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PHYSICAL CHEMISTRY OF SOLUTIONS

Dissociation Constant of Nitric Acid

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Abstract—The composition of nitric acid solutions is investigated by means Raman spectroscopy (RS). The results are compared to critically selected data from other authors. The value of the thermodynamic dissociation constant in an aqueous nitric acid solution at 25°C ($K_a = [H^+][NO_3^-]\gamma'^2_{\pm}/[HNO_3]\gamma'_{HNO_3} = 35.5 \pm 1.5$ M) is determined by analyzing an extensive set of reliable and consistent literature and original data. Expressions for the dependences of the activity coefficient of undissociated HNO_3 molecules (γ'_{HNO_3}) and the mean ionic coefficient ($\gamma'_{\pm} = \sqrt{\gamma'_{H^+}\gamma'_{NO_3^-}}$) on the stoichiometric concentration of nitric acid in the range of 0–18 M are found.

Keywords: nitric acid, dissociation constant, concentration dependence

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INTRODUCTION

Nitric acid is widely used in human economic activity and plays a significant role in different natural processes. One of the most important fields of its industrial use is reprocessing spent nuclear fuel [1]. In nature, nitric acid is the main component of polar stratospheric clouds, which have an enormous impact on the concentration of atmospheric ozone [2]. In the troposphere, it leads to the generation of reactive halogens of marine aerosol according to the mechanism of acid displacement [3]. In all of these processes, the strong acidic properties of nitric acid and its dissociation according to the following reaction play a key role:

$$HNO_3(aq) \rightleftarrows H^+(aq) + NO_3^-(aq). \qquad (1)$$

The values of the dissociation constants of nitric acid (the equilibrium constant for reaction (1)), available in the literature, are shown in the table. Approximately half of the original data lie in the range of 20–27, but a considerable number of results deviate from these values. The value of the constant is thus currently unknown.

The dissociation of nitric acid in aqueous solutions has been studied by many authors, and great progress has been made due to Raman spectroscopy (RS) and nuclear magnetic resonance (NMR).

Nevertheless, there are still doubts about the degree of accuracy in the dissociation values [29]. In addition, the nature of the particles is not completely clear even in dilute solutions. Some authors [18, 21, 22] believe that along with HNO_3(aq) molecules, H^+(aq), and NO_3^-(aq) ions, we must consider that there are also $H^+\cdots NO_3^-$ ionic pairs. It was noted in [30–33], dedicated to thermodynamic modeling of processes in marine aerosols, that it is difficult to find the dissociation constants of HNO_3 and other strong acids, and it is recommended not to use them as parameters of models of atmospheric chemistry (it has been proposed that Henry constants be used instead). However, this approach does not allow us to consider the reaction of undissociated molecules of strong acids. The data on nitric acid dissociation thus require clarification.

The aim of this work was to construct a thermodynamic model of a nitric acid aqueous solution (at 25°C) in terms of the classical approach. More specifically, our aim was to determine reliable and fair values of the thermodynamic dissociation constant of nitric acid and the activity coefficients of neutral molecules and ions in an aqueous solution (with a concentration of 18 M or 79 wt %). More concentrated solutions were not studied, because in addition to main reaction (1) there could be such secondary processes as the formation and autoprotolysis of nitronium ions [34].

EXPERIMENTAL

The composition of our nitric acid aqueous solution (with concentrations up to 15 M or 67 wt %) was

Dissociation constants of nitric acid at 25°C, according to the literature data

K_a	Method	Reference	K_a	Method	Reference
40	(*1*)	[4]	25	(*4*)	[17]
21 ± 4	RS	[5]	24.4	RS	[18]
22	NMR	[6]	174–195	RS, UV	[19]
23.5	RS	[7]	4–16	RS	[20]
23.5	(*2*)	[8]	12	RS, (*5*)	[21]
27	RS, EP	[9, 10]	31; 40	RS, (*5*)	[22]
25	NMR	[11]	20	(*6*)	[23]
10–20	(*2*)	[12]	1.2–25	(*6*)	[24]
28; 120; 6000	(*3*)	[13]	43.6	(*6*)	[25]
15.4; 18.8	(*2*)	[14]	$10-6 \times 10^3$	(*6*)	[26]
33.5	(*2*)	[15]	34	(*6*)	[27]
20	NMR	[16]	23.5	(*6*)	[28]

The above are dimensionless quantities for an infinitely dilute aqueous solution, corresponding to units of concentration of mol/L or mol/kg. (*1*) Extrapolation over the values of the dielectric constant on the basis of data on non-aqueous solutions; (*2*) analysis of data on the activity coefficients and the degree of dissociation of HNO_3(aq); (*3*) analysis of the RS and NMR data, assuming the formation of $HNO_3 \cdot 3H_2O$, $HNO_3 \cdot 2H_2O$, and $HNO_3 \cdot H_2O$ complexes; (*4*) analysis of the experimental data on the solubility of $CaSO_4$ in HNO_3(aq); (*5*) analysis of the RS experimental data in terms of the thermodynamic model of mean spherical approximation; (*6*) based on the literature data.

determined by means of RS at room temperature (22–23°C). We used a LabRam HR 800 UV (Horiba Jobin Yvon) spectrometer with a diffraction grating of 1800 g/mm. Helium neon (632.81 nm) or argon ion (514.53 nm) lasers were used for excitation; the radiation power on the sample was about 5–7 mW. The samples were irradiated and the scattered radiation was collected using an Olympus BX41 optical microscope coupled to the spectrometer with a lens having a focal length of 40 mm. Nitric acid solutions were placed in a special cylindrical glass cell whose working part was positioned horizontally on a microscope stage. The cuvette was secured so that the vertical optical axis passing through the lens intersected with the horizontal axis of symmetry of the cylindrical cell; the distance from the lens to the point of intersection was 40 mm. In all experiments, the cuvette's position under the microscope remained the same.

Typical Raman spectra of solutions of KNO_3 and HNO_3 (conc.) are shown in Fig. 1. Normal vibrations are marked according to [35–37]. The spectra of the dilute nitric acid solutions contain only signals of nitrate ions, and these are very similar to the signals of KNO_3(aq). In the concentrated solutions, the peaks of undissociated nitric acid molecules emerged among those of NO_3^-. The spectrum is a superposition of the well-defined signals of free NO_3^- ($\nu_1 = 1048$ cm^{-1}) and undissociated HNO_3 ($\nu_7 = 638$ cm^{-1}, $\nu_5 = 688$ cm^{-1}, $\nu_6 = 958$ cm^{-1}, $\nu_3 = 1305$ cm^{-1}). In addition, the NO_3^- peak with a maximum at 1048 cm^{-1} is asymmetric and broadened in the low-frequency region.

The concentration of free nitrate ions was determined from the area of the peak at 1048 cm^{-1}. In fairly concentrated solutions, this peak is superimposed with the peak of HNO_3 molecules at 958 cm^{-1}. For precise determination of the area, the spectrum was therefore deconvoluted in the range of 850–1150 cm^{-1} using special software [38] (see Fig. 2). Spectral profiles were approximated using Voigt functions. Good agreement between calculated and experimental values was achieved only if the spectrum was treated as a set of three signals with peaks at 958, 1048, and ~1034 cm^{-1}. It was assumed that similar undefined signals at 1048 and ~1034 cm^{-1} correspond to NO_3^- ions. Note that in [18, 21, 22], an undefined signal with a frequency of ~1034 cm^{-1} was attributed to $H^+\cdots NO_3^-$ ion pairs. Allowing for experimental error, the hypothetical existence of ion pairs is unimportant when determining the composition of an aqueous solution of HNO_3, as is shown below.

Dissociation is almost complete in dilute solutions, and the concentration of free nitrate ions $[NO_3^-]$ is the same as the stoichiometric concentration of nitric acid C_{HNO_3}. There is thus a direct proportionality between the area of the signal $S_{1048+1034}$ and the stoichiometric concentration (Fig. 3); $S_{1048+1034} = kC_{HNO_3}$ when $C_{HNO_3} \leq 2$ M. The initial linear part of the dependence between $S_{1048+1034}$ and C_{HNO_3} was used to determine calibration coefficient k, allowing us to recalculate the value of $S_{1048+1034}$ relative to $[NO_3^-]$. In more concentrated solutions ($C_{HNO_3} > 3$ M), where deviations from

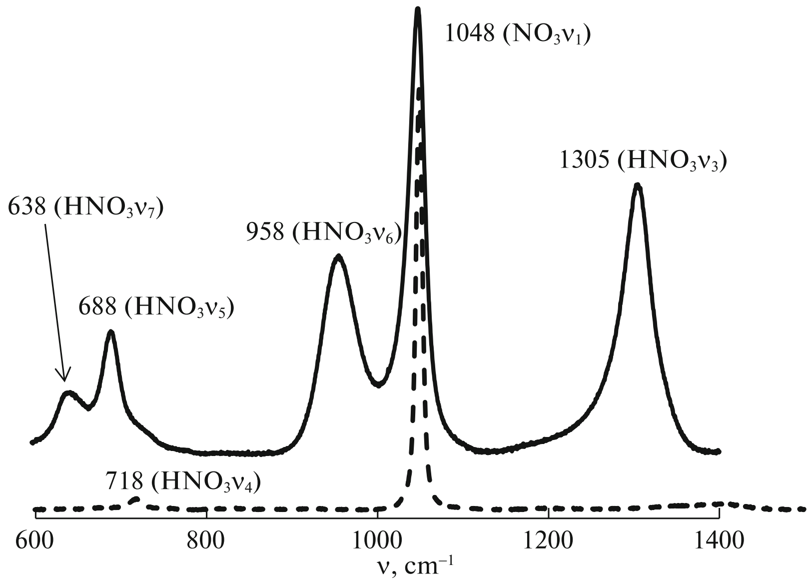

Fig. 1. Raman spectra of 2.4 M aqueous solution of KNO_3 (dashed) and 14.5 M aqueous solutions of nitric acid (solid).

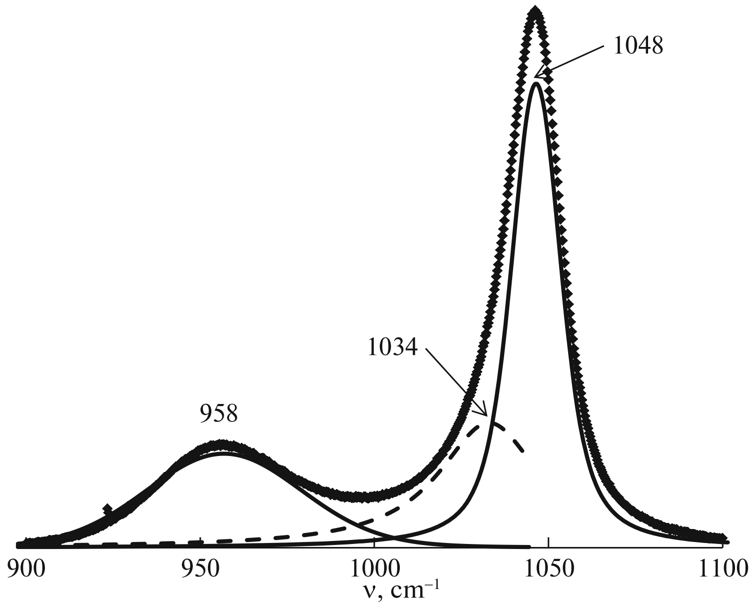

Fig. 2. Decomposition of the complex signal into component peaks in the spectra of nitric acid aqueous solutions in the range of 900–1100 cm^{-1}.

the linear dependence become noticeable because of incomplete dissociation (Fig. 3), the concentration of the free nitrate ion was found using the equation $[NO_3^-] = S_{1048 + 1034}/k$. The concentration of undissociated HNO_3 molecules was calculated under the conditions of material balance: $[HNO_3] = C_{HNO_3} - [NO_3^-]$. In addition, the concentration of free ions and undissociated HNO_3 molecules and/or the degree of dissociation of the nitric acid in the solution were determined using critically selected literature data.

The activity coefficients of nitric acid were taken from the literature. In [39, 40] we can find the mean ionic activity coefficients of nitric acid as a strong electrolyte (assuming the complete dissociation of HNO_3 molecules into H^+ and NO_3^- ions) for an asymmetric version of the standard state in the concentration scales of molality [40] ($m(HNO_3) = 0–28$ mol/kg) and

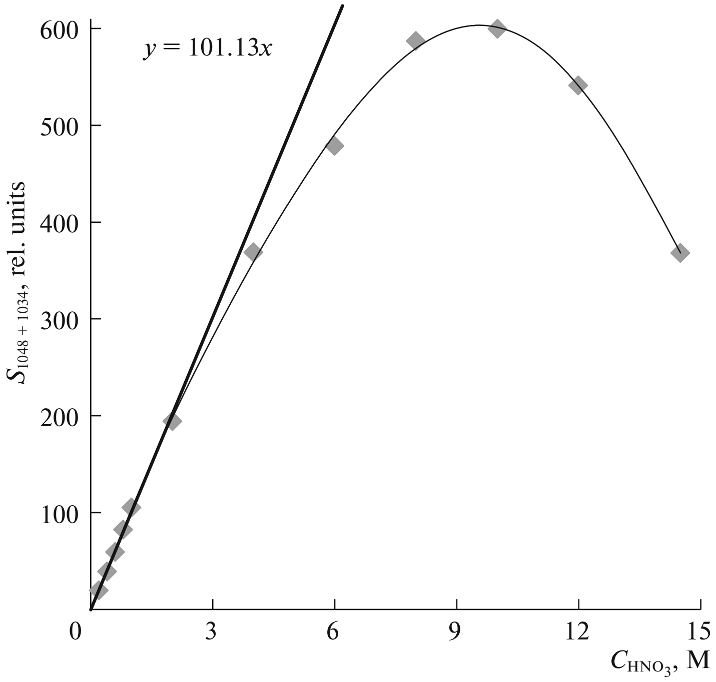

Fig. 3. Dependence of the peak area of the free nitrate ion on the stoichiometric concentration of nitric acid in a series of experiments in this work.

mole fraction [39] ($x(HNO_3) = 0{-}1$). In this work, the molar volume concentration (molarity) was used in units of mol/L (M) to express the composition of solutions. The activity coefficients were converted to molar scale using the familiar equations in [34]; the density of nitric acid aqueous solutions was taken from [41]. To express the mean ionic activity coefficients of HNO_3 (assuming complete dissociation) in the range of 0–18 M at 25°C using data from [39, 40] we derived the interpolation equation

$$\begin{aligned}\log\gamma_{\pm} = &-\frac{0.51156\sqrt{C_{HNO_3}}}{1+1.5847\sqrt{C_{HNO_3}}} \\ &+ 4.3296\times10^{-2}C_{HNO_3} \\ &+ 8.5493\times10^{-3}(C_{HNO_3})^2 \\ &- 1.0181\times10^{-3}(C_{HNO_3})^3 \\ &+ 5.9345\times10^{-5}(C_{HNO_3})^4 \\ &- 9.0342\times10^{-7}(C_{HNO_3})^5,\end{aligned} \tag{2}$$

where C_{HNO_3} is the stoichiometric concentration of nitric acid in M.

Let us describe the method for determining the dissociation constant of nitric acid K_a, the activity coefficients of the undissociated molecules (γ'_{HNO_3}), and the mean ionic one ($\gamma'_{\pm} = \sqrt{\gamma'_{H^+}\gamma'_{NO_3^-}}$). The prime mark means that these coefficients are determined by assuming balance conditions (1), i.e., the incomplete dissociation of nitric acid. Coefficient $\gamma_{\pm}$, calculated according to Eq. (2) using the reference data, is determined by assuming the full dissociation of HNO_3. According to [34], we obtain the relation

$$\gamma_{\pm} = \alpha\gamma'_{\pm}, \tag{3}$$

where α is the degree of dissociation ($\alpha = [H^+]/C_{HNO_3} = [NO_3^-]/C_{HNO_3}$). The thermodynamic dissociation constant of nitric acid is determined using the equation

$$K_a = \frac{[H^+][NO_3^-](\gamma'_{\pm})^2}{[HNO_3]\gamma'_{HNO_3}}, \tag{4}$$

where $[H^+]$, $[NO_3^-]$, and $[HNO_3]$ are the real concentrations of H^+, NO_3^-, and HNO_3 particles in the solution, expressed in M. Substituting expressions $[H^+] = \alpha C_{HNO_3}$ and $[NO_3^-] = \alpha C_{HNO_3}$ into Eq. (4) and allowing for Eq. (3), we obtain the relation

$$K_a = \frac{(C_{HNO_3}\gamma_{\pm})^2}{[HNO_3]\gamma'_{HNO_3}},$$

which can be transformed into

$$K_a\gamma'_{HNO_3} = \frac{(C_{HNO_3}\gamma_\pm)^2}{[HNO_3]}. \qquad (5)$$

The right side of Eq. (5) is calculated using the experimental concentrations of undissociated HNO_3 and reference values of $\gamma_\pm$ (Eq. (2)). The product of $(K_a\gamma'_{HNO_3})$ is therefore a fully-defined quantity. In an infinitely dilute solution, $\lim_{C_{HNO_3}\to 0}\gamma'_{HNO_3} = 1$, so $\lim_{C_{HNO_3}\to 0}(K_a\gamma'_{HNO_3}) = K_a$. To find thermodynamic dissociation constants K_a, we must therefore plot the dependence of $(K_a\gamma'_{HNO_3})$ on C_{HNO_3} using the experimental and reference data and extrapolate it to $C_{HNO_3} \to 0$. If the value of K_a is known, the activity coefficient can be determined from the dependence of $(K_a\gamma'_{HNO_3})$ on C_{HNO_3} as a function of the stoichiometric concentration of nitric acid. The dependence of $\gamma'_\pm$ on C_{HNO_3} can be obtained using Eq. (4) and the known values of K_a, γ'_{HNO_3}, and the concentrations of $[H^+]$, $[NO_3^-]$, and $[HNO_3]$.

The principle behind this method is rather obvious and has been used before [5, 15]. We now have a substantial amount of original and published data on the dissociation of nitric acid, allowing us to perform a full critical analysis and obtain the most reliable estimates of $HNO_3(aq)$ thermodynamic characteristics.

RESULTS AND DISCUSSION

Figure 4 shows the concentrations of NO_3^- free ions as a function of the stoichiometric concentration of our HNO_3 aqueous solution. Along with the results of this work, we present the most reliable and recognized data in [7] (this work was performed under the supervision of Prof. T.F. Young; the concentrations were determined by RS), [16] (the final work of a series of studies conducted under the supervision of Prof. Redlich in 1943–1968 via NMR), [18] (RS studies), and the results from recent works [21, 22, 42] (RS studies). We can see that all of the data are in good agreement with one another, but are characterized by noticeable errors. Even if a random data error in any work is minor, a comparison with the results of other authors thus shows there are systematic errors. Note that in [18, 21, 22], the concentration of nitrate was calculated by assuming there were $H^+\cdots NO_3^-$ ion pairs in solutions, along with H^+, NO_3^-, and HNO_3. We may conclude from Fig. 4 that in light of the error, this assumption does not affect the value of the concentration of NO_3^- (or those of H^+ and HNO_3) and it is therefore not considered in this work.

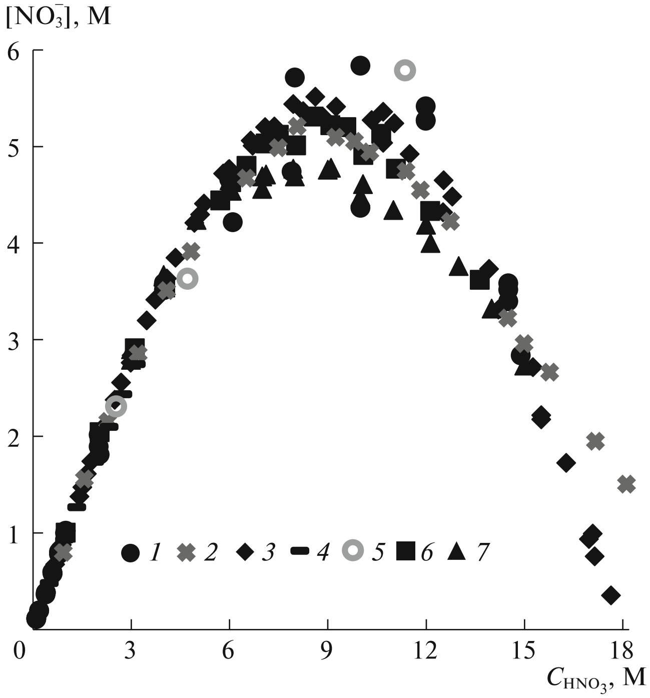

Fig. 4. Dependences of the concentration of free nitrate ions on the stoichiometric concentration of nitric acid aqueous solutions: (*1*) this work, 22–23°C; (*2*) [7]; (*3*) [16]; (*4*) [18]; (*5*) [42]; (*6*) [21]; (*7*) [22]. The literature data are for a temperature of 25°C.

Based on the data in Fig. 4, we calculated the values of the $(K_a\gamma'_{HNO_3})$ product using Eqs. (2) and (5). We used the NO_3^- concentrations obtained in this work at $C_{HNO_3} \geq 4$ M to perform our calculations, since the values at $C_{HNO_3} \leq 2$ M were used to plot the calibration curve. With $C_{HNO_3} < 4$ M, these values lead to great errors in determining the concentration of the undissociated molecules of HNO_3. For the same reason, we did not include data from [7] corresponding to $C_{HNO_3} < 4$ M, or any of the data from [42] (three values). The results from [16, 21, 22] were taken without exception. We did not use the results from [18], since they contradict the fairly obvious idea accepted in this work of an strict rise in the activity coefficient of the undissociated nitric acid molecule γ'_{HNO_3} as C_{HNO_3} grows. The resulting dependence of $\log(K_a\gamma'_{HNO_3})$ on C_{HNO_3} is shown in Fig. 5. It should be noted that there is very good agreement between the values obtained using results of various authors.

For reliable extrapolation of $(K_a\gamma'_{HNO_3})$ to the area of infinite dilution, it is important to know the type of dependence between γ'_{HNO_3} and C_{HNO_3}. It is known that at fairly low ionic strengths I, the activity coefficients of nonelectrolytes γ_{NE} in electrolyte solutions

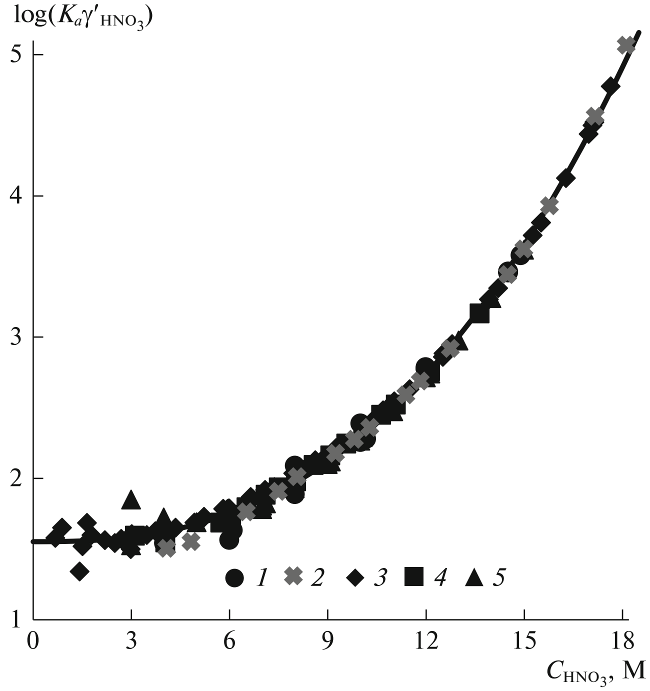

Fig. 5. Dependences of the common logarithm of the $K_a\gamma'_{HNO_3}$ product on the stoichiometric concentration of nitric acid: (*1*) this work, 22–23°C; (*2*) [7]; (*3*) [16]; (*4*) [21]; (*5*) [22].

are described by the Sechenov equation $\log\gamma_{NE} = \kappa_S I$, where κ_S is Sechenov's coefficient [43, 44]. Most of these solutions are characterized by salting out, so $\kappa_S > 0$. In this work, we used a more general form of the dependence,

$$\log\gamma'_{HNO_3} = B(C_{HNO_3})^n, \tag{6}$$

and was considered that the function in Eq. (6) must rise strictly. According to Eq. (6), the dependence of the logarithm of the product ($K_a\gamma'_{HNO_3}$) on the stoichiometric concentration of nitric acid should be described by the equation

$$\log(K_a\gamma'_{HNO_3}) = A + B(C_{HNO_3})^n. \tag{7}$$

Parameters *A*, *B,* and *n* are determined from the condition that the theoretical curve matches the experimental values, calculated from Eq. (5). One criterion of the validity of Eqs. (6) and (7) is the possibility of presenting the dependence of $\log(K_a\gamma'_{HNO_3})$ on $(C_{HNO_3})^n$ as a straight line, while the correct value of *n* corresponds to the highest correlation coefficient.

It turns out that the dependence between $\log(K_a\gamma'_{HNO_3})$ and $(C_{HNO_3})^n$ can be plotted by a straight line (see Fig. 6); the optimum value of *n* is 2.57 (R^2 = 0.9953), while $A = 1.552 \pm 0.014$, and $B = (1.996 \pm 0.025) \times 10^{-3}$. The uncertainty intervals correspond to a 95% confidence level. The line in Fig. 5, calculated according to Eq. (7) with the aforementioned parameters, approximates the experimental results very well. Since $A \equiv \log K_a$, we obtain the value of the dissociation constant of nitric acid:

$$K_a = 35.5 \pm 1.5. \tag{8}$$

We believe that the value in Eq. (8) is the most reliable estimate of the thermodynamic dissociation constant of HNO_3 in aqueous solution at 25°C, since it relies on a large set of data from different researchers, obtained in careful experiments and consistent with one another.

Inserting the values of parameters *B* and *n* into Eq. (6), we obtain the expression for the activity coefficient of the HNO_3 undissociated molecules at 25°C:

$$\begin{gathered}\log\gamma'_{HNO_3} = 1.996\times10^{-3}(C_{HNO_3})^{2.57} \\ (C_{HNO_3} = 0\text{–}18\ \mathrm{M}).\end{gathered} \tag{9}$$

Equation (4) allows us to calculate mean ionic activity coefficient $\gamma'_\pm$ depending on C_{HNO_3} (based on the concentrations of $[H^+]$, $[NO_3^-]$, and $[HNO_3]$, which are determined using the data in Fig. 4), the values of K_a from Eq. (8), and γ'_{HNO_3} from Eq. (9). The results are described by the interpolation equation ($R^2 = 0.9982$)

$$\begin{aligned}\log\gamma'_\pm = {} & -\frac{0.51156\sqrt{C_{HNO_3}}}{1+1.5847\sqrt{C_{HNO_3}}} + 7.4259\times10^{-2}C_{HNO_3} \\ & - 7.5724\times10^{-3}(C_{HNO_3})^2 \\ & + 2.9820\times10^{-3}(C_{HNO_3})^3 \\ & - 2.7778\times10^{-4}(C_{HNO_3})^4 \\ & + 8.9028\times10^{-6}(C_{HNO_3})^5 (C_{HNO_3} = 0\text{–}18\,\mathrm{M}).\end{aligned} \tag{10}$$

Note that according to the available information, equations for activity coefficients γ'_{HNO_3} and $\gamma'_\pm$ (analogous to those from Eqs. (9) and (10)) have yet to be published in the literature.

CONCLUSIONS

The value of thermodynamic dissociation constant of nitric acid in an aqueous solution at 25°C was determined by comparing our results and the critically selected data of other authors, and the dependences of the activity coefficients γ'_{HNO_3} and $\gamma'_\pm$ on the stoichiometric concentration of nitric acid in the range of 0–18 M were obtained.

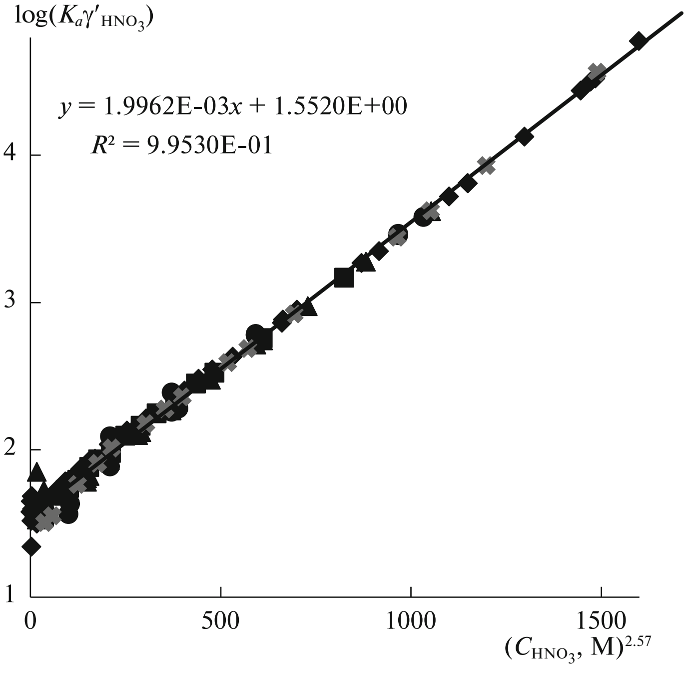

Fig. 6. Straightening of the dependences between $\log(K_a\gamma'_{HNO_3})$ and C_{HNO_3}. The denotation is the same as in Fig. 5.

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