

THE BOUGUER-LAMBERT-BEER ABSORPTION LAW. SPECTROPHOTOMETRY IN ELECTROLYTE SOLUTIONS

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Abstract

The Bouguer—Lambert—Beer absorption law is one of the key laws in spectrophotometric research. Over the last few decades, the researchers have pointed out a number of factors that influence the validity of this law. The paper shows that for the weak electrolytes with the formal linear dependence of the absorbance on the concentration $A = f(C)$ with the correlation coefficient $r \approx 0.99$ and more, a major deviation of the experimentally determined molar absorption coefficient ε_{obs} from the Bouguer—Lambert—Beer Absorption Law can be observed, and it depends on the concentration of the electrolyte solution.

The article presents experimental and calculated data illustrating the reasons that lead to the instability of the molar absorption coefficient $\varepsilon_{\text{obs}} \neq \text{const}$ in the solutions with the concentration of less than 10^{-3} mol/l. It is suggested that, for precise spectrophotometric measurements, the $\varepsilon_{\text{obs}} = f(C)$ dependence, rather than $A = f(C)$, is the most informative one. First time a theoretical model is offered, that reliably describes the $\varepsilon_{\text{obs}} = f(C)$ dependences for the electrolytes of various strengths, which can be instrumental for the further, more detailed studies and analyses of equilibrium in electrolyte solutions, using the innovative method of concentration spectrophotometry.

Keywords: Bouguer—Lambert—Beer Absorption Law, UV/Vis spectrophotometry, strong and weak electrolytes, molar absorption coefficient.

Introduction

The unified Bouguer—Lambert and Beer Law was first presented in 1913 [1, 2]:

$$\log\left(\frac{I_0}{I}\right) = A = \varepsilon \cdot C \cdot l, (1)$$

where ε is the molar absorption coefficient, A is the absorbance, C is the concentration of solution, l is the optical path length.

Ever since, the semi-empirical Bouguer—Lambert—Beer Law (BLB Law) has been productively used as one of the fundamental admissions in optical spectroscopy and, in particular, in absorption spectrometry [3–5]. The main expectation of the researchers is that the BLB Law offers a reliable description of the effects produced by the interaction of light and matter [1, 6, 7]. With the lack of any theoretical arguments against its validity, the BLB Law is used as the basis of the mandatory procedure of establishing the photometric linearity for the spectrophotometers, based on the relationship $A = f(C)$ [8].

And yet, the validity of the BLB Law depends on the conditions of the experiment [9]; thus, it can be rendered invalid when:

- (a) the source of light is far from monochromatic;
- (b) the environment has a prominent dissipating ability;
- (c) the concentrations of the analyzed compounds are very high.

Besides, it is believed that the non-linearity of the $A = f(C)$ dependence might be caused by the chemical interaction between the particles of the analyzed compound (processes of dissociation, dimerization, aggregation) [10], i. e. for the BLB Law to remain valid at a given wavelength, only the particles of a certain type should absorb, with no side chemical reactions that change the concentration of the absorbing compounds being in place.

As the result of dissociation, several particles that have various absorption spectra might be present in the weak electrolyte solutions. Thus, an attempt is made either to suppress dissociation (the way it is done in analyzing the compounds by the HPLC method [11–13]) or to fixate it by using buffer systems, as done in conducting the traditional spectrophotometric pH-titration for

determining the dissociation constants pK_a^T [14–16]. In other words, the researchers have agreed on using just the spectrophotometric data received for the systems where the $A = f(C)$ dependence is linear, and thus $\varepsilon = const$ does not depend on the concentration of the compound in the solution.

We have earlier presented our results on using the concentration spectrophotometry method for determining the thermodynamic dissociation constants pK_a^T for the compounds of various structuring and strength in water and organic solutions [17–20]. The method offers several advantages over the standard spectrophotometric pH-titration:

- no need to measure the pH environment values;
- no need to use buffer systems with the constant ionic strength;
- no need to introduce the adjustments for the ionic strength, using the Debye–Hückel, Stokes—Robinson equations, etc.;
- values of the molar absorption coefficients for the neutral and ionized forms can be obtained without the use of the concentrated acid and alkaline solutions;
- accurate values of the thermodynamic dissociation constants can be obtained for the compounds with the poor spectral resolutions of the prototropic forms, which is always a challenge in the spectrophotometric pH-titration.

While conducting spectrophotometric measurements, we constantly ran into a situation when the experimentally determined molar absorption coefficient ε_{obs} of a weak electrolyte would not have a constant value and would change depending on the concentration of the analyzed compound, with the $A = f(C)$ being almost linear with the correlation coefficient $r \approx 0.99$. It should be pointed out that the analyzed concentrations in many cases would not exceed 10^{-3} mol/l. For the solutions of this type, with very low ionic strength ($I \rightarrow 0$), intermolecular interactions and association are routinely neglected, and the solutions are treated as close to ideal.

Thus, in this paper we have analyzed this phenomenon and, using the electrolyte dissociation as the case study, we have shown, both theoretically and experimentally, that in the solutions with the ongoing dissociation process, the BLB Law loses its validity for the mixture of particles, but remains valid for each

type of particle. We have also shown the importance of checking the validity of the BLB Law not only in the coordinates of the dependence of absorbance A on concentration C , but in the coordinates of the ε_{obs} dependence on C as well; we have suggested a theoretical model that allows to reliably describe the dependence of $\varepsilon_{\text{obs}} = f(C)$ for the electrolytes of various strengths.

Theory

As the result of the dissociation and ionization processes (e. g. formation of ions in protonation of bases), more than one particle is formed in the electrolyte solutions, and their spectra might differ. Thus, in measuring the intensity of the passing light, it's imperative to take into account that it is being absorbed by a mixture of particles, where each one has its own spectrum and its own molar absorption coefficient. As the concentration changes, the ratio of the quantity of the electrolyte forms, with the equilibrium between them, varies, and for the extremely diluted solutions it is determined by the Ostwald's Dilution Law:

$$K_a^T = \frac{C \cdot \alpha^2}{1 - \alpha}, (2)$$

where K_a^T is the thermodynamic dissociation constant, α is the degree of the electrolyte dissociation, C is its molar concentration.

With the concentration values being in the same order as the value of K , the ratio of the various forms of electrolyte will markedly depend on the changes of the electrolyte content in the solution.

The additivity of light absorption law states that in the absence of interaction between the absorbing components, the absorbance is described as:

$$A_{\text{cm}} = \sum A_i = \sum \varepsilon_i \cdot C_i \cdot l, (3)$$

And for the binary electrolyte with the uniform optical path length it looks like:

$$A = (x \cdot \varepsilon_1 + (1 - x) \cdot \varepsilon_2) \cdot C \cdot l, (4)$$

where C is the concentration of the solution, x is the molar share of 1 component.

As applied to the 1,1-valency electrolytes that dissociate in the solution, the expression would look like:

$$A = (\alpha \cdot \varepsilon_{\pm} + (1 - \alpha) \cdot \varepsilon_0) \cdot C \cdot l, (5)$$

where ε_{\pm} and ε_0 are the molar absorption coefficients for the dissociated form and the neutral non-dissociated form of the electrolyte. In accordance with the BLB Law, we can find with the expression for the observed molar absorption coefficient for the mixture, ε_{obs} :

$$A = \varepsilon_{obs} \cdot C \cdot l = (\alpha \cdot (\varepsilon_{\pm} - \varepsilon_0) + \varepsilon_0) \cdot C \cdot l. (6)$$

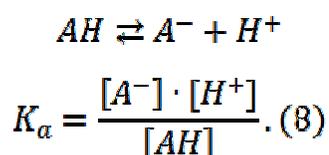
Thus,

$$\varepsilon_{obs} = \varepsilon_0 + \alpha \cdot (\varepsilon_{\pm} - \varepsilon_0). (7)$$

In accordance with Ostwald's Dilution Law, the dissociation degree α does not have a constant value, being dependent on the electrolyte concentration C , and thus ε_{obs} does not have a constant value either, depending on concentration.

Below is the theoretical model of calculating ε_{obs} for the water solutions of acids and bases of various strengths.

The process of the acid dissociation, as well as its equilibrium constant, can be presented as:



Balanced concentrations of the dissociated and the neutral form of the acid can be expressed through its dissociation degree α :

$$[A^{-}] = [H^{+}] = \alpha \cdot C (9)$$

$$[AH] = (1 - \alpha) \cdot C.$$

For the weak acids, where the following correlation is true:

$$K_a \gg \sqrt{K_w},$$

where K_w is the solvent's autoprotolysis constant, the solvent dissociation might be treated as negligible, and thus the expression for calculating the dissociation degree is obtained:

$$\alpha = \frac{K_a}{2 \cdot C} \cdot \left(\sqrt{1 + \frac{4 \cdot C}{K_a}} - 1 \right). \quad (10)$$

For the weak acid solution ε_{obs} can be defined, based on (7):

$$\varepsilon_{obs} = \varepsilon_0 + \frac{K_a}{2 \cdot C} \cdot \left(\sqrt{1 + \frac{4 \cdot C}{K_a}} - 1 \right) \cdot (\varepsilon_- - \varepsilon_0). \quad (11)$$

For the very weak acids, where the following correlation is true:

$$K_a \ll \sqrt{K_w},$$

the solution would predominantly contain only the neutral form, and ε_{obs} would not change and would be equal to the molar absorption coefficient of the acid's neutral form:

$$\varepsilon_{obs} \rightarrow \varepsilon_0.$$

Dissociation of protonated base and its equilibrium constant could be expressed as:

$$BH^+ \rightleftharpoons B + H^+$$

$$K_{BH^+} = \frac{[B] \cdot [H^+]}{[BH^+]} = \frac{1 - \alpha}{\alpha} \cdot [H^+] = \frac{1 - \alpha}{\alpha} \cdot \frac{K_w}{\alpha \cdot C} \quad (12)$$

$$\frac{K_w}{K_{BH^+}} = \frac{\alpha^2 \cdot C}{1 - \alpha}.$$

For the weak bases, where the following is true:

$$K_{BH^+} \ll \sqrt{K_w},$$

we obtain, based on (7), the following expression for calculating ε_{obs} :

$$\varepsilon_{obs} = \varepsilon_0 + \frac{K_w}{2 \cdot C \cdot K_{BH^+}} \cdot \left(\sqrt{1 + \frac{4 \cdot C \cdot K_{BH^+}}{K_w}} - 1 \right) \cdot (\varepsilon_+ - \varepsilon_0). \quad (13)$$

For the very weak bases, where the following correlation is true:

$$K_{BH^+} \gg \sqrt{K_w},$$

the solution would predominantly contain the neutral form of the base's molecular, and thus:

$$\varepsilon_{obs} \rightarrow \varepsilon_0.$$

The calculations are based on the BLB Law and on the surmise that, for each individual particle in the weak electrolyte solution, the $A = f(C)$ dependence is linear, and $\varepsilon = const$. Since the analyzed system is a mixture of particles, certain deviations from linearity might be observed in depicting the $A = f(C)$ dependence, as shown in Figure 1.

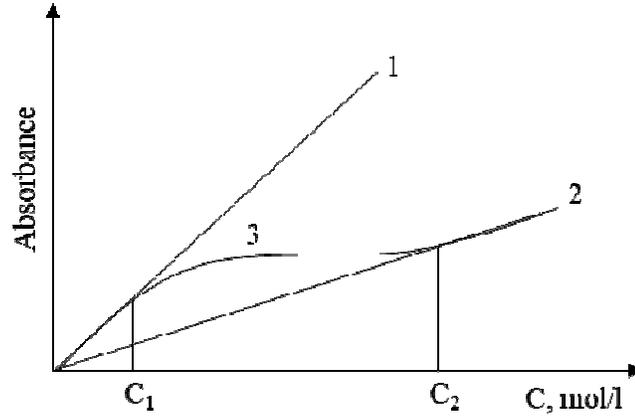


Fig. 1 Absorbance dependence on concentration:

- 1) fully dissociated electrolyte: $A = \varepsilon_{\pm} \cdot C \cdot l$,
- 2) electrolyte with very low dissociation: $A = \varepsilon_0 \cdot C \cdot l$,
- 3) weak electrolyte, with A calculated by formula (6)

Curve 3 describes the $A = f(C)$ dependence for the cases where $C_1 < C < C_2$, $0 < \alpha < 1$, and thus $\varepsilon_{obs} \neq const$. For low concentrations, the absorbance of the weak electrolytes is equal to Line 1 ($C < C_1$, $\alpha \rightarrow 1$ and $\varepsilon_{obs} = const$), for the big ones it approaches Line 2 ($C > C_2$, $\alpha \rightarrow 0$ and $\varepsilon_{obs} = const$).

As a method for processing the results of concentration spectrophotometric measurements, the method of linear regression has proven itself in the best way [17-20]. In the context of this work, its application is a procedure for minimizing the sum of squares of different theoretical and experimental values. Suppose there are N pairs of experimental values of the absorbance A_i^{ex} at the corresponding concentrations C_i . According to equation (11), for each C_i , it is possible to write down the theoretical value of the absorbance:

$$A_i^{th} = l \left(\varepsilon_0 \cdot C_i + \frac{K_a}{2} \cdot \left(\sqrt{\left(1 + \frac{4 \cdot C_i}{K_a}\right)} \right) \cdot (\varepsilon_- - \varepsilon_0) \right) \quad (14),$$

which is a function of three independent unknowns: the dissociation constant K_a , ε_0 - the molar absorption coefficient of the undissociated and ε_- - the molar absorption coefficient of the dissociated form of the electrolyte. Thus, it is required to minimize the sum:

$$S = \sum_i^N (A_i^{ex} - A_i^{th})^2 \quad (15),$$

which is also a function of three unknowns: K_a , ε_0 , ε_- .

A necessary condition for the existence of a global minimum of the function S is the joint observance of three equalities:

$$\frac{\partial S}{\partial K_a} = \frac{\partial S}{\partial \varepsilon_0} = \frac{\partial S}{\partial \varepsilon_-} = 0 \quad (16)$$

In this work, to solve the system (16), we used the numerical methods of the MATHCAD software package [21]. As a result, the most reliable values of the constant K_a and the coefficients ε_0 and ε_- were obtained [17-20].

Further, several model systems (strong electrolyte, weak and very weak electrolyte) will be considered, with both experimental and theoretical dependences $A = f(C)$ and $\varepsilon_{obs} = f(C)$ established for each.

Results and discussion

Impact of concentration on spectrophotometric properties

1) Strong electrolytes

We took the water solution of potassium permanganate as the sample of the strong electrolyte solution:



In potassium permanganate solution, only one particle, permanganate ion, is absorbant. Predictably, this case is in full compliance with the BLB Law, the $A = f(C)$ dependence is linear, with the correlation coefficient $r = 1.0000$, while $\varepsilon_{obs} = const$ (Fig. 2).

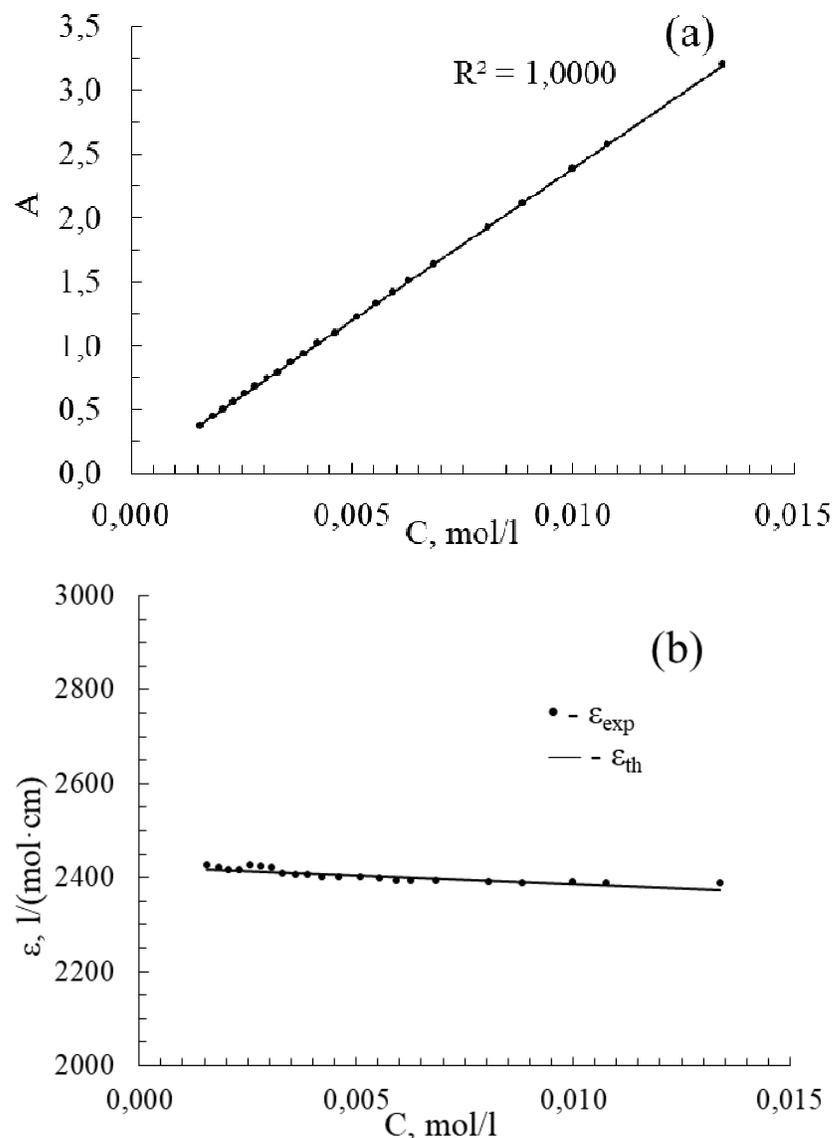


Fig. 2 Absorbance (a) and molar absorption coefficient (b) dependence on the concentration of water solution of potassium permanganate ($\lambda=525.5$ nm, $l=0.1$ cm)

Besides, Fig. 2 features the model view of the $\epsilon_{obs} = f(C)$ dependence, obtained by calculation method that provides reliable correlation between the experimental and calculated data. Similar dependencies for other compounds were modeled in the same way.

Here strong electrolytes are treated as specific cases, since in real life they're fairly rare. The main study objects are weak electrolytes, the widespread chemical compounds of various structuring, that are of practical interest.

2) Weak electrolytes

We took the water solution of benzoic acid as the sample of the weak electrolyte solution:



$A = f(C)$ and $\varepsilon_{\text{obs}} = f(C)$ dependencies are shown in Fig. 3.

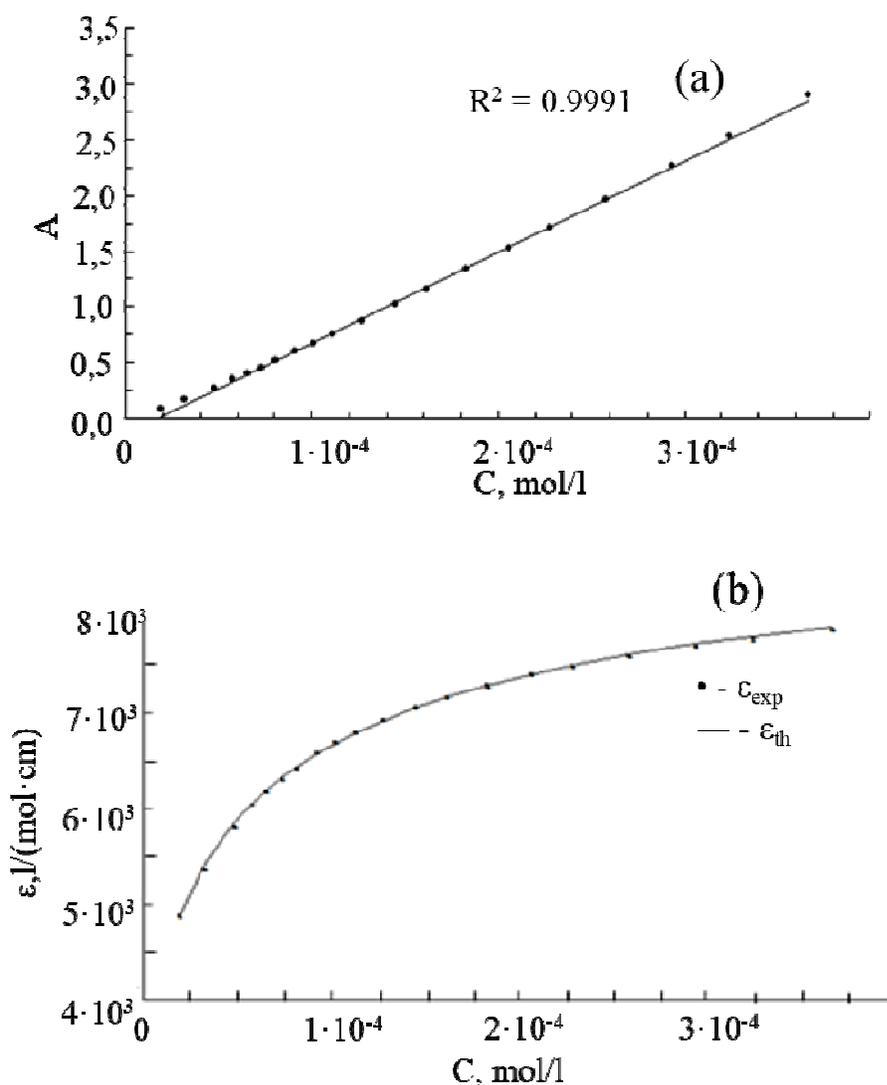
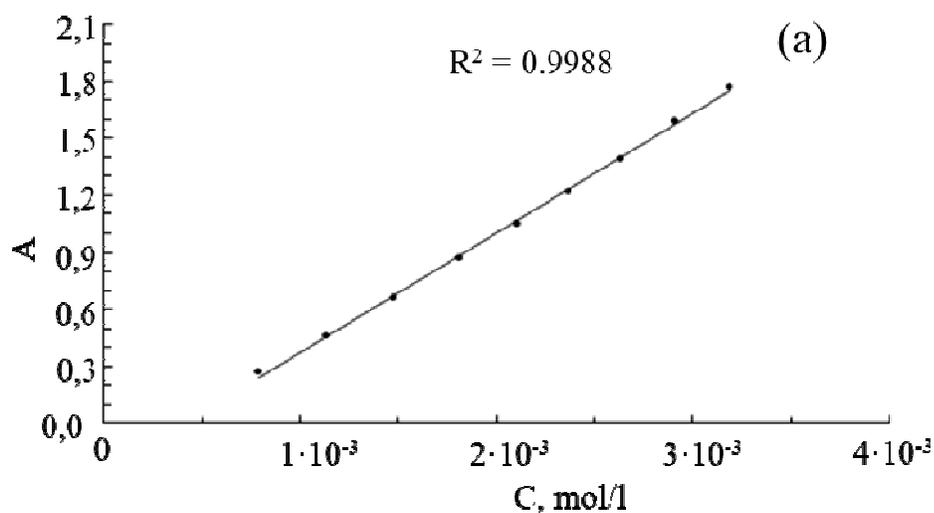
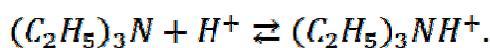


Fig. 3 Absorbance (a) and molar absorption coefficient (b) dependence on the concentration of water solution of benzoic acid ($\lambda=235.5$ nm, $l=1.0$ cm)

Figure 3 makes it clear that, with the $A = f(C)$ dependence for benzoic acid ($r = 0.9991$) being almost linear, ε_{obs} at low concentrations ($2 \cdot 10^{-5} \div 2 \cdot 10^{-4}$ mol/l) of the analyzed compounds changes significantly, because there're two types of absorbing particles, the anion and the neutral form, in the water solution of benzoic acid. Obviously, if ε_{obs} could be measured in a wider range of concentrations, the dependence would be sigmoid, featuring the $\varepsilon(A^-)$ values at low concentrations and $\varepsilon(AH)$ values at high concentrations, as shown in Figure 1 (Curve 3).

We took the water solution of triethylamine as the sample of the weak base solution (Fig. 4):



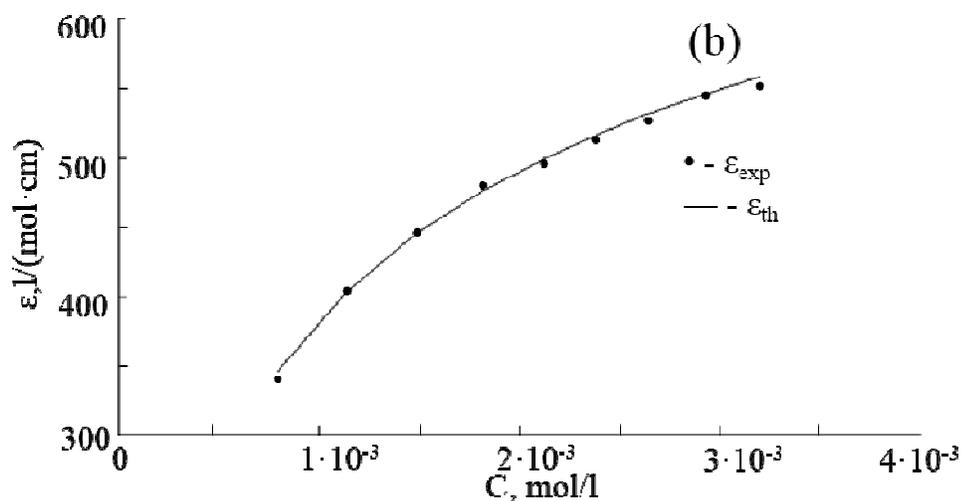
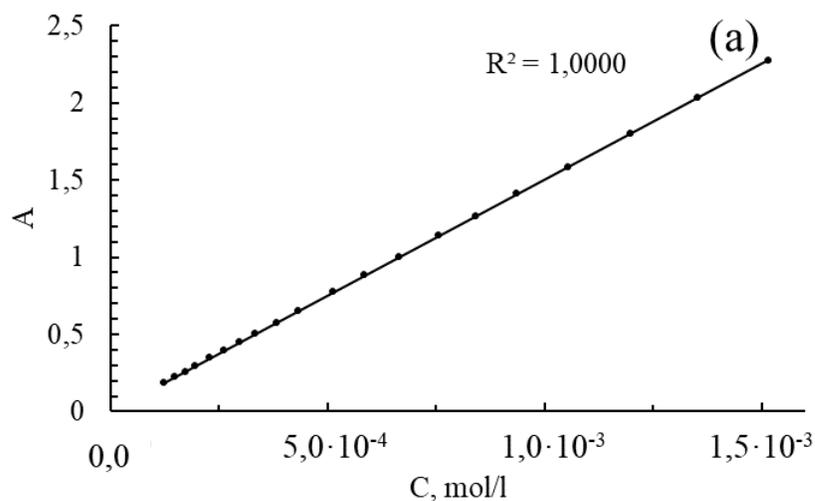


Fig. 4 Absorbance (a) and molar absorption coefficient (b) dependence on the concentration of water solution of triethylamine ($\lambda=262.0$ nm, $l=1.0$ cm)

Figure 4 shows that, for triethylamine, $A = f(C)$ and $\varepsilon_{\text{obs}} = f(C)$ dependence looks the same as the one for benzoic acid.

3) *Very weak electrolytes*

The case of a very weak electrolyte solution, e. g. phenol, should also be considered (Fig. 5):



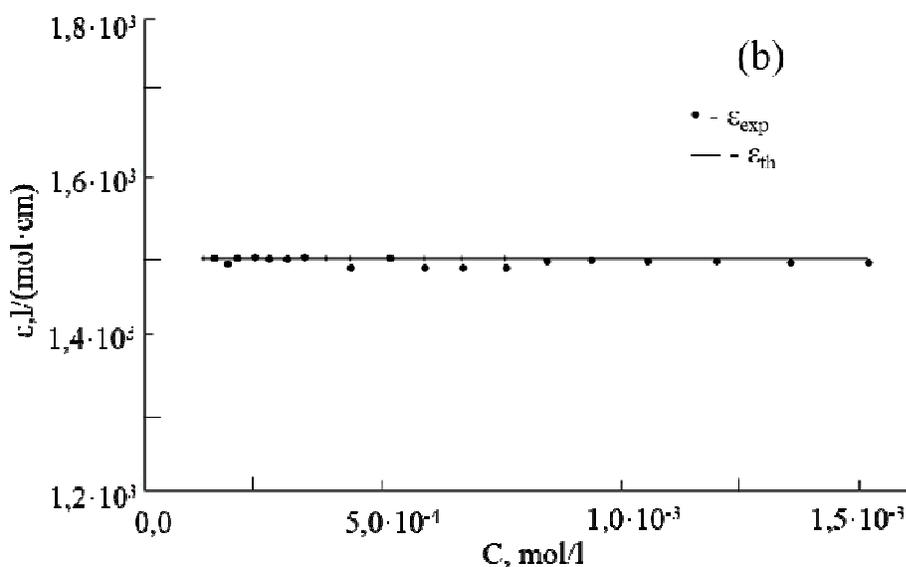


Fig. 5 Absorbance (a) and molar absorption coefficient (b) dependence on the concentration of water solution of phenol ($\lambda=269.5$ nm, $l=1.0$ cm)

Here the equilibrium is shifted significantly to the left, the $A = f(C)$ dependence for the compound is linear, and $\epsilon_{\text{obs}} = \text{const}$. The data obtained lead to believe that, in an water solution, this electrolyte is predominantly present in a neutral form.

Experiment

Study Objects

Triethylamine (Sigma-Aldrich; $\geq 99.5\%$) was used without additional purification. Benzoic acid (Sigma-Aldrich; $\geq 99.5\%$) and phenol (Sigma-Aldrich; $\geq 99\%$) were purified through sublimation at atmospheric pressure. Potassium permanganate solution was prepared from fixanal 0.1 H (Uralkhiminvest). The standard titer of oxalic acid 0.1 H ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) (Uralkhiminvest) was used as the primary standard for determining the exact concentration of the potassium permanganate solution.

Equipment

Analytical balance MB 210-A with the electronic precision of ± 0.1 mg was used for measuring the samples of the analyzed compound and its solutions. The

absorbance of the water solutions of the analyzed compounds was measured by the density meter DMA-5000 M Anton Paar (Austria) with the electronic precision of $\pm 5 \times 10^{-5} \text{ g} \cdot \text{cm}^{-3}$. UV/Vis spectra of each water solution with various concentrations of the analyzed compounds were registered by Shimadzu UV 2700 spectrophotometer (Japan). The temperature of the analyzed solution and of the reference cell was controlled by the cell holder Shimadzu TCC-100 with the precision of $\pm 0.1^\circ\text{C}$.

Concentration UV/Vis Spectrophotometry Method

A series of the titrant solutions of each compound was prepared, using the weight dilution method; its advantages have been shown in [21, 22]. Small samples of the analyzed compounds and solutions were weighed on the MB 210-A analytical balance. The storage time of the solutions never exceeded 24 hours.

Each experimental series began with registering the UV/Vis spectrum of the solution with the highest concentration, using the UV 2700 spectrophotometer (Shimadzu, Japan) with photometric accuracy $\pm 0.002 \text{ Abs}$ (at 0.5 Abs). Then, by gradual dilution with fresh distilled water, the concentration of the initial solution was lowered, and the absorbance of the resulting solution was measured all the time.

Molar concentrations of the solutions were calculated based on the results of the solutions' density and the weighing of the solutions before and after dilution. Density meter DMA-5000 M (Anton Paar, Austria) was used to measure the density of the solutions. All the measurements were carried out at 25°C .

Quartz cells, 0.1 and 1.0 cm, were used for spectrophotometric measurements. The analytical wavelength was selected at the maximum absorption of the analyzed compound.

Conclusion

It has been shown that, in the absence of buffer systems and with the variable concentration of the weak electrolyte solutions where $0 < \alpha < 1$, the observed molar absorption coefficient is not a constant. At the same time, for the electrolyte

solutions where the dissociation degree tends to one or zero with the change of the concentration, the observed molar absorption coefficient is a constant.

First time a mathematical apparatus for the calculation of the $\varepsilon_{obs} = f(C)$ dependence has been suggested for the electrolytes of various strengths, in the absence of the buffer systems.

In spectrophotometric measurements, the $\varepsilon_{obs} = f(C)$ dependence, rather than $A = f(C)$, seems to be more sensitive to the deviations from the Bouguer—Lambert—Beer Absorption Law.

The $E_{obs} = f(C)$ dependence can be used for detailed studies of equilibria in the weak electrolyte solutions, e. g. for determining the constants of polythic equilibria [17-20].

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