

Original article

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DIELECTRIC AND THERMAL PROPERTIES OF NANOCOMPOSITES BASED ON DIISOPROPYLAMMONIUM IODIDE AND ALUMINA NANOPARTICLES

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Abstract. In the paper, nanocomposites based on diisopropylammonium iodide (DIPAI) and Al₂O₃ nanoparticles (sized 100 nm) have been synthesized with varying a volume fraction of the latter (0.05, 0.10, 0.20). Temperature dependences of the permittivity and a signal of differential thermal analysis of the samples (in the range of 300 – 400 K) were studied in heating and cooling modes. Their infrared spectra were recorded at room temperature as well. An analysis of the measured temperature dependences indicated a change in the sequence of phase transitions in nanocomposites compared with the pure DIPAI. The totality of the results obtained allowed us to conclude that a change in hydrogen bonds with the participation of amino groups manifested itself in the nanocomposite structure took place. This can lead to the appearance of a ferroelectric state in the DIPAI.

Keywords: ferroelectric, permittivity, nanocomposite, phase transition, DIPAI, alumina

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ДИЭЛЕКТРИЧЕСКИЕ И ТЕПЛОВЫЕ СВОЙСТВА НАНОКОМПОЗИТОВ НА ОСНОВЕ ИОДИДА ДИИЗОПРОПИЛАММОНИЯ И НАНОЧАСТИЦ ОКСИДА АЛЮМИНИЯ

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Аннотация. В работе синтезированы нанокomпозиты на основе иодида диизопропиламмония (DIPAI) и наночастиц Al₂O₃ (размер наночастиц 100 нм) с варьированием объемной доли последнего (0,05; 0,10; 0,20). Исследованы температурные зависимости диэлектрической проницаемости и сигнала дифференциального термического анализа синтезированных соединений (в интервале 300 – 400 К) в режимах нагрева и охлаждения. Зарегистрированы также их инфракрасные спектры при

комнатной температуре. Анализ измеренных температурных зависимостей указывает на изменение последовательности фазовых переходов в композитах по сравнению с чистым DIPAI. Совокупность полученных результатов позволяет сделать вывод, что в структуре нанокompозита проявляется изменение водородных связей с участием аминогрупп, что может приводить к появлению сегнетоэлектрического состояния в DIPAI.

Ключевые слова: сегнетоэлектрик, диэлектрическая проницаемость, нанокompозит, фазовый переход, DIPAI, оксид алюминия

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Introduction

Ferroelectrics whose spontaneous polarization P_s can be switched by an external electric field have been studied for over 100 years [1]. In this time, thousands of inorganic and organic materials with spontaneous polarization have been discovered. However, inorganic oxide perovskites have mainly been used in practice, due to their high stability, high permittivity and spontaneous polarization. Despite this, there is an extensive search for organic ferroelectrics, due to their low cost, easy preparation, environmental friendliness and biocompatibility. Successful cases include croconic acid [2], diisopropylammonium chloride and bromide [3,4]. These substances compete with inorganic perovskite ferroelectrics in terms of spontaneous polarization and Curie temperature. There are contradictor reports in the literature on diisopropylammonium iodide (DIPAI), which is the third representative of ferroelectrics based on diisopropylamine [5, 6]. The spontaneous polarization of DIPAI crystals grown in [5] was approximately $33 \mu\text{C}\cdot\text{cm}^{-2}$. However, a polar state was not detected in the crystals synthesized in [6]. It was established in [7] that DIPAI must be heated above a temperature of 423 K to be converted to a ferroelectric state.

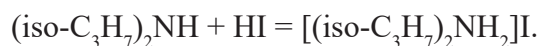
One of the well-known techniques for modifying the properties of ferroelectric materials is to create composites based on them [8]. Since DIPAI is an organic ferroelectric with a complex molecular structure, adding aluminum oxide (Al_2O_3) nanoparticles to it can lead to interaction at the molecular level, contributing to polarization pinning of ferroelectric molecules (this has been confirmed, for example, for triglycine sulfate [9]).

This paper presents the findings of a study on the dielectric and thermal properties of $(\text{DIPAI})_{1-x}/(\text{Al}_2\text{O}_3)_x$ nanocomposites obtained by mixing DIPAI powder with 100 nm alumina nanoparticles. A comparison with bulk DIPAI samples was also carried out.

Samples and experimental procedure

DIPAI crystals have orthorhombic nonpolar symmetry with the space group $P2_12_12_1$ [5, 6]. One or two phase transitions occur in DIPAI heated above room temperatures (depending on the production technology and thermal history). Reporting on ferroelectricity in DIPAI, Ravi et al. [5] established that a ferroelectric monoclinic phase with $P2_1$ symmetry is formed in DIPAI crystals heated to 369 K, remaining stable up to 415 K. The DIPAI structure transforms into a nonpolar monoclinic phase $P2_1/m$ above this temperature. A transition to a monoclinic ferroelectric phase with $P2_1$ symmetry is observed in the region of about 407 K upon further cooling from a temperature above 415 K. The authors of another paper [6] did not detect a ferroelectric phase in the DIPAI crystals they obtained. A transition from the orthorhombic phase $P2_12_12_1$ to the monoclinic phase $P2_1/m$ was observed in DIPAI crystals at 376 K.

In our experiment, DIPAI (diisopropylammonium iodide[(iso- C_3H_7) $_2\text{NH}_2$]I) was prepared based on the interaction of equimolar amounts of diisopropylamine (iso- C_3H_7) $_2\text{NH}$ (Sigma) and hydrochloric acid (57%, AcrosOrganics) in an aqueous solution [8]. In general, the reaction proceeds by the following equation:



The synthesis started with dissolving 0.1 mol of hydroiodide (HI) in 10 ml of distilled water. Next, 0.1 mol of diisopropylamine was slowly added to the resulting solution under cooling. After the solvent was evaporated at room temperature, large colorless transparent needle-like crystals were obtained, which were recrystallized from hot ethanol. The crystals were washed with diethyl ether and dried in the dark in a desiccator over calcium chloride. The yield was 68%.

Samples of $(\text{DIPAI})_{1-x}/(\text{Al}_2\text{O}_3)_x$ composites ($x = 0.05; 0.10; 0.20$; x are the volume fractions) were obtained using DIPAI ($\sim 10 \mu\text{m}$) and Al_2O_3 powders with a particle size of 100 nm. The composites were prepared by thoroughly mixing these powders in an agate mortar. To study the dielectric properties, the powder was pressed into disks measuring $\varnothing 10 \times 1.5$ mm under a pressure of 7500 kg/cm^2 .

An E7-25 meter with the frequency range from 20 to 10^6 Hz was used to measure the dielectric properties (permittivity ϵ'). Silver paste was applied to the sample to make the electrodes before the measurements. The temperature was recorded with an accuracy of 0.1 K using a TS-6621 thermometer. Measurements were carried out in the temperature range from 300 to 440 K, in automatic mode at a rate of 1 K/min. The error in determining the permittivity of the studied samples did not exceed 5%. Temperature dependences of the DTA signal were measured with an STA PT 1600 simultaneous thermal analyzer (Linseis, USA) with the maximum resolution of 0.3 MW. The heating and cooling rate of the samples in our experiment was 1 K/min.

Infrared (IR) spectra for samples of DIPAI, Al_2O_3 and $(\text{DIPAI})_{0.80}/(\text{Al}_2\text{O}_3)_{0.20}$ composite pressed into pellets with KBr were recorded with an FSM-2202 FTIR spectrometer (OKB Spectr, Russia) in the range of $400\text{--}4000 \text{ cm}^{-1}$. The FSpec program (version 4.0.0.2 for Windows, Monitoring LLC, Russia) was used to control the device and process the spectra.

Experimental results and discussion

Judging from the temperature dependences of ϵ' and the DTA signal, the DIPAI sample we obtained exhibits one phase transition under heating and one under cooling (Fig. 1). This allows to conclude that the ferroelectric phase does not appear, and the transition occurs between two paraelectric phases at temperatures of 376 and 360 K under heating and cooling. The values of the transition temperature are consistent with the data in [7]. The temperature dependences of the dielectric loss tangent $\text{tg}\delta$ have minima at 381 and 361 K in heating and cooling modes, respectively. The quantities $\epsilon'(\omega)$ and $\text{tg}\delta(\omega)$ decrease with increasing frequency, which is consistent with the data obtained for single crystals [6]. Further studies found that the thermal history does not change the sequence of phase transitions in DIPAI, unlike the samples obtained in [7].

The measurements of permittivity and DTA signal for $(\text{DIPAI})_{0.95}/(\text{Al}_2\text{O}_3)_{0.05}$ composites also point to the presence of two phase transitions (Fig. 2). The maximum of the DTA signal is observed at a temperature of 360 K under heating, and at about 358 K under cooling.

As the content of Al_2O_3 nanoparticles is increased to 10%, the data for the DTA signal and the permittivity $\epsilon'(T)$ indicate two phase transitions in heating mode: at 362 and 376 K (Fig. 3). This corresponds to the formation of a ferroelectric phase, according to the data from [7]. One phase transition is observed in cooling mode at 358 K.

Two phase transitions are also observed under heating in the $(\text{DIPAI})_{0.80}/(\text{Al}_2\text{O}_3)_{0.20}$ composite, at about 363 and 378 K; one phase transition is observed under cooling, at about 358 K (Fig. 4).

Alumina is a linear dielectric, and Al_2O_3 particles do not have dipole moments that could induce the ferroelectric phase in DIPAI. Therefore, the only possible mechanism of interaction between DIPAI and Al_2O_3 nanoparticles is their interaction at the molecular level. To verify this assumption, we obtained IR spectra of pure DIPAI and $(\text{DIPAI})_{0.80}/(\text{Al}_2\text{O}_3)_{0.20}$ composite.

The supramolecular structure of diisopropylammonium halides is due to the $\text{N-H}\cdots\text{Hlg}$ hydrogen bonds, where $\text{Hlg} = \text{Cl}, \text{Br}, \text{I}$. The distance $\text{H}\cdots\text{Hlg}$ in the series of halide ions $\text{Cl}^-, \text{Br}^-, \text{I}^-$ increases with a corresponding increase in the atomic radius of halogen. The iodide ions I^- in the DIPAI crystal, serving as bridges, combine diisopropylammonium cations. The phase transition observed under heating in DIPAI crystals is accompanied by their complex structural rearrangement, which is caused by the formation of optical isomers by DIPA cations, their regrouping in the crystal, and transformation in the chain packing. The strength of $\text{N-H}\cdots\text{I}$ hydrogen bonds is significantly different in the two phases.

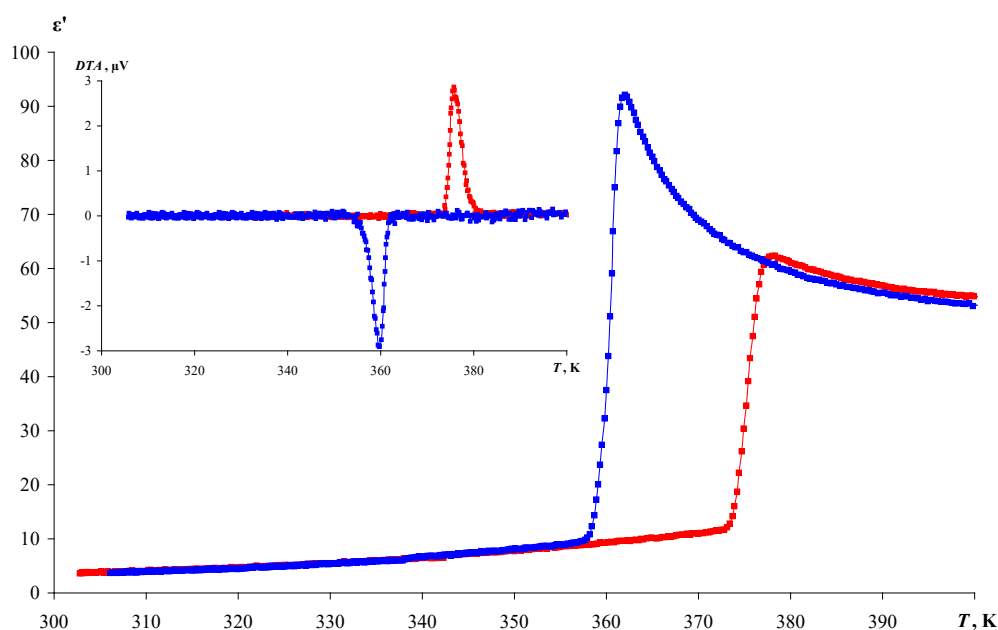


Fig. 1. Temperature dependences of permittivity ϵ' and DTA signal (in the inset) obtained at a frequency of 10 kHz for the DIPAI sample under heating (red symbols) and cooling (blue)

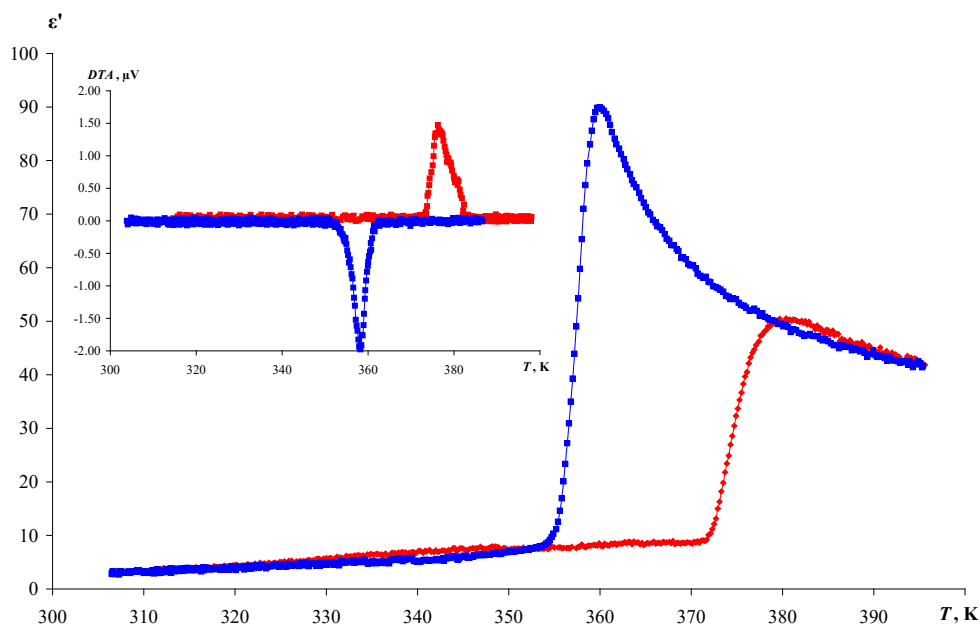


Fig. 2. Temperature dependences of permittivity ϵ' and DTA signal (in the inset) obtained at a frequency of 10 kHz for the $(\text{DIPAI})_{0.95}/(\text{Al}_2\text{O}_3)_{0.05}$ sample under heating (red symbols) and cooling (blue symbols)

The N–H...I distances in the DIPAI crystal of monoclinic syngony (room temperature) are 2.81 E (across the chains) and 2.67 E (along the chains), amounting to 3.03, and 2.82 E in orthorhombic syngony (405 K) [9, 6]. The hydrogen bonds of N–H...I are much longer and consequently more labile in DIPAI than those in chloride and bromide.

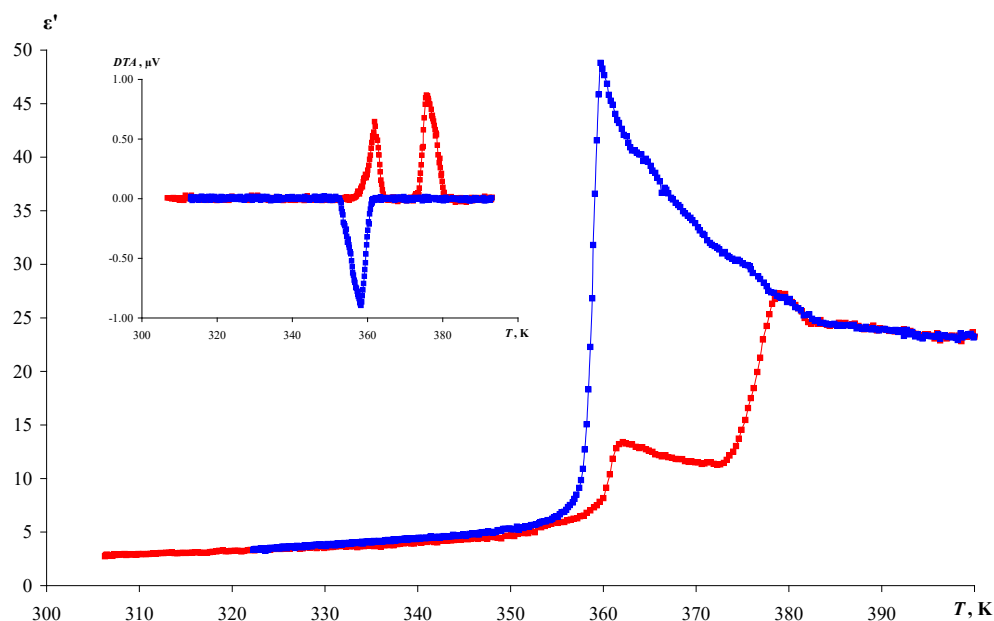


Fig. 3. Temperature dependences of permittivity ϵ' and DTA signal (in the inset) obtained at a frequency of 10 kHz for the $(\text{DIPAI})_{0.90}/(\text{Al}_2\text{O}_3)_{0.10}$ sample under heating (red symbols) and cooling (blue symbols)

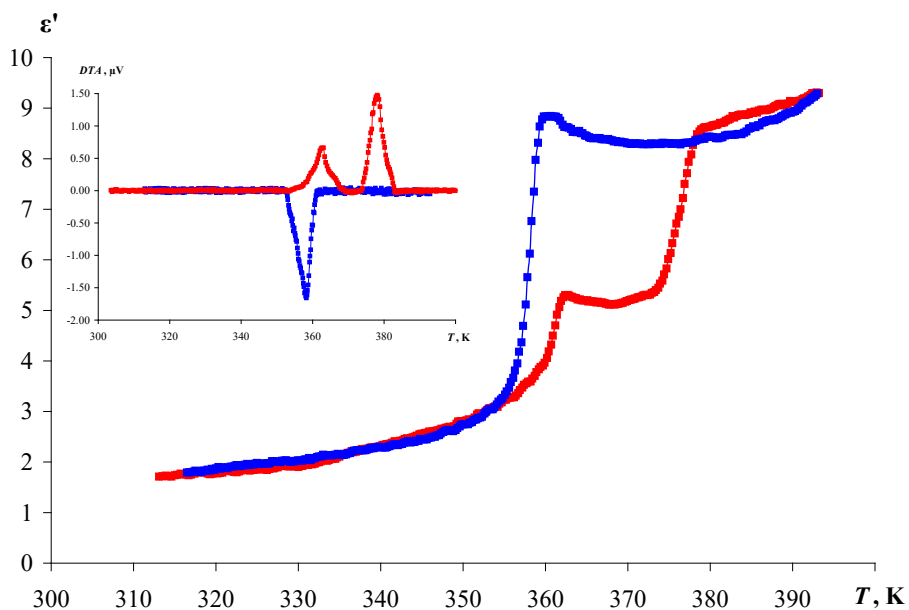


Fig. 4. Temperature dependences of permittivity ϵ' and DTA signal (in the inset) obtained at a frequency of 10 kHz for the $(\text{DIPAI})_{0.80}/(\text{Al}_2\text{O}_3)_{0.20}$ sample under heating (red symbols) and cooling (blue symbols)

We identified the absorption bands in the IR spectra of the compounds in accordance with the data in [10, 11]. The symmetric and asymmetric stretching vibrations of C–H bonds of the structural fragment $\text{C}(\text{CH}_3)$ in the IR spectrum of DIPAI correspond to absorption bands at 2810.3 , 2833.3 and 2967.7 cm^{-1} , symmetric and asymmetric bending vibrations of the C–H bonds correspond to absorption bands at 1379.6 and 1469.6 cm^{-1} . Stretching vibrations of N–H bonds appear as a group of absorption bands at

3296.8, 3385.0, 3410.4, 3424.4, 3438.8, 3447.8 cm^{-1} .

The bands at 1573.9 cm^{-1} (1579.1 cm^{-1} , shoulder) and 1325.4 cm^{-1} are caused by bending vibrations of N–H bonds. An absorption band appears in the IR spectra of the DIPAI- Al_2O_3 and DIPAI samples in the region of symmetric stretching vibrations of the CNC fragment with a maximum at 1182.7 cm^{-1} .

The lability of hydrogen bonds in the DIPAI structure contributes to their rearrangement and the formation of new bonds in the $(\text{DIPAI})_{0.80}/(\text{Al}_2\text{O}_3)_{0.20}$ composite. The absorption band at $3770\text{--}3730 \text{ cm}^{-1}$ in the IR spectrum of aluminum oxide (Fig. 5,*a*) corresponds to the Lewis basic site [12]. Considerable smoothing and disappearance of the absorption band at 3760.0 cm^{-1} in this region of the spectrum may be evidence for the formation of hydrogen bonds N–H \cdots O–Al between hydrogen atoms of NH_2 groups and coordinatively unsaturated oxygen atoms, which are the basic sites, in the DIPAI– Al_2O_3 system. The presence of Lewis acid sites in aluminum oxide, i.e., aluminum atoms, suggests that they may participate in (Al \cdots I) interactions leading to a significant decrease in the intensity of absorption bands at 3566.8 and 3588.1 cm^{-1} . Interactions of this type are discussed in [13, 14].

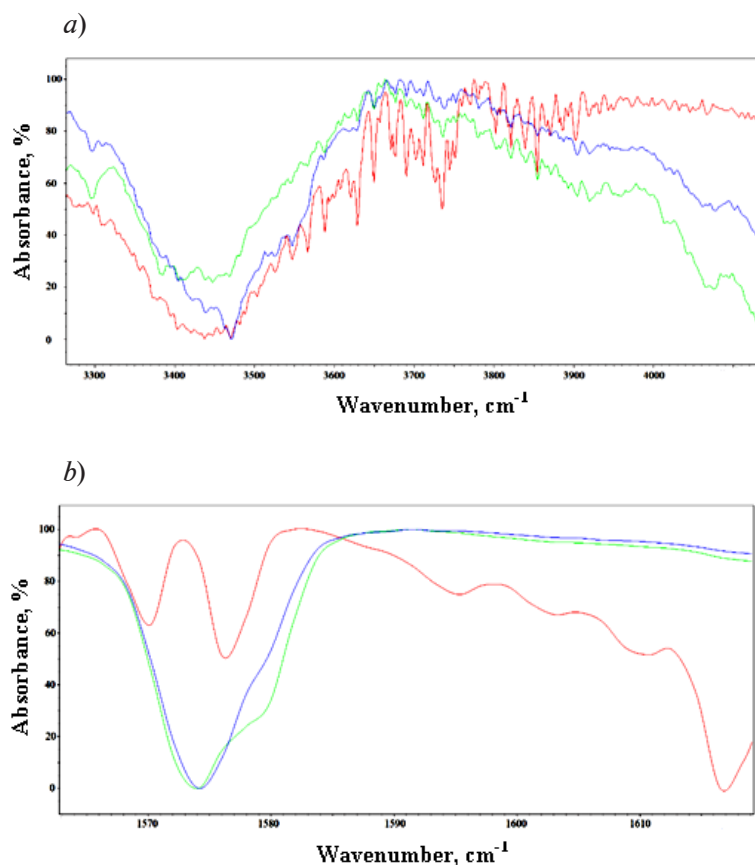


Fig. 5. IR absorption spectra for Al_2O_3 (red curve), DIPAI (green curve) and $(\text{DIPAI})_{0.80}/(\text{Al}_2\text{O}_3)_{0.20}$ composite (blue curve) samples in the wavenumber ranges of $4000\text{--}3100 \text{ cm}^{-1}$ (a) and $1610\text{--}1560 \text{ cm}^{-1}$ (b)

The frequencies of stretching vibrations of NH_2 – and *i*-Pr-groups depend on the degree of their participation in the hydrogen bond. The formation of hydrogen bonds is manifested by a shift of the bands corresponding to $\nu(\text{N–H})$ and $\nu(\text{C–H})$ towards low frequencies [10, 11]. The absorption band of N–H stretching vibrations (3424.4 cm^{-1}) in the DIPAI– Al_2O_3 spectrum is shifted to the low-frequency region of the spectrum (3422.5 cm^{-1}). In addition, the absorption band of bending vibrations (1573.9 cm^{-1}) is shifted and appears at 1574.3 cm^{-1} , the shoulder at 1579.1 cm^{-1} is not pronounced, which indicates the suppression of bending vibrations of N–H



bonds (Fig. 5,*b*). This confirms the assumption about the strengthening of hydrogen bonds that the amino groups participate in forming. At the same time, there is no shift in the absorption band of stretching and bending vibrations of (C–H)-bonds of *i*-Pr groups.

Considering the aggregate results obtained, we can conclude that a transformation in hydrogen bonds with the participation of amino groups can be observed in the structure of the DIPAI–Al₂O₃ sample, potentially leading to the appearance of a ferroelectric state in DIPAI.

Conclusion

As established in [6], the phase transition in DIPAI crystals is due to rearrangement of the cationic substructure, where *i*-(C₃H₇)₂NH₂⁺ are reoriented by about 90° along the long axis and rotate along the orthorhombic direction [100]. These ions are simultaneously split into two equivalent positions related to each other by a mirror plane. As a result, the dipole moments of the *i*-(C₃H₇)₂NH₂⁺ ion are oriented along one of the metastable directions. This process changes the packing of the chains and causes deformation of the anionic structure.

DIPAI and DIPAB are isostructured at room temperature and have a similar crystal packing. Larger ions cause an increase in interatomic distances and an increase in the volume of the unit cell, which, in turn, affects the strength of interactions between the hydrogen bonds. An increase in all distances from the donor to the acceptor is observed along with the increase in ion radii. A phase transition to the disordered state *P*2₁/*m* occurs in DIPAI, and the phase is shifted by about 50 K towards lower temperatures, compared with DIPAB; this indicates significantly weaker N–H–I bonds, compared with N–H–Br bonds.

Analyzing the overall results obtained, we can conclude that the transformation in the hydrogen bonds of N–H–I with the participation of amino groups within the structure of the DIPAI–Al₂O₃ sample can stabilize the ferroelectric state in the DIPAI compound.

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