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Dielectric Properties of Polyhydric Alcohols: Butanediols

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Abstract—The equilibrium and dynamic properties of 1,3-, 1,4-, and 2,3-butanediols were studied experimentally in the frequency range from 1 MHz to 36 GHz at temperatures from 283 to 423 K. The densities of the compounds were measured. The Kirkwood correlation factor was calculated with various (experimentally measured and calculated) dipole moments of the molecules.

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Many liquid systems are widely used as structural and functional materials. Therefore, it becomes necessary to determine the structure of liquids from the experimental data and to develop model concepts for liquids. This is one of the most complex problems of the modern chemistry. As a rule, the structure of liquids is analyzed using data obtained by various physical methods. One of useful methods is dielectrometry. Modern devices allow measurements of the complex dielectric constant $\varepsilon^*(\omega)$ in a wide frequency range with a high accuracy.

This paper continues our studies of polyhydric alcohols and their solutions and of molecular mechanisms of structural rearrangements occurring in the course of thermal motion [1–3].

Diols are liquids whose molecules can participate in the formation of network structures. Owing to the presence of two hydroxy groups in the molecule, butanediols are capable of forming intermolecular and intramolecular hydrogen bonds, which gives rise to a diversity of structures existing in the liquid. One of the methods suitable for studying the molecular structure of such liquids and its rearrangements is dielectric radiospectroscopy [4–6]. In this study we experimentally examined the equilibrium and dynamic properties of 1,3-, 1,4-, and 2,3-butanediols. The procedures for measuring the dielectric constant ε' and dielectric loss ε'' are given in [4], and those for measuring the static dielectric constant ε_s and density ρ, in [6].

As experimental objects we chose 2,3-, 1,4-, and 1,3-butanediols. The compounds were dried and subjected to fractional distillation in a vacuum. The physicochemical properties of 2.3-, 1,4-, and 1,3-butanediols after the distillation are given in Table 1.

Dielectric radiospectroscopy as one of methods for structural studies of liquids allows description of the molecular structure of liquids in terms of the Onsager–Kirkwood–Fröhlich model [2, 5–6, 8]:

$$g_{\text{exp}} = \frac{9V_m k_B T(\varepsilon_S - \varepsilon_\infty)(2\varepsilon_S + \varepsilon_\infty)}{4\pi N_A \mu_v^2 \varepsilon_S(\varepsilon_\infty + 2)^2}, \quad (1)$$

Table 1. Physicochemical properties of butanediols

Substance	bp, °C (p, mm Hg)		n_d (T, °C)		ρ, g cm^{-2} (T, °C)	
	experiment	[7]	experiment	[7]	experiment	[7]
2,3-Butanediol	91–91.5 (23)	182.5 (760) 86 (16)	1.4310 (25)	1.4310 (25)	0.988 (25)	1.0033 (25)
1,4-Butanediol	120 (10)	120 (10)	1.4470 (20)	1.4467 (20)	1.016 (20/4)	1.020 (20/4)
1,3-Butanediol	120–120.5 (25) 105–106 (10)	207.5 (760) 109 (14)	1.4399 (20)	1.4401 (20)	1.0043 (20)	1.0041 (20)

where ε_s is the static dielectric constant; μ_v, dipole moment of the molecule in a vacuum; V_m, molar volume; k_B, Boltzmann constant; N_A, Avogadro number; T, temperature; and ε_∞, dielectric constant caused by deformation polarization.

We showed in [9] that the error in calculating the correlation factor g is mainly determined by the choice of a method for calculating ε_∞ and of the μ_v value used. The deformation dielectric constant ε_∞ is usually calculated by Eq. (2) or (3):

$$\varepsilon_\infty = 1.1 n_D, \quad (2)$$

$$\varepsilon_\infty = (V_m + 2P_\infty)/(V_m - P_\infty), \quad (3)$$

where n_D is the refractive index, and P_∞, molar deformation polarization calculated by the additive scheme from data on the polarization of bonds. In this study, to determine ε_∞, we used Eq. (3) and the bond polarization values suggested in [10]:

$$P_\infty = 4.76 n_{C-O(OH)} + 1.22\, n_{C-C} + 1.70\, n_{C-H},$$

where n_i is the number of bonds of the corresponding types. The calculated value of P_∞ is 26.78 $cm^3\ mol^{-1}$.

The dipole moment of 1,4-butanediol, μ 2.46 D, was taken from [11]. In the case of 1,3-butanediol, the mean dipole moment was compared to that of 1,3-propanediol, because formally 1,3-butanediol can be obtained by replacement of a hydrogen atom in the 1,3-propanediol molecule by the CH_3 group [12]. Buc demonstrated in the same study [12] that the dipole moment of β-diols (1,3-, 2,4-) is strongly influenced by the intramolecular hydrogen bond. The mean dipole moment of β-diols is μ_v 2.7 D (2.4–2.9 D). For our calculations, we took the values of μ 2.4 and 2.46 D [12]. For 2,3-butanediol, we found no experimental value of the dipole moment in the literature. By analogy with pentanediols, we took μ 2.13 D [13]. In this connection, it is interesting to consider the results of DFT conformation analysis of butanediol isomers at 298.15 K, performed by Lopes et al. [14]. Taking into account the statistical weights of the most stable isomers, Lopes et al. calculated the weighted-average enthalpy for each isomer in the gas phase. By combining these results with the experimental enthalpies of vaporization at 298.15 K, they estimated the enthalpy of each butanediol isomer in the liquid state. We, in turn, estimated the dipole moments of 2,3-butanediol taking into account various molecular conformations. As a result, for 2,3-butanediol we obtained μ 2.54 D, which agrees with μ 2.53 D of the *gauche* conformer *tGg'* of (*R*,*S*)-2,3-butanediol [14].

The dielectric dispersion $\varepsilon^*(\omega) = \varepsilon' + i\varepsilon''$ of strongly associated liquids such as diols and triols and of weakly associated liquids such as acetonitrile is described by different forms of the Havriliak–Negami equation [15]:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_S - \varepsilon_\infty}{[1 + (i\omega\tau)^{1-\alpha}]^\beta}. \quad (4)$$

Here the parameters α and β characterize the distribution function of the relaxation time; ε_s is the static dielectric constant; ε_∞, high-frequency limit of the dispersion range; and τ, relaxation time. At α 0 and β 1, Eq. (1) transforms into the Debye equation; at β 1 and $0 \le \alpha < 1$, into the Cole–Cole equation; and at α 0 and $0 < \beta \le 1$, into the Davidson–Cole equation.

The first paper on the dielectric properties of 1,4-butanediol was published by Sagal in 1962 [16]. The frequency range was from 100 kHz to 14 GHz, and the temperature range, 288–323 K. Sagal [16] described the dispersion $\varepsilon^*(\omega)$ with the Cole–Cole equation with the parameter α 0.09 at T 298 K. Lux and Stockhausen [17] studied aqueous solutions of diols: ethanediol, 1,2- and 1,3-propanediols, and 1,2-, 1,3-, 1,4-, and 2,3-butanediols in the frequency range from 5 MHz to 70 GHz at T 293 K. For all the diols studied, the dispersion $\varepsilon^*(\omega)$ was described by the Davidson–Cole equation. The following parameters of the equation were obtained for 1,4-butanediol: β 0.69, τ 1282 ps. Becker and Stockhausen [18] studied binary solutions of the same diols in 1-butanol and tert-butanol in the same frequency range at T 293 K. The dielectric spectra obtained were described by using several Debye regions. Hanna et al. [19, 20] studied a series of α,ω-alkanediols: 1,2-ethanediol, 1,3-propanediol, 1,4-butanediol, and 1,5-pentanediol in the range from 10 MHz to 10 GHz at 293 K. For all these diols, the dispersion $\varepsilon^*(\omega)$ was also described by the Davidson–Cole equation. The parameters of the equation for 1,4-butanediol were as follows: ε_s 30.9; β 0.86; $\beta\tau$ 820 ps.

The first paper on the dielectric properties of 2,3-butanediol was, apparently, published in 1978 [21]. In [6, 8, 9], we studied the equilibrium properties of pure diols and of their aqueous solutions. The investigation objects were ethanediol, 1,2- and 1,3-propanediols, and 1,2-, 1,3-, 1,4-, and 2,3-butanediols at a frequency of 1 MHz in the temperature range from 283 to 383 K.

Table 2. Temperature dependence of physicochemical characteristics of 1,3-butanediol

T, K	ρ, g cm^{-3}	V_M, ml mol^{-1}	ε_∞	ε_s	g_{exp}(μ_v 2.4 D)	μ_l^2	μ_l, D	g_{exp}(μ_v 2.46 D)
293	1.006	89.58	2.28	29.45	2.31	13.309	3.648	2.19
303	0.999	90.21	2.27	27.92	2.28	13.187	3.631	2.18
313	0.991	90.94	2.25	26.27	2.25	12.972	3.602	2.14
323	0.983	91.68	2.24	24.86	2.22	12.823	3.581	2.13
333	0.975	92.43	2.22	23.68	2.21	12.750	3.571	2.11
343	0.967	93.20	2.21	22.59	2.20	12.685	3.562	2.09
353	0.960	93.88	2.20	21.41	2.16	12.497	3.535	2.06
363	0.952	94.66	2.18	20.41	2.15	12.401	3.522	2.05
373	0.944	95.47	2.17	19.41	2.12	12.264	3.502	2.02
383	0.936	96.28	2.16	18.36	2.08	12.048	3.471	1.99
393	0.928	97.11	2.14	17.50	2.07	11.924	3.453	1.97
403	0.920	97.96	2.13	16.64	2.04	11.760	3.429	1.95
413	0.912	98.82	2.12	15.82	2.00	11.589	3.404	1.91
423	0.905	99.58	2.10	15.00	1.97	11.354	3.370	1.88

Szabat et al. [22] reported the parameters of the Davidson–Cole equation obtained for four butanediol isomers (2,3-, 1,2-, 1,3-, and 1,4-butanediols) at 293 K in the frequency range from 10 MHz to 4 GHz. For 2,3-butanediol, they obtained the following parameters of the Davidson–Cole equation: ε_s 23.1, β 0.58, βτ 853 ps.

The first paper on the dielectric properties of 1,3-butanediol was published by Moriamez in 1958 [23]. The frequency range was 0.1–2500 MHz, and the temperature range, 253–298 K. The authors distinguished two regions of the dispersion. The high-frequency region was described by the Cole–Cole equation with the parameter α 0.21 at T 298 K. In 1964, Moriamez et al. [24] extended the frequency range to 14 GHz and obtained the parameter α 0.05 at T 298 K. In 1962, McDuffie and Litovitz [25] performed measurements in the frequency range 0.1–1200 MHz and temperature range 283.3–297 K. The dielectric dispersion was described by the Davidson–Cole equation with the parameter β varying from 0.85 to 0.7 in the examined temperature range. Sudo et al. [26] studied aqueous solutions of 1,3-butanediol in the frequency range from 10 MHz to 20 GHz at 298 K.

The experimental values of ε_s and ρ and the calculated values of g_{exp}, obtained in this study, are given in Tables 2–4. It can be seen that the results we obtained are in good agreement with the data obtained in [8, 27–30]. This primarily concerns the experimental data on the density ρ [27, 28] and static dielectric constant ε_s [8, 29, 30]. We have already noted in [9] that, for correct interpretation of the correlation factor g_{exp}, it is necessary to take into account that diol molecules can have essentially different conformations whose dipole moments differ considerably. Apparently, the energetically favorable conformations for which the dipole moments were determined in the gas phase may be quite different in the neat liquid. In [3] we emphasized that, in the liquids, all the conformations occur in a dynamic equilibrium, but with increasing temperature this equilibrium is disturbed, and the relative content of conformers changes. Thus, the correlation factor, in principle, can reflect more complex processes than it is commonly noted. It is seen from Tables 2–4 that the use of different μ_v values leads to noticeable changes in g_{exp}; therefore, in this case we can analyze the behavior of μ_l, because Eq. (5) is valid:

$$g_{exp} = \frac{\mu_l^2}{\mu_v^2}. \quad (5)$$

The product $g_{exp}\mu_v^2 = \mu_l^2$ is independent of the choice of the vacuum dipole moment, and it becomes possible to analyze the dipole moment of a molecule in a liquid μ_l.

It should be noted here that, commonly, if $g_{exp} > 1$, the orientation of dipole moments is considered to be predominantly parallel, which usually corresponds to the formation of chain associates. If $g_{exp} \approx 1$, this suggests disordered mutual orientation of the dipole moments of molecules or formation of cyclic associates.

As seen from Tables 2–4 and Fig. 1, the correlation factor g_{exp} of butanediols shows a smooth monotonic

Table 3. Temperature dependence of physicochemical characteristics of 1,4-butanediol

T, K	ρ, g cm^{-3}	V_m	ε_∞	ε_s	Calculation for μ_v 2.46 D			Calculation for μ_v 2.4 D		
					g_{exp}	μ_l^2	μ_l, D	g_{exp}	μ_l^2	μ_l, D
293	1.020	88.35	2.30	31.70	2.31	14.00	3.74	2.40	14.00	3.74
303	1.014	88.86	2.29	30.20	2.30	13.92	3.73	2.42	13.83	3.72
313	1.007	89.49	2.28	28.72	2.28	13.82	3.72	2.40	13.64	3.69
323	1.000	90.12	2.27	27.23	2.26	13.68	3.70	2.37	13.45	3.67
333	0.993	90.76	2.26	25.27	2.18	13.20	3.63	2.28	13.21	3.63
343	0.986	91.40	2.24	23.91	2.15	12.99	3.61	2.26	12.99	3.60
353	0.979	92.05	2.23	22.77	2.13	12.87	3.59	2.23	12.85	3.58
363	0.972	92.72	2.22	21.64	2.10	12.70	3.56	2.20	12.67	3.56
373	0.965	93.39	2.21	20.50	2.06	12.49	3.53	2.16	12.44	3.53
383	0.958	94.07	2.19	19.36	2.02	12.22	3.50	2.12	12.23	3.50
393	0.951	94.76	2.18	18.41	1.99	12.04	3.47	2.09	12.03	3.47
403	0.944	95.47	2.17	17.59	1.97	11.92	3.45	2.06	11.90	3.45
413	0.936	96.28	2.16	16.68	1.94	11.71	3.42	2.02	11.67	3.42
423	0.929	97.01	2.14	15.73	1.89	11.41	3.38	1.98	11.41	3.38

Table 4. Temperature dependence of physicochemical characteristics of 2,3-butanediol

T, K	ρ, g cm^{-3} [27, 28]	V_m	ε_∞	ε_s	Calculation for μ_v 2.13 D			Calculation for μ_v 2.54 D		
					g_{exp}	μ_l^2	μ_l, D	g_{exp}	μ_l^2	μ_l, D
283	1.0054	89.636	2.275	22.60	2.145	9.734	3.120	1.509	9.734	3.120
293	0.9970	90.391	2.260	21.48	2.138	9.699	3.144	1.503	9.699	3.114
298	0.9928	90.774	2.253	20.91	2.130	9.662	3.108	1.498	9.662	3.108
303	0.9885	91.168	2.245	20.44	2.131	9.667	3.109	1.498	9.667	3.109
313	0.9810	91.865	2.232	19.45	2.117	9.604	3.099	1.498	9.604	3.099
323	0.9716	92.754	2.215	18.45	2.101	9.534	3.088	1.478	9.534	3.088
333	0.9632	93.563	2.200	17.73	2.109	9.569	3.093	1.483	9.569	3.093
343	0.9548	94.386	2.186	16.95	2.103	9.540	3.089	1.479	9.540	3.089
353	0.9463	95.234	2.171	16.00	2.066	9.374	3.062	1.453	9.374	3.062
363	0.9379	96.087	2.157	15.45	2.079	9.431	3.071	1.462	9.431	3.071
373	0. 9294	96.966	2.142	14. 73	2.061	9.351	3.058	1.449	9.351	3.058
383	0.9210	97.850	2.128	14.14	2.057	9.331	3.055	1.446	9.331	3.055
393	0. 9125	98.762	2.114	13. 36	2.015	9.144	3.024	1.417	9.144	3.024
403	0.9041	99.679	2.100	12.86	2.014	9.138	3.023	1.416	9.138	3.023
413	0.8956	100.625	2.086	12.23	1.985	9.004	3.001	1.396	9.004	3.001
423	0.8872	101.578	2.072	11.64	1.955	8.870	2.978	1.375	8.870	2.978

Table 5. Dielectric characteristics of 1,3-butanediol

T, °C	ε_s	λ 15 cm		λ 10.3 cm		λ 3.2 cm		λ 0.8 cm		ε_∞
		ε'	ε''	ε'	ε''	ε'	ε''	ε'	ε''	
20	29.45									
25	28.81	4.56	2.67	4.07	2.23	3.56	1.00	3.15	0.51	2.27
30	27.92									
40	26.27	5.50	4.42	4.74	3.30	3.78	1.61	3.23	0.75	2.25
50	24.86	6.35	6.04	5.42	4.20	4.01	2.06	3.30	0.94	2.24
60	23.68	8.25	7.28	6.45	5.35	4.37	2.68	3.38	1.15	2.22
70	22.59	10.85	7.92	7.74	6.62	4.77	3.42	3.47	1.40	2.21
80	21.41	14.00	7.65	9.45	7.58	5.29	4.19	3.58	1.70	2.20
90	20.41	15.15	7.00	11.70	7.61	5.96	4.98	3.70	2.02	2.18
100	19.41	15.86	6.24	13.62	6.75	6.81	5.57	3.84	2.40	2.17
110	18.36	16.15	5.00	14.68	5.90	7.80	6.06	4.00	2.78	2.16
120	17.50	16.25	3.60	15.20	5.14	8.98	6.24	4.19	3.11	2.14
130	16.64	15.85	2.82	15.23	4.25	9.94	5.71	4.39	3.39	2.13
140	15.82	15.25	2.20	15.04	3.40	10.66	5.25	4.62	3.65	2.12
150	15.00	14.65	1.75	14.60	2.43	11.32	4.96	4.92	3.95	2.10

temperature dependence, remaining considerably higher than unity throughout the examined temperature range. A decrease in g_{exp} with increasing temperature is less pronounced for diols than for monohydric alcohols [9]. This fact suggests existence of 3D network structures, which are broken to a lesser extent than linear associates. A decrease in g_{exp} with increasing temperature is the least pronounced for 2,3-butanediol. This is apparently associated with a considerable fraction of intramolecular hydrogen bonds, which varies with temperature insignificantly [3, 9, 14].

The experimental values that we obtained for the dielectric constant ε' and dielectric loss ε'' are given in Tables 5–7 and in Figs. 2–4. As seen from these plots, all the curves are described by the Davidson–Cole equation [31]. The parameters of the Davidson–Cole equation were determined by the least-squares method [32].

Figure 2 shows the ε''(ε') plots for 1,4-butanediol at three temperatures. These plots are described by the Davidson–Cole equation. At 298 K, β 0.59, ε_s 31.23, ε_∞ 2.69, and τ 2.06×10^{-9} s. The rms error of the description is 1.33×10^{-2}.

Figure 3 shows the ε''(ε') plots for 1,3-butanediol at three temperatures. All the curves are described by the Davidson–Cole equation. At 298 K, β 0.59, ε_∞ 2.65,

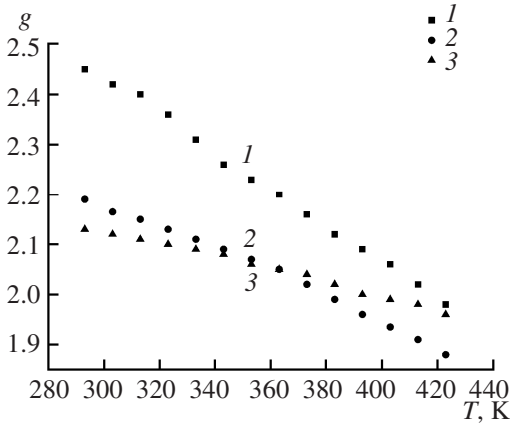

Fig. 1. Temperature dependence of the correlation factor g_{exp} for butanediols: (*1*) 1,4-butanediol, (*2*) 1,3-butanediol, and (*3*) 2,3-butanediol.

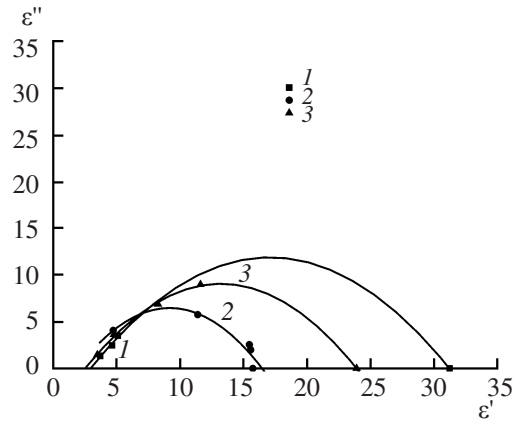

Fig. 2. Presentation of the dielectric spectrum of 1,4-butanediol on the complex plane at different temperatures: (*1*) 298, (*2*) 423, and (*3*) 343 K.

Table 6. Dielectric characteristics of 2,3-butanediol

T, °C	ε_s	λ 15 cm		λ 10.3 cm		λ 3.2 cm		λ 0.8 cm		ε_∞
		ε'	ε''	ε'	ε''	ε'	ε''	ε'	ε''	
20	21.42									
25	20.91	5.63	4.05	5.15	2.43	3.90	1.45	3.27	0.77	2.26
30	20.30									
40	19.45	7.10	5.85	6.15	3.55	4.31	2.20	3.39	1.08	2.23
50	18.45	9.30	5.83	7.17	4.39	4.67	2.90	3.48	1.30	2.22
60	17.73	12.00	5.32	8.75	5.04	5.16	3.54	3.57	1.54	2.21
70	16.95	13.30	4.48	10.50	5.12	5.76	4.20	3.68	1.81	2.19
80	16.00	13.96	3.73	11.76	4.97	6.50	4.65	3.79	2.10	2.17
90	15.45	13.90	3.05	12.65	4.27	7.36	5.02	3.92	2.40	2.16
100	14.73	13.60	2.45	13.05	3.45	8.25	5.08	4.08	2.75	2.15
110	14.14	13.08	1.98	12.90	2.76	8.84	4.89	4.26	3.08	2.13
120	13.36	12.58	1.60	12.67	2.07	9.73	4.47	4.46	3.39	2.12
130	12.86	12.10	1.30	12.40	1.58	10.00	3.96	4.68	3.57	2.10
140	12.23	11.66	1.05	11.90	1.26	10.14	3.44	4.95	3.72	2.09
150	11.64	11.30	0.85	11.30	1.06	10.21	2.87	5.30	3.83	2.07

Table 7. Dielectric characteristics of 1,4-butanediol

T, °C	ε_s	λ 15 cm		λ 10.3 cm		λ 3.2 cm		λ 0.8 cm		ε_∞
		ε'	ε''	ε'	ε''	ε'	ε''	ε'	ε''	
20	31.70									
25	31.23	5.08	3.50	4.63	2.45	3.70	1.34	3.25	0.60	2.29
30	30.03									
40	28.40	6.08	4.90	5.30	3.60	3.97	1.92	3.30	0.83	2.28
50	26.90	7.40	6.60	5.95	4.57	4.14	2.50	3.34	1.01	2.27
60	25.40	9.40	8.50	6.88	5.86	4.40	3.05	4.40	1.22	2.26
70	23.91	11.60	8.95	8.25	6.86	4.78	3.62	3.47	1.46	2.24
80	22.77	13.90	8.98	9.60	7.48	5.08	4.48	3.56	1.72	2.23
90	21.64	15.50	8.00	12.25	7.63	5.82	5.06	3.66	2.02	2.22
100	20.50	17.00	6.47	14.30	6.80	6.44	5.96	3.78	2.30	2.21
110	19.36	17.38	5.43	15.43	5.85	7.30	6.42	3.91	2.60	2.19
120	18.41	17.40	4.45	16.18	4.94	8.52	6.80	4.07	2.96	2.18
130	17.59	16.90	3.60	16.15	4.10	9.81	6.76	4.27	3.43	2.17
140	16.68	16.20	2.63	15.90	3.07	10.62	6.32	4.47	3.80	2.16
150	15.73	15.55	1.98	15.45	2.54	11.38	5.74	4.72	4.06	2.14

and τ 2.59×10^{-9} s. The rms error of the description is 1.91×10^{-2}.

Figure 4 shows the ε''(ε') plots for 2,3-butanediol at three temperatures. These plots are described by the Davidson–Cole equation. At 298 K, β 0.526, ε_s 20.91, ε_∞ 2.51, and τ 1.08×10^{-9} s. The rms error of the description is 5.6×10^{-2}.

The results obtained will make it possible in future to find the relationship of the empirical parameters of the Davidson–Cole equation with the molecular characteristics of the liquids within the framework of the Dissado–Hill relaxation theory [5, 33].

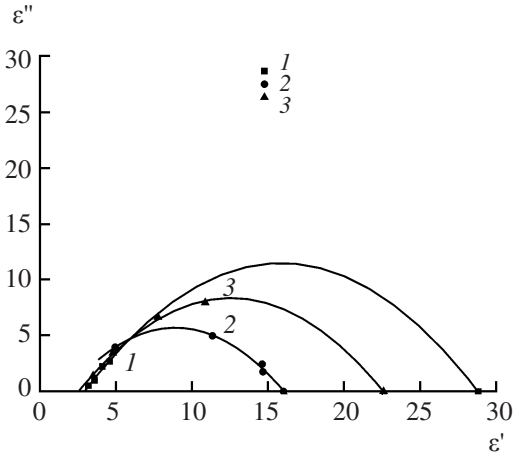

Fig. 3. Presentation of the dielectric spectrum of 1,3-butanediol on the complex plane at different temperatures: (*1*) 298, (*2*) 423, and (*3*) 343 K.

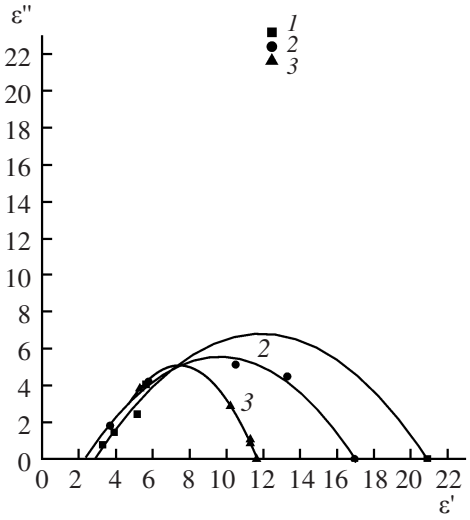

Fig. 4. Presentation of the dielectric spectrum of 2,3-butanediol on the complex plane at different temperatures: (*1*) 298, (*2*) 343, and (*3*) 423 K.

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