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LASER METHOD OF REFRACTIVE INDEX DETERMINATION WITHIN THE SELECTIVE REFLECTION BAND OF CHOLESTERIC LIQUID CRYSTALS

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Abstract A principally new method of simultaneous determination of refractive index and small values of helical pitch of cholesteric liquid crystals is proposed. Its accuracy, limitations and possible use are also discussed.

INTRODUCTION

A determination of precise values of helical pitch and optical constants of cholesteric liquid crystals (CLC) is very urgent for purpose of development of information display systems based on liquid crystals (LC) and for synthesis of new chiral LC materials.

Determination of refractive index n within the selective reflection (SR) band of CLC and small values of helical pitch P (less than $1\ \mu\text{m}$) is connected with a number of difficulties conditioned by principle and techniques. For example, measurement of helical pitch or average value of refractive index of CLC \bar{n} by SR band characteristics using the known relation $\lambda = P \cdot \bar{n}$, where λ is wavelength of the centre of SR band, requires in turn the knowledge of the accurate magnitude either of quantity \bar{n} or P obtained by another independent method. A measurement of \bar{n} by the oscillations in transmission spectrum of the cell, with its walls adjusted strongly parallel, filled by the CLC (a variant of the interferometrical method) could not ensure high accuracy. This is caused by the following reasons: a) faint Fresnel reflection on the glass/CLC boundaries because of small difference in the values of their refractive indices leads to the interference pattern with small modulation of transmittance; b) analogous decrease in the pattern's contrast is due to strong scattering of probe light beam in the close neighbourhood (and within) of SR band and near the CLC absorption band. Refractometrical determination of n for CLC with small value of helical pitch, e.g. by means of standard Abbe refractometer, widely applied for liquids, possesses significant drawbacks, namely: a) CLC between the

prisms of refractometer does not form well-ordered structure; b) attenuation of light intensity due to its scattering when measuring under illumination of the work plane of measuring prism through the CLC layer; c) use of white light that leads to the value n corresponding to wavelength which the optical scheme (exactly a compensator of dispersion) is adjusted on, e.g. that of yellow Na line 589 nm; d) really the very low accuracy (worse than 0.02) while illuminating the work plane of measuring prism (light does not pass through the layer); e) disadvantage of using interference filters for selection of spectrally narrow band of light (with bandwidth 10÷20 nm) owing to the low brightness and contrast of image in the lunette of refractometer.

Methods of direct determination of P , based on the measurement of the distance between disclinations in planar texture, formed either in the wedge-shaped CLC-cell^{1,2} or between the lens and flat surface² (the variants of Cano-Grandjean method), or immediate measurements of P by the microscope using the "finger print" texture^{1,2} do not provide high accuracy and are actually applicable to the CLC with a value of P not less than 1.5÷2 μm . Method of visual measurement of the scattering angle of parallel beam of monochromatic light (e.g. laser beam) on focal conic texture of CLC^{1,2} does not also ensure necessary accuracy and can be used for CLC with $P > 1 \mu\text{m}$.

METHOD AND ITS JUSTIFICATION

In this work we present a method of simultaneous determination of magnitudes of \bar{n} and P for CLC with the value of P less than 1 μm . The value of \bar{n} is determined through the analysis of the spectral composition of generation of distributed feedback (DF) laser on basis of CLC doped by generating dye. Then, a value of P is calculated from the relation $P = \lambda_B / \bar{n}$, where λ_B is a wavelength of longitudinal generation mode with index 0, coinciding with Bragg one.

This DF-laser is pulsed dye laser, with flat CLC layer of thickness L , doped by generating dye, being used as an active medium. In such type of laser DF is brought about by the coherent scattering of dye fluorescence radiation on the permanent phase grating of refractive index. Its period and amplitude of modulation are determined by the helical pitch and birefringence Δn of CLC, respectively.

Intermodal interval in spectrum of the DF-laser resonance frequencies (wavelengths) under excitation conditions close to generation threshold is determined by the following relation³

$$\frac{(\lambda_B^2 - \lambda_{\pm 1}^2)}{\lambda_{\pm 1} \cdot \lambda_B^2} = \frac{1}{\bar{n} \cdot L} \left(\pm 1 + \frac{1}{\pi} \cdot \arg(\kappa) \right), \quad (1)$$

where $\lambda_{\pm 1}$ is a wavelength of longitudinal mode with index ± 1 , and $\arg(\kappa)$ is a phase of coupling constant of counter-propagating gained waves κ , which is determined as

$$\kappa = \frac{\pi \cdot \Delta n}{\lambda_B} + i \cdot \frac{\Delta \alpha}{2}. \quad (2)$$

In Eq. (2) $\Delta \alpha$ is the amplitude of gain modulation. To estimate the magnitude of $\arg(\kappa)$ we can use an evident relationship $\Delta \alpha \leq S \cdot \alpha_{th}$, where S is the order parameter for dye in CLC and α_{th} is the threshold gain value. As was established,⁴ the order parameter for cholesterol derivatives, used in DF-laser,⁵ is nearly the same as for nematic LC 4-methoxybenzylidene-4'-butylaniline (MBBA). This gives some basis for an assumption about dye order parameter, determined by the linear dichroism of its absorption in MBBA ($S=0.25$ for the phenalenone dye⁵ used in our DF-laser), to be approximately equal to that for CLC. An estimation of the threshold gain using relation from^{6,7} under the typical values of DF-laser parameters ($\Delta n=0.05$, $\bar{n}=1.5$, $L=50 \mu m$, threshold pumping intensity $J=60 \text{ kW/cm}^2$) gives the magnitude of $\alpha_{th} \approx 10 \text{ cm}^{-1}$. According to this $\Delta \alpha \leq 2.5 \text{ cm}^{-1}$, hence, $\arg(\kappa) \approx 5 \cdot 10^{-4} \text{ rad}$ and its contribution to the value \bar{n} (see Eq. (1)) under the near-threshold conditions is negligible. The relation (1) may be transformed for \bar{n} calculation into the form:

$$\bar{n} = \frac{\pm 1 \cdot \lambda_{\pm 1} \cdot \lambda_B^2}{L \cdot (\lambda_B^2 - \lambda_{\pm 1}^2)} \quad (3)$$

Relationship (1) may be modified in a more simple form coinciding with that for lower-index eigenmodes of flat plate made from isotropic material with the same L and $n=\bar{n}$

$$\frac{\lambda_B - \lambda_{\pm 1}}{\lambda_{\pm 1} \cdot \lambda_B} = \frac{\pm 1}{2n \cdot L} \quad (4)$$

The simplification error for \bar{n} is big enough, $\delta n = 3 \cdot 10^{-3}$, and is comparable with the accuracy of our measurements. That is why use of (1) and (3) is preferable. The discrepancy between (1) and (4) is connected with the particularly different mechanisms of mode formation. For DF-laser it is the Bragg scattering, distributed in the bulk CLC film, while for the second case it is usual Fresnel reflection on plate boundaries.

EXPERIMENTAL SETUP AND RESULTS

A DF-laser on the basis of CLC was described in details in^{5,8}. A three-component mixture of cholesterol derivatives of following composition was used: cholesteryloleate 43.5 wt.%, cholesterylpelargonate 30 wt.% and cholesterylchloride 26.2 wt.%. In this

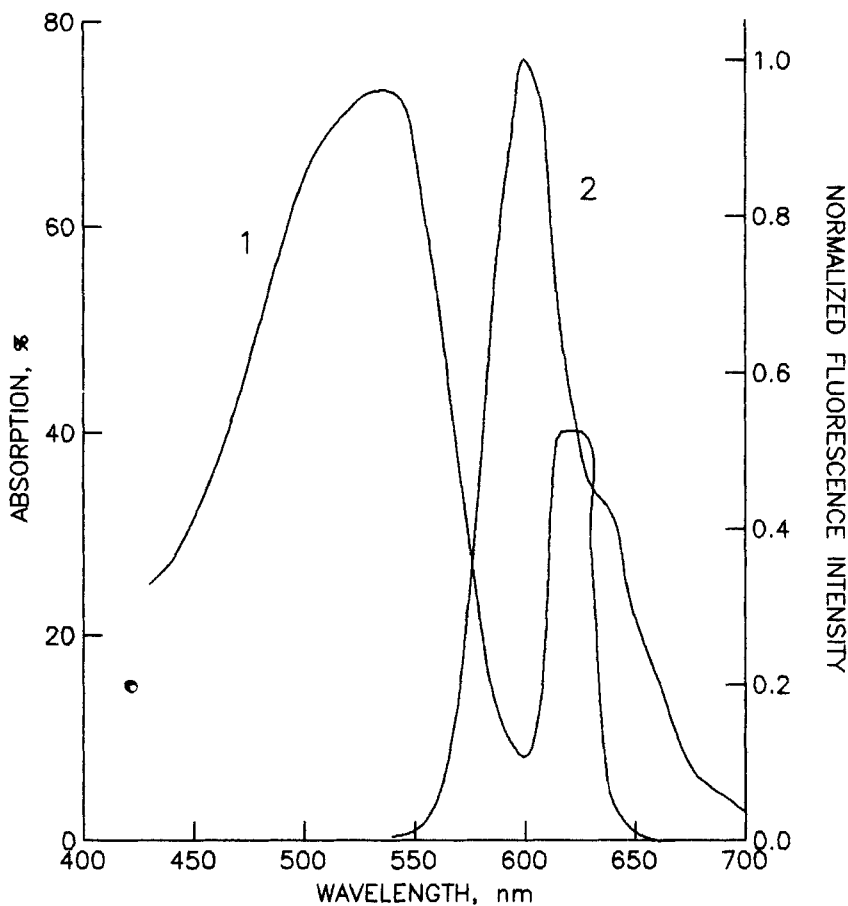


FIGURE 1 Absorption (1) and normalized fluorescence (2) spectra of 0.3 wt.% dye-doped CLC ($L=50\ \mu\text{m}$).

mixture a generating dye 3-threefluorine-6-cyclohexyl-aminophenalenone was solved in concentration 0.3 wt.%. A transmission spectrum of this mixture, shown in Fig.1, indicates the dye absorption maximum near 530 nm and the wavelength of SR band centre above 600 nm.

The CLC layer with thickness ranging from 30 to 60 μm was prepared between two substrates covered by the polyimide lacquer and uniaxially rubbed in order to form a

planar texture. For obtaining better planar orientation, namely, a homogeneous Grandjean texture with helical axis perpendicular to the cell walls, a relative shifting of substrates along the rubbing direction was made. The maximum dimensions of domain of qualitative planar texture are about 3 mm × 3 mm.

For successfully using this method the thickness of CLC layer should be in range 30÷60 μm. For the layers thicker than 100 μm intermodal interval in generation spectrum is small, and, as a rule, a quality of planar texture is worse that gives rise to broadening of generation lines. These causes decrease substantially the accuracy of intermodal interval measurement, which mainly determines that of method in whole. For the layers thinner than 20 μm a generation threshold intensity and, hence, heating of excited area, increases essentially.

Preparing the CLC cells a special care was taken for the substrates were mutually parallel. It is essential for precise determination of CLC layer thickness which is roughly specified by that of the calibrated spacers. Then, a cell was held in a special frame with three thumb screws that allowed the substrates to be adjusted parallel by observing the interference fringes (by this way one fringe or less per one centimeter of length may be achieved). A cell was subdivided by these spacers in two compartments and only one of them was filled by the CLC. The unfilled half of the cell was used for thickness determination. The thickness was measured by the interference pattern in transmission spectrum, monitored by the SP-20 double beam spectrophotometer. For thickness determination the known relationship

$$L = \frac{(i-j)\lambda_i\lambda_j}{2(\lambda_j - \lambda_i)}, \quad (5)$$

was used, in which i and j are the interference orders for two selected peaks of interferogram, λ_i and λ_j - their wavelength, respectively. The accuracy of thickness measurement while using many interference maximums ($|\lambda_i - \lambda_j| > 200$ nm) was down to 0.05 μm.

A generation of DF-laser was excited by the second harmonic radiation of Nd³⁺-glass Q-switched laser ($\lambda_{exc}=530$ nm), operated in a single pulse mode. Pumping power was 1.5÷2.5 times higher than the threshold one. Generation spectra were recorded photographically at the spectrograph with the dispersion 0.6 nm/mm. Experimental setup is sketched in Fig.2.

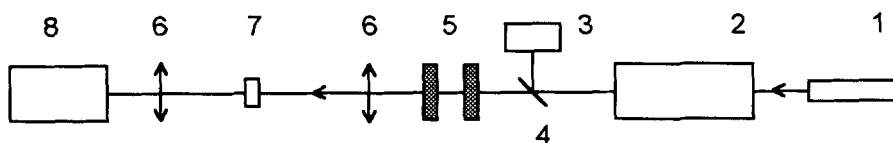


FIGURE 2 Experimental setup: 1 - He-Ne-laser, 2 - Nd-glass-laser, 3 - power meter, 4 - beam splitter, 5 - attenuation filters, 6 - lenses, 7 - CLC-cell, 8 - spectrograph.

The generation line of a He-Ne laser ($\lambda_{\text{ref}} = 632.8 \text{ nm}$) was chosen as a reference one. The distance between lines on the film was measured either by the comparator of spacing IZA-2 or using densitograms of a film transparency (Fig.3). The error of generation wavelength measurements was about $0.005 \div 0.02 \text{ nm}$. The accuracy of P determination was given, mainly, by that for \bar{n} and was $0.3 \div 1.0 \text{ nm}$.

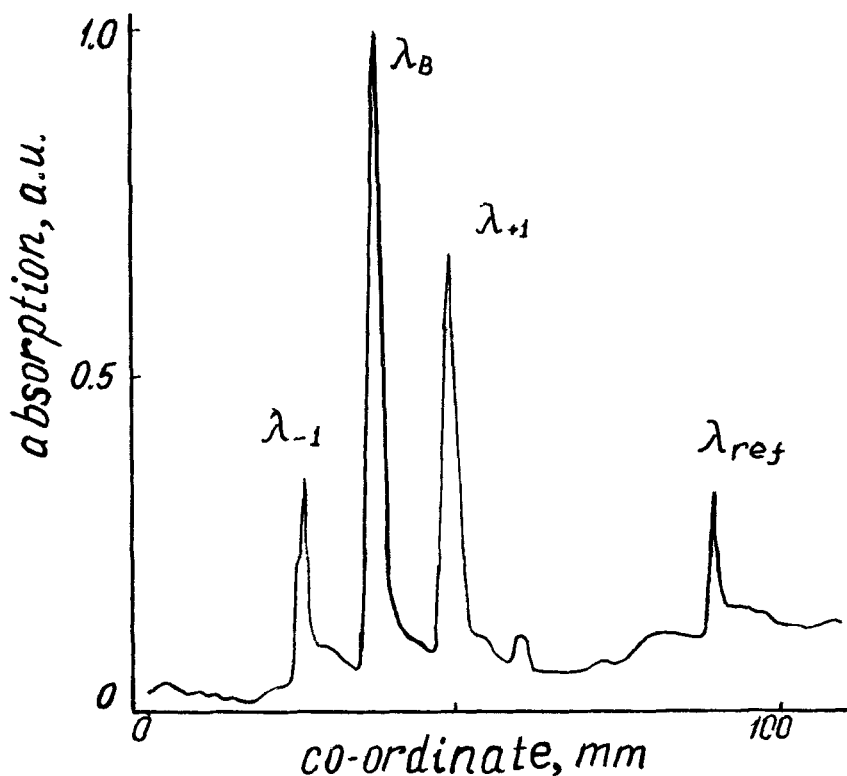


FIGURE 3 Transparency densitogram of the film with DF-laser generation spectrum recorded.

It should be noted that the diameter of generation zone was $0.1 \div 0.3$ mm, therefore measured values of \bar{n} and P were local ones (average for this area, not for the whole sample). This is the reason for a considerable spatial discrepancy of measured values (about 2 nm for P and 0.005 for \bar{n}).

The method was validated on a number of samples with variable cell thickness and dye concentration under the different experimental conditions.

It was very interesting to compare our data for \bar{n} with those obtained by the other independent technique for the same mixture. A standard Abbe refractometer URL-1 with the white light source and outcoming beam analyzer was used for this purpose. Measurements were made for two mutually perpendicular analyzer positions (parallel (\parallel) and orthogonal (\perp) to the normal to the working plane of a measuring prism): $n_{\parallel}=1.4980 \pm 0.0008$, $n_{\perp}=1.5170 \pm 0.0005$ and $\bar{n}=(n_{\parallel}+n_{\perp})/2=1.5071 \pm 0.0010$ for undoped CLC; $n_{\parallel}=1.4972 \pm 0.0010$, $n_{\perp}=1.5172 \pm 0.0005$ and $\bar{n}=1.507 \pm 0.001$ for 0.3 wt.% doped CLC. They are roughly consistent with results obtained for the same compound with the help of laser technique: $\bar{n}=1.516 \pm 0.002$, $P=404.9 \pm 0.6$ nm ($L=51.55 \pm 0.05$ μ m). Besides that, they are the experimental evidence to the fact that dye doping has no effect on the value of refractive index of CLC matrix.

THEORETICAL ESTIMATION OF THE EFFECT OF EXPERIMENTAL CONDITIONS ON THE ACCURACY OF THE METHOD

Pumping energy is lost partially in the radiationless transitions that results in the temperature increase of⁸ (disregarding heat transfer from CLC layer to substrates)

$$\Delta T \approx \frac{E_{th}(1-\eta)}{C_v \cdot V \cdot \rho} \cdot \frac{\lambda_{exc}}{\lambda_g}, \quad (6)$$

where E_{th} is the threshold excitation energy, λ_g is the generation wavelength, η is the quantum efficiency of the dye fluorescence, C_v - constant volume specific heat capacity of CLC, V - volume of the excited zone, ρ is the CLC density. Under $E_{th}=3 \cdot 10^{-6}$ J, $\lambda_g=600$ nm, $C_v \approx 1$ J/g·K, $\rho \approx 1$ g/cm³, $V=1.5 \cdot 10^{-5}$ cm³, $\eta=0.55$, the value of temperature rise mounts up to 0.1 K. Note, the value of ΔT will be still smaller if heat transfer being taken into account. Such small heating could not produce considerable variation in n owing to the change in density (an effect analogous to "thermal lens" formation). Besides, characteristic time of density variation of the order of $L/v \sim 400$ ns (where $v \sim 1.3 \cdot 10^4$ cm/s is sound velocity in CLC⁹ and $L=50$ μ m is characteristic value of CLC layer thickness) is much greater than duration of pumping pulse 15 ns. During the time of laser pulse there is no variation of the pitch due to its temperature dependence

($dP/dT \sim 2 \div 3$ nm/K for such a CLC) as well, because the time of orientational diffusion of molecules is much greater and comparable with that of energy dissipation⁷ $\tau = C_V \cdot r / \chi \sim 1$ ms (here χ is thermal conductivity and r is linear size of excitation zone). Hence, heating of the generation area does not affect the accuracy of \bar{n} determination under the threshold conditions.

A correction to the value of \bar{n} should be also made for the variations in density, molecular polarizability γ , molecular weight M , number of molecules per unit volume N (or average effective volume per one molecule v) of CLC mixture due to the dye doping. Well-known Lorenz-Lorentz's relationship for isotropic fluid can be written for pure CLC in the form of (through the values N or v)

$$\frac{n_C^2 - 1}{n_C^2 + 2} = \frac{4\pi}{3} \cdot N_C \cdot \gamma_C = \frac{4\pi}{3} \cdot \frac{\gamma_C}{v_C}, \quad (7)$$

and for doped CLC with a per-unit molar fraction of dye x (taking into account an additivity of molecular refractions of components) as

$$\frac{n^2 - 1}{n^2 + 2} = \frac{4\pi}{3} \cdot N \cdot ((1-x) \cdot \gamma_C + x \cdot \gamma_D) = \frac{4\pi}{3} \cdot \frac{((1-x) \cdot \gamma_C + x \cdot \gamma_D)}{v}, \quad (8)$$

where index D corresponds to pure dye. Subtracting Eq. (7) from Eq. (8), then, dividing the resulting equation by the Eq. (7) and taking into account the relation $v = (1-x) \cdot v_C + x \cdot v_D$, we obtain after simplification the following estimation (in the linear approximation)

$$\delta n \cong \frac{(n^2 + 2)(n^2 - 1)}{6n} \cdot \left(\left(1 - \frac{v_D}{v_C} \right) + \left(\frac{\gamma_D}{\gamma_C} - 1 \right) \right) \cdot x. \quad (9)$$

A value of x may be expressed through a per-unit mass fraction of dye in the mixture y by the following relationship

$$x = \frac{y}{\frac{M_D}{M_C} + \left(1 - \frac{M_D}{M_C} \right) \cdot y} \approx \frac{M_C}{M_D} \cdot y. \quad (10)$$

Under the $y = 1 \div 3 \cdot 10^{-3}$, $v_D/v_C \approx 0.7$ and $|\gamma_D/\gamma_C - 1| \leq 1.0$ we calculate $\delta n \approx 2 \div 5 \cdot 10^{-4}$, that doesn't exceed the accuracy of our method and, so, we may neglect it.

Since there is not any considerable bleaching of the dye under the excitation power close to the threshold one (threshold of bleaching is near 10^6 W/cm²), a theoretical estimation of dye absorption effect on the refractive index value is to be made. We regard dye-doped CLC layer as completely uniform and isotropic absorbing medium, with absorption coefficient α equal to that of the real layer, refractive index n^* and resonance frequency of electron transition Ω . For the plane electro-magnetic wave $E = E_0 \cdot \exp\{i(kz - \omega t)\}$ with circular frequency ω , vacuum wavenumber $k_0 = \omega/c$ (where c is

the light velocity in vacuum) and wavenumber in the medium $\mathbf{k}=\mathbf{k}'+i\cdot\mathbf{k}''$ the following obvious relations can be written:

$$\mathbf{k}' = \mathbf{n}^* \cdot \mathbf{k}_0, \quad (11a)$$

$$\mathbf{k}'' = -\alpha/2. \quad (11b)$$

From the other hand, solving the wave equation for this wave, which is propagating in the medium indicated above, the following characteristic equation is obtained in ¹⁰

$$\mathbf{k}^2 = \mathbf{k}_0^2 \cdot (\mathbf{n}^2 + \gamma), \quad (12)$$

where $\gamma=\gamma'+i\cdot\gamma''$ is a polarizability and \mathbf{n} is refractive index of the same medium without absorbing centres. Comparing Eqs. (11) with the Eq. (12) we derive the relations

$$\mathbf{k}'^2 - \mathbf{k}''^2 = \mathbf{k}_0^2 \cdot (\mathbf{n}^2 + \gamma'), \quad (13a)$$

$$2 \cdot \mathbf{k}' \cdot \mathbf{k}'' = \mathbf{k}_0^2 \cdot \gamma''. \quad (13b)$$

Substituting Eqs. (11) into the Eq. (13b) we obtain

$$\mathbf{n}^* = -\mathbf{k}_0 \cdot \gamma'' / \alpha. \quad (14)$$

From a system of equations (13), taking into account Eq. (11b) and a relation between the γ' and γ'' for the case of Lorentzian profile of absorption band¹⁰ $\gamma'=\gamma'' \cdot \{2 \cdot (\omega-\Omega) / \Delta\omega_L\} = \gamma'' \cdot \sigma$ (where $\Delta\omega_L$ is a halfwidth of the resonance curve), we can derive an equation

$$4\mathbf{k}_0^4 \cdot \gamma''^2 - 4\mathbf{k}_0^2 \alpha^2 \sigma \cdot \gamma'' - \alpha^2 \cdot (4\mathbf{k}_0^2 \mathbf{n}^2 + \alpha^2) = 0 \quad (15)$$

Solving Eq. (15) for γ'' and making appropriate selection of the solutions ($\gamma'' < 0$) we find

$$\gamma'' = \mathbf{n} \cdot \frac{\alpha}{\mathbf{k}_0} \cdot \left(\frac{\alpha}{2\mathbf{n}\mathbf{k}_0} \cdot \sigma - \sqrt{1 + \left(\frac{\alpha}{2\mathbf{n}\mathbf{k}_0} \right)^2 \cdot (1 + \sigma^2)} \right). \quad (16)$$

Hence, for $\mathbf{n}^* = -\mathbf{k}_0 \cdot \gamma'' / \alpha$, we have

$$\mathbf{n}^* = \mathbf{n} \cdot \left(\sqrt{1 + \left(\frac{\alpha}{2\mathbf{n}\mathbf{k}_0} \right)^2 (1 + \sigma^2)} - \frac{\alpha}{2\mathbf{n}\mathbf{k}_0} \cdot \sigma \right), \quad (17)$$

and for variation of \mathbf{n} due to anomalous dispersion of the dye absorption $\delta\mathbf{n}^* = \mathbf{n}^* - \mathbf{n}$, we can derive from Eq. (17)

$$\delta\mathbf{n}^* = -\frac{\alpha}{\mathbf{k}_0} \cdot \frac{\sigma - \frac{\alpha}{4\mathbf{n}\mathbf{k}_0}}{1 + \frac{\alpha}{2\mathbf{n}\mathbf{k}_0} \cdot \sigma + \sqrt{1 + \left(\frac{\alpha}{2\mathbf{n}\mathbf{k}_0} \right)^2 (1 + \sigma^2)}} \approx -\frac{\alpha \cdot \sigma}{2 \cdot \mathbf{k}_0}. \quad (18)$$

For Lorentzian absorption profile it takes place $\alpha(\omega) = \alpha_{\max} / (1 + \sigma^2)$, where α_{\max} - absorption coefficient in the maximum. For $\alpha_{\max} = 173 \text{ cm}^{-1}$ (that corresponds to $\lambda_{\max} = 530 \text{ nm}$), $\lambda = 2\pi/\mathbf{k}_0 = 600 \text{ nm}$, $\sigma = -2$, $\mathbf{n} = 1.5$ we can calculate $\delta\mathbf{n}^* = 3 \cdot 10^{-4}$ that is nearly one order of magnitude lower than pretended accuracy of \mathbf{n} measurement by the laser method.

CONCLUDING REMARKS

The method proposed is, naturally, not free of limitations. Some of them are caused by the apparatus potentialities, others are based on fundamental restrictions, which may be divided into three groups. The inaccuracy produced by absorber adding was discussed above. Besides, the range of P and \bar{n} values' measurement is conditioned by the width of dye fluorescence band (<100 nm) and, thereby, when changing a range of pitch variation then other dye, with corresponding spectral position of gain band, must be selected. Under this, the following requirements should be taken into account: a) only the substances of superior purity must be used; b) it is preferable, that dye molecules are isomorphic to the CLC ones, have comparable dimensions, are chemically stable and don't aggregate; c) there must be substantial Stocks shift of fluorescence band (≥ 50 nm) to avoid reabsorption of radiation.

The quality of planar texture has to be as high as possible because any disturbance of helicity gives rise to the pitch gradient that leads to generation lines broadening and, hence, to decrease of the measurement accuracy.

Such laser effect as transverse-mode generation can also worsen the quality of spectrum registration through the appearance of additional lines, and the proper selection is needed in this case.

In spite of all limitations mentioned above the method proposed could be used as a reliable and a sensitive one for the determination of small local variations of helical pitch and refractive index of CLC. Our technique provides rather high accuracy accompanied by the negligible disturbance of CLC structure.

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