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Research Article

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DEVELOPMENT OF LOW-TEMPERATURE CELL FOR IR FOURIER-SPECTROSCOPY OF HYDROCARBON MATERIALS

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Abstract. *This work introduces a technology for Fourier-Transform InfraRed spectroscopy of hydrocarbon materials at low temperatures and atmospheric pressure. This device allows to study optical properties of various substances at temperature range of 77 – 300 K without need of a vacuum and to obtain new fundamental data since there is insufficient research in this area. Described new techniques and methods are working with Fourier-Transform InfraRed spectrometer, diffuse reflection attachment and two Dewar vessels using for cooling with liquid nitrogen inside cryogenic capillary system and blowing with gaseous nitrogen to create InfraRed inactive environment. It makes this new technique a valuable method for obtaining fundamentally new data useful for various energy and infrastructure spheres along with education field as a theoretical information.*

Keywords: Fourier-Transform InfraRed spectroscopy, low temperature, cell, cryogenic capillary system, hydrocarbon materials.

1. Introduction

At the dawn of spectrometry and since the creation of technologies that allow studying hydrocarbon compounds using spectroscopic methods at low temperatures, the process was accompanied by the formation of a vacuum, without which it was difficult to study the nanostructure [1-2], molecular composition [3-4] and physical, chemical, thermal, and optical properties [5-9] of a substance due to the presence of moisture and other impurities in the air that prevent high-quality study and analysis of a substance by any of the spectroscopic methods, including FTIR (Fourier-Transform InfraRed) [10-13].

Low-temperature study of substances [14], in particular hydrocarbon compounds, which involves the use of InfraRed (IR) spectroscopy, presupposes the presence of ultra-low pressure [15], i.e. vacuum, in consequence of which the results of high-quality fundamental studies of the optical properties of hydrocarbon substances under atmospheric pressure are still very rare, since this issue is associated with a number of problems, such as the complexity of developing, maintaining and subsequently upgrading technologies for spectroscopic study that allow achieving low temperatures at atmospheric conditions, the difficulty of creating an inert and IR-inactive or IR-transparent environment [16-17] that helps to achieve accuracy due to the low noise level of spectra and high radiation intensity, as well as the lack of a developed accurate step-by-step research methodology.

Overall, there are very few technologies in the world to study hydrocarbon substances at low temperature and atmospheric pressure. Even though some research studies [18-21] include experiments of spectroscopy at atmospheric pressure and low temperatures, there is limited scientific research in this direction, due to the issues described above. We offer a technology [22] for FTIR spectroscopic study of hydrocarbon fuels under atmospheric pressure, but at low (77 K) temperatures, which presents solutions to the above problems when studying outside of a vacuum.

The idea of the article is the proposed experimental setup of a cryogenic-capillary system with a low-temperature cell and a measurement technique on it, allowing to obtain profoundly new fundamental data on the properties and behavior of hydrocarbon materials at low temperatures and atmospheric pressure, representing valuable information that can contribute to the subsequent development of high-quality research in this direction, including more factual data related to the action of molecules under these conditions, which can further contribute to the development of more accurate technological equipment.

In this paper, we study kerosene – a hydrocarbon used in energy, technology and other industries as a fuel or solvent and this compound as a test sample, as well as comparison of its spectra with the results of other authors were chosen and carried out in order to confirm the effectiveness of this technique and setup. So, this research corresponds to the goals according to the Sustainable Development Goal (SDG) [23] SDG 4 – Quality education and SDG 9 – Industry, innovation and infrastructure adopted by the United Nations (UN). These new data can be used in writing academic textbooks for higher education students in technical universities with Science, Technology, Engineering, Mathematics (STEM) direction as well as theoretical data for innovative energy industry sphere.

2. Materials and methods

In order to obtain high-quality results of low-temperature spectroscopic research of hydrocarbon materials at atmospheric pressure, we conduct experiments using cryogenic capillary system with low-temperature measuring cell. To provide accurate information about spectroscopic data of hydrocarbons we use high-precision mid-IR Fourier-spectrometer from “Infraspec” with a spectral resolution of 0.125 cm^{-1} in the range of 370–7800 cm^{-1} (figure 1).

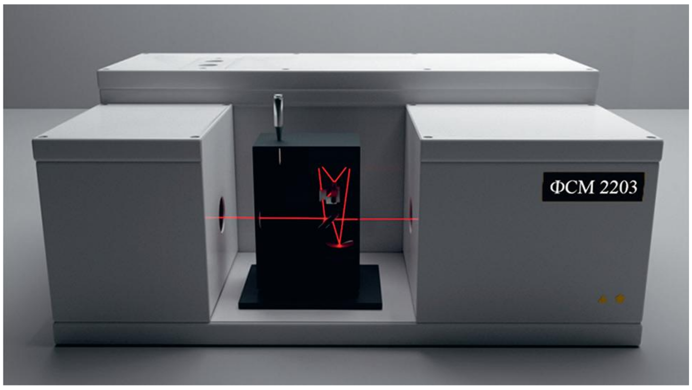


Fig.1. Infrared Fourier spectrometer “FSM 2203” with a diffuse reflection attachment.

To be able to conduct experiment of low-temperature diffuse reflection spectroscopy at atmospheric pressure we had to develop the installation allowing us to study hydrocarbon substances in these conditions. The focus shifts to enabling temperature control of the test sample within the diffuse reflection attachment (figure 2), where the sample is positioned in the cuvette compartment of the FSM 2203 spectrometer. Of utmost significance is the capability to lower the temperature to 77 K. The use of the diffuse reflection method entails minimal sample preparation requirements and allows for the examination of irregular surfaces and various coatings, including polymers. Furthermore, this method enables spectrum analysis across a broad range, offering an advantage over traditional transmission measurement techniques.

The central components of this accessory consist of a mirror system and an retractable sample holder (fig. 2, item 4) where the test samples are housed. A micrometer screw (fig. 2, item 1) is utilized to modify the vertical position of the sample holder, by moving the directive (fig. 2, item 5) along its way we can modify horizontal position. The radiation entering the accessory passes through its inlet and strikes a flat double-sided mirror (fig. 2, item 2). This beam is then reflected towards an elliptical mirror (fig. 2, item 3), which focuses it onto the test sample's surface within the retractable holder. The diffusely reflected light from the sample's surface is redirected back towards the elliptical mirror, which guides it to a spherical mirror (fig. 2, item 6). The reflected beam further encounters the opposite side of the double-sided flat mirror before entering the detector chamber of the FSM 2203 spectrometer.

Initial processing of the obtained IR spectra is conducted using the FSpec software, with subsequent analysis carried out through the Origin software. In order to achieve temperature reduction of the test sample within the retractable holder's cell of this accessory, a specially designed modification was implemented based on the standard sample holder (figure 3). The enhanced holder's construction comprises two primary materials: polylactide and copper. The selection of these materials for specific holder components is based on their respective thermal conductivity properties. Polylactide, with a thermal conductivity ranging from about 0.11 to 0.19 W/m*K, is chosen to counteract the adverse impact of external atmospheric temperatures on both the copper bar and the samples housed in the measuring cells.

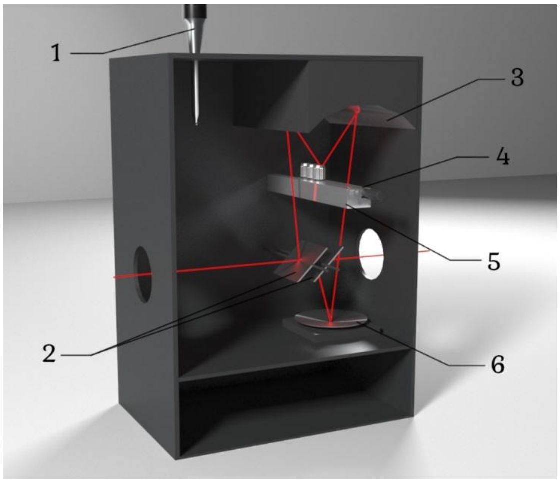
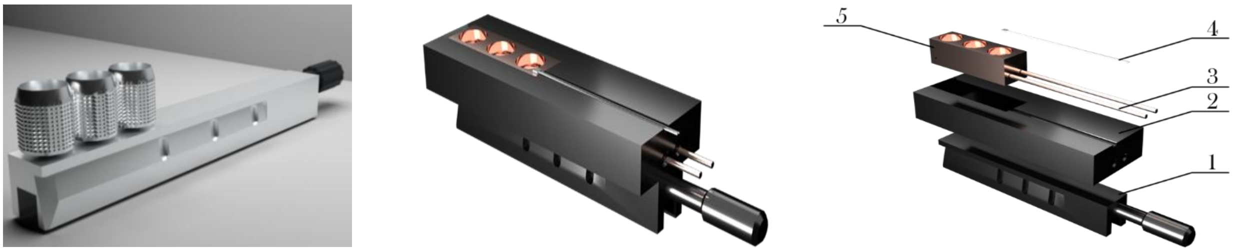


Fig.2. Schematic representation of the diffuse reflection attachment: 1 — micrometer screw; 2 — double-sided flat mirror; 3 — elliptical mirror; 4 — retractable sample holder; 5 — directive; 6 — spherical mirror.



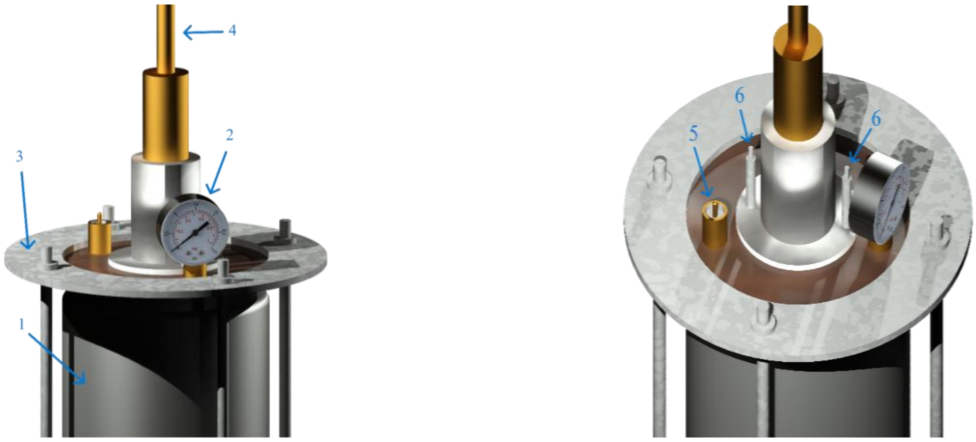
a) b) c)

Fig.3. Three-dimensional model of a retractable holder. a) standard retractable sample holder, b) modernized specialized retractable sample holder, assembled, c) modernized specialized retractable sample holder, disassembled. 1 – holder handle, 2 – heat insulating cover of the holder, 3 – cryogenic capillary system, 4 – temperature sensor (thermocouple), 5 – container

The bar with cylindrical cavities (cells) for sample placement (fig. 3, item 5) is crafted from copper, a high thermal conductivity material, to facilitate rapid and even distribution of temperature provided by the cryogenic capillary system. The cryogenic capillary system itself (fig. 3, item 3) is also constructed from copper for similar reasons.

Cooling the samples under study to a temperature of 77 K is facilitated by the continuous flow of liquid and gaseous nitrogen through the copper tube of the cryogenic capillary system. The close contact between the copper tube, copper bar, and the cells, along with copper's high thermal conductivity, enables swift cooling of the test material (such as hydrocarbon) to the desired temperature. The copper bar temperature is monitored using a thermocouple attached to the surface of the bar by the LakeShore PID controller (fig. 3, item 4). Heating the test samples is accomplished by halting the nitrogen circulation through the cryogenic capillary system, thereby restoring thermodynamic equilibrium between the sample and the surroundings. To circulate nitrogen through the copper tubes of the cryogenic capillary system, high pressure is established through heating in a specialized Dewar vessel. This process causes nitrogen to be expelled from the vessel into the cryogenic capillary system, enabling the cooling of the samples.

The cryogenic capillary system is connected to the Dewar vessel mentioned above through a hermetically sealing fastening system (figure 4). This installation is made to supply and cool samples at atmospheric pressure in the low-temperature cell with permanent liquid and gaseous nitrogen circulation (77 K) via copper capillaries. The installation consists of Dewar vessel (vacuum flask) (fig. 4, item 1), fastening system (fig. 4, item 3), copper capillary (fig. 4, item 4) covered with thermal insulating coating, manometer or pressure gauge (fig. 4, item 2), contacts for electrical clamp (fig. 4, item 6) to create electrical connection and provide with voltage from heater, check valve (fig. 4, item 5) to prevent exceeding pressure inside a flask.



a) b)

Fig.4. Cryogenic capillary system and Dewar vessel (vacuum flask)

To start an experiment, we first need to ensure that the installation is ready:

1) Dewar vessel which is connected to the cryogenic capillary system is firmly hermetically sealed, a check valve is not stuck and the manometer (pressure gauge) indicates pressure at least 6 psi (0,4 atm)

2) Voltage supplied from the heater is reaching a contact element inside the vessel and heating a liquid nitrogen which is supposed to circulate inside capillary system by thermal expansion

3) There is no electrical contact with other metal parts of the installation to avoid short circuit

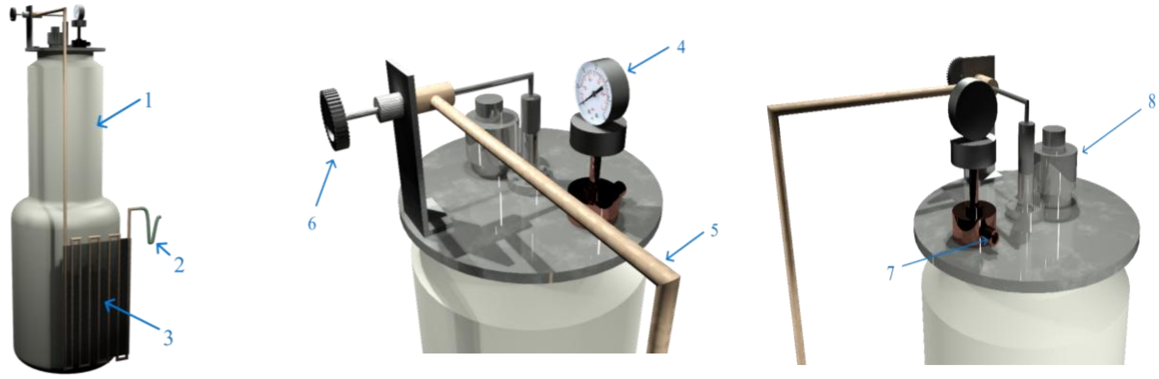
4) Thermocouple is working with LakeShore PID-controller

5) Capillaries are thoroughly covered with thermal insulating coating to avoid thermal conductivity with ambient environment while nitrogen is circulating inside them.

To ensure the experiment's purity, a blowing system was incorporated into the diffuse reflection attachment. By introducing gaseous nitrogen into the attachment, an inert environment is created, which not only safeguards the samples and the copper bar from frost formation but also supports the experiment's wholeness by creating IR-inactive environment. The next step of our experiment technique is preparation above mentioned blowing system, gasifier – ensuring hermetical sealing of the gasifier, pouring nitrogen into the gasifier's vessel, connecting it to the spectrometer's cuvette compartment with rubber hose in order to provide the diffuse reflection attachment, inside of which the IR radiation will pass, with inert IR-inactive environment that, in addition, lets us to avoid water vapor deposition on the samples while cooling. Preparation of gasifier is accurately described below:

1. Ensuring that the gasifier's Dewar vessel is empty and ready to get filled with nitrogen, being aware that manometer (pressure gauge) indicator is pointing to zero and only then opening the vessel's lid
2. The hose has to be attached firmly to both outlets and has no holes or another opening to avoid outflows
3. To check all radiators for frost absence, in order to provide quality gaseous nitrogen flow
4. When the gasifier is ready to be filled up, starting carefully pouring nitrogen into its vessel from another Dewar vessel
5. Closing the vessel's lid tightly and ensuring its manometer shows some additional pressure
6. Check the hose's outlet for the nitrogen to be flowing unhindered.

The gasifier's structure is shown in figure 5 – the vessel (fig. 5, item 1) is dedicated to store nitrogen since it is also a Dewar vessel, the manometer (pressure gauge) (fig. 5, item 4) shows overpressure inside the vessel and helps us to calculate approximate time of nitrogen exhaustion, the rubber hose (fig. 5, item 2) connected to the spectrometer to provide diffuse reflection attachment with a nitrogen inert IR-inactive environment, the capillary tube (fig. 5, item 5) for nitrogen transfer, the radiators (fig. 5, item 3) that are meant to conduct a heat and help to provide with gaseous nitrogen particularly, the lid (fig. 5, item 8) to prevent nitrogen outflow, the valve (fig. 5, item 6) to regulate nitrogen flow and the check valve (fig. 5, item 7) to avoid excess pressure.



a) b) c)

Fig.5. Gasifier generating nitrogen for inert IR-inactive environment

Temperature of the samples in container's cells changes as we cool them with nitrogen flow or heat them by stopping nitrogen circulation. We need to know exact temperature of the samples while taking a spectrum. To measure the temperature of the sample we use E-type thermocouple (chromel and constantan) connected to the LakeShore Model 325 cryogenic temperature PID-controller. Within the controller's interface we can set initial temperature, room temperature, thermocouple type, an output (A or B) of thermocouple contact, etc.

The controller provides two separate proportional-integral-derivative (PID) control loops. The PID algorithm determines the control output by considering the temperature setpoint and feedback from the control sensor. With broad tuning parameters, it is compatible with most cryogenic cooling systems and many small high-temperature ovens. A high-resolution digital-to-analog converter ensures smooth control output. Users can manually set the PID values, or use the Autotuning feature to automatically adjust the tuning process.

By connecting cryogenic capillary system to the cell and putting it into diffuse reflection attachment inside of "FSM 2203" FTIR-spectrometer, we start our experiment. As temperature is going down after reaching crystallization point of hydrocarbon substance, we are taking spectra at every few certain temperatures. One of our goals is to study hydrocarbons' physical and chemical properties at atmospheric pressure and nitrogen boiling temperature (77 K) by taking spectra of samples in these conditions. Once the samples' temperature has reached its minimal point and the PID-controller shows 77 K we are taking Fourier-transform infrared spectra and starting heating process by stopping nitrogen flow and letting thermodynamical equilibrium to take place, and in the same way take spectra at every few certain temperatures.

After ensuring that all installations and instrumentation we need are ready, we start our experiment of studying optical properties of test substances (hydrocarbon compounds) at low temperature (77 K) and at atmospheric pressure. To conduct the experiment, we perform the following steps:

Stage 1: Filling up the gasifier and cryogenic capillary system with nitrogen and sealing them properly. Connect them to the following instrumentations: 1) gasifier to the spectrometer's inlet of cuvette compartment with rubber hose to supply with inert environment, 2) cryogenic capillary system to the LakeShore 325 temperature PID-controller with E-type (chromel-constantan) thermocouple, and to heater with wires via alligator clips to create additional pressure inside the flask and thereby enhance nitrogen circulation through capillary.

Stage 2: Covering cuvette compartment with polypropylene and polyethylene film to keep IR-inactive inert nitrogen environment inside the cuvette for obtaining more quality IR-spectra, with only some space for micrometer screw and retractable holder. Due to comparatively similar molecular weight of air and N_2, it does not seem difficult to keep such an environment.

Stage 3: Putting pre-pestled potassium bromide sample, which absorbs moisture from ambient, into first cell of retractable holder's container bar so we can obtain its spectrum and use as base that will then divide test substance's spectra. Preparation of the test sample (kerosene in this case) includes only few steps – opening properly sealed vessel, picking up 1 ml kerosene using new disposable syringe, putting it into second and third cells (0.5 ml each) and inserting the holder into diffuse reflection attachment. Syringe is used to take equal doses of kerosene for each experiment. The kerosene used in our experiment is from brand "Derzhava" with sulfur content 1% according to GOST 18499-73.

Stage 4: Finding out the holder's position with the strongest intensity of IR-beam by adjusting vertical micrometer screw for every cell separately. Once it is found, we stop adjusting and further use this position to register samples' spectra by "FSpec" and analyze them in "Origin" software.

Stage 5: Taking spectra while cooling samples starting from room temperature (290-300 K) and all the way down to the nitrogen boiling point (77 K), using temperature controller to real-time determine the sample temperature and applying heater to force nitrogen inside the flask flowing more intensively through capillary. It is important not to miss test substance's phase transition and record its spectra in each phase to further describe the differences. After passing phase transition temperature taking a few spectra every certain temperature until reaching lowest point – 77 K and registering samples spectra at this point. After that we stop the heater thereby reducing nitrogen circulation and let the thermal equilibrium to take place. This will force a sample to get heated and increase its temperature while we are taking spectra. Afterwards we compare sample's cooling and heating spectra and describe their differences.

Stage 6: After obtaining spectra we needed, stopping the experiment by removing the holder from attachment, electrical contacts from vacuum flask and PID-controllers thermocouple. Blowing spectrometers compartment with nitrogen to get rid of possible residual hydrocarbon vapors and thereby cleaning inside of diffuse reflection attachment and FSM 2203 spectrometer.

Afterwards we need to analyze spectra obtained by this experiment and draw up the results. These research and experiment can be used as fundamental information for many spectroscopic studies of hydrocarbon materials since results are obtained at low temperature but at atmospheric pressure. But before that it is important to compare our results with another data of obtaining spectra of hydrocarbons using low-temperature setups.

3. Results and discussion

Results of the experiment are shown in figure 6. Test substance is kerosene, a hydrocarbon used in energy, technological and other industries as a fuel or solvent. In this transmission spectrum, characteristic peaks of the functional groups of kerosene are visible, in particular, the stretching and bending of the C-H bonds, one can also notice a tendency to shift the peak of the C-H bond bending relative to the results of other authors, a difference in wave numbers is obvious during the stretching of the above-mentioned bond in the kerosene molecule, that is, a hypsochromic shift.

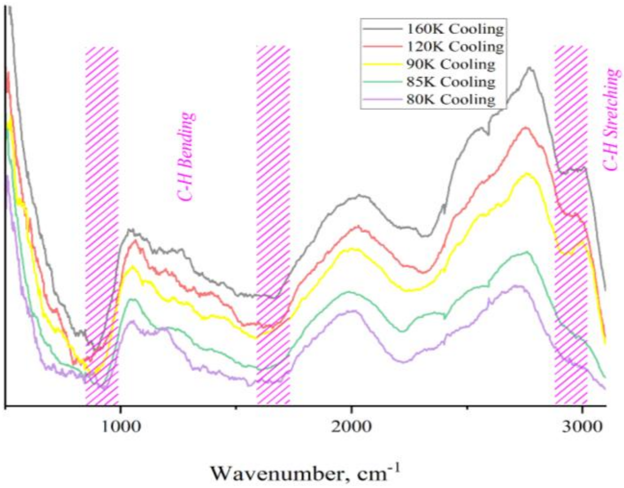


Fig.6. Spectra of kerosene while cooling from room temperature (only few temperatures are shown)

As we can see, there is the C-H stretching in the wavenumber range of 2800-3000 cm^{-1}, mild hypsochromic shift in the range of 1500-1700 cm^{-1} for the C-H bending (scissoring) and almost no shift in the range of 600-900 cm^{-1} for C-H bending (rocking and out-of-plane) relative to the results of other authors. Highlighted with dotted lines areas represent the peaks that indicate presence of kerosene key C-H group and its vibrations at specific wavenumber range. These lines are meant to give importance and significance to the vibrations. To show data in more clear way we are providing the table of our research and other authors research results of kerosene spectra (tab. 1).

Table 1. Comparison of spectra results

№	Authors	C-H stretching (cm^{-1})	C-H bending (scissoring) (cm^{-1})	C-H bending (rocking and out-of-plane) (cm^{-1})
1.	Our data	2800-3000	1500-1700	600-900
2.	Dollah, A., Zainol Rashid, Z. et al [24]	2800-3000	1300-1500	800
3.	Mirea, R., & Cican, G. [25]	2800-3100	1300-1500	650-800
4.	Biaktluanga, L., Lalhruaitluanga, J. et al [26]	2800-3000	1500	800

Results of authors [24] correspond to the wavelength under normal conditions: C-H stretching at 2800-3000 cm^{-1}, C-H bending (scissoring) at 1300-1500 cm^{-1} and C-H bending (rocking and out-of-plane) at 800 cm^{-1}. Other researchers' results [25] are given as well; they correlate well with our data what additionally confirms effectiveness of the measurement technique used in our research. More of other authors' results in this field [26] are given. We can see the same characteristic peaks we observed in previous comparisons, particularly C-H stretching at 2800-3000 cm^{-1}, C-H bending (scissoring) at 1500 cm^{-1} and C-H bending (rocking and out-of-plane) at 800 cm^{-1}. The physical meaning of this comparison is that we obtained spectral results of characteristic peaks of kerosene molecules' key functional groups, well-correlated with results of other respected authors in this field. This comparison confirms the effectiveness of our new cryogenic setup, used for obtaining FTIR-spectra of hydrocarbon fuels at low temperature and atmospheric pressure.

The kerosene spectra at low temperature shown in fig. 6 are obtained at atmospheric pressure, which undoubtedly influences the spectra and vibration wavenumbers of the sample, and that is proved by hypsochromic shift of C-H stretching, which was mentioned before. This result shows that conditions of test sample as well as the way of obtaining solid kerosene (below 226 K) directly affect FTIR-spectra, since other researchers' spectra being compared with our data [24-26] are received in vacuum.

We are truly sure that given comparison analysis proves the operability and effectiveness of our setup with low-temperature cell and cryogenic-capillary system being used in our experiments at low temperatures (down to 80 K) and atmospheric pressure (101,325 Pa, 1 atm).

4. Conclusions

In this paper, we have described an experimental setup and measurement technique for studying hydrocarbon compounds, particularly a kerosene, at low-temperatures and atmospheric pressure using the cryogenic-capillary system with nitrogen blowing system, the gasifier, and high resolution FTIR spectrometer. The verification performed by comparing the FTIR spectra of kerosene obtained using our setup with the results of FTIR spectra reported by other researchers allows us to conclude that there is a good correlation.

Also, we can say that kerosene being studied at atmospheric pressure has slightly different result in one characteristic peak of one vibration, related to the C-H stretching, from research results of kerosene under vacuum. That can be valuable fundamental information about kerosene (may be not only) properties and behavior. The presented setup has a number of advantages, such as a possibility to study low-temperature samples at atmospheric pressure, to obtain profoundly new fundamental data about properties and structure of hydrocarbon substances under unusual conditions and opportunity to present it to scientific community as a new operable device for cryogenic studies at atmospheric pressure.

Conflict of interest statement

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

CRediT author statement

Kenbay A: Methodology, Formal analysis, Investigation, Writing – Original Draft, Visualization; **Yerezhep D:** Writing – Review & Editing, Validation, Data Curation, Project administration; **Aldiyarov A:** Conceptualization, Resources, Supervision, Funding acquisition. The final manuscript was read and approved by all authors.

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