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Improvement of the automatic temperature stabilisation process in the cryovacuum unit

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Abstract

This study concerns the issues of temperature stabilization in units used to research the properties of molecules at low and ultra-low temperatures. This research is relevant due to the need to increase the speed and accuracy of the data obtained. Using the LabView graphical programming environment tools, a control program was created for the LakeShore 325 thermocontroller which reacts when the current temperature is close to the control point temperature set by the researcher. By adding controls for the heating element power and PID controller boot times, it is possible to use them more flexibly. The method was verified for the temperature control points of 40 K, 100 K, 150 K and 200 K. A comparison of the proposed temperature stabilization program with the standard PID controller solution demonstrates the advantages of the former. The speed of reaching the control points was doubled. The digitalization of the LakeShore 325 thermocontroller makes it possible to work further on improving temperature stabilization. The resulting increase in the accuracy–time stabilization ratio makes it possible for those who conduct low-temperature experiments to improve the quality of their measurements dramatically. The introduction of a digital version of the temperature control device opens up possibilities for further automation of cryovacuum units by linking the thermal control program with other programs, for example, recording the spectra at specific temperature values.

Keywords

automation, temperature control, low temperatures, PID-controllers, IR spectroscopy, programming

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Улучшение процесса автоматической стабилизации температуры в криовакуумной установке

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Аннотация

Предмет исследования. Рассмотрены вопросы температурной стабилизации в установках, предназначенных для выполнения исследований свойств молекул при низких и сверхнизких температурах. Актуальность работы обусловлена необходимостью повышения скорости и точности получаемых данных, на которые в основном оказывает влияние температура исследования. **Метод.** С помощью инструментов программирования графической среды LabView создана управляющая программа для термоконтроллера LakeShore 325, реагирующая на приближение (рабочей) температуры к температуре (заданной) контрольной точки. Добавление элементов управления мощностью нагревательного элемента и временем включения PID-регулятора позволяет использовать их более гибко. Проведена верификация метода стабилизации для контрольных точек температуры 40, 100, 150 и 200 K. **Основные результаты.** Сравнение предложенной программы стабилизации температуры со стандартным решением в виде PID-регулятора показало его преимущество. Получено увеличение скорости достижения контрольных точек до двух порядков. Цифровизация термоконтроллера LakeShore 325 дала возможность выполнять дальнейшие работы по совершенствованию температурной стабилизации. **Практическая значимость.** Полученное увеличение соотношения точность–время при стабилизации позволило в разы улучшить качество проводимых измерений в области низких температур. Внедрение цифровой версии терморегулирующего прибора открывает возможности для дальнейшей автоматизации криовакуумных установок с помощью объединения программы термоконтроля с другими программами, регистрирующими, например, спектры при определенных значениях температуры.

Ключевые слова

автоматизация, контроль температуры, низкие температуры, PID-регуляторы, ИК спектроскопия, программирование

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Introduction

Over the last decades, studying the behavior of various molecules at low and ultra-low temperatures has been of great scientific interest [1, 2]. Firstly, observing molecular complexes at negative temperatures allows the thermodynamics of the processes involved to be determined, the simplest stages of complex reactions to be determined, and the selectivity of the processes to be ensured. The processes of the formation of glass-like states of various molecules are important and relevant [3–7]. Secondly, ultra-low temperatures significantly affect the physical and mechanical (ultimate strength and endurance) as well as thermophysical (heat capacity, thermal conductivity, linear thermal expansion coefficient) properties of some substances [8, 9]. Due to low temperatures, the material experiences brittle fracture or changes one or more of its thermophysical properties. And thirdly, the study of infrared (IR) spectra of molecules and molecular compounds at low temperatures allows us to answer a number of questions and describe a number of processes occurring in open space [10–21].

So far, astrochemical studies conducted in ground-based and space laboratories have provided IR evidence for about a dozen extraterrestrial molecules and ions in the solid phase [19]. H_2O ice and ice mixtures consisting primarily of H_2O compounds have been found to be important constituents of interstellar particles, comets and a number of planetary satellites [21].

Identification of extraterrestrial H_2O [14, 20], CH_3OH [11, 12], C_2H_5OH [13, 15, 16], NH_3 [22], CO_2 [23] and CH_4 in their condensed state was performed by comparing the spectra taken in the near- and mid-infrared ranges (100 m^{-1} to 4 m^{-1}) with the spectra of the same molecules obtained in the ground-based laboratories. The obtained IR spectra of low molecular weight amorphous and crystalline solids, focusing on amorphous ice below 100 K, are used to study the low-temperature chemistry of interstellar clouds and extraterrestrial solar system objects such as ice-covered moons and trans-Neptunian objects [17, 18].

In spite of the fact that the IR spectra of molecules in various low-temperature ranges have been studied, an important challenge is the stabilization of the temperature in the research unit and the minimum temperature step of obtaining the IR spectra of the sample under study. At the moment, the problem of stabilization is solved by using a proportional–integral–derivative (PID) temperature controllers with a heating element until the temperature points set by the experimenter are reached. However, such temperature controllers are not flexible enough and often lack a graphical interface and special software that would make it possible to configure them more flexibly for specific tasks. As a result, the accuracy of the experiment is negatively affected, and it is not possible to obtain infrared spectra at certain temperatures or with small temperature steps; or it takes the researcher a lot of time to do so.

The aim of this research is the temperature stabilization of the cryogenic vacuum spectrophotometer unit at Al-

Farabi Kazakh National University in order to solve the problems highlighted above. The results pursued include reducing the time of the experiment, reducing the temperature step of obtaining IR spectra of various molecules at low temperatures and creating more flexible conditions for future experiments.

Methodology

Experiments to verify the work done were performed on a universal cryogenic vacuum spectrophotometer unit in the cryolaboratory at Al-Farabi Kazakh National University. LakeShore 325 thermal controller (LakeShore Cryotronics, USA) is used as a temperature controller in this cryovacuum unit. This temperature controller model is a two-channel regulator, capable of working with various types of temperature sensors (diodes, resistance temperature detectors, thermocouples). In addition to the sensors, the controller also features two independent PID control loops with 25W and 2W heater output, controlling either 50-ohm or 25-ohm loads for optimal flexibility in temperature control of the cryogenic unit.

The PID controller integrated into the thermal controller plays a special role in achieving our objectives as it is used for the initial stabilization of the heating element temperature. The PID controller is a feedback device in the control loop of the system which generates the control signal to produce an output signal with the required accuracy. The control signal is the sum of three other signals: the error signal (proportional to the difference between the input and feedback signals), the integral of the error signal and its derivative.

Therefore, the output signal of the controller u is defined as

$$u(t) = P + I + D = K_p e(t) + K_i \int_0^t e(\tau)d\tau + K_d \frac{de}{dt},$$

$$e(t) = r(t) - y(t),$$

where $r(t)$ is the input signal; $y(t)$ is the output sum signal; $e(t)$ is the discrepancy or deviation of the value from the original signal; P is the difference component; I is the time-integral component; D is the time-differentiation component; t is the current moment in time; τ is the time step; and K_p, K_i and K_d are the amplification factors of the corresponding controller components.

Despite the advantages and seemingly complete operability of the PID controller as a stabilizing component of the temperature controller, it was observed during the experiments that it took quite a long time to reach the set temperature. Besides, when approaching the set temperature, significant fluctuations were observed near it, sometimes up to 5 K, which can cause significant deviations in the final results when conducting an experiment. The hypothesis was made that the insufficient accuracy and speed of the temperature stabilization performed by the PID controller is related to its inability to “predict” the behavior of the heating element within a wide range. Most likely due to the uneven heating after switching off the cooling unit of the machine, the PID controller is unable to reduce the heating power in time, constantly “jumping over” the set temperature.

Therefore, the automation element to be introduced into the system must first address the issue of predicting the speed at which the sample substrate is heated in order to minimize the time it takes for the PID controller to stabilize the temperature near the control point set by the experimenter. So, this automation element must have direct control over the LakeShore 325 thermal controller, be able to change the power of the heater, the gains of the PID controller and, if possible, switch on and off the McMahon machine that is used to lower the temperature of the substrate and the experimental chamber of the unit.

The software suite LabVIEW (National Instruments, USA) was chosen as the environment in which the automation would be performed. This development environment allows executing programs written in graphical programming language G. This choice was motivated by the simplicity of the development of applications in the LabVIEW environment, a wide range of features as well as the availability of a large number of ready solutions, which can simplify the process of writing the program required to fulfill the task.

Results

The stabilization of the PID controller was improved by adding elements to the program that react to the current temperature of the heated substrate approaching the temperature set by the experimenter. It is known that PID controllers also react to approaching the control point by reducing the power of the heater if P, I and D parameters are properly adjusted. However, this process either takes too long or is characterized by “jumping over” the control point (up to 1.5–2 K) followed by harmonic equalization of temperature. Both options are not good enough for our research carried out on a cryogenic vacuum spectrophotometer, since in the first case, the duration of the experiment would be significantly increased by the slow speed of reaching the control points, and in the second case, the spectrum close to the control point would be obtained with fluctuations of more than 1 K which is also undesirable.

Thus, the proposed solution solves the two disadvantages of the options mentioned above: it allows reaching the reference temperature quickly and at the same time, makes the fluctuations near it less severe. This is achieved by activating the PID controller later in relation to the modes with the default settings of the P, I and D parameters. As a result, the control temperature is reached more quickly (due to the longer operation of the heater at 100 % power). The PID controller is activated by comparing the control temperature with the current temperature, incremented by a small value, which has been calculated experimentally and assumed to be 0.5 K. When the current temperature reaches the reference temperature with an increment value, the heating power is reset to 5 % and then, after a short delay, the PID-controller is activated. After the power of the heater is reduced, the temperature of the substrate starts to decrease, but the activated PID controller soon stabilizes the temperature close to the set control point.

To confirm the improved performance of the modified temperature stabilization algorithm, it is necessary to compare it with the traditional method of regulation using the PID controller integrated in the LakeShore 325 thermocontroller. It is important to compare the performance of the two stabilizing components over a wide range, capturing both low (below 100 K) and high (100–200 K) temperature ranges. Fig. 1 and 2 show the graphs comparing the temperature stabilization of the standard PID controller of the LakeShore 325 thermocontroller with the default settings of $P = 50$, $I = 20$, and $D = 5$ and the improved (i-PID) controller with the adjustments mentioned above for the temperatures of 40 K, 100 K, 150 K, and 200 K. The temperature-time dependencies were obtained 10 K before the control point in order to investigate a more complex stabilization process than with a small step.

As can be seen from the graphs, in three of the four cases, the heating up to the control point was faster with the improved PID controller. Slower heating was observed only when the reference point of 100 K was reached. Obviously, faster heating of the substrate is achieved by activating the PID controller later, thus keeping its power at 100 % until the very last moment, and by using it more flexibly close to the control point.

In all cases, stabilization near the control point was faster with the improved PID controller. The advantage of the i-PID controller is particularly noticeable in Fig. 1 where stabilization near 40 K was achieved about three times more quickly than with the standard PID controller. The difference in the speed of reaching the control points in Fig. 1 and 2 is presented in the form of timestamps. It can be seen that in the case of stabilization to 40 K, the method has an advantage of 18 seconds (Fig. 1, *a*); for stabilization to 100 K, it has an advantage of 165 seconds (Fig. 1, *b*); for 150 K, it has an advantage of 60 seconds (Fig. 2, *a*); and for 200 K, it has an advantage of 70 seconds (Fig. 2, *b*).

Reaching the 100 K and 200 K control points (Fig. 1, *b* and Fig. 2, *b*) with the i-PID controller was about twice as fast as with the standard PID controller. Once the control point of 150 K was reached, there was only a small advantage in using the i-PID controller — primarily the smaller deviation. The faster stabilization of the i-PID controller can be explained by the correctly selected timing

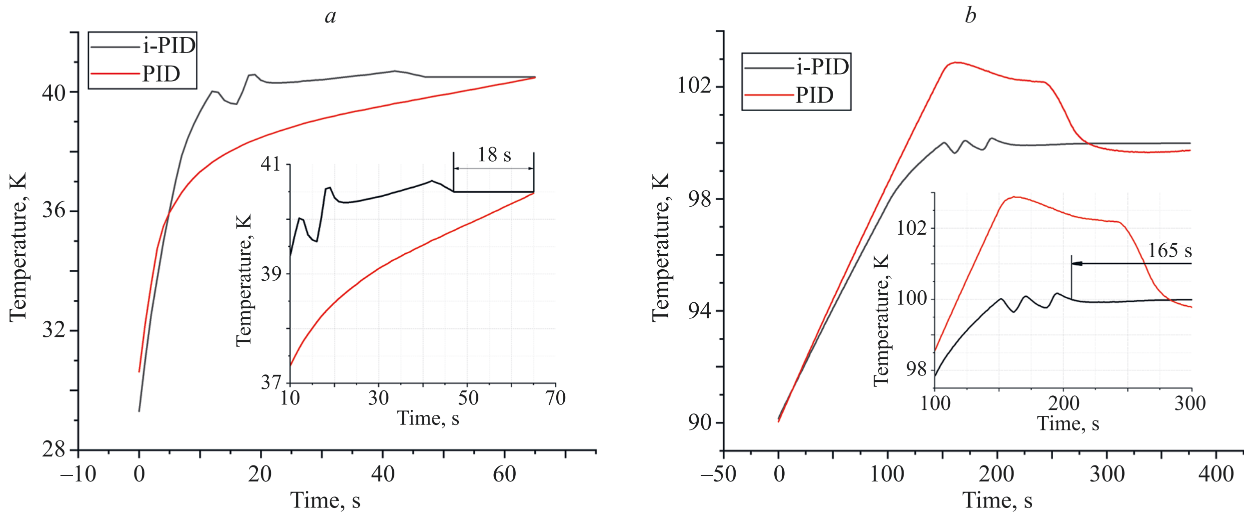

Fig. 1. Standard (PID) and improved (i-PID) thermocontrollers stabilization speed close to the control points of 40 K (*a*) and 100 K (*b*)

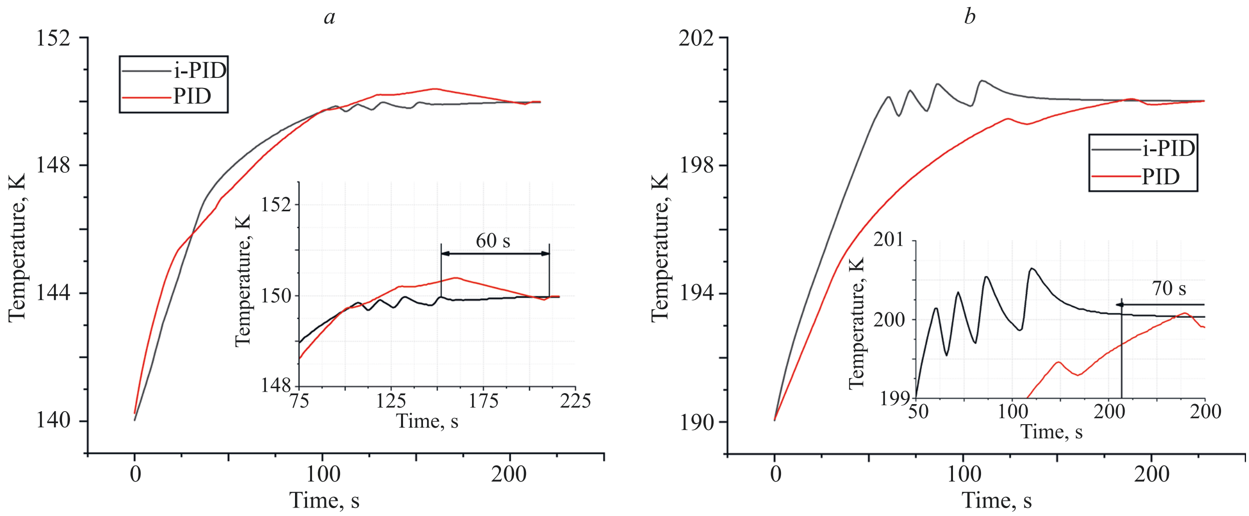

Fig. 2. Standard (PID) and improved (i-PID) thermocontrollers stabilization speed close to the control points of 150 K (*a*) and 200 K (*b*)

of the increase and decrease of the power of the heating element, simultaneously with controlling the PID controller by turning it on and off at the right time.

It is worth noting that it is also possible to reach the control points more quickly when using a standard PID controller by changing the *P* and *I* values and removing the *D* component. However, with this approach, the fluctuation was found to be too long and significant (about 1–2 K), this prevented the IR spectra from being obtained immediately after reaching the control point and required an equally long wait for the stabilization as with the option of the parameters $P = 50$, $I = 20$ and $D = 5$ explored in this article.

Conclusion

The results achieved in this research may seem insignificant. However, for further work on the automation and improvement of the operation of the experimental cryovacuum unit, the obtained gain of 20–160 seconds is significant, not to mention obtaining the temperature change not exceeding 0.5 K at the moment of reaching the control points.

Despite the versatility and accuracy of stabilization, the PID controller can still be improved by incorporating a control element capable of switching the PID controller on and off within designated ranges and independently (without relying on the PID controller) changing the power of the heating element. This has been demonstrated in this research.

An important issue is the response of the demonstrated i-PID controller to the heating step value (the temperature range between the current temperature and the set control point) on which the stabilization time strongly depends. In this research, only the case of a relatively large heating step has been considered implying more complex stabilization conditions. Nevertheless, it is worth investigating whether it is possible to reach and stabilize the temperature near the control point even faster with smaller heating steps by varying the selected stabilization factor.

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