Condition monitoring of current media using a differential refractometer

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Abstract. A new design of a differential flow refractometer has been developed to monitor the condition of flowing media in a pipeline. A new method of refractive index measurement has been implemented, taking into account the specifics of flowing and closed cuvette arrangement, as well as the angles of incidence of laser radiation on their walls. The effect of changes in the optical density in the flowing liquid on the refractive index measurement result is determined. The results of experimental investigations of different media are presented.

1. Introduction
Currently, the number of tasks associated with monitoring the state of condensed matter is constantly increasing [1-10]. Depending on the tasks to be solved, different methods and instruments are used [10-18]. The greatest difficulties arise while monitoring the liquid flowing through the pipeline, for example, during scientific research or monitoring the operation of cooling systems [13-15, 19-25]. The use of hostile requirements or severization for the stability of the measurement process has led to the preference for non-contact measurement methods when monitoring the state of the current environment [26-32]. In some cases, like food& beverages, chemical or pharma industry, apart from strong requirements for accuracy and reliability of measurements, there is a number of arrangements [33-35]. The measurements shall not change the physical and chemical properties of the environment [34-38]. All these requirements are met only by nuclear magnetic flowmeters - relaxometers or flow refractometers [8, 13, 15, 24, 34, 35, 39].

Refractometers, unlike nuclear-magnetic flow meters - relaxometers, allow one measuring device to monitor both the current and stationary state of the matter [34, 35, 39-44]. In addition, refractometers are easier to operate, etc. [34, 35, 39-45]. The most frequently used types of fractometers are differential refractometers and refractometers, based on the use of the phenomenon of complete internal reflection of laser radiation from the boundary of two matters [39-41]. A number of problems arise in refractometers with complete internal reflection. As the temperature changes, the refractive indexes of the current liquid and prism do change, so it requires a quick automatic adjustment of the optical part of the device. With fast fluid flows, the refractive index measurement error is impacted by the Hus-Hengchen optical beam shift phenomenon, which complicates the operation of the device and limits the measurement range n.
Therefore, the development of new designs of flow refractometers of the differential type and methods for implementing in them the measurement of the refractive index \( n \) with an error of less than \( 10^{-4} \) is an urgent task.

2. Differential type refractometer designs and refractive index measurement errors

The study of the effect of the change in optical density on the measurement error of the current fluid index [45] made it possible to develop a new design of a flow refractometer of the differential type (Fig. 1). In this refractometer design, a differential method is used to detect laser radiation. Therefore, the refractometer can be considered differential.

![Figure 1. The Block diagram of a laboratory model of a differential refractometer: 1 - semiconductor laser; 2 - power source; 3 - optical system; 4 - prism Dove; 5 - closed cuvette; 6 - flowing cuvette; 7 - dividing prism; 8 - photodiode array; 9 - ADC; 10 - processing device; 11 - indicating device; 12 - temperature sensor; 13 - device for processing information with a temperature sensor; 14 - specialized power supply ADC and photodiode arrays; 15 - laptop; 16 - power supply unit for processing and indication devices.](image)

The radiation from the semiconductor laser 1 with a \( \lambda = 632.8 \) nm is transmitted to the optical system 3 (prism collimator). The collimator is an anamorphic system consisting of two prisms. In this system, unlike lens systems, there is no axial symmetry when converting a laser beam. The laser kit includes a long-focus and short-focus lens. Their use allows narrowing the laser beam to 0.4 mm. Overall makes it possible to obtain at the exit of the collimator a parallel beam in the form of a narrow strip measuring 24 mm by 1 mm. The laboratory model of the refractometer we have developed uses cylindrical cuvettes made of ARCUNA glass with \( n = 1.4953 \). Flow cell 6 is placed inside closed 5 (Fig. 1). In our design of the refractometer, we have proposed a new method for placing ditches in an upright position (Figure 1). Its use makes it possible to eliminate the formation of voids completely in the flow cell at low liquid flow rates and vortex flows at high flow rates \( q \), which were previously used in the differential cells. The use of this method made it possible to use cells of large diameter (20 and 28 cm) in the design of the refractometer - voids in the flow cells are not formed at low flow rates \( q \).
This arrangement of cells 6 and 5 also provides a more efficient heat exchange between fluids (current and reference) than previously used differential cells designs. This significantly reduces the impact of the temperature error contribution. The refractive index value \( n_f \) of the current liquid is determined from the ratio:

\[
\beta = \Delta n \tan(\gamma) \tag{1}
\]

where \( \gamma \) is the angle between the incoming beam and the perpendicular to the boundary of the flow and closed cell, \( \Delta n = n_f - n_r \) (\( n_r \) is the refractive index of the reference liquid located in the closed cell).

The conducted researches showed that the main contribution to the uncertainty value of the measurement \( \delta n_f \) of refraction index will only change the optical density of the current liquid, the impact of other deviations on measurement \( n_f \) result will be insignificant. In this case, the value of \( \delta n_f \) can be determined from the following formula:

\[
\delta n_f = 0.993 \frac{a^2 L \Delta d}{\Delta n} \tag{2}
\]

where \( \Delta d = d_f - d_0 \) is the change of optical density \( d_f \) relative to initial value \( d_0 \), \( a \) - half-width of light beam, \( L \) - distance from wall of cylindrical cell 5 to linear photodiode array 8.

Taking into account the features of the placement of cells and the effect on the current and reference liquid of laser radiation, we have developed a method for measuring the refractive index of \( n_f \) using a differential optical signal recording circuit. Magnitude of amplitudes difference of normalized output signals from photodiode lines 8 is proportional to value \( \beta \) and \( L \) (distance from cell wall 6 to linear photodiode array 8. In this case, formula (1) for determining the informative parameter \( \beta \) is converted into the following form:

\[
\beta = \frac{A_1 - A_2}{A_{max}} - L K_{ar} \Delta n = L K_{ar} (n_f - n_r) \tag{3}
\]

where \( K_{ar} \) - proportionality coefficient, \( A_1, A_2 \) - normalized output signals from photodiode lines 1 and 2.

Normalization of output signal \( A \) from photodiode ruler 8 is performed according to the following principle:

\[
A = \frac{\sum_{i=1}^{512} A_i}{A_{max}} \tag{4}
\]

where \( A_{max} \) is the maximum value of the illumination signal from the element of the photodiode line, \( A_i \) is the amplitude of the signal from the element of the photodiode cell, \( i \) is the element number.

From the photodiode lines 8, signals are transmitted through analog-to-digital converters to the information processing device 10, developed on the basis of the microcontroller STM32 (ARM Cortex M3 core - STM32F100RBT6B). Information on \( n_f \) value is transmitted from this device to indication device 11. In addition, a laptop 15 was used to monitor the operation of the information processing device in the laboratory layout, which received information from the microcontroller. It was designed to determine the value of the \( n_f \) and visualize the change in the parameters of the matter during long monitoring of its condition.

3. Results of experimental data and their discussion
The validity of \( n \) measurements using the developed refractometer design was tested on several liquid matters for different temperatures \( T \). Figure 2 shows the \( n \) (T) dependencies for several current matters.
Figure 2. The dependence of the refractive index $n_f$ from the temperature $T$. Charts 1, 2, 3 correspond to the following environments: distilled water, tap water, ethyl alcohol.

The refractive indices of these matters were also measured using the industrial flow refractometer PRM-100 alpha (company ATAGO, Japan) - measurement deviation $10^{-4}$. The obtained refractive index values using two instruments coincided within the measurement deviation range. In addition, the refractive index values of these matters, measured on the laboratory model of the differential-type refractometer developed by us, coincided with the refractive index values obtained earlier by scientists using other refractometer designs [34, 35, 39-41, 44, 45]. In some cases, the liquids were stationary during measurements, so the measurement error $\eta$ was less than for the flowing liquid.

An important parameter when considering the error associated with the change in optical density of the liquid medium is transparency. Especially many liquid matters with low transparency are used in the food and pharmaceutical industries. Refractometers are often used to monitor the condition of these matters. Figure 3 shows as an example the relationship of the refractive index change $n_f$ different oil grades to the temperature change $T$.

Figure 3. The dependence of the refractive index $n_f$ of edible oils from the temperature $T$. Charts 1, 2, 3, 4 correspond to the following oils: olive, sunflower, castor, cedar.
Refractive indices of oils were measured using an industrial flow refractometer PRM-100 alpha (company ATAGO, Japan) - measurement error $10^{-4}$. The refractive index results obtained coincided within the measurement error range. Besides, comparison of measured values of refractive indices of $n_f$ oils using the design of refractometer developed by us with data obtained on stationary refractometers [46] showed their coincidence within the limits of measurement deviation.

4. Conclusion
The results of the studies showed that the use of the design of a flow refractometer developed by us with a differential method for measuring the refractive index of $n_f$ implemented in it allows us to monitor the state of the current matter in the entire range of changes in its flow $q$ and temperature $T$, unlike refractometers, the principle of operation of which is based on the use of the total internal reflection phenomenon from the boundary of two matters.

In the newly developed refractometer design, there are no limits on the measuring range $n_f$ in comparison with refractometers operating on the basis of total internal reflection phenomenon, in which there are restrictions on changing the angle of complete internal reflection depending on the temperature of the current matter $T$, etc. Its use allows you to control, for example, the state of the current flow of phoenylhydrazine $n_f = 1.6105$, used in the medical industry in the production of drugs (for example, antipyrin or amidopyrin). Currently, control of the state of this substance in the flowing stream by industrial refractometers is not implemented. All limitations in the work of the refractometer we have developed are related to the transparency of the liquid and the presence of insoluble particles in it. They were considered in detail in the discussion of the experimental results.

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