

Full length article

A differential phase spectropolarimeter for measuring optical rotatory dispersion

Gennady Vishnyakov^{a,b}, Alexander Yurin^{c,a,*}, Alexander Goloposov^a, Vladimir Minaev^{a,c}

^a All-Russian Research Institute for Optical and Physical Measurements, 46, Ozernaya Street, 119361 Moscow, Russia

^b Bauman Moscow State Technical University, 5/4, 2 Baumanskaya Street, 105005 Moscow, Russia

^c HSE University, 20, Myasnitskaya Street, 101000 Moscow, Russia

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ABSTRACT

This paper considers the features of polarimetric methods used for the analysis of optically active substances. It presents a design of a differential spectropolarimeter operating by analyzing the phase difference between two harmonical signals received when splitting a laser beam from a common source into separate reference and object beams. The expediency of using the Fourier transform to calculate the phase difference of differential spectropolarimeter signals is noted. The results of calculating the parameters of the measuring method and the results of an experimental study of reference polarimetric plates at various wavelengths are presented. The error in measuring the optical rotation angle in the wavelength range 405–780 nm did not exceed $\pm 0.005^\circ$.

1. Introduction

Polarimetric methods for studying substances are based on measuring the optical rotation angle (ORA) [1]. The optical activity of substances consists in the rotation of the plane of the polarization of light and is due to the structure of the crystal lattice or molecules, therefore, by measuring the ORA, it is possible to determine the composition of the substance, its molecular structure, the concentration of solutions, etc. [2]. Substances that rotate the plane of the polarization clockwise for the observer to which the light is directed are called dextrorotatory. Those substances that rotate the plane of polarization in the opposite direction are called levorotatory. Cuvettes with a length of 100 mm are usually used for the analysis of optically active liquid substances.

For a layer of substance with thickness d , the value of the ORA $\Delta\phi$ will be [3]

$$\Delta\phi = \alpha(\lambda)d \quad (1)$$

where $\alpha(\lambda)$ is the specific rotation of the substance for a certain wavelength λ .

The specific rotation also depends on the temperature of the sample, therefore, for high-precision measurements of the ORA, it is necessary to provide thermal stabilization. As a rule, the values of $\alpha(\lambda)$ are calculated at a temperature of $t = 20^\circ\text{C}$.

Polarimetry methods in comparison with chromatographic, X-ray, mass-spectrometric or nuclear magnetic resonance methods have advantages such as high measurement accuracy, high resolution, contactless and non-destructive testing, as well as the relative simplicity of the measurement procedures, compactness, and the safety of polarimetric equipment.

Broader information about the physicochemical nature of the substance under study can be obtained using spectropolarimetric methods [4,5], which uses the phenomenon of optical rotatory dispersion (ORD), i.e. changes of ORA depending on the wavelength of light. For example, for a solution of two optically active substances, it is possible to measure ORA at two wavelengths, compose and solve two equations and calculate the concentrations of the analyzed substances. Particularly interesting is the ultraviolet region of the spectrum, in which an anomaly of ORD is observed in many substances, called the Cotton effect [6].

For substances with a disordered structure, the dependence of ORD can be described using the Drude equation [7]

$$\alpha(\lambda) = \frac{K_0}{\lambda^2 - \lambda_0^2} \quad (2)$$

where K_0 is a constant and λ_0 is the wavelength corresponding to the absorption band.

It should be noted that the Drude equation is only suitable for substances with smooth curves of the ORD far from the value of λ_0 . To

* Corresponding author at: HSE University, 20, Myasnitskaya Street, 101000 Moscow, Russia.

E-mail address: ayurin@hse.ru (A. Yurin).

account for the Cotton effect, the empirical two-term Moffitt-Young equation is used [8].

ORD is widely used in analytical chemistry in determining the structure of organic compounds (including the analysis of the chirality of molecules, the secondary structure of proteins, the absolute configuration of compounds, the chemical structure and conformation of macromolecules, etc.) [9–11]. ORD dependence analysis is an effective method of identifying optically active substances in the food industry, perfumery, pharmacology, and medicine [12–19]. However, for the wide dissemination of these methods, it is necessary to create inexpensive, highly sensitive and high-precision spectropolarimeters using the existing element base of optical and electronic components. This article describes a simple and promising design of a differential phase spectropolarimeter for measuring ORD and the physicochemical analysis of substances.

2. Polarimetry methods

The main elements of any device for polarimetric analysis are a polarizer and an analyzer, which are plates or prisms made of special materials, placed one after another [20]. The action of a linear polarizer is based on the separation of one linearly polarized component from incident light. When the polarization axes of polarizer and analyzer are in parallel, the light beam passes through them unhindered, and when crossed, it does not. If a cuvette with an optically active substance is placed between the crossed polarizer and the analyzer, the plane of polarization will rotate by a certain angle $\Delta\varphi$ and some light can pass through the analyzer. To achieve a “zero position”, it is necessary to rotate the analyzer at same angle $\Delta\varphi$ which will be equal to ORA. The simplest polarimeters are built based on a similar design, the measurement is possible due to the visual alignment of the intensities of the two sectors of the field of view and the reading of the ORA on a scale with a vernier [21].

Modern polarimeters implement various options for determining the moment of the “zero position” of linearly polarized light passed through a polarizer and analyzer [22–26]. Most often, light is directed to the photodetector, the plane of polarization of which fluctuates by several degrees near the “zero position” with some f_m frequency set by the magneto-optical modulator (Faraday oscillator) [27]. In that case, a signal with a frequency of $2f_m$ is received by the photodetector. If an optically active sample is introduced, then due to the additional rotation of the ORA, the working point will change and an additional signal with an f_m frequency will appear on the photodetector. By turning the analyzer with a servo drive, a position is achieved in which the harmonic with the f_m frequency disappears again. The measurement of the ORA is carried out using an angle measuring device based on the difference in the angular positions of the analyzer in the absence and the presence of a sample. Typical error values for this method are ± 0.01 – 0.001° , depending on the measurement range.

However, such measurements are strongly influenced by electrical noise and fluctuations in the intensity of the light source. In addition, the need to use optical modulators, precision angle measuring devices and servos increases the cost of equipment. Methods based on the analysis of frequency-phase characteristics have a lower sensitivity to noise and have been developed in differential phase polarimetry [28], in which the ORA is measured using the phase difference. The essence of the method is using linearly polarized light, the plane of polarization of which continuously rotates around the optical axis. This light is divided into two beams, one of which passes through the analyzer and hits the photodetector, forming a harmonic reference signal. The second beam passes through the cuvette with the test substance and receives an additional ORA, after which it also passes through the analyzer and hits another photodetector, forming an object signal shifted in phase relative to the reference one. Thus, to measure the ORA, it is necessary to determine the phase difference of harmonic signals.

To obtain light with a rotating plane of polarization, light from a

conventional source is usually passed through a uniformly rotating linear polarizer. Various options for implementing the idea of the method are possible – by rotating the polarizer [28], the analyzer [29,30] or with two rotating analyzers [31]. Considering the current level of optoelectronics, it is possible to design an inexpensive high-precision differential phase spectropolarimeter with low sensitivity to fluctuations of the wavelength and intensity of the light sources, the unevenness of analyzer rotation, differences in the characteristics of polarization elements, or temperature drift, based on such a method of measuring the ORA.

3. The differential phase spectropolarimeter design

The optical design of the differential spectropolarimeter (Fig. 1) uses the two-channel photoelectric registration of optical signals with subsequent phase measurements. To analyze the spectral dependence of the ORA, laser diode modules 1 of different wavelengths with fiber-optic output are used, which can be switched on alternately. The light from the laser diode modules is directed using a collimator 2.

The light beam is divided into two beams of approximately equal intensity using a beam-splitting cube 2. The object beam then passes through a stationary polarizer 4 and a cuvette with the test substance 6, and the reference one is directed through a stationary mirror 12 and a stationary polarizer 5 to a uniformly rotating analyzer 7 and a photodiode 8. The object beam also passes through the analyzer 7 and is recorded by a second photodiode 9. Also, an electric motor 11 is used to rotate the analyzer, and a high-precision analog-to-digital converter (ADC) 10 connected to a personal computer (PC) 13 implementing all calculations.

To modulate the beam, an analyzer rotating at a constant speed is used, so the light intensity at its output is described by Malus' law [1]. Then the signals $i_1(t)$ and $i_2(t)$ from the photodetectors can be written as

$$i_1(t) = k_1[\Phi_0 + \delta\Phi(t)]S_1(\lambda)[\cos^2(\omega_0 t + \phi_1 + \Delta\phi + \delta\phi(t)) + T] \quad (3)$$

$$i_2(t) = k_2[\Phi_0 + \delta\Phi(t)]S_2(\lambda)[\cos^2(\omega_0 t + \phi_2 + \delta\phi(t)) + T] \quad (4)$$

where k_1, k_2 are the light beam division coefficients; Φ_0 is the constant component of the laser light intensity; $\delta\Phi(t)$ is the function of the laser light intensity fluctuation; $S_1(\lambda), S_2(\lambda)$ are the spectral characteristics of photodetectors; ω_0 is the analyzer rotation frequency; ϕ_1, ϕ_2 are the initial phases of the signals; $\Delta\phi$ is the ORA; $\delta\phi(t)$ is a function of the instability of the rotation of the analyzer; T_{\perp} is the transmission of the analyzer in a state perpendicular to the polarizer.

The distinctive features of the proposed design are that two parallel polarizers are used, and the reference and object beams pass through the same section of the rotating analyzer coinciding with the axis of rotation, so the inhomogeneity of the direction of the polarization plane over the area of the polarization plates will not affect the signals from the photodiodes. Since both beams are formed from the same diode laser, the light intensity fluctuation will be the same in both channels and can be compensated using a signal processing algorithm. Since the design uses one rotating analyzer, the unevenness of its rotation will also equally affect the shape of the electrical signal from both photodiodes, and the phase shift between the signals will remain constant for each wavelength.

Note that there may be a measurement error associated with the passage of the reference beam through the analyzer at some angle. The fact is that Malus' law is not strictly valid if there is an angle between the beam and the polarizer plane [32]. But in our case this angle is small (does not exceed 10 – 12°), and does not have a significant effect, which is confirmed by experimental results. The main difficulty of setting up our optical design is that it is necessary to attain the passage of both beams through the same section of the analyzer coinciding with the center of rotation to achieve high measurement accuracy.

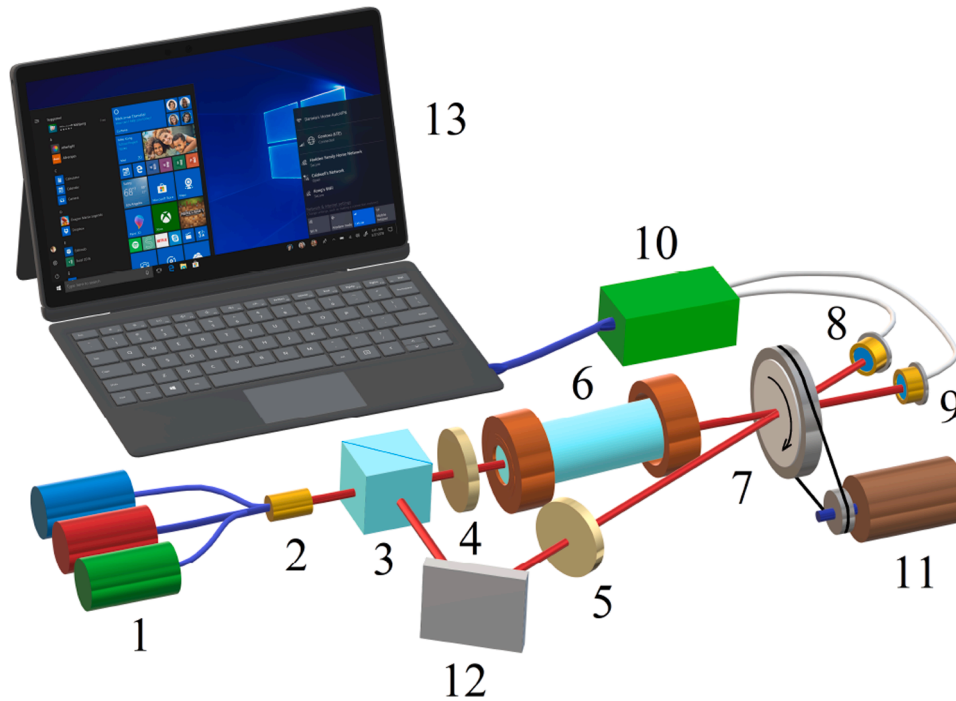


Fig. 1. Diagram of a differential phase spectropolarimeter 1 – laser diodes; 2 – collimator; 3 - beam-splitting cube; 4,5 – polarizers; 6 – cuvette with the test substance; 7 – rotating analyzer; 8,9 – photodiodes; 10 – analog-to-digital converter; 11 – electric motor; 12 – fixed mirror; 13 – personal computer.

4. The operating modes

Since the signal of the spectropolarimeter has a very narrow spectrum, it is advisable to use the Fourier transform to calculate the phase difference [33]. The results of calculating the phase difference by the described method are influenced by various signal parameters – sampling rate, number of analyzed periods, signal-to-noise ratio, etc. Therefore, in order to select the parameters and operating modes of the spectropolarimeter, a simulation of the operation of the algorithm for calculating the phase difference in the MATLAB environment with different signal parameters was carried out. As criteria for evaluating the accuracy of the algorithm, the error of restoring the phase difference $\varphi_{1,2}$ was used, i.e. the difference between the restored phase and the one originally set in the model.

The parameters of the measuring method recommended for obtaining high accuracy and acceptable analysis time are given in Table 1.

The simulation results of the algorithm at the values of the parameters given in Table 1 showed an error in restoring the phase difference $\Delta\varphi$ of no more than $\pm 0.0001^\circ$, which is sufficient for high-precision measurements of the ORA.

Table 1

Recommended operating modes of the differential phase spectropolarimeter.

Parameter	Value
ADC bitrate	12
ADC sampling rate, kHz	100
Analyzer rotation speed, rpm	300
Signal amplitude, V	2.0
Signal period, s	0.1
The number of counts for a period	1000
Duration of signal accumulation, s	20
The total number of counts	200,000
Signal-to-noise ratio, dB	50
Unevenness of the analyzer rotation	0.01

5. Implementation

The proposed design of the spectropolarimeter was implemented in the form of a measuring installation. The electrical signals of the photodetectors (InGaAs *Hamamatsu* photodiodes, Japan) were digitized by a 12-bit two-channel ADC (*Rudnev-Shilyaev*, Russia) with a sampling frequency of 200 kHz at an analyzer rotation speed of about 300 rpm.

Inexpensive thin-film polarizers were used as polarizers and analyzer, and laser diodes with fiber-optic output manufactured by *Laserscom LLC* (Belarus) with wavelengths from 405 to 780 nm were used, the light of which was directed to the object using a collimator.

To assess the accuracy of measurements, reference quartz polarimetric plates [34] from *Bellingham-Stanley Ltd* (Great Britain) were used. The temperature of the samples during the measurement was controlled with an error of no more than $\pm 0.01^\circ\text{C}$.

We used the original SpectroPolarimeter software for processing all measured data.

6. Calculation of the phase difference

Since a cuvette with the test substance is installed in the first channel in front of the analyzer, rotating the plane of light polarization by a certain angle $\Delta\varphi$, the phase of the signal $i_1(t)$ will differ by $2\Delta\varphi$, provided that the initial phases φ_1 and φ_2 are equal. The intensity of the light may also differ, therefore, before calculating the phase difference, the amplitudes of the digitized signals are normalized (Fig. 2).

Signal processing begins with multiplication by the apodization function of the modified fourth-order Blackman-Harris window [35] (Fig. 3).

Next, the Fourier transform of the signals is calculated and bandpass filtering of the first order in the spectrum is performed by the Butterworth filter of the fifth order [35]. After applying the filter and calculating the inverse Fourier transform, we get the processed signals $i_1(t)^\sim$, $i_2(t)^\sim$ in complex form. To find the phase difference between signals $i_1(t)^\sim$ and $i_2(t)^\sim$ we need to multiply one of the signals by the complex conjugation of the other and calculate the argument of the result:

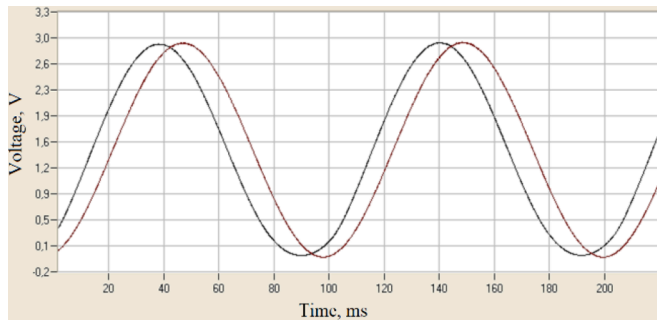


Fig. 2. Time diagram of the normalized signals of a differential phase spectropolarimeter.

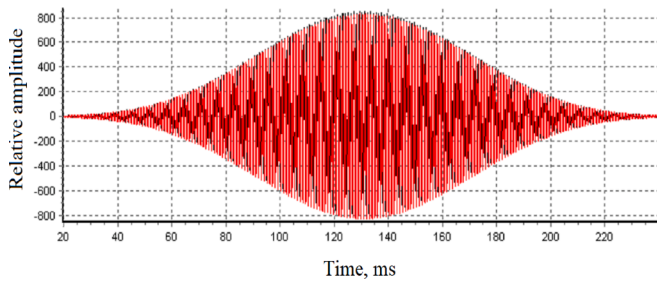


Fig. 3. Diagram of the apodized signals of a differential phase spectropolarimeter.

$$\begin{aligned} \phi_{1,2} &= \text{Arg}[i_1(t) i_2(t)^*] = \\ &= 2(\phi_1 - \phi_2) + \text{Arg}[d(t)] + \text{Arg}[d(t)^*] \end{aligned} \quad (5)$$

where * means the complex conjugate value.

Expression (5) allows us to calculate the phase difference of the signals (Fig. 4), while the instability of the laser light and the rotation of the analyzer are compensated for by the fact that the last two terms of expression (5) turn to zero when added together.

The ORA value for most optically active substances is units or tens of degrees. For example, the most popular sucrose concentration measurements, it is about tens of degrees. But if ORA of the sample may be greater than 180° , it is necessary to take this fact into account. For example, to investigate that substance at different concentration, or to use cuvettes with different length.

7. Experimental

To measure the ORA at a certain wavelength, it is necessary to make two exposures. During the first exposure, the object under study is missing. A small initial phase shift of ϕ_0 between the harmonical signals from the photodetectors is caused by a mismatch of the polarization direction set by the stationary polarizers 4 and 5. The value of ϕ_0 is calculated according to the described algorithm and considered in

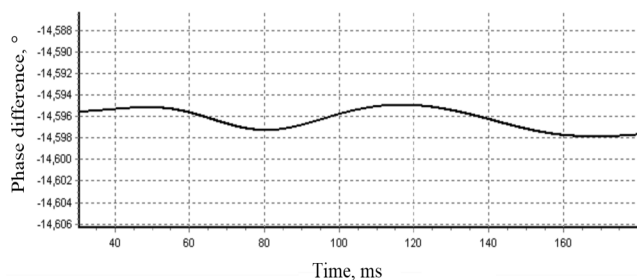


Fig. 4. Time diagram of the calculated phase difference of the signals.

subsequent measurements to improve the accuracy. Then the object under study is added to the optical scheme and a second exposure is performed. After the second exposure, the phase difference $\phi_{1,2}$ is also calculated. The half-difference between the phases during the second and first exposures gives the ORA for the wavelength used, i.e. $\Delta\phi = (\phi_{12} - \phi_0)/2$

It is important that no more than a minute passed between recording the first exposure without a sample and the second one with a sample. In this case, the experimental conditions will not change significantly, so high accuracy and repeatability of measurements is achieved.

Since reference quartz polarimetric plates were used as the objects under study, it is possible to estimate the measurement error in comparison with the reference values of the ORA for specific wavelengths. The reference values are given for the wavelength of the He-Ne laser (632.8 nm), therefore, it is necessary to recalculate using the formula for the specific rotation of a quartz plate with a thickness of 1 mm [34]:

$$\alpha(\lambda) = \alpha_1 + \alpha_2\lambda^{-2} + \alpha_3\lambda^{-4} + \alpha_4\lambda^{-6} \quad (6)$$

where $\alpha_1 = -0.1963657^\circ/\text{mm}$; $\alpha_2 = 7.262667^\circ\mu\text{m}^2/\text{mm}$;
 $\alpha_3 = 0.1171867^\circ\mu\text{m}^4/\text{mm}$; $\alpha_4 = 0.0019554^\circ\mu\text{m}^6/\text{mm}$.

The recalculation of the measured values of the ORA $\phi(\lambda)$ is performed according to the formula [34]:

$$\phi_{\text{HeNe}} = \phi(\lambda)\alpha_{\text{HeNe}}/\alpha(\lambda) \quad (7)$$

where ϕ_{HeNe} is the ORA for a wavelength of 632.8 nm; $\phi(\lambda)$ is the ORA for the wavelength used.

To account for the temperature, the formula was used [34]:

$$\alpha_{T^\circ\text{C}} = \alpha_{20^\circ\text{C}} [1 + (b_1 + b_2\lambda^{-1} + b_3\lambda^{-2})(t - 20^\circ\text{C})] \quad (8)$$

where $b_1 = 1.280924 \cdot 10^{-4} 1/^\circ\text{C}$; $b_2 = 1.0852636 \cdot 10^{-5} \mu\text{m}/^\circ\text{C}$;
 $b_3 = -9.0311692 \cdot 10^{-7} \mu\text{m}^2/^\circ\text{C}$.

Table 2 shows the results of measurements of the ORA $\Delta\phi_i$ for a polarimetric plates N \circ 1, N \circ 2, and N \circ 3 with a reference value of $\phi_{\text{HeNe}1} = 29.757^\circ$, $\phi_{\text{HeNe}2} = -9.053^\circ$ and $\phi_{\text{HeNe}3} = 4.513^\circ$ obtained from the results of international comparisons [34].

Fig. 5 shows the ORD for a polarimetric plate N \circ 1.

Table 3 shows the results of recalculation of the ORA $\Delta\phi_i^*$ according to (6), (7), and (8).

Fig. 6 shows the results of the measurements error Δ calculation compared with a reference value of ORA.

According to the calculated error values, the differential phase spectropolarimeter, built according to the presented design, allows determining the ORA in the wavelength range from 405 nm to 780 nm with an error of no more than $\pm 0.004^\circ$ and standard deviation of 0.0005° , which corresponds to the accuracy of the best reference polarimetric instruments [36].

8. Conclusion

The phase spectropolarimeter proposed in the article has a high accuracy, since the design uses a differential measurement method that compensates for the effect of the analyzer rotation unevenness and fluctuations in light intensity due to the separation of the beam from a common source into reference and object beams. This optical design based on phase measurements does not require high-precision angle

Table 2
The ORA measurement results.

λ , nm	$\Delta\phi_1$	$\Delta\phi_2$	$\Delta\phi_3$
405	77.8273	-23.6716	11.8024
450	61.7148	-18.7737	9.3516
520	45.1659	-13.7375	6.8419
638	29.2704	-8.9039	4.4383
780	19.2120	-5.8455	2.9137

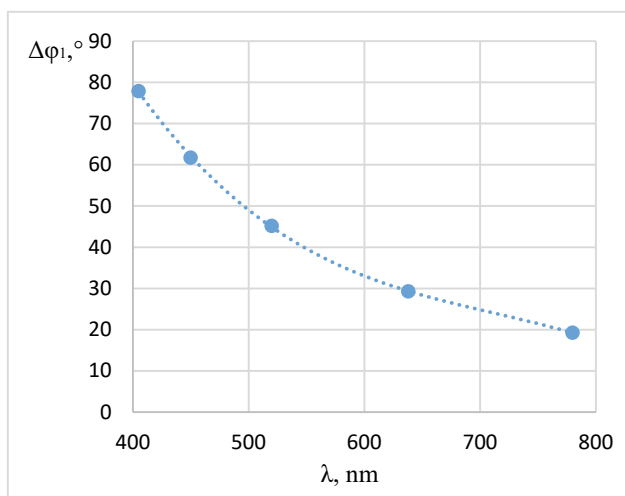


Fig. 5. The ORD for quartz polarimetric plate with reference value $\varphi_{\text{HeNeI}} = 29.757^\circ$.

Table 3

Results of recalculation of ORA reduced to 20 °C and 632.8 nm.

λ , nm	$\Delta\varphi_1^*$	$\Delta\varphi_2^*$	$\Delta\varphi_3^*$
405	29.7582	-9.0511	4.5138
450	29.7572	-9.0522	4.5091
520	29.7609	-9.0520	4.5083
638	29.7601	-9.0529	4.5125
780	29.7583	-9.0543	4.5132

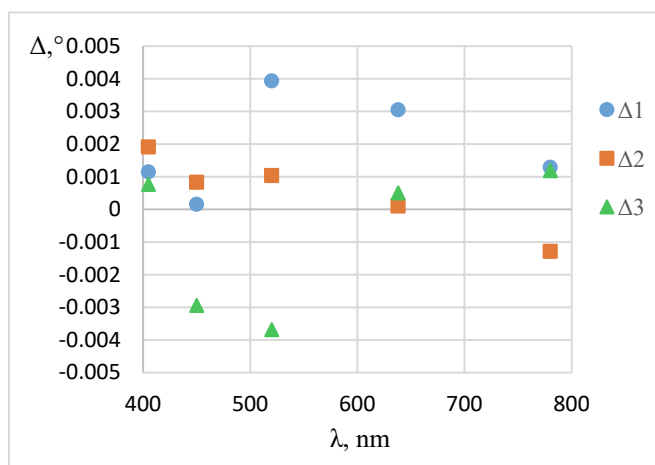


Fig. 6. The measurements error Δ calculation compared with a reference value of ORA for quartz polarimetric plates.

measuring devices, encoders or servos and can be implemented using a few off-the-shelf optical and electronic components - laser diodes, film polarizers, photodiodes, an electric motor, and a PC with ADC. The simplicity of the design of such a spectropolarimeter, low cost, compact size and high accuracy make it a powerful and versatile instrument for ORD measurements and the physicochemical analysis of optically active substances.

CRediT authorship contribution statement

Gennady Vishnyakov: Conceptualization, Methodology, Formal analysis, Writing – review & editing. **Alexander Yurin:** Conceptualization, Methodology, Validation, Data curation. **Alexander**

Golopolosov: Data curation, Validation, Formal analysis. **Vladimir Minaev:** Project administration, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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