ISSN 0036-0244, Russian Journal of Physical Chemistry A, 2014, Vol. 88, No. 4, pp. 685–690. © Pleiades Publishing, Ltd., 2014.
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The Use of Positron Spectroscopy for Revealing the Nanosized Structures in Liquid Mixtures. Identification of *n*-Propanol Nanoagglomerates in Aqueous Solutions

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Received May 14, 2013

Abstract—The potential of positron spectroscopy for identification of nanosized structures in liquid mixtures, which is a difficult problem that still remains a challenge in physical chemistry of liquids, was demonstrated. The water–*n*-propanol mixtures were chosen as an example. An analysis of the concentration dependences of the mean lifetime of *ortho*-positronium atoms in water–*n*-propanol binary mixtures and water–propanol mixtures containing $CoCl_2$ (0.4 M) and $Co(ClO_4)_2$ (0.6 M) suggested that the water–*n*-propanol mixture with ~0.1 to ~0.4 mole fractions of propanol resembled an emulsion of alcohol nanodrops suspended in water.

Keywords: positronium, water–alcohol mixtures, *n*-propanol agglomerates in water.

DOI: 10.1134/S003602441404027X

INTRODUCTION

In studies of liquids, the question arises whether a mixture of liquids is a real solution or a nanoheterogeneous structure. This question often remains unanswered because of the limitations of modern procedures for investigation. However, identification of nanostructures has become increasingly important in view of the development of nanotechnologies. The problem of nanostructure identification in liquid mixtures can obviously be solved only by combining the traditional and modern physicochemical approaches. Positron spectroscopy is one of the promising methods for this.

This paper demonstrates the potential of positron spectroscopy for identification of the nanoheterogeneous structure of binary mixtures based on positron annihilation data for aqueous propanol solutions reported in [1].

To confirm the positron spectroscopy data, we used additional data on the concentration dependences of viscosity, light scattering intensity, and some other physicochemical properties of aqueous propanol mixtures.

EXPERIMENTAL

Ps atoms are formed in the terminal parts of the tracks of fast positrons injected in condensed media. Every positron that lost its energy is surrounded by a group of approximately 30–40 ion–electron pairs that occupy a spheroid with a diameter of up to 100 Å. This agglomerate of ion–electron pairs is called a blob because of its shape (suspended drop). This term is used in the literature on radiation chemistry and positron spectroscopy. The blob has favorable conditions inside of it for the occurrence of a reaction between the positron and the electrons that were thermalized but not yet solvated [2–5]:[1]

$$e^+ + e^- \rightarrow (3/4)o\text{-Ps} \rightarrow (1/4)p\text{-Ps}. \quad (1)$$

If the reagents react to form a positronium atom, the *ortho*-positronium (*o*-Ps) atom with the same orientation of the positron and electron spins is formed at a probability of 3/4, while the *para*-positronium (*p*-Ps) atom with oppositely directed positron and electron spins is formed at a probability of 1/4. The life of both varieties of the Ps atom ends with two-photon annihilation in condensed media. The positron of the *p*-Ps atom is annihilated with its own electron within ≈0.1 ns on average, while the positron of the *o*-Ps atom prefers to be annihilated with one of the electrons of the surrounding molecules whose spin is opposite to its own one. This so-called pick-off annihilation of *o*-Ps

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[1] In contrast to the quasifree electrons, the solvated electrons do not markedly contribute to Ps formation. Therefore, in polar liquids (water, alcohols, ammonia, etc.), in which the time of positron recombination with track electrons is limited by their solvation, the probability of Ps formation is two or three times lower than in nonpolar liquids (hydrocarbons, dioxane, etc.).

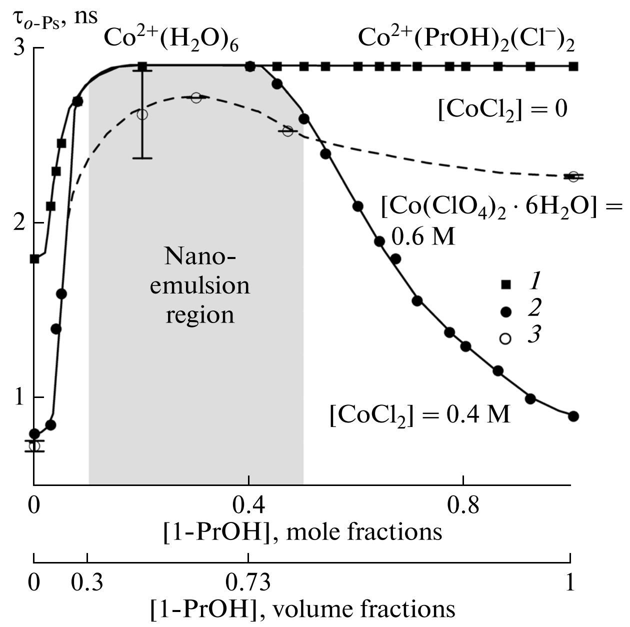

Fig. 1. Variation of the lifetime $\tau_{o\text{-Pr}}$ of o-Ps in (*1*) water–1-propanol binary mixtures, (*2*) water–propanol mixtures with an addition of 0.4 M $CoCl_2$, and (*3*) water–propanol mixtures with an addition of 0.6 M $Co(ClO_4)_2 \cdot 6H_2O$ [1].

occurs within much longer times, from two to two dozen nanoseconds.

The o-Ps annihilation rate increases substantially in solutions with paramagnetic species (molecules or ions) with odd electrons, which can cause the conversion of o-Ps to p-Ps (the reverse process is unlikely because of the much smaller lifetime of p-Ps). For example, when the solution contains Co^{2+} ions, the reaction is [6, 7]

$$o\text{-Ps}(\uparrow\uparrow) + \mathrm{Co}^{2+}(\downarrow) \to p\text{-Ps}(\uparrow\downarrow) + \mathrm{Co}^{2+}(\uparrow). \tag{2}$$

The formation of a microheterogeneous structure in liquid nonpolar media (hydrocarbons) after the addition of polar molecules (e.g., water and alcohols) is generally attributed to the ability of these molecules to form more stable (hydrogen) bonds with one another than with the solvent (hydrocarbon) molecules and to create agglomerates due to this. It is not clear whether the alcohol molecules can form similar agglomerates in such a highly polar medium as water.

The first evidence for the emulsion structure of water–alcohol mixtures based on the specific behavior of their thermodynamic parameters appeared in the late 1930s [8] and were confirmed later [9, 10]. Some physicochemical parameters of the water–1-propanol system were analyzed in [10]; it was concluded that at ~0.1 to ~0.3 mole fractions of 1-propanol, the mixture resembled an emulsion of alcohol "nanodrops" suspended in water.

While the set of given data strongly suggest that 1-propanol molecules can agglomerate in water, it should be borne in mind that the results were obtained by indirect methods. Therefore, it is highly desirable to be able to confirm the existence of agglomerates by another technique. It seemed reasonable to try to give additional arguments in favor of the formation of 1-propanol agglomerates by analyzing the data of [1] on the lifetimes of o-Ps atoms in water–1-propanol binary mixtures and water–propanol mixtures containing the dissolved $CoCl_2$ and $Co(ClO_4)_2$ additives.

RESULTS AND DISCUSSION

Let us trace how the lifetime of *ortho*-positronium $\tau_{o\text{-Ps}}$ in water–1-propanol mixtures changes at increasing mole fraction of alcohol x_{PrOH} (Fig. 1). The change is nonlinear. At first, in the range of mole fractions $0 < x_{PrOH} < 0.1$, $\tau_{o\text{-Ps}}$ increases from the minimum value of 1.8 ns in pure water to the maximum (in this system) value of ~2.9 ns, which is characteristic of the lifetime of o-Ps in pure 1-propanol (at $x_{PrOH} = 1$). Remarkably, the limiting value of $\tau_{o\text{-Ps}}$ is reached already at $x_{PrOH} \simeq 0.1$, which is ten times smaller. Then $\tau_{o\text{-Ps}}$ remains constant as the 1-propanol content increases further to $x_{PrOH} = 1$.

The longer lifetime of o-Ps in alcohol is due to the fact that Ps is a solvophobic particle. The exchange repulsion of its electron from the electron shells of the surrounding molecules leads to the formation of a nanovoid (or nanobubble) around the Ps atom. The radius of the bubble (R_b) is determined by the competition between the repulsive pressure of the Ps atom on the bubble wall and the bubble-tightening counter pressure, $2\sigma/R_b$ (σ is the surface tension of the liquid). The lower the σ value, the larger the Ps bubble and the longer the lifetime of o-Ps because of the weaker contact of its positron with the electrons of the surrounding molecules: $\tau_{o\text{-Ps}} \propto \sigma^{-1/2}$ [11]. The longer lifetime of o-Ps in 1-propanol than in water is direct evidence for the bubble state of the Ps atom in liquid and, as a consequence, for the surface tension of this alcohol that is three times lower than that in water ($\approx$23 erg/cm^2).

What is the reason for $\tau_{o\text{-Ps}}$ quickly reaching the limiting value (~2.9 ns)?

The first reason could be rapid replacement of water molecules on the surface of the Ps bubble by alcohol molecules. Their adsorption decreases the surface tension on the boundary of the bubble, increasing its size and the o-Ps lifetime. However, as follows from the discussion below, this effect is important at high alcohol concentrations but unimportant at low concentrations.

We will try to show that the most plausible reason for the drastic increase in the lifetime of o-Ps is the above-mentioned nanoheterogeneous structure of water–alcohol mixtures. At 0.1–0.4 mole fractions of propanol, the mixture is an emulsion of nanomicelles ("nanodrops") of alcohol suspended in water.

In positron experiments, the emulsion structure of water–propanol solutions is clearly reflected in the character of variation of the lifetime of o-Ps when water and the water–propanol mixtures contain, e.g., some fixed amount (0.4 M) of $CoCl_2$. The paramagnetic Co^{2+} ions effectively interact with *o*-Ps atoms by reaction (2), leading to their conversion into short-lived *p*-Ps atoms. In water, this process leads to a twofold reduction of the lifetime of *o*-Ps. In a mixture with alcohol, however, the conversion rate rapidly decreases and at 0.1–0.4 mole fractions of 1-propanol, the lifetime of *o*-Ps lifetime becomes the same as in pure propanol (table). *o*-Ps here is not affected by the presence of neither Co^{2+} ions, nor water molecules. This means that either the surface of *o*-Ps bubbles is covered with a rather thick layer of adsorbed alcohol molecules impenetrable for Co^{2+} ions or water molecules. At the indicated 1-propanol contents or the *o*-Ps atoms and Co^{2+} ions are distributed in different phases: alcohol micelles and the surrounding water medium, respectively.

The possibility of segregation is caused by the fact that Co^{2+} ions are hydrophilic particles that form stable $Co^{2+}(H_2O)_6$ aqua complexes at propanol contents of 0–0.5 mole fractions [1].

On the other hand, even the simplest model of a Ps bubble in the form of an infinitely deep potential well shows that the sum of the "zero" energy of the Ps atom $E_0 = \pi^2\hbar^2/4m_eR_b^2$ and the surface energy $E_s = 4\pi R_b^2\sigma$ of the positronium bubble $E = (E_s + E_0) \propto \sigma^{-1/2}$ decreases when the Ps atom passes from the water phase to the alcohol medium. Consequently, the alcohol micelles can accept the Ps atoms. If the *o*-Ps atoms that appeared in water starting from $x_{\mathrm{PrOH}} \approx 0.1$ concentrate in micelles within a period of time that is considerably smaller than $\tau_{o\text{-Ps}}$, this prevents a reaction of *o*-Ps with Co^{2+} ions. The validity of this approach to the change in $\tau_{o\text{-Ps}}$ is proven below.

The effect of Co^{2+} ions (Fig. 1) almost completely vanishing within the lifetime of *o*-Ps in the range of 0.1–0.4 mole fractions of 1-propanol is regarded as direct evidence for the emulsion structure of the water–propanol mixture in this range and the impossibility for Co^{2+} ions to penetrate in alcohol micelles.[2]

Let us now answer the natural question about the different behaviors of the lifetime of *o*-Ps at high mole fractions of 1-propanol in the presence and absence of Co^{2+} ions in solution. Based on the aforesaid, it is evident that in mixtures containing Co^{2+} ions, the decrease in $\tau_{o\text{-Ps}}$ at $x_{\mathrm{PrOH}} > 0.4$ should be interpreted as the consequence of the decomposition of the emulsion structure of the mixture and hence of the renewed contacts of Co^{2+} ions with *o*-Ps atoms, leading to their conversion into short-lived *p*-Ps atoms. But if so, then why does not the emulsion decomposition, which certainly occurs in the absence of Co^{2+} ions too, affect the constancy of $\tau_{o\text{-Ps}}$ in the water–1-propanol mixture at $x_{\mathrm{PrOH}} > 0.4$ when the surface of the positronium bubble seems to be also accessible for water molecules? The presence of water molecules would have necessarily affected the surface tension and hence the lifetime of *o*-Ps, but it does not.

Lifetimes of *o*-Ps (ns) in water–propanol mixtures with $CoCl_2$ and $Co(ClO_4)_2 \cdot 6H_2O$ additions (marked with * for the latter) and without them [1]

x_{PrOH}	Binary mixture	0.4 M $CoCl_2$ 0.6 M $(CoCl)_4)_2 \cdot 6H_2O$
0.00	1.80	0.80
0.00	–	0.73 ± 0.03*
0.03	2.10	0.85
0.04	2.30	1.40
0.05	2.46	1.60
0.08	2.70	2.70
0.20	2.90	2.625 ± 0.25*
0.30	2.90	0.72*
0.40	2.90	2.90
0.45	2.90	2.80
0.47	2.90	2.53*
0.50	2.90	2.60
0.54	2.90	2.40
0.60	2.90	2.10
0.64	2.90	1.90
0.67	2.90	1.80
0.71	2.90	1.56
0.77	2.90	1.38
0.80	2.90	1.30
0.86	2.90	1.16
0.92	2.90	1.00
1.00	–	2.27 ± 0.01*
1.00	2.90	0.90

The answer to this question is as follows. When the volume fraction of 1-propanol exceeds 0.73 ($x_{\mathrm{PrOH}} >$ 0.4), the water molecules have no chance to penetrate into the boundary layer of the positronium bubble and thus to markedly affect the surface tension and the lifetime of *o*-Ps. This is hindered by the surface-active 1-propanol molecules lying on the surface of the positronium bubble. The constant lifetime of *o*-Ps at $0.4 < x_{\mathrm{PrOH}} < 1$ is determined by the surface-active properties of the alcohol molecules adsorbed on the surface of the positronium bubble. At the same time, the adsorbed alcohol molecules cannot hinder the electron exchange between the *o*-Ps atom and Co^{2+} ions. The conversion is facilitated by the gradual transformation of the cobalt aqua complexes at $x_{\mathrm{PrOH}} > 0.5$ [1]:

$$Co^{2+}(H_2O)_6 \to Co^{2+}(PrOH)_2(Cl^-)_2. \quad (3)$$

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[2] The micelles can contain nondissociated $CoCl_2$ molecules, which have no odd electrons and do not cause *ortho*,*para*-conversion of Ps.

At high alcohol concentrations, the Co^{2+} aqua complexes lose their charge, incorporating the chloride anions in their solvation shells, and have water molecules replaced by propanol molecules. Both processes provide access to the Ps bubble for Co^{2+} ions.

Here it is reasonable to discuss the effect of the presence of $Co(ClO_4)_2$ in the water–propanol mixture on the lifetime of *o*-Ps. Figure 1 shows that in contrast to $CoCl_2$, the presence of 0.6 M of $Co(ClO_4)_2$ slightly decreases the lifetime of *o*-Ps at $0.1 < x_{PrOH} < 0.4$, where the mixture is an emulsion of 1-propanol nanodrops in a water medium. This means that the Co^{2+} ions of $Co(ClO_4)_2$ penetrate into the 1-propanol nanodrops. This probably occurs because the dissociation of $Co(ClO_4)_2$ is weaker than that of $CoCl_2$. Indeed, if the dissociation of $CoCl_2$ molecules requires that the total solvation energy of the Co^{2+} cation and two Cl^- anions should be higher than the Cl–Co–Cl bond cleavage energy, then the dissociation of the $Co(ClO_4)_2$ molecule requires that the total solvation energy of the Co^{2+} cation and two $(ClO_4)^{2-}$ anions should exceed the ClO_4–Co–ClO_4 bond cleavage energy. This is a much more stringent requirement because the radius of the $(ClO_4)^{2-}$ anion is almost two times (more exactly, 1.7 times) larger than the radius of the Cl^- anion. The nondissociated $Co(ClO_4)_2$ molecules probably penetrate into the 1-propanol nanodrops and dissociate there into ions, causing the conversion of *o*-Ps into *p*-Ps, though, certainly, to a smaller degree than in water.

Referring to the emulsion structure of water–alcohol solutions above, we always meant that alcohol micelles were nanosized. The estimations below confirm this.

As is known, each Ps atom is formed at first in a quasifree, highly delocalized state. After approximately 50–100 ps, the quasifree Ps forms a nanobubble. The transformation occurs on a nanovoid or another appropriate inhomogeneity. Suppose the ratio between the aqueous and alcohol components is such ($0 < x_{PrOH} < 0.4$) that the mixture acquires a micellar structure. The probability of quasifree and bubble states of Ps in the aqueous and alcohol phases is believed to be proportional to the volume fraction of the corresponding component.

The motion of the Ps bubble in a liquid has the character of diffusion with a coefficient (in the Rybchinski–Hadamard approximation)

$$D_{Ps}^{RH} = \frac{k_B T}{4\pi a_{Ps}\eta}, \tag{4}$$

where k_B is Boltzmann's constant, and a_{Ps} is the radius of the Ps bubble. In water, $a_{Ps} = 3.2$ Å [3] and $D_{Ps}^{RH} \approx 1 \times 10^{-5}$ cm^2/s.

On the other hand, the diffusion coefficient can be determined as $D_{Ps} = vl_{tr}/3$, where l_{tr} is the transport length, i.e. the distance to which the Ps bubble is transferred and at which its velocity v changes direction to $\pi/2$. Evidently, l_{tr} is comparable to the radius of the bubble a_{Ps} in liquid. Equating the two relations for the diffusion coefficient, we determine the rate of the Ps bubble in water, $v_{Ps} \approx 3D_{Ps}^{RH}/a_{Ps} \approx 10$ m/s.

Let us divide the whole volume of the micellar mixture into spheroidal cells such that each cell contains only one alcohol micelle. Suppose R_{cell} is the average radius of the cell, and a_{mic} is the average radius of the micelle ($R_{cell} > a_{mic}$). The average time τ_{dif} required for a Ps bubble that appeared and diffused in the water phase to meet one of the surrounding alcohol micelles can be evaluated as $\tau_{dif} \approx R_{cell}^2/6D_{Ps}^{RH}$.

Each moving Ps bubble covers the volume $\pi(a_{Ps} + a_{mic})^2 v_{Ps}\tau_{dif} \approx \pi a_{mic}^2 v_{Ps}\tau_{dif}$ during the time τ_{dif}. If this volume approximately equals the average volume $(4/3)R_{cell}^3$ that contains one micelle, we can consider that during the time τ_{dif} the *o*-Ps atom will encounter one of the micelles and pass into the alcohol phase. Equating the two volumes and using the evident equation for the volume fraction of alcohol $\phi_{PrOH} = (a_{mic}/R_{cell})^3$, we have

$$R_{cell} \approx (24/9)\phi_{PrOH}^{-2/3} a_{Ps}. \tag{5}$$

Assuming that the micellar structure appears when $\phi_{PrOH} \approx 0.3$ (Fig. 1), we determine $R_{cell} \approx 18.6$ Å and $a_{mic} \approx 12.6$ Å and the residence time of *o*-Ps in the aqueous phase is $\tau_{dif} \approx 0.6 \times 10^{-9}$ s.

The τ_{dif} value is five times smaller than the lifetime of *o*-Ps (2.9×10^{-9} s) as it should be. Dividing the average volume of the micelle (8300 Å^3) by the volume of the propanol molecule (120 Å^3), we obtain that the average micelle contains about 70 alcohol molecules. The given estimates are certainly valid only in the order of magnitude.

To confirm the conclusions made with the use of positron spectroscopy, it is helpful to use additional independent data on the concentration dependences of viscosity, light scattering intensity, and reactivity of solvated electrons in the solutions under study.

The nonmonotonous character of the concentration dependence of viscosity of the water–propanol mixture is an independent proof of the emulsion structure with alcohol nanodrops in the aqueous propanol solution in the range $\sim 0.1 < x_{PrOH} < \sim 0.4$. The viscosity reaches maximum exactly in this range of alcohol concentrations (Fig. 2).

Indeed, the existence of a nanoemulsion with alcohol nanodrops should manifest itself as increased viscosity of the water–alcohol mixture. Einstein derived a relation for the viscosity η of a suspension of spherical particles depending on their volume fraction ϕ ($\phi \ll 1$) [13]. For our case,

$$\frac{\eta(\phi_{PrOH})}{\eta_{H_2O}} = 1 + A\phi_{PrOH} = 1 + \frac{5}{2}\phi_{PrOH}. \tag{6}$$

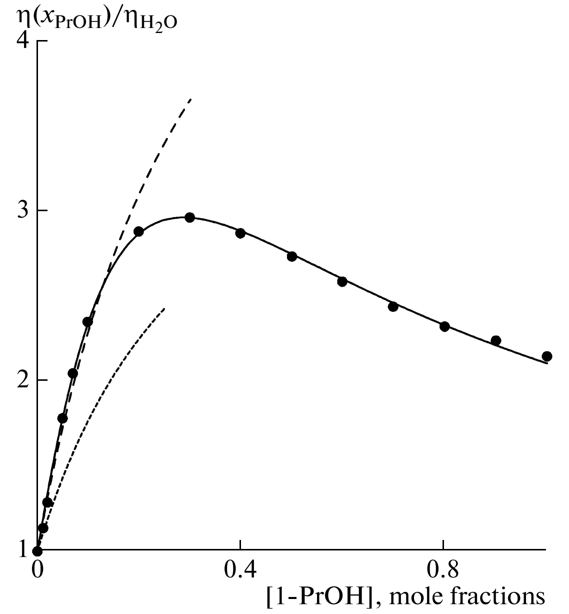

Fig. 2. Dependence of the relative viscosity of the water–*n*-propanol mixture $\eta(x_{\mathrm{PrOH}})/\eta_{\mathrm{H_2O}}$ on the mole fraction of propanol at 298 K [12]. Dotted line: viscosity predicted by Eq. (6) with the numerical coefficient $A = 2.5$. Dashed line: viscosity predicted by the same equation with $A = 4.2$.

Taking into account the fourfold excess of the volume of the PrOH molecule over that of the H_2O molecule, from (6) we obtain a relation that allows us to predict the increase in the relative viscosity of the water–propanol mixture, $\eta(x_{\mathrm{PrOH}})/\eta_{\mathrm{H_2O}}$ at low x_{PrOH} values (Fig. 2). For better coincidence of the theoretical curve and the experimental data, the numerical coefficient $A = 2.5$ for spherical particles should be increased to 4.2 for flattened and elongated rotation ellipsoids (Fig. 3, [13]), which are more suitable for modeling the emulsion particles subjected to continuous deformation by the solvent. Satisfactory agreement between the theoretical prediction and the experimental data confirms the above conclusion about the emulsion structure of the water–propanol mixture.

Another argument in favor of the nanoemulsion structure of the solution in the range of 1-propanol concentrations $0.1 < x_{\mathrm{PrOH}} < 0.4$ is the presence of a wide peak of light scattering intensity with a maximum at $x_{\mathrm{PrOH}} \approx 0.2$ (Fig. 4). The appearance of this peak was attributed in the literature to wide fluctuations of the concentration of the mixture components [14].[3] This view is quite consistent with our concept about the emulsion structure of the solution.

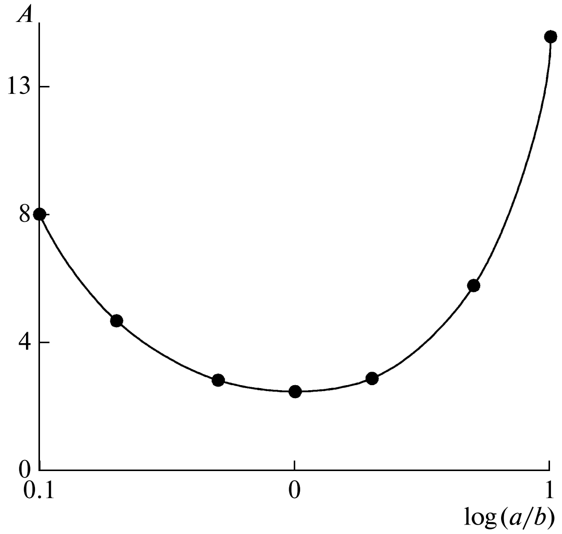

Fig. 3. Numerical values of the correction coefficient A in Einstein equation $\eta/\eta_0 = 1 + A\phi$ (6) for suspension with particles in the form of rotation ellipsoids depending on the a/b ratio (a and $b = c$ are the ellipsoid half-axes [13].

The extremum character of the dependence of the rate of the reaction of the solvated electron with oxygen on the alcohol concentration in aerated aqueous solutions of methanol and ethanol found by pulse radiolysis is another evidence for the emulsion structure of water–alcohol solutions [15]. According to Fig. 5, the rate constant of the reaction

$$e_s^- + O_2 \rightarrow O_2^-$$

at methanol and ethanol contents of 0.2–0.5 mole fractions has the smallest value (the same in methanol and ethanol) and 1.5 times lower than in pure water,

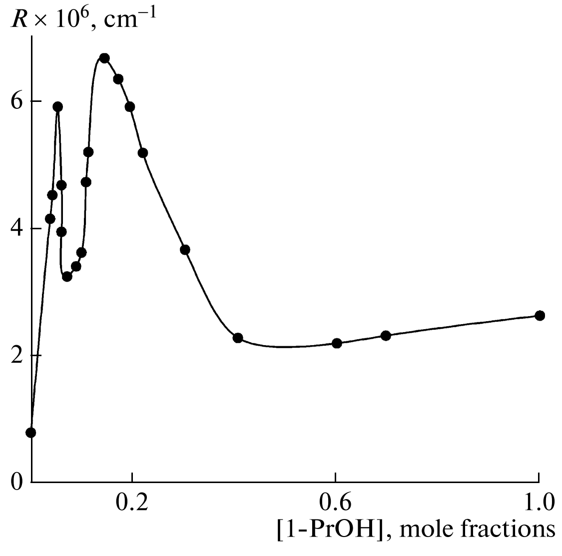

Fig. 4. Variation of the intensity of the concentration light scattering in water–1-propanol mixtures [14].

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[3] Another (narrow) peak near $x_{\mathrm{PrOH}} \approx 0.05$ is not related to these fluctuations according to [14].

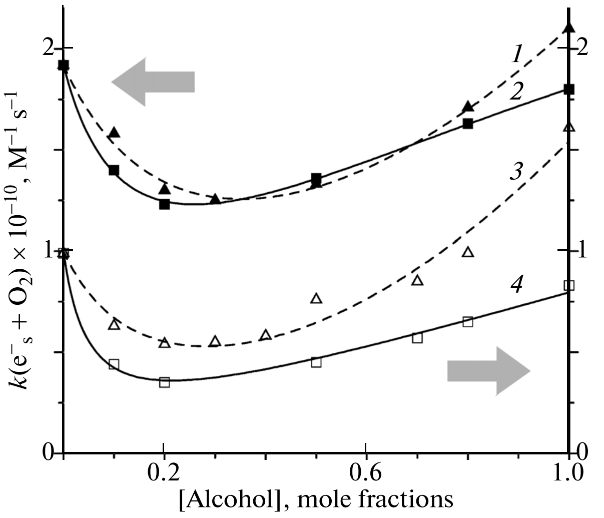

Fig. 5. The *k* coefficient of the reaction rate of the solvated electron with oxygen in aerated aqueous solutions of (*1*) methanol and (*2*) ethanol and the dependences of the relative viscosity of these solutions on the mole fraction of (*3*) methanol and (*4*) ethanol [15].

though the viscosities of the solutions differ in this range of concentrations. The reason for this seems to be partial segregation of the solvated electrons e_s^- and O_2 molecules similar to the segregation of Co^{2+} ions and *o*-Ps atoms in 1-PrOH solutions. The solubility of oxygen in alcohols is higher, while solvated electrons concentrate in the aqueous phase. Water around the alcohol nanomicelles is a drain for radiolytic quasifree electrons that lost their energy because the minimum energy of the bottom of the conduction band in water is lower than in alcohols: $V_0(H_2O) \approx -1.3$ eV, $V_0(MeOH) \approx -0.7$ eV, and $V_0(EtOH) \approx -0.5$ eV [16]. Having approached the inner surface of the alcohol nanomicelle, the electron migrates to the water phase surrounding the micelle. To get into the micelle from the water phase, it has to overcome the potential barrier ~0.5 eV.

CONCLUSIONS

To summarize, the above discussion of experiments on positron annihilation in water–1-PrOH systems in the presence of the paramagnetic Co^{2+} ions gives sound arguments in favor of the existence of a nanoemulsion structure of the solution consisting of micelles of the polar 1-propanol molecules in the polar water solvent at $\sim 0.1 < x_{PrOH} < \sim 0.4$.

The independent arguments for the existence of a nanoemulsion structure at $\sim 0.1 < x_{PrOH} < \sim 0.4$ are the increased viscosity of the water–alcohol mixture and the decreased rate constants of the reaction of the solvated electron with dissolved oxygen.

ACKNOWLEDGMENTS

This study was financially supported by the Russian Foundation for Basic Research, project no. 08-03-00571 and Rosatom.

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Translated by L. Smolina