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Interference method for determination of the refractive index and thickness

Title	Interference method for determination of the refractive index and thickness
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Publication Date	2000-09-01
Publisher	Society of Photo-optical Instrumentation Engineers
Repository DOI	10.1117/1.1287583

Interference method for determination of the refractive index and thickness

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Abstract. The interference method for simultaneous measurement of the absolute refractive index and the thickness of plane parallel samples is developed. This method enables us to test solid (including optical glasses, crystals, plastics, and other sheet materials), liquid, and gaseous media over a wide spectral range. The experimental model for the realization of this method, the laser interference refractometer and thicknessmeter (IRT), is manufactured and investigated. The IRT accuracy of the refractive index measurement is not worse than that of the best modern goniometers and the accuracy of the thickness measurement corresponds to the interference accuracy. © *2000 Society of Photo-Optical Instrumentation Engineers.* [S0091-3286(00)01809-2]

Subject terms: refractive index; thickness; interferometry; optical measurements; optical materials; plane parallel plates.

Paper 990270 received July 12, 1999; revised manuscript received Feb. 25, 2000; accepted for publication Mar. 20, 2000.

1 Introduction

All methods for determination of the refractive index can be divided into two groups: methods to determine the absolute refractive index and differential methods, which enable us to determine the variations of the refractive index under the influence of different factors or in comparison with a reference sample.

The refractometric and goniometric methods are the methods for absolute measurements of the refractive index of optical materials that are most frequently used in practice. These methods enable us to attain accuracy of 10^{-5}. But the refractometric method has a limited range of measurements; requires application of samples with a prism shape; and requires the application of immersion oils, which are toxic as a rule. The goniometric method also requires an application of considerable sized samples with a prism shape. Therefore using these samples to produce optical elements from them is either impossible or economically nonprofitable. Sometimes it is a significant deficiency, for example, in the research of new synthesized materials.

Some of the other refractive index measurement methods include a fiber-optic refractometer that enables us to determine the refractive index of solid and liquid media[1,2] with accuracy up to 10^{-5}. The method for measuring the refractive index of a liquid, which is based on determining the displacement of a laser beam going through a cell containing a liquid under investigation was described in Ref. 3.

The interference methods are some of the most precise. These include an interference method for measuring the refractive index of transparent materials using reflective grating[4] and a method using Fizeau fringes,[5] which enables us to measure the refractive index for liquids with an accuracy from 0.02 to 0.0006. Some authors use interference methods to measure the refractive index of air[6] and for the simultaneous measurement of the refractive index of air and of displacement of the cube corner reflector.[7]

Two groups of methods used to measure the thickness and refractive index of films are constant angle reflectance and transmittance interference spectroscopy (CARIS and CATIS, respectively) and a variable angle reflectance and transmittance interference spectroscopy[8] (VARIS and VATIS, respectively). In the most common usage mode, the interference fringe pattern obtained from these methods is combined with previous knowledge or measurement of the refractive index of the sample to determine its thickness. When the refractive index of the sample is unknown, at least two measurements at different incidence angles using the VARIS and VATIS methods or two measurements at different wavelengths using the CARIS and CATIS methods are required. For example, one of these methods, the method to determine the refractive index and thickness of films, was proposed in Ref. 9. This method involves the use of a microscope equipped with a monochromatic filter on the objective and a stage that can be rotated so that the reflected light is observed at various angles. Some other interference spectroscopy methods to determine the refractive index and thickness of films were developed later.[10–13]

The variable incidence angle was used to measure the refractive index of thick (with thickness up to some tens of millimeters) plane parallel plates in Refs. 14–16. But applying known interference methods to determine the absolute refractive index of thick samples gives low accuracy. Therefore in practice, differential interference methods are applied in most cases. The measurement accuracy for differential methods is up to 10^{-8}.

One of the advantages of the interference method in the investigation of optical materials is the possibility to use the samples with plane parallel surfaces.[17–19] This simplifies the process of making samples and conserves them for further utilization.

A new method that provides the combination in one instrument of the advantages of two traditional methods, such as goniometric and interference, and a device for its real-

ization were proposed recently.[20–23] This device, the laser interferometer for determination of refractive index and thickness, we called the interference refractometer and thicknessmeter (IRT). The IRT requires samples with plane parallel surfaces and provides the possibility to determine the geometrical thickness of the sample (with thickness from one to some tens of millimeters) simultaneously with the absolute refractive index. In this paper, we consider the theory of this method and present some results of experimental research with the IRT model that realized the proposed method.

2 Theory and Method

2.1 *Basic Principles*

If the plane parallel sample is placed into one beam of the two-beams interferometer, then the phase difference between interferometer beams depends on the refractive index n of the sample, on the geometrical thickness d of the sample, on light wavelength λ, and on incidence angle a, i.e., the angle between the light beam and the normal to the sample surface. If the sample is turned, then the incidence angle a is changed and so the phase difference is also changed. The change of the phase difference can be written as

$$\Delta\varphi(a)=2\pi d/\lambda[(n^2+n_a^2\cos^2 a-1)^{1/2}-n_a\cos a -(n^2+n_a^2\cos^2 a_0-1)^{1/2}+n_a\cos a_0], \quad (1)$$

where n_a is the refractive index of air, and a_0 is the initial incidence angle. Equation (1) is correct if a and a_0 have the same sign. If d and a are known and $\Delta\varphi$ is measured, then it is possible to determine n from this expression.

A similar method to determine the refractive index based on the Mach–Zehnder interferometer was proposed in Ref. 15. But in this interferometer, a light beam that has passed through the sample is shifted respective to the reference light beam when the sample is turned. This shift limits either the range of the turned angles or the maximal thickness of the sample under investigation and causes the result of measurements to depend on the wavefront shapes of both light beams, which form the interference pattern. In addition, this method requires measuring the thickness of the sample with high accuracy and with a special technique before the measurement of the refractive index is begun.[15] Owing to that, the measurement accuracy of this method is low.

In the proposed method the autocollimation interferometer scheme is used, i.e, the interferometer provides double light passing through the sample being studied. Thus the shift of the object light beam to the reference light beam is eliminated. This makes it possible to increase the measurement accuracy both by double passing through the sample and by increasing the maximum possible incidence angle (because the angle of the sample turning is increased) and also to eliminate the influence of the quality of the optical details of the interferometer on the measurement accuracy.

In addition, in the proposed method, the geometrical thickness is calculated simultaneously with the refractive index with the same experimental data. In fact, it can be done by measurements of the change of phase difference $\Delta\varphi$ for two or more angles a and by solving of the system of Eq. (1). The proposed method, as shown in the following, enables us to attain the high accuracy in the determination of n and d because the incidence angles are large enough to realize this possibility.

2.2 *Principal Scheme of IRT*

The concrete schemes of interferometers for practical realizations of the proposed method may be different. We propose and studied two basic IRT schemes. The first is based on the autocollimation Rayleigh interferometer.[17–20] This scheme has a good vibration stability and enables us to decrease the requirements for interferometer optical element quality. The research on this interferometer revealed its two main disadvantages. One is connected with the low-level light intensity in the photocell plane. Owing to this there is no possibility for precision measurement of the samples with small dimensions (less than 10 mm). The second disadvantage results from the dependence of the Rayleigh scheme element parameters on the light wavelength. This hampers measurements for different wavelengths.

In the second scheme, these disadvantages are eliminated.[21–23] The refractive and diffraction optical elements are absent in this scheme. This makes operative measurements with different light wavelengths possible. The absence of the diffraction elements and diaphragms in the scheme increases the useful light intensity of the photocell more than in 200 times. It increases measurement accuracy and enables us to use light beams with small diameters for the measurement of the small size samples.

The IRT scheme is shown in Fig. 1. A laser with the adjusted light wave or a complete set of lasers with different wavelengths can be used as a light source. The light beam from laser 1 is divided into beams I and II by prism (2). Beam II goes through the tested sample (3). Beams I and II are reflected from mirror (6) and come to the photocell (7) plane. The sample (3) is placed on a rotary table (4). The angle of the table (4) rotation is measured by the angle sensor (5). Smooth rotation of the sample (3) causes the periodic change of the light intensity on the photocell. The electric signal from photocell (7) varies in proportion to the phase difference between the beams I and II. The number of signal periods is measured by counter (8). Control of measurements and calculations of n and d is performed with a PC.

2.3 *Deviation of the Basic Principles for Real Conditions*

Equation (1) is valid for ideal experimental conditions. In real conditions, the phase difference is dependent on parameters that are connected with adjustment of the sample under investigation in the interferometer scheme and with interferometer elements. According to scheme shown in Fig. 1, these parameters are nonperpendicularity of the sample rotation axis to the incident light beam, nonparallelism of the sample plane to the sample rotation axis, and nonperpendicularity of the plane of the autocollimation mirror to the light beam and deviation of the sample from plane parallel shape. Thus for real experimental conditions, the change of the phase difference will have the form

$$\Delta\varphi(a'')=\Delta\varphi_1[a(a'')]+\Delta\varphi_2[a'(a'')], \quad (2)$$

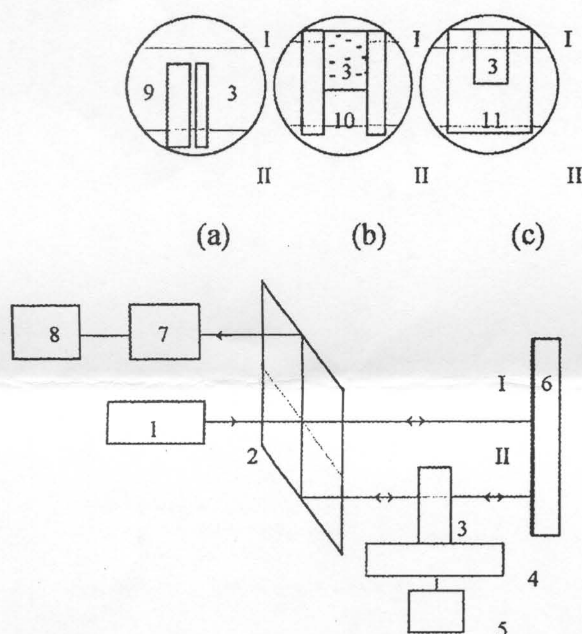


Fig. 1 Principal scheme of the laser interferometer for determination of refractive index and thickness: 1, light source; 2, prism; 3, tested sample; 4, rotary table; 5, angle sensor; 6, mirror; 7, photocell; 8, signal period counter; 9, additional plate or film base; 10, cell for liquid media; and 11, cell for gaseous media. (a) Scheme for thin films, (b) scheme for liquid media, and (c) scheme for gaseous media.

where $\Delta\varphi_1$ and $\Delta\varphi_2$ are the phase differences at the direct and back passing of light through the sample, a and a' are the incidence angles at the direct and back passing of light through the sample, and a'' is the projection of the incidence angle a on the horizontal plane. We are considering the horizontal plane as the sample rotation plane and the vertical plane as the perpendicular plane that contains the incidence beam. The values $\Delta\varphi_1$ and $\Delta\varphi_2$ are determined with Eq. (1) and the values a and a' are determined using

$$\cos a = \cos W \frac{\cos(a'') + \tan P \tan W}{(1+\tan^2 P)^{1/2}},$$

$$\cos a' = \cos W \frac{\cos(a''-2E) - \cos 2E \tan(P-2U) \tan W}{[1+\tan^2(P-2U)\cos^2 2E]^{1/2}}, \tag{3}$$

where W is the angle between the normal to the sample plane and the horizontal plane, P is the angle between light beam and the horizontal plane, E is the projection of the angle between light beam and the normal to the autocollimation mirror on the horizontal plane, and U is the projection of the angle between light beam and the normal to the autocollimation mirror on the vertical plane.

The phase difference also depends on the value of the sample wedge β and on the sample surfaces quality N_P (fringe count). We obtained the mathematical expressions for description of the dependence of the phase difference on these parameters, but the expressions are very bulky and so are not presented in this paper.

2.4 *Theoretical Analyses of Errors*

The mathematical expressions obtained to determine the phase difference that takes into account of all the mentioned parameters were taken as a basis for mathematical model of the IRT. This model was used to analyze the errors in determining the refractive index and thickness depending on these parameters. The model was realized as a computer program where the initial data are the parameters of the sample n and d and the parameters λ, n_a, E, P, U, W, N_P, β, a_m, and i, where a_m is the maximal incidence angle (maximal rotation angle of the sample), and i is the number of rotation angles that are uniformly distributed within range $-a_m + a_m$ and are used to calculate the phase difference. The output data are the dependence of the phase difference $\Delta\varphi$ on the rotation angle a'', so this model simulates the work of the IRT, namely, it provides the same information as is obtained from the counter, (8) and from the angle sensor (5) of the IRT (see Fig. 1). Next, the refractive index and the thickness are calculated with the specially developed algorithm using these output data. The differences between initial values and calculated values n and d provide a simulation of the measurement errors.

It is obvious that if the parameters λ, n_a, E, P, U, W, N_P, and β do not change during the measurement process, then they are the cause of the systematic errors of the n and d determination. To investigate the influence of random errors, additional random oscillations were introduced into the phase difference output values. These oscillations simulated the errors of the counter (8). We used the normal distribution of the random errors and root mean square value δN to describe these errors.

To analyze the influence of each parameter on the measurement accuracy the dependences of the errors in determining n and d on the value of this parameter at different values of n, d, a_m, and i were established. These dependences were obtained as numeral data and were approximated with empirical expressions so that the deviations of the values that were calculated with these empirical expressions from initial numeral values were not more than 30%.

Random errors (root mean square deviation) of the determined n and d values could be estimated by the following empirical expressions:

$$\delta n \cong \frac{30 n^2 \lambda}{a_m^4 d \sqrt{i}} \delta N,$$

$$\delta d \cong \frac{60\lambda}{a_m^5 \sqrt{i}} \delta N. \tag{4}$$

As already noted, the systematic errors of n and d determination also depend on the interferometer adjustment errors and errors resulting from the construction of the interferometer. The influence of these errors is great and is determined by Eqs. (3). The theoretical analysis of the mathematical model shows that our algorithm for data processing enables us to decrease the influence of these

errors if the value of these errors is not more than 30 arcsec. In this case, the systematic errors of n determination are not more than 10^{-7} and errors of d determination are not more than 0.005 m km. The examined experimental model of the IRT was adjusted so that the angles P, E, and U [see Eqs. (3)] were not more than 10 arcsec and angle W was not more than 30 arcsec.

Thus the systematic errors can be expressed by

$$\Delta n \cong C_1 \frac{n^3}{d} \beta^2 + C_2 \frac{n^3 \lambda^2}{d^3 (n-1)^2} N_P^2,$$
$$\Delta d \cong 2 C_1 n^2 \beta^2 + 2 C_2 \frac{n^2 \lambda^2}{d^2 (n-1)^2} N_P^2 + d \frac{\delta \lambda}{\lambda} + d \frac{\delta n_a}{n_a}, \tag{5}$$

where C_1 and C_2 are the coefficients, $C_1 = 2 \times 10^{-6}$ mm/s^2, $C_2 = 2 \times 10^4$ mm; β is the optical wedge angle (in arcseconds); N_P is the maximal deviation of the sample surface from the plane [in interference fringes (fringe count)]; $\delta\lambda$ is the error of the light wavelength; and δn_a is the error of refractive index of air determination.

Equations (4) and (5) were designed for the following ranges: $\delta N = 0$ to 1, $n = 1.01$ to 10.0, $d = 0.1$ to 200 mm, $\lambda = 200$ to 2000 nm, $a_m = 20$ to 85 deg, $i = 10$ to 2000, $N_P = 0$ to 10, and $\beta = 0$ to 1 arcmin.

2.5 *Possibilities for Measuring of Liquid Media, Gaseous Media, Thin Samples, and Films*

The proposed method in addition to examining solid media also enables us to research liquid and gaseous media. Special plane parallel cells should be used for measurements of the refractive index of liquid and gaseous media. The principal schemes for the investigation of thin samples and films [Fig. 1(a)], liquid media [Fig. 1(b)], and gaseous media [Fig. 1(c)] are also shown in Fig. 1. As we can see from Fig. 1(b), each wall of the cell gives the same phase retardation in both interferometer branches. Therefore the measuring process for liquid media is the same as for solid media.

The measuring process for thin samples, films, and gaseous media has some differences from the preceding process because of the additional phase retardation that takes place. The cause of this phase retardation is an additional plate or film base [9 in Fig. 1(a)] at the measurement of thin samples or films and are cell walls [11 in Fig. 1(c)] at the measurement of gaseous media. The change of the phase retardation in these cases is given by

$$\Delta \varphi_\Sigma = \Delta \varphi (n_p, d_p, \lambda, a) + \Delta \varphi (n, d, \lambda, a), \tag{6}$$

where n_p is the refractive index of the additional plate (film base) or of the cell material; d_p is the thickness of the additional plate (film base) or the difference between thickness of the cell (11) base and walls.

The necessity to use the additional plate to measure thin samples arises because the error of the phase difference measurement with the signal period counter (8) increases for the samples with thickness less than 0.3 mm. It is not the principal limitation of the proposed method for the refractive index and thickness determination, but it follows from the limitation of the method used in the experimental model for measuring of the phase difference with counter (8).

From Eq. (6) we can see that the measurement process should have two stages. In the first stage, the values n_p and d_p are measured while the thin sample, film, or gaseous medium are absent. The measurement process is the same as for solid media. In the second stage, the values n and d are measured with the counting of measured n_p and d_p.

The mathematical model of the measuring process shows that a simple substitution of n_p and d_p in Eq. (6) does not guarantee high accuracy for n and d determination. The measurement errors δn_p and δd_p lead to considerable increases in the errors δn and δd of n and d determination. Therefore, a special algorithm for the determination of the n and d was developed. In this algorithm, more accurate values n_p and d_p are calculated simultaneously with values of n and d, while the measured values of n_p and d_p and the measured errors δn_p δd_p give the ranges where these accurate values can be found. This way enables us to attain a sufficiently high accuracy for n and d determination (as for the best analogs).

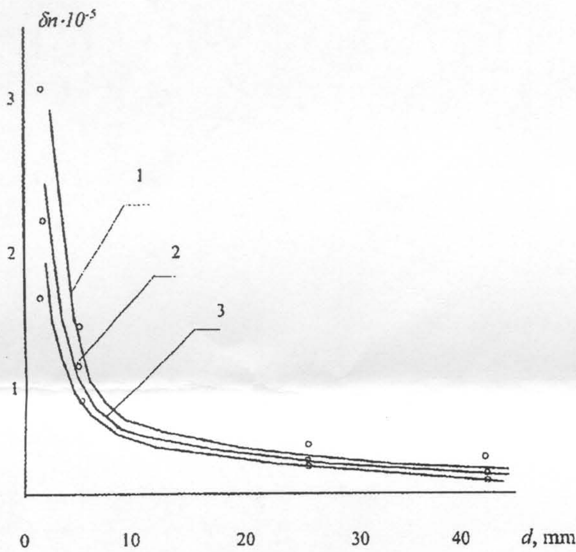
3 Experiments and Results

The experimental model of the IRT was developed, manufactured, and investigated. In our experimental model, three lasers with nominal wavelengths 441.6, 632.8, and 1150 nm were used. The error of the angle sensor (5) is about 0.6 arcsec. The error of the counter (8) is about 0.01 of the signal period. The range of sample turning is ±63 deg. The deviation of the mirror (6) surface from plane (fringe count) is not more than 0.02λ for $\lambda = 632.8$ nm. The control of measurements and processing of measured signals was performed with PC. The time of one measurement is not more than 1 min.

The experimental investigation of the model was performed using three complete sets of reference glass samples with different thicknesses from 2 to 45 mm. The complete sample sets were composed of three different glass grades with different refractive indices from 1.45 to 1.8. The samples were certified with an accuracy equal to 10^{-5} for n and 0.2 m km for d. The sample optical wedge angle did not exceed 1.5 arcsec and the surface fringe count did not exceed $\lambda/20$ for all samples. This provides the possibility to determine the refractive index with errors up to 10^{-6} and of the thickness with errors up to 0.01 m km.

Unfortunately we could not certify our complete sample set with this accuracy, so we present the results of investigations according to the certificate accuracy. The influence of the refractive index of air on the measurement results was counted by measuring the temperature, pressure, and humidity and by calculating the real value of the refractive index of air according to the Edlen expression.[24]

Each of the samples was measured not less than 50 times. The mean values of the refractive indices did not exceed the limits of the certified errors for all the samples. Thus the systematic errors of the experimental model for the refractive index determination are not more than 10^{-5}. The obtained thickness values for different samples had significant (up to some micrometers) distinctions from the certified values. Even for the same sample at different



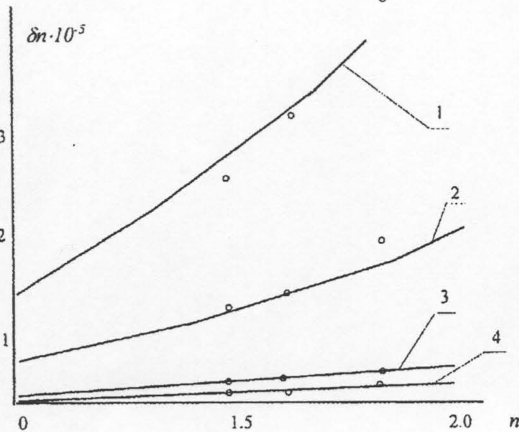
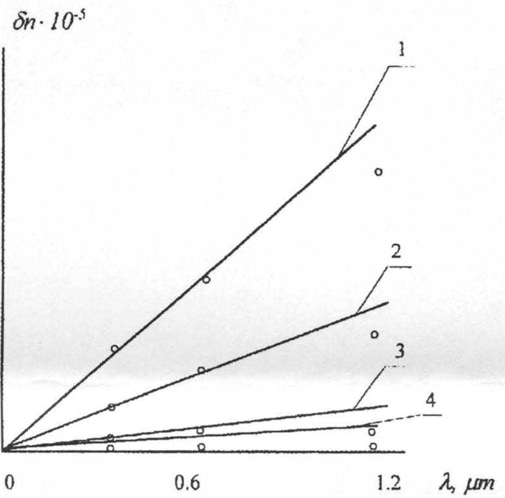
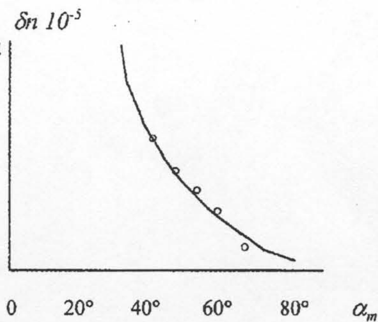


Fig. 2 Error δn as a function (a) of d, where curve 1 is for $n=1.8$, curve 2 is for $n=1.61$, and curve 3 is for $n=1.477$; and (b) of n, where curve 1 is for $d=2$ mm, curve 2 is for $d=5$ mm, curve 3 is for $d=25$ mm, and curve 4 is for $d=45$ mm. The dots correspond to the experimental values of the errors and the lines correspond to the estimated values of the errors.





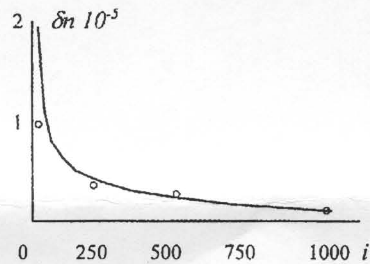
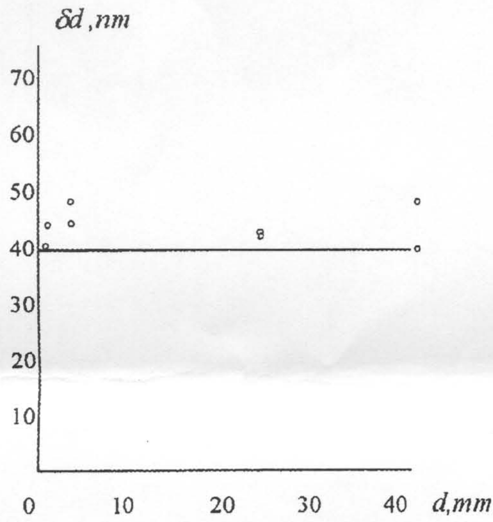


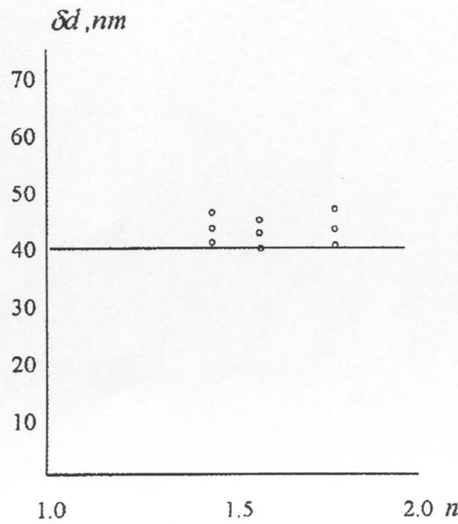
Fig. 3 Error δn as a function (a) of λ, where curve 1 is for $d=2$ mm, curve 2 is for $d=5$ mm, curve 3 is for $d=25$ mm, and curve 4 is for $d=45$ mm; (b) of a_m; and (c) of i. The dots correspond to the experimental values of the errors and the lines correspond to the estimated values of the errors.

wavelengths, the mean thickness values had different values. At the same time, the character of these differences (value and sign depend on the wavelength) was similar for all the samples. The main reasons for these differences, in our opinion, are the deviations of the nominal laser wavelengths from its real values and the dependence Δd on λ according to Eq. (5). All the differences became less than the certified errors when the next changes in lasers wavelengths were achieved: 441.6813 instead of 441.6 nm; 632.9867 instead of 632.8 mm, and 1152.56 instead of 1150 nm. All these changes are less than admissible values for the lasers that were used in experimental model. The obtained results showed that it is necessary to use at least one laser with a high wavelength stability. The wavelength of this laser must be known with the relative error not more than 10^{-7}.

The errors (root mean square deviations) of n and d determination that were obtained experimentally for all the samples are represented in Figs. 2–5. The curvatures of errors that were obtained by theoretical analysis of the mathematical IRT model are also represented in these figures for comparison with the experimental data. The dots in

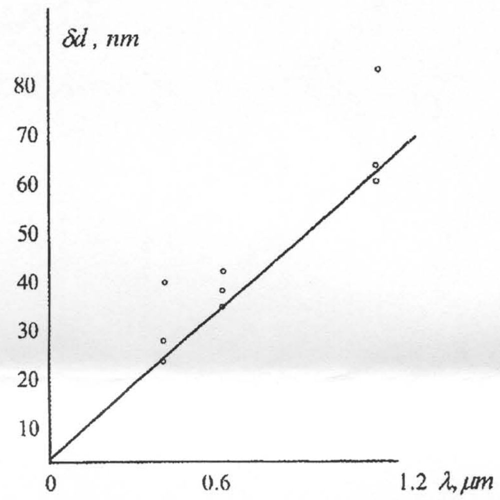


(a)

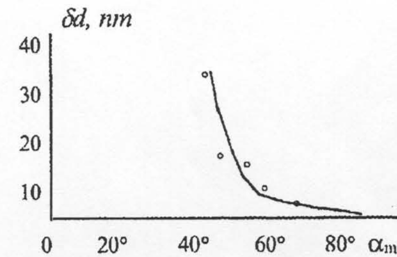


(b)

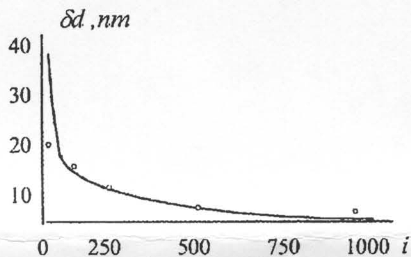
Fig. 4 Error δd as a function (a) of d and (b) of n. The dots correspond to the experimental values of the errors and the lines correspond to the estimated values of the errors.



(a)



(b)



(c)

Fig. 5 Error δd as a function (a) of λ, (b) of a_m, and (c) of i. The dots correspond to the experimental values of the errors and the lines correspond to the estimated values of the errors.

Figs. 2–5 correspond to the experimental values of the errors and the lines correspond to the estimated values of the errors. The estimated values of the errors were calculated by Eqs. (4) for $\delta N=0.05$ because this value corresponded to the real errors of the experimental model (noise of photocell, vibrations, etc.). Under these conditions, a good correspondence between estimated and experimental data is observed. Thus one can use Eqs. (4) to find out the technical requirements for the device parameters.

Requirements for the accuracy of the sample surfaces and the wedge angle can be found from Eqs. (5). For example, if $\Delta n=10^{-5}$, then β should be less than 5 arcsec and N_p- less than 1.

The analysis of results obtained revealed that δn and δd mostly depended on the maximal angle of rotation a_m [see Figs. 3(b) and 5(b)]. If $a_m<40$ deg, then the use of the device is not efficient. The same conclusion can be reached for $i<100$ [see Figs. 3(c) and 5(c)]. In our experiments, we found $i=1000$.

The errors δn and δd were directly proportional to the light wavelength λ. The error δd did not depend on both d

and n and did not exceed 0.1 m km. At the same time, the error of n determination did not depend on the error of light wavelength $\delta\lambda$. The error δn was proportional to n^2 and was inversely proportional to d. For $d=5$ mm, the error δn did not exceed 1.5×10^{-5} and for $d>25$ mm, it did not exceed 4×10^{-6}. The mean error for d determination for all samples was 0.045 m km.

4 Conclusion

A new method for simultaneous determination of absolute refractive index and geometrical thickness of the plane parallel samples was proposed. This method enables us to test solid, liquid, and gaseous media over a wide spectral range. Mathematical simulations of the measuring processes with a PC showed the high precision possibilities of the proposed method.

The experimental model of IRT for practical realization of this method was developed and constructed. The experimental examination of the experimental model of the IRT with a complete set of reference samples that were certified for refractive index and thickness were performed. The experiment showed that the accuracy of absolute refractive index determination with the IRT for the thin (thickness less than 30 mm) samples corresponds to the best goniometer accuracy and exceeds it for the thick (thickness more than 30 mm) samples. The error of absolute thickness measurement with the IRT corresponds to the error of known interference methods. This device, in our opinion, can be used in the optical industry, scientific research, metrology, pharmacy, etc.

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