Conference materials
UDC 535,015
DOI: https://doi.org/10.18721/JPM.173.233

A new method of managing the discretization of the scale in a mobile differential refractometer

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Abstract. The necessity of developing a new method for managing the discretization of the scale in a mobile differential refractometer has been justified. The implementation of this method is necessary to expand the functional capabilities of the developed mobile differential refractometer (providing a measurement mode of the refractive index of a liquid medium ranging from 1.23 to 2.63 with an error of 0.0001). All existing liquid media and their mixtures worldwide fall within this measurement range. When using other models of compact and mobile refractometers for express control, such a measurement range of n cannot be provided. Within the range of change of *n* from 1.23 to 2.63, a new management method has been implemented, which ensured a measurement error of 0.0001. Studies of various media have been conducted, confirming the adequacy of our development.

Keywords: refraction, liquid, refractive index, Anderson cuvette, laser radiation, photodiode array, measurement error

Funding: This research was funded by Russian Science Foundation (RSF), grant 21-72-20029.

Citation: Provodin D.S., Yakusheva M.A., Davydov V.V., A new method of managing the discretization of the scale in a mobile differential refractometer, St. Petersburg State Polytechnical University Journal. Physics and Mathematics. 17 (3.2) (2024) 166–172. DOI: https://doi.org/10.18721/JPM.173.233

Материалы конференции
УДК 535,015
DOI: https://doi.org/10.18721/JPM.173.233

Новая методика управления дискретностью шкалы в мобильном дифференциальном рефрактометре

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Abstract. Обоснована необходимость разработки новой методики управления дискретностью шкалы в дифференциальной кювете Андерсона. Для расширения функциональных возможностей использования разработанного мобильного дифференциального рефрактометра в его конструкции обеспечен режим измерения показателя преломления жидкой среды от 1,23 до 2,63 с погрешностью 0,0001. В этот диапазон измерения n попадают все существующие в мире жидкие среды и их смеси. Другим моделям малогабаритных и мобильных рефрактометров для экспресс-контроля данный диапазон измерения *n* не доступен. Для реализации контроля значения *n* в

диапазоне изменения от 1,23 до 2,63 был разработан новый метод измерения. Проведены исследования различных сред, которые подтвердили адекватность нашей разработки.

Keywords: рефракция, показатель преломления жидкости, кювета Андерсона, лазерное излучения, фотодиодная линейка, погрешность измерения

Финансирование: Работа выполнена при финансовой поддержке Российского научного фонда в рамках программы «Суперкомпьютерное моделирование и технология биомолекулярных пленочных структур» (грант № 20029-72-21).

Ссылка при цитировании: Проводин Д.С., Якушева М.А., Давыдов В.В. Новая методика управления дискретностью шкалы в мобильном дифференциальном рефрактометре // Научно-технические ведомости СПбГПУ. Физико-математические науки. 2024. Т. 17. № 3.2. С. 166–172. DOI: https://doi.org/10.18721/JPM.173.233

Introduction

The development of scientific and technological progress has led to an increase in the number of liquid media and their mixtures used in various research, industrial, and pharmaceutical production, among other fields [1–4]. To obtain reliable results, both in research and in ensuring the technological cycle in production, it is necessary to monitor the state of the liquid medium [1, 2, 5–8]. The greatest difficulties arise in the express monitoring of liquid media. These difficulties are related to the fact that the device for express monitoring must, on the one hand, be mobile, and on the other hand, provide measurements for monitoring the state of a large number of liquid media and their mixtures [8–11]. Currently, only two devices meet these requirements: nuclear magnetic resonance (NMR) meters and refractometers [9–15].

Refractometers are the most preferred among these two devices due to their ease of use, lightweight design, and cost. To date, a large number of different mobile refractometers have been developed for the express monitoring of liquid media. All of these devices have limitations regarding the type of liquid being measured and the measurement accuracy of liquid medium parameters, which restricts their application in various fields. One of the most promising solutions to these problems is the use of our developed differential refractometer [8, 15], which allows measuring the refractive index n_m of a liquid medium in the range from 1.23 to 2.63 (covering all existing liquid media and their mixtures).

When solving the problem of measuring n_m in this range (from 1.23 to 2.63), measurements using a mobile differential refractometer must ensure a measurement error of 0.0001 (in relative units) or less, as this is a requirement for the rapid control of liquid media and their mixtures [7, 8, 10, 11]. The problem of conducting n_m measurements with such accuracy lies in the following. Direct measurement of refractive index in differential refractometer designs is extremely difficult, as the photodiode array contains 4096 pixels, which, with a measurement step of refractive index at 0.0001, will not allow for measurements across the entire range. Therefore, the goal of our work is to develop a new measurement methodology that will allow managing the discretization of the refractive index measurement scale to ensure a measurement error of 0.0001 in the range from 1.23 to 2.63.

Differential Anderson cuvette and new method for managing the optical axis of laser radiation

The main measuring elements in the design of the differential refractometer are the Anderson cuvette and the photodiode array, on which the position of the optical axis of the laser radiation is registered. Fig. 1 shows the design of the Anderson cuvette and the change in the trajectory of the optical axis of the laser radiation for calculating its displacement on the photodiode array. To register the position of the laser radiation axis in the developed refractometer design [8, 15], a photodiode array with 2048 sensors (pixels) is used. This allows, at the first measurement step, to

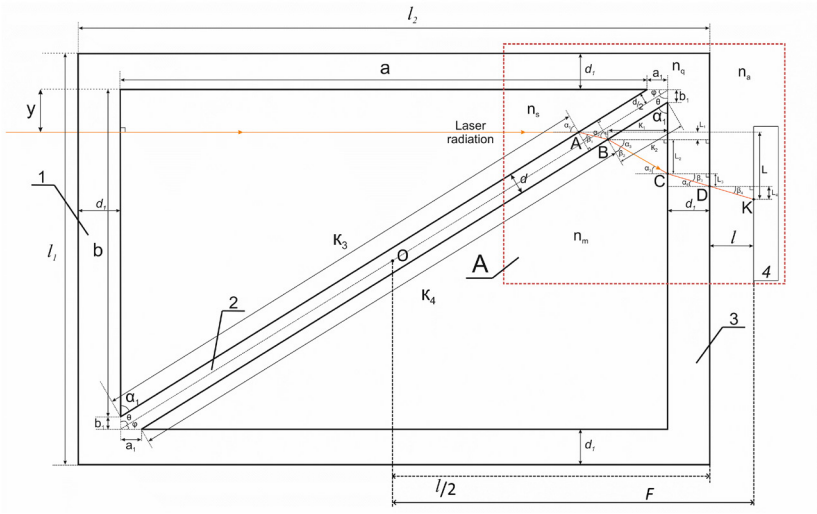

Fig. 1. Structural diagram of optical part of laboratory model of differential refractometer for measuring refractive index: Anderson cuvette wall *1* through which laser radiation is introduced; partition *2* in differential Anderson cuvette; cuvette face *3* for laser radiation output; photodiode array *4*

control the change in the refractive index n_m in the range from 1.230 to 2.630 with a step of 0.001 (with a margin of 600 sensors on each end of the array).

This construction ensures the necessary measurement range of n_m with a 20% margin, which meets modern requirements for measuring instruments. This construction of the laser radiation axis registration scheme allows measurements of n_m to start from the 310th sensor, significantly reducing edge effects that previously occurred when using 1024 sensors in the photodiode array. To determine the value of n_m (of the studied liquid) in the two compartments of the Anderson cuvette, reference and test liquids are used (Fig. 1). Then, laser radiation is introduced into the cuvette at a right angle (Fig. 1), and its axis, after several refractions, is registered by the photodiode array 4. Relative to the initial position of the laser radiation axis on the photodiode array (without the Anderson cuvette), the displacement L is determined considering the parameters of the cuvette, the distance l, and the values of n_m and n_s (reference liquid). The works [8, 15] thoroughly examine the processes of laser radiation axis refraction and the various conditions for its reception by the photodiode array sensors, deriving a relation for determining L:

$$L = L_1 + L_2 + L_3 + L_4 = \sin\alpha_1 \left(d\left(1 - \frac{n_2 \cos\alpha_1}{\sqrt{n_q^2 - n_s^2 \sin^2\alpha_1}}\right) + \left(\sqrt{n_m^2 - n_s^2 \sin^2\alpha_1} - n_s \cos\alpha_1\right) \cdot \right.$$

$$\cdot \left(\frac{l}{\sqrt{n_a^2 - \sin^2\alpha_1 \left(n_m^2 - n_s^2 \sin^2\alpha_1 + n_s^2 \cos^2\alpha_1 - 2n_s \cos\alpha_1 \sqrt{n_m^2 - n_s^2 \sin^2\alpha_1}\right)}} + \right. \quad (1)$$

$$+ \frac{d_1}{\sqrt{n_q^2 - \sin^2\alpha_1 \left(n_m^2 + n_s^2 \cos^2\alpha_1 - n_s^2 \sin^2\alpha_1 - 2n_s \cos\alpha_1 \sqrt{n_m^2 - n_s^2 \sin^2\alpha_1}\right)}} +$$

$$\left. \left. + \frac{K_1}{\cos\alpha_1 \sqrt{n_m^2 - n_s^2 \sin^2\alpha_1} + n_s \sin^2\alpha_1} \right) \right),$$

where α_1–α_4, β_1–β_4 are the angles of laser beam refraction at the boundaries of the cuvette; L_1–L_4 are the distances between the points (A, B, C, D, K) of laser beam refraction on the photodiode array, which are summed into the desired value L; n_s is the refractive index of the standard liquid;

n_m is the refractive index of the measurement liquid; n_a is the refractive index of the air; n_q is the refractive index of the cuvette material (quartz); l_1, l_2 are the external dimensions of the Anderson cuvette; l is the distance between the wall of the Anderson cuvette and the photodiode array; d, d_1 are the thicknesses of the partition and the cuvette wall; K_1 is the distance from point B to the inner right wall of the Anderson cuvette; a, b, a_1, b_2 are the internal dimensions of the Anderson cuvette.

For obtaining reliable values, in accordance with express control requirements, the refractive index n_m of the liquid medium must be measured with an error of 0.0001 or less in range from 1.23 to 2.63. In the refractometer design we developed for measuring n_m, a 2048-pixel array is used. For the measurements, we can use 1600 pixels (with 220 pixels reserved to ensure the safety limit at the boundary of the measurement range). This allows us to determine the position of the laser beam peak at the edges of the n_m measurement range. The smallest step with which we can cover the range from 1.23 to 2.63 using this number of pixels for measurement is 0.001. The refractometer design also has a reserve of 200–300 pixels in case new liquid media are introduced and the n_m values increase. The measurement step in this case defines the measurement error. Other factors (tolerances in the manufacturing of optical materials, pixel size of the photodiode array and distance between pixels, error in determining distance), which influence the n_m measurement error, according to data from industrial refractometers using photodiode arrays for measuring the angle of total internal reflection or the position of the light-shadow boundary, begin to affect the refractive index measurement error starting from a value of 0.00005 or less.

To ensure a refractive index measurement error of n_m in the differential refractometer at 0.0001, we developed a new method for modifying the scale resolution without changing the position of the optical elements, photodiode array, or laser, as well as without altering the laser radiation registration principle. For the n_m value measured in the first stage, it is necessary to select a new design for the Anderson cuvette and a new reference liquid (new ns value) so that a change in n_m by 0.0001 corresponds to the shift of the laser beam peak of one sensor on the photodiode matrix (Fig. 2).

Previously, the value of n_m was determined to the third decimal place (e.g., n_m = 1.753). After such replacement, the measurement range of n_m will be from 1.6730 to 1.8330 with a step of 0.0001 (measurement error of 0.0001). The remaining factors, as noted earlier, do not have a

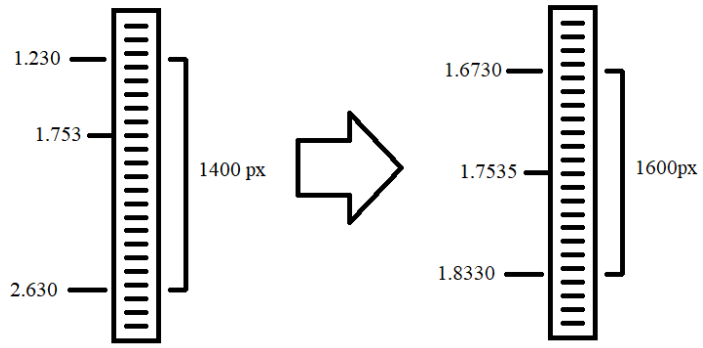

Fig. 2. Method for changing the measurement scale discreteness of refractive index n_m on the photodiode array

significant impact on the measurement error of n_m. This pixel reserve, when changing the scale resolution of the measurement range, is necessary for sharp temperature changes, which will lead to a change in the refractive index value of n_m. The optical axis of the laser beam will not go beyond the measurement range, and rapid control will be maintained. This fundamentally distinguishes this work from what was presented earlier in [16–18] based on the developments for the mobile differential refractometer. This work is a continuation of the research and developments in [8, 16–18] for the mobile differential refractometer.

Results and discussion

The developed methodology for changing the scale discreteness of the refractive index was tested on the previously developed design of the differential refractometer during the study of hydrocarbon media and their mixtures. Fig. 3, as an example, shows the results of the study of the refractive index changes of various gasoline brands with changes in temperature T.

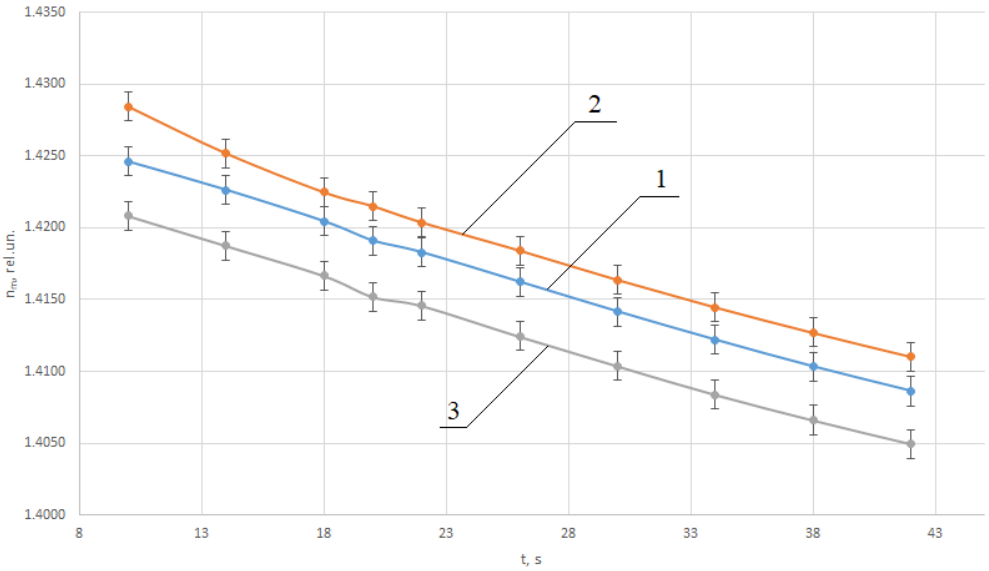

Fig. 3. Change in refractive index n_m with temperature T for various gasoline brands. Graphs *1*, *2*, and *3* correspond to gasoline brands: AI-95+, AI-98, and AI-100

Analysis of the obtained dependencies shows that the nature of the change in n_m corresponds to the previously obtained results on stationary refractometers and reflects the physical processes occurring in gasoline during heating.

To verify the reliability of the developed methodology for changing the scale discreteness of the refractive index n_m, studies of gasoline mixtures at various temperatures were conducted and compared with the results of measurements on the industrial refractometer SNEL-105 (measurement error of 0.00005). Tables 1 and 2 present the research results using both devices.

Analysis of the data in Tables 1 and 2 shows that the measurement results of n_m match within the measurement error, confirming the reliability of our developed measurement methodology using scale discreteness changes. This allows for a measurement error of 0.0001 or less.

Table 1

Change in refractive index n_m of mixture of AI-95 and AI-95+ gasolines in 50% to 50% ratio with temperature T

T, °C	Laboratory layout of differential refractometer	Industrial refractometer SNEL-105
10.0 ± 0.1	1.4239 ± 0.0001	1.42384 ± 0.00005
14.0 ± 0.1	1.4217 ± 0.0001	1.42165 ± 0.00005
18.0 ± 0.1	1.4194 ± 0.0001	1.41936 ± 0.00005
20.0 ± 0.1	1.4185 ± 0.0001	1.41844 ± 0.00005
22.0 ± 0.1	1.4173 ± 0.0001	1.41723 ± 0.00005
26.0 ± 0.1	1.4151 ± 0.0001	1.41493 ± 0.00005
30.0 ± 0.1	1.4131 ± 0.0001	1.41305 ± 0.00005
34.0 ± 0.1	1.4110 ± 0.0001	1.41093 ± 0.00005
38.0 ± 0.1	1.4092 ± 0.0001	1.40914 ± 0.00005
42.0 ± 0.1	1.4074 ± 0.0001	1.40733 ± 0.00005

Table 2

Change in refractive index n_m of mixture of three gasolines in following ratio: 60% AI-100, 20% AI-98, 20% AI-95 with temperature T

T, °C	Laboratory layout of differential refractometer	Industrial refractometer SNEL-105
10.0 ± 0.1	1.4262 ± 0.0001	1.42613 ± 0.00005
14.0 ± 0.1	1.4244 ± 0.0001	1.42434 ± 0.00005
18.0 ± 0.1	1.4222 ± 0.0001	1.42214 ± 0.00005
20.0 ± 0.1	1.4205 ± 0.0001	1.42043 ± 0.00005
22.0 ± 0.1	1.4202 ± 0.0001	1.42014 ± 0.00005
26.0 ± 0.1	1.4182 ± 0.0001	1.41815 ± 0.00005
30.0 ± 0.1	1.4162 ± 0.0001	1.41613 ± 0.00005
34.0 ± 0.1	1.4144 ± 0.0001	1.41434 ± 0.00005
38.0 ± 0.1	1.4125 ± 0.0001	1.41244 ± 0.00005
42.0 ± 0.1	1.4110 ± 0.0001	1.41093 ± 0.00005

Conclusion

The results of the study showed that changing the parameters of the differential Anderson cuvette and the refractive index n_s of the reference liquid allows for managing the scale discreteness of the measurement n_m within various ranges. This ensures a measurement error of 0.0001 in the range of the refractive index of the tested medium from 1.23 to 2.63, fully meeting the requirements for express control.

In the future, there is a possibility to achieve a measurement error of about 0.00005 or less by selecting the parameters of the Anderson cuvette and the refractive index n_s. For this, it is necessary to determine the tolerances for the fabrication of the faces and partitions in the Anderson cuvette and to ensure the value of n_s of the reference liquid with an accuracy of no worse than 0.00001. In such a case, the developed model of the differential refractometer can be used for scientific research and other applications.

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Received 15.09.2024. Approved after reviewing 14.10.2024. Accepted 14.10.2024.