressure of $P = 1.0 \cdot 10^{-5}$ Torr while maintaining the substrate temperature at 150 K. IR radiation supplied from an external source reflects from sample, passes through a system of mirrors and goes into FSM 2203 where it is being processed and transformed into spectra we receive.

Results and discussion

In our researches, the main attention was paid to the IR spectra of low molecular weight amorphous and crystalline ethanol in the range from 2700 to 4000 cm^{-1} , obtained under normal conditions at a temperature of 90–200 K, with an emphasis on amorphous ices at temperature of

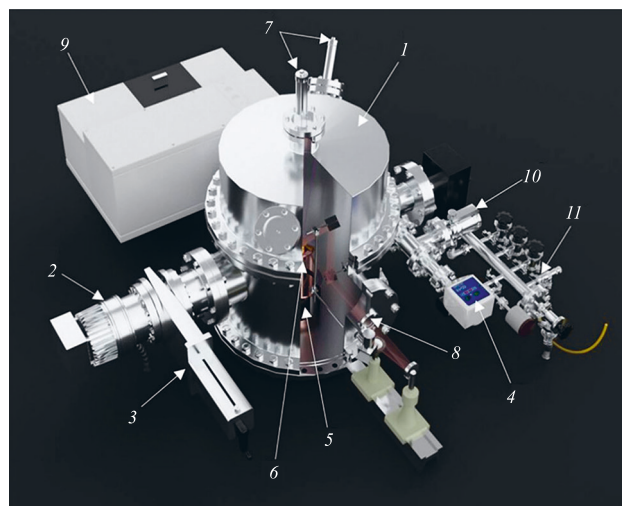


Fig. 4. Experimental setup for cryovacuum condensation: 1 — vacuum chamber; 2 — vacuum pump Turbo-V-301; 3 — vacuum gate valve CFF-100; 4 — pressure detector FRG-700; 5 — Gifford-McMahon refrigerator; 6 — substrate; 7 — photo multiplier, laser interferometer; 8 — light source, optical channel; 9 — IR spectrometer; 10 — high-precision gas supply leak into the chamber; and 11 — gas leak into the mixture production system

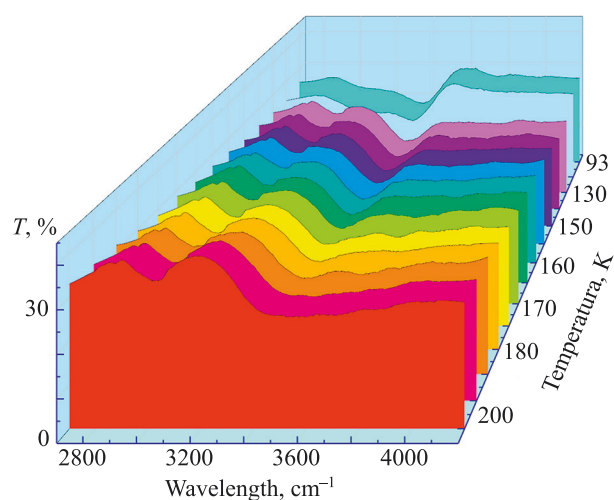


Fig. 5. A family of transmittance (T) of the IR spectra of ethanol in the temperature range of 93–200 K

150 K. The family of obtained spectra and their temperature evolution are shown on Fig. 5. IR spectra were recorded from a sample placed in a measuring low-temperature cell at room temperature and subsequently cooled with cryogenic capillary system in a special attachment to a temperature of 93 K. Direct recording was carried out by heating the sample from the minimum obtained temperature to room temperature. The temperature interval from 200 K to 300 K was cut out because of the absence of any important structural-phase changes in the sample in this interval. The inner part of the diffuse reflection attachment, as mentioned earlier, was continuously blowing with nitrogen to create the most inert environment and exclude the possibility of foreign substances getting inside the sample under research.

Main result is shown on the Fig. 6. How you can see on the figure, we conducted two type of experiments and now present comparing and analysis of them. First experiment was conducted under normal atmospheric pressure using developed low-temperature cell with cryogenic capillary system which provides cooling of ethanol, and second one in vacuum uses cryovacuum chamber with PVD method. In the experiment with designed low-temperature cell, we can observe hypsochromic shift at 150 K relative to the experiment in vacuum. This is due to the method of obtaining a solid sample, since the vacuum sample is obtained using PVD, and the atmospheric sample is obtained by cooling the liquid phase ethanol. Despite the shift, we observe the peaks in absorption bands from 2850 cm^{-1} to 3000 cm^{-1} and from 2950 cm^{-1} to 3100 cm^{-1} , which correspond to the C–H stretching vibrations of ethanol in experiment with PVD and with Cell, respectively, from 3150 cm^{-1} to 3400 cm^{-1} and from

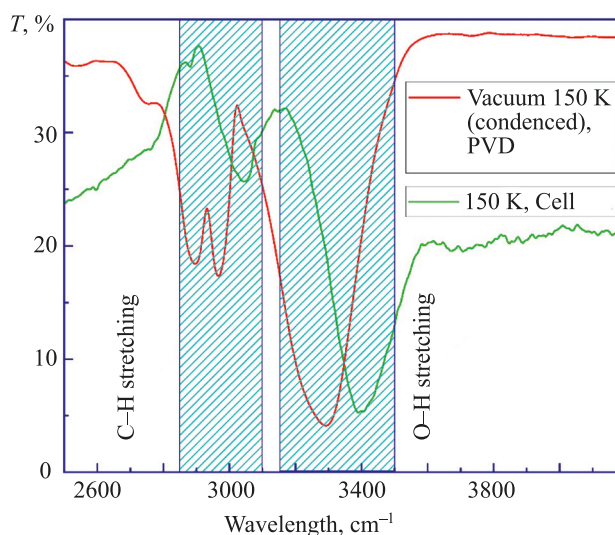


Fig. 6. Transmittance (T) of the IR spectrum of ethanol at 150 K from two experiments

3300 cm^{-1} to 3500 cm^{-1} , which correspond to the O–H stretching vibrations in the experiments with PVD and Cell, respectively.

Presence of ethanol functional groups characteristic peaks confirms working capability and efficiency of measurement technique with our cryogenic capillary system and particularly the low-temperature cell.

Conclusion

The initial motivation for this article arose from desire to modernize existing methods in the study of hydrocarbons with habitual equipment and create fundamentally new installation to research them at low temperatures but under normal atmospheric pressure. The present work contains useful results about successful modernizing of diffuse reflection attachment for IR Fourier spectrometer. The developed at Al-Farabi University cryogenic capillary system with low-temperature cell is completely workable. These results can be used, for example, for a new researching works in the sphere of low-temperature spectroscopy at normal pressure. This cell and measurement technique made it possible to obtain low-temperature spectra of ethanol under atmospheric conditions. This makes it possible to state the operability of this method as well as the possibility of conducting a number of further researches aimed at studying the thermophysical properties of many hydrocarbons including monohydric alcohols at low temperatures. Using the developed low-temperature cell will help to understand how many types of hydrocarbons behave at low temperatures and atmospheric pressure.

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