ANNOTATION

of the dissertations thesis for the degree of Doctor of Philosophy (PhD) in the educational program “8D07502 – Standardization and certification (by industry)” by Akylbaeva Aigerim “Regulatory and technical documentation development for IR spectrometric studies of cryocrystals molecules properties”

General description of the work

The dissertation work presents the results of the development of regulatory and technical documentation for IR-spectrometric studies of the properties of molecular cryocrystals.

Relevance of the dissertation theme

Standardization and certification is aimed at establishing technical requirements for reusable tasks in science, technology, industry, and economics. The result is a regulatory and technical document in the field of technical regulation, that is, a state standard.

Standardization, as a type of activity, is aimed at achieving an optimal degree of ordering in a certain area. Thus, the modern industrial world strives for an optimal degree of ordering and special attention is paid to the accuracy and quality of scientific and technological production, which is directly related to standardization and metrology. This is because scientific components are becoming more complex, and quality control requirements are increasing. Therefore, new technologies are being developed to meet high regulatory requirements.

According to the Law of the Republic of Kazakhstan “On commercialization of the results of scientific and (or) scientific and technical activities”, commercialization of the results of scientific and (or) scientific and technical activities along with scientific and educational activities is a priority activity of scientific organizations.

There is a need to develop a number of regulatory and technical documents for conducting qualitative research in the field of modern condensed matter physics. This is because the actual problem of the formation of disordered systems is the study of polymorphic transformations, which are accompanied by the formation of intermediate metastable states. The metastable state largely depends on the state of the thermodynamic system and physicochemical properties. As an example of this kind of condensed matter, we can consider molecular cryocrystals that exist only at low temperatures.

From a fundamental point of view, molecular cryocrystals are interesting in many areas, such as condensed matter physics, astrophysics, low temperature physics and materials science. Thus, cryocrystals include crystals of hardened inert gases in which Van der Waals forces bind atoms and molecular cryocrystals, such as nitrogen, carbon monoxide, carbon dioxide, methane. Molecular cryocrystals are interesting as ideal objects for studying a number of fundamental problems of low temperature physics and condensed matter physics. It should be noted that there are cryocrystals whose particle motion energies are comparable to the energy of intermolecular interaction. Such cryocrystals form macroscopic systems whose behavior is determined by quantum physics and are interesting during both thermosetting and thermal cycling.

Thus, it is necessary to improve the control of the external parameters of cryocondensation and the release of the test substance for further detailed study of the isolated form of the polymorphic state of molecular cryocrystals. The development of regulatory and technical documents (techniques) will allow precise control and standardization of experimental conditions to establish an unambiguous correlation between the properties of molecular cryocrystals and the conditions of their formation.

Based on the above, the dissertation work is devoted to improving the quality of research and the development of regulatory and technical documentation for IR spectrometric studies of the properties of molecular cryocrystals.

Connection of the dissertation topic with the plan of scientific works

The dissertation work was carried out in accordance with the plan of fundamental research works of the Ministry of Internal Affairs of the Republic of Kazakhstan "Grant financing of scientific research" on the topics: "Structural-phase transformations and relaxation processes in thin films of cryovacuum condensates of glass-forming organic molecules" (2020-2022, code AP08855738, State registration No. 0120RK00576); "Study of the effect of cryocondensed radiation on optical characteristics working surfaces of cryogenic equipment" (2020-2022, code AP08052736); "Investigation of the processes of cryosacception and desorption of linear molecules in a water film at low temperatures" (2020-2022, code AP08855681, State registration no. 0120RK00577); "Study of the sintering mechanism of bulk nanostructured thermoelectric materials in the process of their formation" (2022-2024, code AP15473758); "Development of a universal cryosurface for cooling and scientific research of physico-chemical and optical properties of substances from room temperatures to 80 K" (2023-2025, code AP19576644).

The purpose of the dissertation is to solve the scientific and technical problem of ensuring the effectiveness of standardization by developing regulatory and technical documentation for conducting IR spectrometric studies of the properties of molecular cryocrystals on a universal cryovacuum installation by gas-phase deposition.

To achieve this goal, it was necessary to solve the following tasks:

1. Comprehensive analysis of the problems of obtaining high quality and standardized results of IR spectrometric studies of the properties of molecular cryocrystals;
2. Upgrade the universal cryovacuum plant to improve the quality of the experiments;
3. To develop a method for calibration of the IR-Fourier spectrometer and a method for obtaining a thin film of a cryocrystal for conducting IR-spectrometric studies of the properties of molecular cryocrystals on a universal cryovacuum installation by gas-phase deposition;
4. To develop a methodology for performing measurements on a universal cryovacuum installation to obtain standardized test results with a given uncertainty interval.

The object of the study is thin films deposited on a cooled substrate in the temperature range from 10 to 200 K and pressures from 10^{-6} to 10^{-3} Torr, leading to a change in the transmission coefficient of infrared radiation during thermal cycling.

The subject of the study is the thermophysical and optical properties of molecular cryocrystals.

Research methods

When solving the tasks necessary to achieve the goals, the following methods were used:

Laser interferometric method for measuring the condensation rate, thickness and refractive index of cryocapacitors of water, carbon dioxide, freon 134a and a mixture of methane;

IR spectroscopic method for analyzing the processes of structural-phase transformations of freon 134a and a mixture of methane using an IR Fourier spectrometer;

Thermodesorption method for determination of thermophysical and optical parameters in samples of freon 134a and a mixture of methane.

The scientific novelty of the work lies in the fact that for the first time

1. The universal cryovacuum installation has been modernized by retrofitting with modern equipment, computer technology and software, allowing for a better study of fundamental characteristics related to the formation processes and properties of cryocondensed systems at low temperatures.

2. A method of calibration of the IR-Fourier spectrometer makes it possible to assess the reliability of experimental data results in the wavenumber range of 400-7800 1/cm with a confidence probability of 95% and a coverage factor of 2.

3. The developed standardized measurement technique is effectively used in the preparation and analysis of thin films of molecular cryocrystals under conditions of ultra-low vacuum, respectively, from 0.1 to 100 μTorr and low temperature from 10 to 50 K.

Provisions submitted for protection

1. The modernized universal cryovacuum installation provides control of the inlet of the test substance at pressures in the range from 1 to 100 μTorr, and increases the spectral range from 420-4200 1/cm to 370-7800 1/cm with a resolution from 1 1/cm to 0.5 1/cm when obtaining IR spectra during the experiment, while reducing the average time for performing a standard experiment by 4 times (from 8 hours to 2 hours).

2. The developed calibration technique for the IR-Fourier spectrometer of a universal cryovacuum installation makes it possible to assess the reliability of the results of experimental data on a scale of wavenumber intervals (400-7800 1/cm) and guarantees a quantitative assessment of the performance with a confidence probability of 95% and a coverage factor of 2.

3. The developed standardized measurement technique with a specified uncertainty of 0.95 1/cm makes it possible to obtain thin films of molecular cryocrystals under conditions of ultra-low vacuum in the range from 0.1 to 100 μTorr and low temperature from 10 to 50 K.

Theoretical and practical significance of the work

The results of this study, the development and justification of regulatory and technical documentation for IR spectrometric studies of the properties of molecular cryocrystals allow us to obtain fundamental results in low-temperature condensed matter physics.

In the course of the dissertation work, a technique for obtaining a thin film of cryocrystals, a technique for measuring the optical properties of thin films of cryocrystals, as well as a calibration technique for an IR-Fourier spectrometer was developed.

With the modernization and introduction of a new measurement methodology, not only an improvement in the quality of the experiment was achieved, but also a significant economic effect was achieved from the introduction of the received regulatory and technical documents, which amounts to 25 million tenge over 30 years.

The results of the work were introduced into the Testing Laboratory of "Ғылыми-зерттеу орталығы «Алматы-Стандарт»", and an implementation document was received.

The practical significance of the results obtained is confirmed by publications in international peer-reviewed publications and participation in regular international conferences, as well as by the patent of the Republic of Kazakhstan No. 7959, the copyright certificate of the Republic of Kazakhstan No. 33250.

The author's personal contribution lies in the fact that the entire volume of the dissertation work, the choice of the research method, problem solving and the development of regulatory and technical documentation was carried out by the author independently. The task statement and discussion of the results were conducted jointly with scientific supervisors.

Reliability and validity of the results obtained

The reliability and validity of the results obtained is confirmed by publications in highly rated journals of the far abroad with a high impact factor and in publications recommended by the Committee for Quality Assurance in the Field of Education and Science of the MSHE of the Republic of Kazakhstan for the publication of the main results of scientific activity, and in the proceedings of international scientific conferences of the near and far abroad.

Publications

Based on the materials of the dissertation, 15 publications were published: 6 papers in the materials of International scientific conferences, 4 in journals from the list of by the Committee for Quality Assurance in Education of the Ministry of Education of the Republic of Kazakhstan for publishing the main results of the dissertation for the PhD degree and 2 articles in the journal of foreign countries with a high impact factor included in the international information resources Web of Science (Clarivate Analytics, USA) and Scopus (Elsevier, Netherlands).

Structure and scope of the dissertation

The dissertation consists of an introduction, five sections, a conclusion, a conclusion; a list of sources used and contains fourteen appendices. The work is presented on 194 pages of typewritten text, illustrated with 52 figures, 18 formulas, 26 tables are given, the list of sources used contains 143 titles and 14 appendices.