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# DIELECTRIC RELAXATION AND MOLECULAR INTERACTIONS IN ACETIC ACID

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This article presents the results of measurements of the dielectric coefficients of acetic acid and its solutions. Measurements were carried out at wavelength $\lambda$ = 40.0; 30.0; 20.0; 10.0; 6.4; 4.4; 2.1; 1.2; and in the temperature range 20 ÷ 50°C. Models of molecular clustering in liquid acetic acid were studied based on analysis of the dielectric absorption spectrum. The results of a study of the radio frequency absorption spectrum of acetic acid indicate the presence of two polymorphic forms of this compound. The characteristic temperature dependences of the dielectric constant $\varepsilon'$ of acetic acid have been determined, from which two isothermal rotational transitions are clearly visible. The first - at the melting point - is accompanied by a sharp decrease in the number of dielectrically active units. The second, at a temperature below the melting point, leads to dielectric constant $\varepsilon'$ values close to the high frequency (HF) limit of the total orientation contribution for microwave absorption in liquid acid. At temperatures above the first and below the second transitions, the values of the dielectric constant $\varepsilon'$ do not depend on the frequency of the electromagnetic wave; on the contrary, pronounced dispersion is observed in the interval between two transitions.

**Keywords:** *Dielectric spectroscopy; Dielectric relaxation; Complex dielectric constant; Dielectric properties; Acetic acid*
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## 1. INTRODUCTION

The main goal of dielectric research is to study the patterns of manifestation of the features of molecular interactions and movements in the condensed state of matter. Effective control of chemical and technological processes, revelation of the nature of physical and chemical processes in biological media, synthesis of new polymeric materials that meet specific requirements, creation of high-speed methods of analysis and control - none of these pressing problems can be successfully solved without a deep understanding of the mechanism of molecular movements and interactions.

The extreme diversity of the molecular structures of polar liquids and their solutions that must be taken into account, as well as the difficulties associated with theoretical inter predation, make it extremely necessary to systematically accumulate experimental data on molecular motion and interactions in connection with the microscopic properties of matter. In this aspect, dielectric information acquires special value due to the exceptionally high resolution of the method on a time scale, which makes it possible to observe the movements of atomic groups, individual molecules, and large supra molecular formations - clusters. This feature distinguishes the dielectric method from other widely used spectroscopy methods. However, its implementation is possible only when setting up an experiment within a wide range of frequency variations, which is associated with overcoming great methodological and technical difficulties.

Dielectric studies were carried out in parallel in two directions:

a) mastering the existing methodology, as well as the development and implementation of a new experimental methodology;

b) direct dielectric studies of substances with various types of interactions and molecular structure in liquid and solid states, under conditions of significant variation in external parameters.

The dynamic dielectric properties of acetic acid [1 – 3], in agreement with X-ray data [4,5], indicate the existence of highly polar clusters based on intermolecular hydrogen bonds, as well as kinetic units with a smaller effective dipole moment. In this work, changes in the spectrum of the dispersion caused by the melting of the acid were traced. Analysis of changes in the dispersion spectrum [6,7] shows that, to a first approximation, the spectrum can be described by a superposition of two regions. The HF region has a relaxation nature and is characterized by a symmetrical distribution of relaxation times, increasing with decreasing temperature. The small value of the absorption amplitude and the relatively low (for the solid phase) reorientation barrier (~4 kCal/mol) give grounds to assert that the rotational movement of individual molecules creates the high-frequency region.

Low frequency (LF) - the region only partially falls within the overlapped frequency range. A notable feature of this area is the strong dependence of dielectric coefficients on the thermal conditions of sample preparation, which requires special precautions to eliminate or minimize the contribution due to the surface effect. Judging by the long relaxation times and large amplitude, comparable to that in the liquid phase, the main contribution to the LF region is made by the movements of molecules forming hydrogen-bonded multiverse, without the disintegration of the latter.

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The purpose of the work was to study the radio frequency absorption spectrum of acetic acid. Determination of the temperature dependences of the dielectric constant ε' of acetic acid.

## 2. MATERIALS AND METHODS

Acetic acid (United States, Keysight Technologies) was chosen as the object of research. Dielectric constant ε' and dielectric losses ε" of liquid acetic acid were measured at wavelengths λ = 40.0; 30.0; 20.0; 10.0; 6.40; 4.40; 2.10; 1.20; 0.75 cm in the temperature range from 20 to 50°C according to the method described in [8 - 11], taking into account amendments [12,13].Equilibrium dielectric constants $\varepsilon_0$ were measured on a Q - meter VM 560, at a frequency of 1.6 MHz and the same temperatures. The errors are about 2% for the dielectric constant ε', 3 – 5% for the dielectric loss ε" and 0.5% for the equilibrium dielectric constant $\varepsilon_0$. Further details about the methodology and constants of the studied sample are given in [14, 15].

To measure the dielectric constant ε' and dielectric losses ε" of liquids and solutions in the range of centimeter and millimeter wavelengths, waveguide methods are widely used, characterized by a relatively high measurement accuracy. The expression for the complex propagation constant γ of an electromagnetic wave in a waveguide fill fused by a polar liquid, is written in the form [11, 15 – 17]:

$$\hat{\gamma} = i\,\frac{2\pi}{\lambda}\sqrt{\varepsilon^* - \left(\frac{\lambda}{\lambda_{\text{кр}}}\right)^2} = i\,\frac{2\pi}{\lambda_g}\left(1 - i\,tg\frac{\Delta}{2}\right). \tag{1}$$

Here$\Delta = arctg\frac{\varepsilon''}{\varepsilon' - \left(\frac{\lambda}{\lambda_{\text{кр}}}\right)^2}$; λ – wavelength in vacuum; $\lambda_{cw}$ – critical wavelength in a waveguide, determined by the dimensions of the waveguide and the type of waves propagating in the waveguide [18], $\lambda_g$ – wavelength in the waveguide with the liquid under study, $\varepsilon^*$ – complex dielectric constant of a polar liquid. From identity (1) it follows that

$$\varepsilon' = \left(\frac{\lambda}{\lambda_{\text{кр}}}\right)^2 + \left(\frac{\lambda}{\lambda_g}\right)^2 \cdot \left(1 - tg^2\frac{\Delta}{2}\right);\;\; \varepsilon'' = 2\left(\frac{\lambda}{\lambda_{\text{кр}}}\right)^2 tg\frac{\Delta}{2}. \tag{2}$$

To determine the quantities $\lambda_g$and $tg\frac{\Delta}{2}$ included in expression (2), the waveguide method of variable thicknesses is often used [19 – 21], which is acceptable only under the condition that$tg\frac{\Delta}{2} \neq 0$. According to this method, a waveguide cell with the liquid under study, the thickness of which in the cell is controlled by the position of the short-circuiting piston at the rear of the cell, is connected to the end of the waveguide path. The dependence of the standing wave coefficient in the waveguide path η on the thickness of the liquid in the cell l is removed. The minimum values η of this dependence are realized at liquid thicknesses in the cell approximately equal to $l = \frac{\lambda_g}{4}(2k - 1)$, where k = 1, 2, 3 … while the ratio of arbitrary maxima $\eta_m$ and $\eta_n$ of the same dependence η on l is related to the value of $tg\frac{\Delta}{2}$ by the relation, which to a first approximation has the form:

$$\frac{\eta_m}{\eta_n} = \frac{th\,(n\pi\, tg\frac{\Delta}{2})}{th\,(m\pi\, tg\frac{\Delta}{2})} \cdot (1 - c_{mn}), \tag{3}$$

where$c_{mn} = 4sin^2\frac{\Delta}{4}\,\left[\frac{1}{1 - \left(\frac{\lambda_g}{\lambda_\text{в}}\right)^2 \cdot cos^2\frac{\Delta}{2} \cdot th^2\left(n\pi\, tg\frac{\Delta}{2}\right)} - \frac{1}{1 - \left(\frac{\lambda_g}{\lambda_\text{в}}\right)^2 \cdot cos^2\frac{\Delta}{2} \cdot th^2\left(m\pi\, tg\frac{\Delta}{2}\right)}\right.,$

here$\lambda_в$ – wavelength in an empty waveguide [1].

The convenience of calculating $tg\frac{\Delta}{2}$using expression (3), the functions $\frac{th\,(n\pi\, tg\frac{\Delta}{2})}{th\,(m\pi\, tg\frac{\Delta}{2})}$are tabulated. It is usually assumed that $c_{mn}$ values are quite small in magnitude. However, as calculations show, this assumption is valid only for values of the indices m > 2 and n > 2. For other values of the indices m and n, the value of $c_m$ should be taken into account. When accurately determining the value of $\lambda_g$, a systematic error is allowed, which, as a rule, is not always taken into account. In some cases, it can exceed the permissible limits and significantly affect the accuracy of measuring the values of ε' and ε" of the liquids under study. To take into account the systematic error in determining the value of $\lambda_g$, we use the following considerations.

The expression for the standing wave coefficient in a waveguide has the following form [11]:

$$\eta = \frac{1+|\rho|}{1-|\rho|}. \tag{4}$$

Where$|\rho| = \left|\frac{th\hat{\gamma}l - \left(\frac{\lambda_\text{в}}{\lambda_g}\right)\cdot\left(1 - i\,tg\frac{\Delta}{2}\right)}{th\hat{\gamma}l + \left(\frac{\lambda_\text{в}}{\lambda_g}\right)\cdot\left(1 - i\,tg\frac{\Delta}{2}\right)}\right|$–modulus of the reflection coefficient of an electromagnetic wave in a waveguide.

The position of the extreme values of the dependence of η on l is determined from the condition.

$$\frac{d\eta}{dl} = \frac{2}{(1-|\rho|)^2} \cdot \frac{d|\rho|}{dl} = 0. \tag{5}$$

We assume that the minimum values of ρ, and therefore η, are realized at liquid thicknesses in the cell

$$l = \frac{\lambda_g}{4} \cdot (2q-1) + \delta, \tag{6}$$

where - δ is a value many times smaller than the thickness of the liquid in cell l.

We substitute expression (6) into (4) and expand the resulting expression for $th\hat{\gamma}l$ into a series in terms of the value of δ. Let us limit ourselves to considering the first three terms of this series. Let us differentiate the resulting equation (4) by the value of δ and use condition (5). Then, discarding terms containing $\delta^2$ as a factor, we obtain in a first approximation

$$\delta_{min} = \frac{\lambda_g}{2\pi} \cdot \frac{tg\frac{\Delta}{2}}{1+tg^2\frac{\Delta}{2}} \cdot \frac{1}{(\frac{\lambda_B}{\lambda_g})^2 \cdot \left(1+tg^2\frac{\Delta}{2}\right)-1} \cdot \frac{1}{th[\frac{\pi}{2}(2p-1)tg\frac{\Delta}{2}]}. \tag{7}$$

Taking into account the correction δ, the first minimum of the dependence of η on l will occur at the thickness of the liquid in the cell:

$$l_1 = \frac{\lambda_g}{4}(1+\delta_1), \tag{8}$$

where$\delta_1 = \frac{2}{\pi} \cdot \frac{tg\frac{\Delta}{2}}{1+tg^2\frac{\Delta}{2}} \cdot \frac{1}{(\frac{\lambda_B}{\lambda_g})^2 \cdot \left(1+tg^2\frac{\Delta}{2}\right)-1} \cdot \frac{1}{th[\frac{\pi}{2}tg\frac{\Delta}{2}]}$

In the general case, the distance between the nearby p and q minima of the dependence of η on l is determined by the relation:

$$l_q - l_p = \frac{\lambda_g}{2} \cdot (1+\delta_{pq}), \tag{9}$$

where$\delta_{pq} = \frac{1}{\pi} \cdot \frac{tg\frac{\Delta}{2}}{1+tg^2\frac{\Delta}{2}} \cdot \frac{1}{(\frac{\lambda_B}{\lambda_g})^3 \cdot \left(1+tg^2\frac{\Delta}{2}\right)-1} \cdot \left\{\frac{1}{th[\frac{\pi}{2}(2q-1)tg\frac{\Delta}{2}]} - \frac{1}{th[\frac{\pi}{2}(2p-1)tg\frac{\Delta}{2}]}\right\}.$

The quantities $\delta_1$ and $\delta_{pq}$ included in formulas (8) and (9) depend on the values of the measured quantities λ and $tg\frac{\Delta}{2}$. For ease of calculation, the values of $\delta_1$, $\delta_{12}$ and $\delta_{23}$ were calculated. According to the calculation data, the correction δ1 always has a positive sign, while $\delta_{pq}$ has negative signs. The values of $\delta_1$ and $\delta_{pq}$ increase sharply with increasing $\lambda_g$ of the measured liquid. However, as the numbers p and q increase, the correction values decrease noticeably; they turn out to be significant only when the wavelength $\lambda_g$ in the waveguide with the liquid under study is measured from the positions of the first three minima of the dependence of η on l.

It should be noted that for most polar liquids with values of $tg\frac{\Delta}{2} > 0{,}25$ in the ultrahigh frequency range, the dependence of η on l has, as a rule, 2–3 well-resolved minima of η.

## 3. RESULTS AND DISCUSSION

Table 1 gives the values of the dielectric constant of acetic acid ε' and dielectric losses ε''. Explicitly, the spectrum contains only one absorption region of the relaxation type (Fig. 1, 2).

**Table 1.** Dielectric coefficients of acetic acid

| T, ºC | 20 | | 30 | | 40 | | 50 | |
|---|---|---|---|---|---|---|---|---|
| λ cm | ε' | ε'' | ε' | ε'' | ε' | ε'' | ε' | ε'' |
| 40.0 | 5.75 | 0.96 | 5.91 | 0.89 | 6.12 | 0.80 | 6.32 | 0.73 |
| 30.0 | 5.48 | 1.15 | 5.70 | 1.08 | 5.93 | 0.99 | 6.17 | 0.91 |
| 20.0 | 5.05 | 1.23 | 5.30 | 1.21 | 5.56 | 1.19 | 5.83 | 1.14 |
| 10.0 | 4.40 | 1.11 | 4.60 | 1.17 | 4.81 | 1.22 | 5.06 | 1.28 |
| 6.40 | 4.11 | 0.97 | 4.25 | 1.06 | 4.44 | 1.14 | 4.63 | 1.23 |
| 4.40 | 3.85 | 0.89 | 4.00 | 0.96 | 4.14 | 1.06 | 4.30 | 1.16 |
| 2.12 | 3.52 | 0.76 | 3.60 | 0.82 | 3.70 | 0.88 | 3.78 | 0.97 |
| 1.20 | 3.28 | 0.62 | 3.34 | 0.70 | 3.41 | 0.77 | 3.50 | 0.85 |
| 0.75 | 3.08 | 0.52 | 3.12 | 0.58 | 3.19 | 0.67 | 3.26 | 0.75 |

The relaxation times corresponding to the peak of the ε''(λ) curves change with temperature according to the relation $\tau = \tau_0 \exp(U/RT)$, where $\tau_0 = 11.8 \cdot 10^{-14}$ cm, U = 4.0 kcal/mol. Found in this way relaxation parameters should be considered effective, which is proven by the clear resolution of the spectrum when using the linear representation method (Fig. 3,4).

To a first approximation, the frequency variation of the values ε', ε'' can be described by a superposition of two independent relaxation processes (Fig. 5):

$$\varepsilon' - i\varepsilon'' = \varepsilon_{\infty 2} + (\varepsilon_{01} - \varepsilon_{\infty 2})\,\{C_1(1 + i\omega\tau_1)^{-1} + C_2\,[(1 + i\omega\tau_2)^{1-\alpha_2}]^{-1}\}. \tag{10}$$

Where: $C_j = \frac{\varepsilon_{0j} - \varepsilon_{\infty j}}{\varepsilon_{01} - \varepsilon_{\infty 2}}$; $C_1 + C_2 = 1$; $\varepsilon_{\infty 1} = \varepsilon_{02}$; $\tau_1 > \tau_2$, $\alpha_2$ – parameter of the symmetric distribution of dielectric relaxation time values around the most probable value $\tau$.

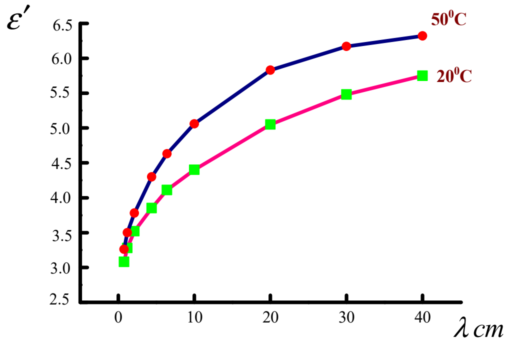

**Figure 1.** Dielectric constants ε' acetic acid depending on the wavelength λ at 20 and 50°C

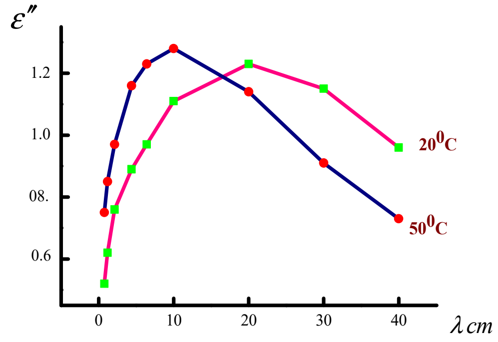

**Figure 2.** Dielectric losses ε'' of acetic acid depending on wavelength λ at 20 and 50°C

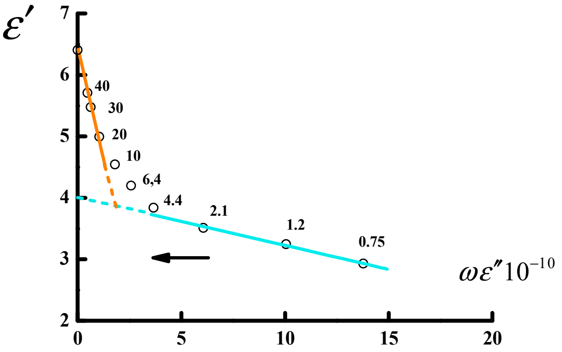

**Figure 3.** Line diagrams of acetic acid at 20°C.
The numbers at the dots are wavelengths (cm)

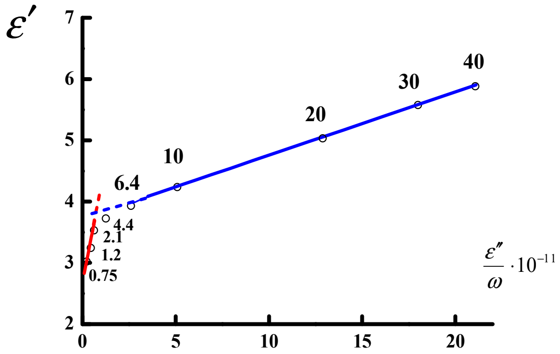

**Figure 4.** Line diagrams of acetic acid at 20°C.
The numbers at the dots are wavelengths (cm)

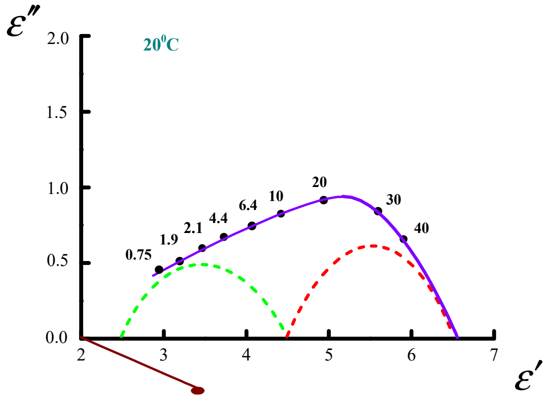

**Figure 5.** Arc diagrams of acetic acid when the spectrum is divided into two regions (20°C).
The numbers at the dots are wavelengths (cm)

Table 2 gives the estimated parameters of equation (1) along with the measured values of $\varepsilon_{01}$. A comparison of the spectrum calculated from these values with the observed one shows [22] that the permissible limits of variation of the estimated parameters, in which the scatter does not exceed the experimental errors, are about 10% for $\tau$ and 0.05 for C.

**Table 2.** Relaxation parameters of the spectrum of acetic acid according to (1)

| T, °C | $\varepsilon_{01}$ | $\varepsilon_{\infty 2}$ | $\tau_1 \cdot 10^{12}$sec | $\tau_2 \cdot 10^{12}$sec | $\alpha_2$ | $C_1$ |
|---|---|---|---|---|---|---|
| 20 | 6.30 | 2.58 | 122 | 11.5 | 0.29 | 0.50 |
| 30 | 6.38 | 2.57 | 100 | 10.0 | 0.28 | 0.49 |
| 40 | 6.46 | 2.56 | 81 | 8.8 | 0.27 | 0.46 |
| 50 | 6.54 | 2.56 | 66 | 7.8 | 0.26 | 0.48 |

Significant broadening of the high-frequency (HF) region makes its further division possible. Due to the large distribution parameter α of the dielectric relaxation time, attempts to describe this region using elementary Debye processes require the introduction of four relaxation time values. By analogy with monohydric alcohols [23], it seems advisable to limit ourselves to separating from the HF region only two contributions, each of which retains a certain distribution of relaxation times of a symmetrical type (Fig. 6). Table 3 gives the spectrum parameters according to the equation

$$\varepsilon' - i\varepsilon'' = \varepsilon_{\infty 3} + (\varepsilon_{01} - \varepsilon_{\infty 3})\sum C_j[\,1 + (i\omega\tau_j)^{1-\alpha}]^{-1} j = 1,2,3. \tag{11}$$

Where $-\varepsilon_{\infty 1} = \varepsilon_{02}$; $\varepsilon_{\infty 2} = \varepsilon_{03}$;$\tau_1 > \tau_2 > \tau_3$, $\alpha_1 = 0$. The values of relaxation activation energy found from the lg graph are also given there $\tau - T^{-1}$

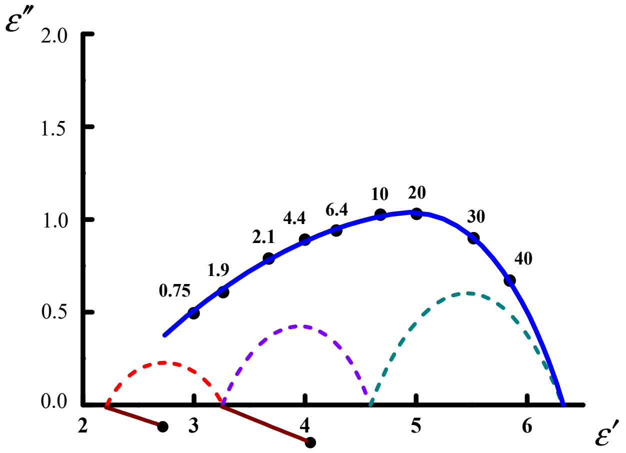

**Figure 6.** Arc diagrams of acetic acid when the spectrum is divided into three regions (20°C). The numbers at the dots are wavelengths (cm).

**Table 3.** Relaxation parameters of the spectrum of acetic acid according to equation (2)

| T, ºC | $\varepsilon_{01}$ | $\varepsilon_{02}$ | $\varepsilon_{03}$ | $\varepsilon_{\infty 3}$ | $\tau_1 \cdot 10^{12}$sec | $\tau_2 \cdot 10^{12}$sec | $\tau_3 \cdot 10^{12}$sec | $\alpha_2$ | $\alpha_3$ |
|---|---|---|---|---|---|---|---|---|---|
| 20 | 6.30 | 4.42 | 3.26 | 2.58 | 122 | 25.0 | 2.8 | 0.11 | 0.13 |
| 30 | 6.38 | 4.48 | 3.28 | 2.57 | 100 | 21.5 | 2.5 | 0.11 | 0.12 |
| 40 | 6.46 | 4.54 | 3.29 | 2.56 | 81 | 19.0 | 2.2 | 0.10 | 0.11 |
| 50 | 6.54 | 4.60 | 3.30 | 2.56 | 66 | 17.0 | 2.0 | 0.10 | 0.10 |

The presence of an absorption spectrum contradicts the widespread opinion about the exclusively nonpolar dimeric type of clustering of liquid acetic acid. The presence of three absorption regions with relaxation times differing by a factor of 40 cannot be explained by the hypothetical interaction of monomers and cyclic dimers. At the same time, the value of the correlation parameter g = 0.4 (20°C) indicates a limited proportion of polar clusters. Taking this into account, as well as summarizing the results of spectrum analysis, identification should be carried out in terms of the equilibrium between non-polar clusters, polar multiverse and monomers.

The characteristics of the HF contribution give reason to believe that it is created by the reorientation of individual molecules as a whole, supplemented by internal rotation. The relative content of kinetic units involved in HF absorption (9%) found from the amplitude $\varepsilon_{03} - \varepsilon_{\infty 3}$ (at $g_{3=1}$) coincides with the concentration of acid monomers (12%) at 20°C, estimated from ultrasonic data. The attribution of the HF contribution to monomers is also confirmed by the closeness of the values of $\tau_3$, $\alpha_3$, and $U_3$ to those for ethyl acetate, where, with a comparable molecular structure, there are no directional interactions.

There is no doubt that the LF contribution is classified as multiverse. As for the intermediate contribution, its identification depends on the specific cluster model. For example, if we proceed from the model of linear highly polar clusters, then the most likely explanation will be the ratio of the intermediate contribution to the terminal molecules. Another model of clusters, proposed for alcohols [24-26], postulates an equilibrium between monomers, weakly polar dimers and highly polar cyclic (non-planar) multiverse (presumably tetramers). In this case, the HF and LF contributions, as before, are created by monomers and multiverse, respectively, and intermediate absorption is associated with polar dimers.

The main difference between linear and cyclic clustering models is that in the first case, the appearance of an intermediate region directly follows from the presence of chain multiverse. On the contrary, with cyclic clustering, the presence of kinetic units responsible for a given absorption region is not due to the existence of other types of relaxers.

## 4. CONCLUSIONS

1. The prerequisites for choosing between the two models are determined by comparing the expected and observed changes in the spectrum caused by the introduction of foreign molecules of acetic acid.

2. The linear clustering model predetermines the correlation between the amplitudes of low-frequency and intermediate contributions upon dilution of acetic acid.

3. It is obvious that the idea of an exclusively nonpolar type of clustering of liquid acetic acid is erroneous, as well as the possibility of two types of clusters, including open or ring polar multiverse.

4. From the dielectric data it is clear that there are two stable modifications in solid acetic acid, differing in orientation freedom.

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# ДІЕЛЕКТРИЧНА РЕЛАКСАЦІЯ ТА МОЛЕКУЛЯРНІ ВЗАЄМОДІЇ В ОЦТОВІЙ КИСЛОТІ

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У цій статті представлені результати вимірювань діелектричних коефіцієнтів оцтової кислоти та її розчинів. Вимірювання проводилися на довжинах хвиль $\lambda$ = 40,0; 30,0; 20,0; 10,0; 6,4; 4,4; 2,1; 1,2; та в діапазоні температур 20 ÷ 500°C. Моделі молекулярної кластеризації в рідкій оцтовій кислоті досліджувалися на основі аналізу спектру діелектричного поглинання. Результати дослідження спектру радіочастотного поглинання оцтової кислоти вказують на наявність двох поліморфних форм цієї сполуки. Визначено характерні температурні залежності діелектричної проникності $\varepsilon'$ оцтової кислоти, з яких чітко видно два ізотермічні обертальні переходи. Перший – у температурі плавлення – супроводжується різким зменшенням кількості діелектрично активних одиниць. Другий, при температурі нижче точки плавлення, призводить до значень діелектричної проникності $\varepsilon'$, близьких до високочастотної (HF) межі загального орієнтаційного внеску для мікрохвильового поглинання в рідкій кислоті. При температурах вище першого та нижче другого переходів значення діелектричної проникності $\varepsilon'$ не залежать від частоти електромагнітної хвилі; навпаки, спостерігається виражена дисперсія в інтервалі між двома переходами.

**Ключові слова:** *діелектрична спектроскопія; діелектрична релаксація; комплексна діелектрична проникність; діелектричні властивості; оцтова кислота*