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Optical activity and electro-optic effect of L-arginine doped KDP single crystals

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Abstract

We have used the modified polarimetric methods to study optical activity (OA) in the potassium dihydrogen phosphate (KDP) crystals doped with 0.7, 1.4 and 3.8 wt % L-arginine (L-arg) amino acid. Crystals were grown by the temperature reduction method. Small changes of the absolute eigen waves ellipticity and OA values in doped crystals were noted. We have experimentally determined the signs of OA in the [100] and [010] directions in KDP type crystals. The electro-optical parameters (half-wave voltage and electro-optical coefficients r_{63}) in L-arg doped crystals for fixed wavelengths of 633, 532 and 405 nm remain unchanged in comparison with the pure KDP.

Keywords: KDP, crystal optics, polarimetry, optical activity, electro-optic effect

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1. Introduction

Potassium dihydrogen phosphate (KH_2PO_4, KDP) and most popular deuterated (DKDP) single crystals are widely used in non-linear optics and optoelectronics, high-energy laser systems, electro-optical applications [1, 2]. One of the methods to improve their nonlinear optical parameters is to introduce organic molecules into the matrix of KDP crystal. The efficiency of second harmonic generation (SHG) for KDP with 0.3 – 1.4 wt % of L-arginine (L-arg) rises up to 2.0 [3, 4], or even up to 2.5 times [5] in comparison with the pure KDP. Alternatively, one can incorporate the TiO_2 nanoparticles into the KDP single crystals in order to improve their nonlinear properties [6].

Considering the improvement of the nonlinear optical characteristics, we have investigated the influence of L-arginine (0.7, 1.4, 3.8 wt % of amino acid in the solution) on the electro-optic effect, phenomenon, that is characterised by large anisotropy and sensitivity to changes in the structure of KDP single crystal. Using the advanced polarimetric technique we expected to detect changes in OA of doped KDP crystals as well. These properties of pure KDP group crystals have been already studied thoroughly in the past [7, 8, 9, 10] however, there is no data about the effect of amino acid doping on nonlinearity in the static electric field. Considering that amino acid molecules and monoclinic L-arginine phosphate single crystals are optically active [11], the interest in the study of OA is justified.

Pure KDP and KDP:L-arg crystals were grown from the aqueous solutions by the temperature reduction method. The temperature was lowered at rates of 0.3 - 0.4 K/day and average crystal growth rate was 1.4 mm/day and 1.0 mm/day for pure KDP and KDP:L-arg, respectively [12]. The study of

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KDP:L-arg crystals has shown that the structural perfection of the doped crystals corresponds to that of pure KDP crystals. Investigations with the X-ray diffractometer using rocking curves and parameters of the crystal cell confirm high quality of the crystal at the level of only $\sim 10^{-4}$ Å in lattice parameter variations [13]. There were no changes observed in the external morphology of the doped KDP crystals, only slight blocking of the 100 faces of the growing crystal as compared to pure KDP was noticed on relatively large crystals ($20 \times 20 \times 25$ mm^3 sizes) at 4.4 wt.% L-arg [13]. Very slight changes in the lattice parameters and no change in the external morphology of KDP:L-arg crystals were reported earlier in [14].

The change of the symmetry of doped crystals was not detected in the optical observations, they are uniaxial and form ideal interference figures, observed along the optical axis. Polished samples were homogeneous and transparent, that allowed us to study their optical properties and, in particular, optical activity, since the crystal quality is vital in polarimetric studies.

KDP is a tetragonal (point group symmetry $\bar{4}2m$) uniaxial crystal, the crystallographic $c-$axis is also the optical axis. Second rank axial gyration tensor for point group symmetry $\bar{4}2m$ includes two nonzero components that are equal in absolute value but opposite in sign ($g_{11} = -g_{22}$) [7, 15] and OA along the optical axis is forbidden by symmetry ($g_{33} = 0$). Therefore, the OA can be measured along directions perpendicular to the optical axis.

2. Polarimetric method

The measurement of the OA in linearly birefringence directions is associated with certain experimental difficulties, since in most cases the eigen

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waves ellipticity k is small [15] and in KDP crystals it does not exceed the 4×10^{-4} value [10]. This explains the fact that optical anisotropy of circular birefringence has been measured for a relatively small number (less than thirty) of crystals [16, 17]. Historically, one of the measurement methods of the OA of crystals along different from the optical axes directions was proposed by Kobayashi et al. [18] and applied to the study of the OA of just KH_2PO_4. However, only after the improvement of the experimental techniques, this method [19, 20], now known as high-accuracy universal polarimeter (HAUP), became very useful in the OA studies, including the KDP crystals [8, 9].

In order to measure the OA in L-arg doped KDP crystal we have applied a dual-wavelength polarimeter [21] which is a modification of HAUP. The polarimetric scheme used for the OA study in birefringent sections is a polarizer-sample-analyzer (PSA), in which polarizer azimuth θ and analyzer azimuth χ, which are measured from the principal crystal axes, are small ($\theta, \chi \ll 1$). One should also consider the imperfections of the polarizers, which are introduced as small ellipticities p and q of the light that passes through the polarizer and analyzer, respectively. Angular error $\delta\chi$ must be taken into account as well [9, 20, 22, 23].

The dual-wavelengths polarimeter has been described in detail in our previous works [21, 24, 25]. Expecting to get a large ellipticity k value, in comparison with the region of 630–650 nm which was used earlier, we chose the semiconductor diode laser ($\lambda_1 = 520$ nm) and diode pumped solid state laser ($\lambda_2 = 532$ nm) in our study. All experiments have been performed in [100] or [010] directions of pure and L-arg doped KDP crystals of about the

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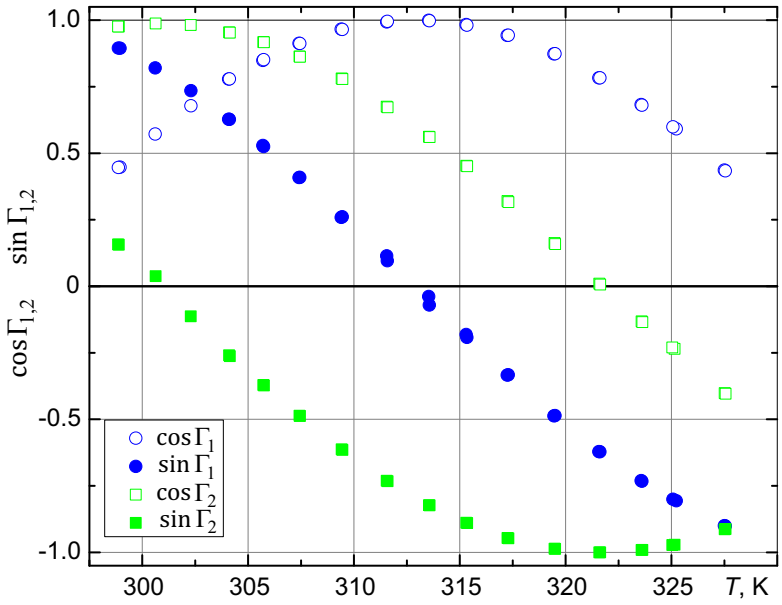

Figure 1: Temperature dependencies of the $\cos\Gamma_{1,2}$ (open symbols) and $\sin\Gamma_{1,2}$ (solid symbols) values for sample KH_2PO_4 doped with 3.8% L-arg for $\lambda_1 = 520$ nm (○, ●) and $\lambda_2 = 532$ nm (□,■).

same $d \approx 0.5$ mm thickness in the temperature region of 295–340 K. Due to the changes in the linear birefringence $\Delta n_{1,2}$ of crystals, such parameters ensured smooth variation of phase retardation $\Gamma_{1,2} = 2\pi\Delta n_{1,2}d/\lambda_{1,2}$ by an amount not exceeding π.

It is easy to measure the cosine of the phase differences $\Gamma_{1,2}$, since in (θ, χ) coordinate system the intensity minima azimuths of the analyzer form a straight line with the tangent of slope angle for each wavelength equal to $\cos\Gamma_{1,2}$. Typical measured dependencies of the $\cos\Gamma_{1,2}$, presented in Figure 1, indicate good wavelength stability of both lasers. For small wavelengths difference ($\lambda_1 - \lambda_2 = 12$ nm) there is a clear shift in the temperature dependencies of $\cos\Gamma_{1,2}$ and $\sin\Gamma_{1,2}$ values.

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For the experimental results processing it is also necessary to obtain the $\sin\Gamma_{1,2}$ correctly which, unlike $\cos\Gamma_{1,2}$, is an odd function of the phase differences $\Gamma_{1,2}$. From the so-called channelled spectra [26, 27] we know that the birefringence values and, therefore, the phase differences, decrease with increasing temperature of the samples. The $\sin\Gamma_{1,2}$ dependencies, based on these KDP crystals properties, are presented in Figure 1. It is also possible to properly calculate the other trigonometric functions which are contained within the works relations in HAUP technique.

In dual-wavelength polarimeter we have used only two laser sources with neighboring λ_1 and λ_2 wavelengths and neglected the dispersion effects of eigen waves ellipticity k value with some approximation, and assumed p, q and $\delta\chi$ to be constant. It allows a simpler solution for the main problem in high accuracy polarimetry, i.e. systematic errors elimination. For wavelengths λ_1 and λ_2 we can obtain three differences $\Delta\theta_{i\lambda} = \theta_i(\lambda_1) - \theta_i(\lambda_2)$ between characteristic azimuths θ_i $(i = 0, 1, 2)$ [21, 24]:

$$\Delta\theta_{0\lambda} = A_0(k - p) - B_0\delta\chi, \tag{1}$$

$$\Delta\theta_{1\lambda} = A_1(k - p) - B_1(k + q), \tag{2}$$

$$\Delta\theta_{2\lambda} = -A_0(p + q)/2. \tag{3}$$

Here $A_0 = \cot(\Gamma_1/2) - \cot(\Gamma_2/2)$, $B_0 = (1 - \cos\Gamma_1)^{-1} - (1 - \cos\Gamma_2)^{-1}$, $A_1 = \cot\Gamma_1 - \cot\Gamma_2$, $B_1 = 1/\sin\Gamma_1 - 1/\sin\Gamma_2$. It is also possible to change the crystal set in the polarization system, i.e. after rotation of the sample by 90 degree around the optical axis of the polarimetric setup the sign of k and Γ in (1)–(3) should be reversed.

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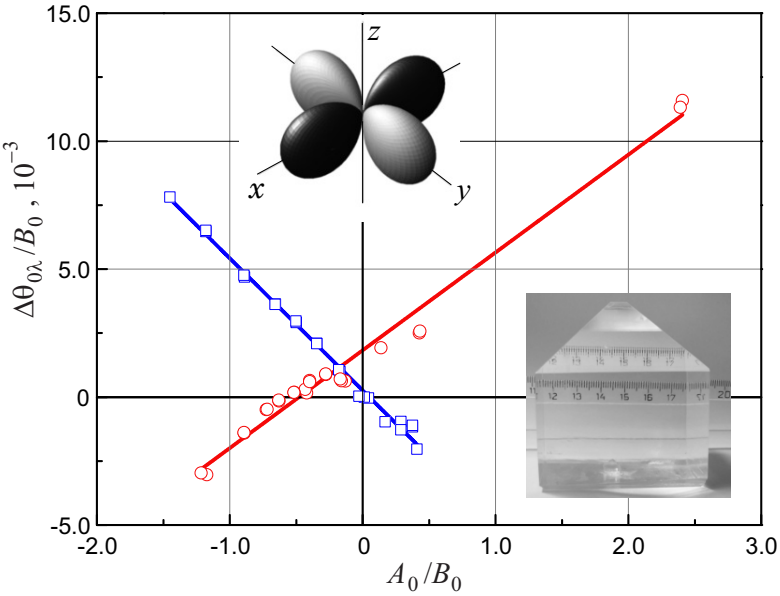

Figure 2: The two plots of the $\Delta\theta_{0\lambda}/B_0$ values versus A_0/B_0 ratio for the (100)-plate of pure KDP correspond to the measurement before (□) and after (○) rotating the sample by 90 degrees around the optical axis of the polarimeter. Solid lines represent the best linear fits and give: $-k-p=-(5.10\pm0.10)\times10^{-3}$ and $k-p=(3.66\pm0.10)\times10^{-3}$. The insets show the gyration surface for $\bar{4}2m$ point group symmetry and grown KDP:L-arg crystal with 1.4 wt % of L-arg concentration viewed normal to one of the enantiomorphous (100) or (010) faces.

The linear fitting of the collected experimental dependencies, according to (1)–(2) similar to those presented in Figure 2, gave us two $\pm k-p$ values for each sample (corresponding to two crystal sets), which are mean values for thermal (295–330 K) and spectral (520–532 nm) intervals. This simplification in the data processing is quite acceptable because of a very weak temperature dependence of the optical activity of KDP crystals close to room temperature which has been observed earlier both experimentally [8, 10] and theoretically [28].

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From the intercepts values in Figure 2 we can also find that the absolute magnitude of the systematic error $\delta\chi < 0.02 \times 10^{-3}$ is within the standard error of the linear fit. Similarly to the previous consideration, we can now obtain linear dependence between characteristic values $\Delta\theta_{1\lambda}/A_1$ and B_1/A_1 ratios for each temperature value of the sample, i.e. $\Delta\theta_{1\lambda}/A_1 = k - p - (k+q)B_1/A_1$. The slopes of these lines, as well as the intersection points, carry information about the required parameters $\pm k$, p, q and also allow us to determine the crystallographic orientation of the sample using the form gyration surface (shown in the inset in Figure 2)

Finally, using the third expression for characteristic differences (3), we find $p + q$ that contains only the sum of parasitic ellipticities of the polarizer and analyzer. Thus, for two crystal sets in PSA system and for three characteristic polarizer azimuth differences, we obtain enough data to find systematic errors p, q, $\delta\chi$ and to calculate the eigen waves ellipticity k values.

3. Optical activity and systematic errors

The relation between eigen waves ellipticity k and gyration tensor component g_{11} for uniaxial crystals is $g_{11} = 2k\Delta n\bar{n}$, where $\bar{n}$ is the mean refractive index [15]. For the mean wavelength of $\bar{\lambda} = 526$ nm the parameters, that we are interested in, are equal to $n_e = 1.4707$, $\bar{n} = 1.4918$ and $\Delta n = 0.0421$ [29]. As the result, we can determine the magnitude of the gyration tensor component g_{11} for pure and doped KDP crystals.

Table 1 presents main results of the measurement that we have conducted on five samples of pure and doped KDP crystals. Each row of the table contains data obtained on dual-wavelength polarimeter for two crystal sets

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in the polarization system. We have also used (100) and (010) crystal plates of pure KDP to confirm the opposite signs of the OA in the x- and y-axis directions, assuming that, according to conventions in [30], the [010] direction is dextrorotatory and eigen waves ellipticity $k > 0$.

Absolute value of the parasitic ellipticity p of polarizer in our polarimetric setup does not exceed 10^{-4} and it is also important that this systematic error does not change its sign in all measurements while parasitic ellipticity q is more sample dependent, what has been noticed by several authors [9, 22]. However, in the HAUP technique the sample is considered to be ideal with two imperfect polarizers.

Grown crystals have well developed growth sectors (100) and (010), but the correct identification of the crystallographic axes is impossible without measurements of the similar to the OA physical effects which, according to the symmetry, should be manifested differently in directions [100] and [010]. Using this property of the KDP crystals and the corresponding experimental dependencies, similar to those in Figure 2, we have determined that the sample with 3.8 wt % L-arg was cut perpendicular to the x-axis whereas with 0.7 and 1.4 wt % concentrations L-arg were cut perpendicular to the y-axis (see Table 1).

In addition to the components of the gyration tensor g_{ij}, the specific optical rotatory power (ORP) is considered to be a practical parameter of the OA. Using the relation $\rho_{\perp} = \pi g_{11}/(\bar{\lambda} n_e)$ formally we get also ORP perpendicular to the optical axis (Table 1). Considering the accuracy of the parameter determination from dependencies, similar to those shown in Figure 2, as well as other possible error sources in polarimetry, we can conclude that the effect

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Crystal	p, 10^{-3}	q, 10^{-3}	$\delta\chi$, 10^{-3}	k, 10^{-3}	g_{11}, 10^{-5}	ρ, deg/mm
Pure KDP, x-cut	–0.068	0.052	0.085	–0.49	–6.14	–14.3
Pure KDP, y-cut	–0.018	–0.072	0.085	0.40	5.06	11.8
0.7% L-arg., y-cut	–0.059	0.210	0.089	0.43	5.40	12.6
1.4% L-arg., y-cut	–0.012	0.005	–0.006	0.41	5.10	11.9
3.8% L-arg., x-cut	–0.030	0.024	0.015	–0.45	–5.63	–13.1

Table 1: Systematic errors of the polarimetric setup and parameters of the pure and doped KDP crystals related to the optical activity

of L-arg additive on the OA of KDP crystals is inconsiderable.

Optical activity in KDP group crystals has a so-called crystalline nature and depends mainly on the inter-atomic interactions. The experimental studies have shown that the replacement of K^+ cation by Rb^+ or NH_4^+ has less impact on optical activity than the replacements, for example, of PO_4^{3-} groups by AsO_4^{3-} [10]. However, tetrahedral symmetry T_d of the PO_4^{3-} groups symmetry does not allow optical activity. Only the interaction of these tetrahedra with protons on hydrogen bonds in H_nPO_4 groups makes the most significant contribution to the total value of the OA for some KDP family crystals and only the [100] and [010] directions have the chiral structure [30]. Measurement of the OA in the UV region, in particular spectroscopic, would be very useful in this case, because spectra of KDP: L-arg crystals contains absorption bands with the maxima at 219 and 270 nm [13], however our transmission method is not applicable close to this spectral region. As shown in [31] by the example of crystals that belong to the same tetragonal

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point group $\bar{4}2m$, the optical activity determination in reflection above the bandgap would be quite sensitive.

4. Longitudinal electro-optic effect

The study is confined to the measurement of electro-optical coefficients r_{63} [7] in the longitudinal mode for three frequently used light wavelengths 405, 520 and 633 nm. Experiments were also carried out on the pure KDP crystal to identify the impact of dopants on electro-optical coefficients. The dimensions of the samples were about $10 \times 10 \times 10$ mm^3. The glass plates coated by indium tin oxide were used as transparent electrodes. The crystals samples with electrodes were placed between two crossed polarizers, and for the given geometry of the experiment the crystallographic axes x and y were parallel to the polarization planes of polarizers. A high voltage (up to 11 kV) supply unit was used in the experiment.

A typical dependence of the transmitted light intensity through the polarizer-sample-analyzer system as a function of applied voltage U at $\lambda = 520$ nm and room temperature is shown in Figure 3 for 0.7 wt % L-arg doped KDP crystal. Similar dependencies for both voltage polarities were found at wavelengths of 633 and 405 nm. The transmittance of such PSA system for polarized light has the following form [7]:

$$T = \sin^2\left(\frac{\pi U}{2U_\pi}\right), \tag{4}$$

where $U_\pi = \frac{\lambda}{2n_o^3 r_{63}}$ is the half-wave voltage and n_o is the ordinary refractive index. The two voltages $U_{-\pi}$ and $U_{+\pi}$, that correspond to the maxima of light intensity, were determined by least squares data fitting independently near both peaks.

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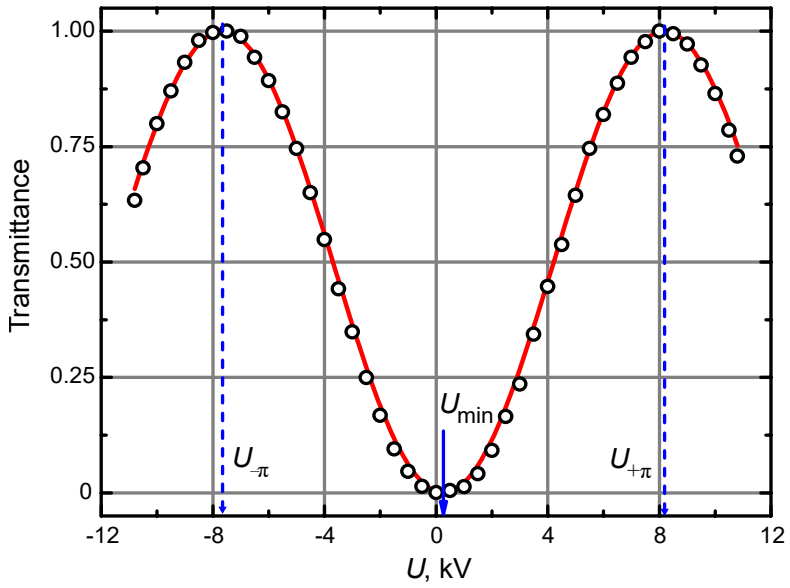

Figure 3: The typical dependence between transmitted through PSA system light ($\lambda = 520$ nm) intensity and applied voltage U of both polarities for pure KDP crystal. $U_{-\pi} = -7.64$ kV, $U_{+\pi} = 8.14$ kV, $U_{\min} = 0.25$ kV. The solid curve is the result of approximation of the measurement data by sine-squared function. The coefficient of determination $R^2 = 0.998$ indicates that the fitted curve is very close to the actual data points.

As seen in Figure 3, the voltage $U_{\min}$, corresponding to the minimum intensity, is not equal zero, and as a result $U_{+\pi} \neq U_{-\pi}$. This is due to the residual stresses in the crystal and the appearance of slight birefringence in the direction of the optical axis at voltage $U = 0$. Typical absolute value of the voltage $U_{\min}$ does not exceed 0.25 kV.

Taking into account the asymmetry of similar dependencies for all crystals, the half-wave voltages were defined as $U_\pi = (U_{+\pi} - U_{-\pi})/2$. The results of half-wave voltages measurement are presented in Table 2. The accuracy of the determination of these values is estimated as ± 0.05 kV. Using the refractive indices for pure KDP [29] and assuming that they do not change

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λ, nm	Pure KDP		0.7% L-arg.		1.4% L-arg.		3.8% L-arg.	
	U_π	r_{63}	U_π	r_{63}	U_π	r_{63}	U_π	r_{63}
633	9.40	9.84	9.57	9.66	9.58	9.65	9.59	9.64
520	7.89	9.51	7.90	9.50	7.80	9.62	7.85	9.56
405	6.08	9.42	6.07	9.44	6.10	9.40	5.96	9.62

Table 2: The half-wave voltages U_π (kV) and electro-optical coefficients r_{63} (10^{-12} m/V) for pure and doped with 0.7, 1.4, 3.8 wt % L-arg KDP crystals, which were measured for three laser wavelengths λ.

in doped crystals, we have obtain also electro-optical coefficients r_{63}.

The present results indicate that the electro-optical parameters (i.e. half-wave voltage and also electro-optic coefficients r_{63}) practically do not change in doped crystals in comparison to the pure KDP crystal. The obtained values of the r_{63} coefficients are very close to the previously determined ones in [32].

5. Conclusions

We have used the modified dual-wavelength polarimetric method to study optical activity of the L-arginine doped KDP crystals and found small differences in the absolute value of the specific optical rotations in birefringent directions [100] and [010]. At the same time, it can be noted that the absolute values of the systematic errors of the polarimeter are very small compared with the average eigen waves ellipticity in crystals. Since optical activity is a phenomenon of spatial dispersion, i.e. it depends mainly on structural properties, this fact, probably, explains the relatively weak influence of L-arginine additive on the OA of KDP crystal in the visible light spectrum region.

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Considering the improvement of the nonlinear optical characteristics we have also investigated the effect of organic material L-arginine on the electro-optical properties of KDP single crystal. The electro-optical parameters (i.e. half-wave voltage and also electro-optic coefficient r_{63}) show very small change in doped crystals in comparison with the pure KDP crystal. This property is a subject for a more thorough study, because the symmetry of the third rank tensor, which describes the linear electro-optic effect, is the same as the nonlinear susceptibility tensor for the effect of SHG. We can assume that in static electric fields the optical nonlinearity is not manifested as much as on the light waves frequencies. One should also note, that in static electric field we observe total effect that consists of primary and secondary effects induced by piezoelectric deformation. Theoretical study of the linear electro-optic effect in KDP type crystals has been presented in [33] and indicates the dominant role of the P–O bonds in the, essentially, ionic electro-optic effect in KDP, in which electronic contribution in the electro-optic coefficient does not exceed 4 %. The L-arginine amino acid contains the amino group with a strong electron acceptor property, which may basically influence the electron density distribution and, consequently, the SHG efficiency in comparison with that of pure KDP. However, this does not greatly change electro-optic effect which is primarily associated with the P–O bonds, which in turn do not contribute to the optical nonlinearity at the second harmonic frequency. Although the linear electro-optic (Pockels) effect can be considered as nonlinear optical phenomenon (such the SHG) in which the intensity of one of the interacting waves is small and the frequency of the other tends to zero, it should be mentioned here that geometry of compared effects is different,

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i.e. directions of the light propagation, external static field and nonlinear polarization.

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