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## DIELECTRIC PROPERTIES OF (R)-3-QUINUCLIDINOL IN THE POROUS MATRIX OF ALUMINUM OXIDE

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The paper presents findings of an investigation of the linear and nonlinear dielectric properties of (R)-3-quinuclidinol embedded in porous aluminum oxide (pores of size 300 nm), in comparison with the properties of bulk (R)-3-quinuclidinol. A decrease in the Curie temperature in the nanocomposite, both upon heating and cooling, in comparison with a bulk sample is revealed. A decrease in the phase transition temperature allows for interpretation on the basis of the known theoretical models for ferroelectric small particles.

**Keywords:** (R)-3-quinuclidinol, aluminum oxide, ferroelectric, dielectric constant, nanocomposite, phase transition

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## ДИЭЛЕКТРИЧЕСКИЕ СВОЙСТВА (R)-3-ХИНУКЛИДИНОЛА В ПОРИСТОЙ МАТРИЦЕ ОКСИДА АЛЮМИНИЯ

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Представлены результаты исследований линейных и нелинейных диэлектрических свойств (R)-3-хинуклидинола, внедренного в пористый оксид алюминия (размер пор – 300 нм), в сравнении со свойствами объемного (R)-3-хинуклидинола. Выявлено понижение температуры Кюри в нанокompозите как при нагреве, так и охлаждении, по сравнению с объемным образцом. Понижение температуры фазового перехода допускает интерпретацию на основе известных теоретических моделей для сегнетоэлектрических малых частиц.

**Ключевые слова:** (R)-3-хинуклидинол, оксид алюминия, сегнетоэлектрик, диэлектрическая проницаемость, нанокompозит, фазовый переход

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## Introduction

Ferroelectric materials have unique properties and wide application in practice. Spontaneous polarization and high permittivity, as well as their dependence on exposure (electric fields, mechanical stress, etc.) makes ferroelectrics a popular solution for the development of various functional electronic devices. Recently, there is a constant search for polar materials with high permittivity  $\epsilon'$ , spontaneous polarization  $P_s$  and Curie temperature  $T_C$ ; moreover, such materials are cheap and environmentally friendly due to the absence of heavy metals in their structure.

Recently, researchers found ferroelectric properties of organic salts  $C_6H_{16}NHal$ , where halogens include Cl, Br and I [1–3]: diisopropylammonium chloride (DIPAC) with Curie temperature of 440 K and spontaneous polarization of  $8.2 \mu C/cm^2$ ; diisopropylammonium bromide (DIPAB) with Curie temperature of 426 K and spontaneous polarization of  $23 \mu C/cm^2$ ; diisopropylammonium iodide (DIPAI) with Curie temperature of 378 K and spontaneous polarization of  $5.17 \mu C/cm^2$ .

Thanks to promising practical application of organic ferroelectrics in nanoelectronics, there is a considerable interest towards the studies of the molecular size influence on the material properties. Ferroelectric phase transitions in the nanocomposites obtained based on DIPAC, DIPAB and DIPAI, and nanoporous matrices were studied in papers [4–7].

Papers [8, 9] reported a discovery of ferroelectric properties of homochiral organic crystals of (R)-3- and (S)-3-quinuclidinol ( $C_7H_{13}NO$ ). These crystals exist in two mirror-isometric (enantiomorphic) forms: homochiral (R)-3- and (S)-3-quinuclidinols. At room temperature, they crystallize in enantiomorphically polar point group 6 ( $C_6$ ) demonstrating a mirror image in the vibrational spectra. The Curie temperature determined using the differential thermal analysis method (DTA) for the single-crystalline samples was  $T_{C1} \approx 398$  K upon heating and  $T_{C2} \approx 360$  K upon cooling [8]. Dielectric permittivity at the phase transition has a dramatic jump anomaly changing approximately from 5 up to 17. Spontaneous polarization at  $T \approx 300$  K

equals approximately  $7 \mu C/cm^2$ , coercive field is 15 kV/cm. It was also discovered that their racemate (Rac)-3-quinuclidinol is crystallizing in a centrosymmetric point group  $2/m$  ( $C2h$ ) which is not ferroelectric.

This discovery shows great significance homochirality plays in occurrence of the ferroelectric state in organic ferroelectrics. As it was found in paper [9], the phase transition temperatures decrease down to  $T_{C1} \approx 338$  K upon heating and  $T_{C2} \approx 324$  K upon cooling for (R)-3-quinuclidinol substrate-supported films (150 nm thick).

This article presents the results of studying dielectric properties of (R)-3-quinuclidinol embedded in porous aluminum oxide  $Al_2O_3$  with 300 nm pores. For a comparison, we also studied similar identical properties in comparison with the properties of bulk polycrystalline (R)-3-quinuclidinol.

## Samples and experiment procedure

To obtain the nanocomposites, we used the (R)-3-quinuclidinol produced by the Acros Organics company (Belgium). According to the sample certificate, the phase temperatures amounted to  $T_{C1} \approx 390$  K upon heating and  $T_{C2} \approx 364$  K upon cooling. The samples for the study were represented by aluminum oxide films 50  $\mu m$  thick with the pores 300 nm in diameter. Fig. 1 shows the photographs of the films obtained using a scanning electron microscope. To fill the aluminum oxide films with the ferroelectric, a saturated (R)-3-quinuclidinol solution in methanol was used. The oxide sample was placed in the solution heated up to 320 K and then cooled down slowly. The remaining methanol was removed by means of vacuum drying. After three repetitions of the described procedure, the pore occupancy measured by the change in the films weight using AND BM-252G balances (accuracy of  $10^{-6}$  g) amounted to 53–55%.

Dielectric properties of bulk and nanostructured (R)-3-quinuclidinol were measured at the frequency of 100 kHz and operating voltage of 0.7 V using E7-25 LCR meter. To electrode the sample surface, we used Gallium–Indium paste. The temperature was determined with the accuracy of 0.1 K by means of TC-6621 electronic

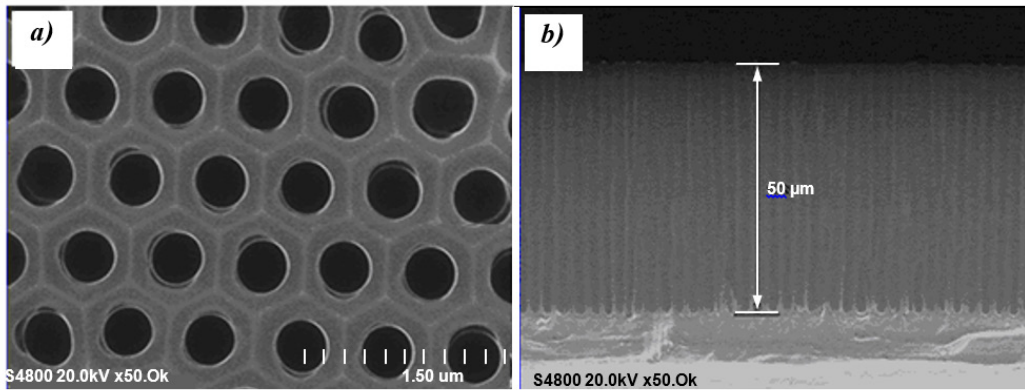


Fig. 1. Photomicrograph of  $\text{Al}_2\text{O}_3$  film: *a* – surface, *b* – end view

thermometer based on a chromel–alumel thermocouple. In the course of the measurements, the samples were heated from 300 to 440 K, and then cooled down. The rate of temperature change equaled 1 DPM (degrees per minute).

To study the non-linear properties, we applied 2 kHz frequency sine-curve voltage to the sample with a series resistor. To determine the amplitude of the multiple frequency signals, we applied sine-curve voltage with the frequency of several kilohertz and the field intensity of approximately  $10^2$  V/mm to the sample with a series resistor. To determine the ferroelectric domain, we used third harmonic coefficient ( $\gamma_{3\omega} = U_{3\omega}/U_{\omega}$ ). Papers [10, 11] describe the methodology of investigating ferroelectrics by means of non-linear dielectric spectroscopy in more detail.

### Experimental results and discussion

As a result of the studying dielectric characteristics of the considered (R)-3-quinuclidinol samples in bulk and (R)-3-quinuclidinol embedded in an aluminum oxide film, we analyzed temperature dependences  $\varepsilon'(T)$  (Fig. 2). The ferroelectric-to-paraelectric phase transition occurs at the temperature of 390 K which corresponds to the maximum of permittivity on the temperature dependence  $\varepsilon'(T)$ . For the bulk sample, when the temperature increases, there is an anomaly  $\varepsilon'$  at 390 K which corresponds to the transition from the ferroelectric phase to the paraelectric one. Upon cooling, the phase transition temperature depends on the temper-

ature up to which the sample was heated to, as well as the cooling rate. If a sample was heated up to 420 K with the cooling rate 1 K/min, it is equal to 372 K. For effective permittivity of the (R)-3-quinuclidinol/ $\text{Al}_2\text{O}_3$  nanocomposite (measured in the same conditions), the anomalies in the neighborhood of the phase transitions are very diffuse and shifted in the lower temperatures domain. As a comparison of the results for the bulk and nanostructured (R)-3-quinuclidinol shows, for the (R)-3-quinuclidinol in the pores of the  $\text{Al}_2\text{O}_3$  films, the transition temperature decreases by 10 K upon heating and by 25 K upon cooling.

At the next stage of the research, in order to determine the temperature interval of the ferroelectric phase in the nano-sized (R)-3-quinuclidinol with more accuracy, we measured the non-linear dielectric characteristics of the bulk and nanostructured (R)-3-quinuclidinol. The phase transition temperatures were determined by temperature dependences of the third harmonic coefficient  $\gamma_{3\omega}$  in the heating-cooling cycle (Fig. 3). In the course of heating, both samples have great values of the  $\gamma_{3\omega}$  coefficient starting from room temperature and up to 391 K (for the bulk sample) and 380 K (for the nanocomposite). Above this temperatures, the third harmonic coefficient changes insignificantly which can be attributed to the transition of the samples into the paraelectric state. Upon cooling, the  $\gamma_{3\omega}$  coefficient begins to grow at around 372 and 347 K for the bulk and the nanocomposite (R)-3-quinuclidinol respectively.

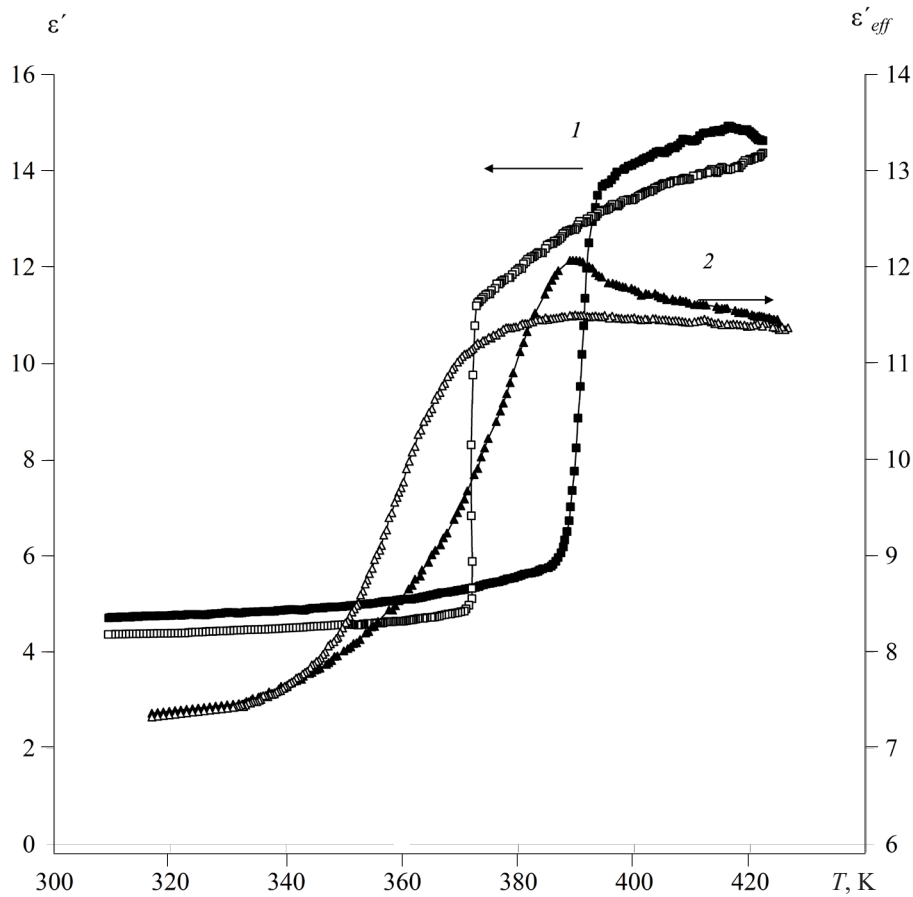


Fig. 2. Dependence  $\varepsilon'(T)$  for bulk (1) and  $\text{Al}_2\text{O}_3$  nanocomposite (2) (R)-3-quinuclidinol samples at the frequency of 100 kHz (black symbols for heating, white symbols for cooling)

A change in the Curie temperature for ferroelectrics located in nanoporous matrices can occur due to several factors. It is primarily connected with size effects observed for isolated nanoparticles. As the size of the particles decreases, the share of surface atoms grows. Free energy  $F$  of nanoparticles is a sum of the volume ( $F_V$ ) and surface ( $F_S$ ) contributions:

$$F = F_V + F_S.$$

The decrease of the phase transition temperature of the (R)-3-quinuclidinol embedded in the pores of aluminum oxide agrees with the conclusion of the theoretical models developed on the basis of the phenomenological Landau theory and Ising model [12–14]. These models predict that the temperature of the structural phase transition for small isolated particles of spherical and

cylindrical shape shifts deeper into the ferroelectric phase as the size of the particles decreases. The conclusions of these models were also experimentally verified for separate small particles of such ferroelectrics as barium titanate (see paper [15] and its references).

For matrix nanocomposites, unlike for separate ferroelectric particles, we need to account for the interaction between the matrix inclusions. In this case, the change in the surface energy is determined as

$$\tilde{F}_S = F_S + \sum_{S_i} \int \sigma_i dS_i + \sum_{S_i} \int \varphi_i \delta_i dS_i,$$

where  $\sigma_i$  is the surface tension;  $S_i$  is the particle surface;  $\varphi_i$  is the electric potential;  $\delta_i$  is the surface-charge density.

The summand  $\sigma dS$  may considerably contribute to the total energy of the system with

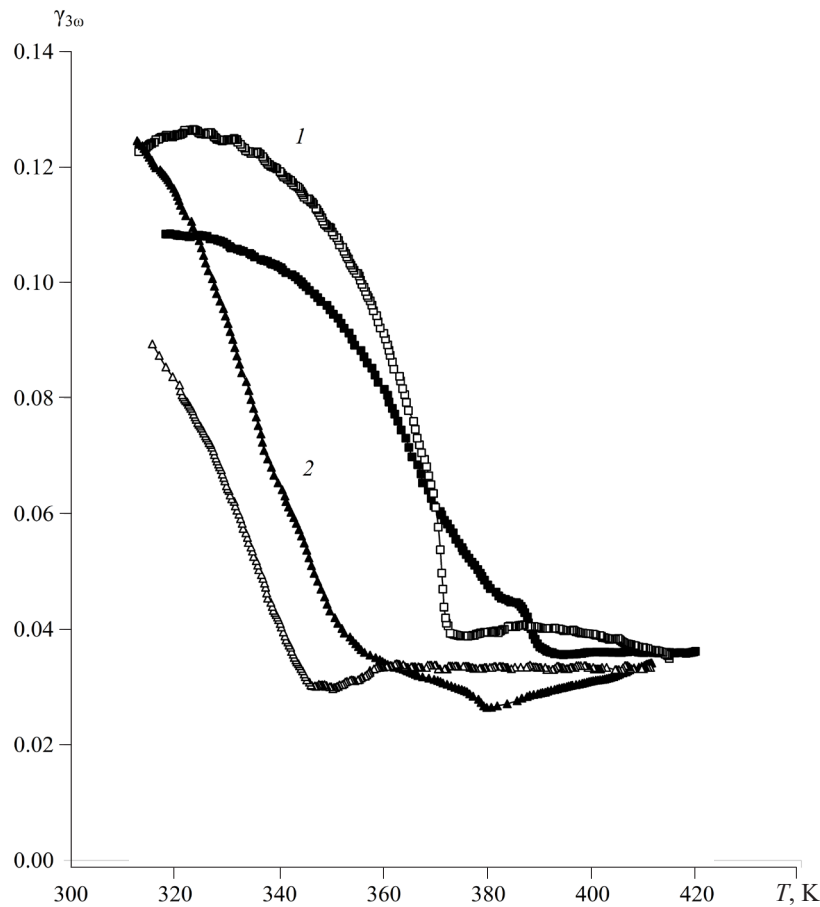


Fig. 3. Temperature dependences of the third harmonic coefficient for bulk (1) and  $\text{Al}_2\text{O}_3$  nanocomposite (2) (R)-3-quinuclidinol samples in the heating-cooling cycles (black symbols for heating, white symbols for cooling)

well developed surface of the phase interface. Formation of a double electric layer at the phase interface as a result electron emission or spontaneous polarization screening leads to emergence of surface conductance and Maxwell-Wagner polarization. The depolarization field depending on permittivity, conductance, shape and size of the particles additionally contributes into the size effect which results in decreased Curie temperature.

Moreover, paper [16] indicated that electric interaction between ferroelectric particles in neighboring pores could influence the phase transition shift in the matrix nanocomposites. However, in our case, the electric interaction between the particles in neighboring pores plays no significant role due to the small value of the spontaneous polarization of (R)-3-quinuclidi-

nol ( $P_s \approx 7 \mu\text{C}/\text{cm}^2$ ) and a considerable distance between the neighboring pores (around 200 nm). Taking mechanical stress for the nanoparticles into account in the measurements is essential in terms of retaining polar properties of the ferroelectric. In this case, pressure under the curved surface is defined by the surface tension tensor  $\mu$ . The dependence of the polar properties of ferroelectric particles on surface tension was evaluated in paper [17–19]. Thus, paper [19] shows that at  $\mu = 0.5 - 50 \text{ N/m}$ , the effect of the transition temperature shift due to electrostriction begins working at the nanoparticle radius of curve  $R = 5 - 50 \text{ nm}$ , which is significantly less than the size of the pores in the composite of interest.

Therefore, a decrease in the ferroelectric phase transition temperature of (R)-3-quinuclidinol in porous matrices of aluminum oxide discovered in

this article is due to the influence of size effects characteristic of free particles.

### Conclusion

The results of the study of (R)-3-quinuclidinol embedded in porous aluminum oxide presented in this article revealed a decrease in the Curie temperature in the nanocomposite, both

upon heating and cooling, in comparison with the bulk sample. A decrease in the phase transition temperature allows interpretation based on the known theoretical models for ferroelectric small particles.

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