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APPLICITATION OF THE NEW KINETIC METHOD FOR PB(II) TRACES Pb(II) TRACES DETERMINATION IN DRINKING WATER

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Abstract: The new kinetic method, which was developed on the model system [1, 2], was used for Pb(II) traces determination in drinking water. The rusults show that this method could be successfuly applied for Pb(II) analyse in the range from 0.8 to 10 $\mu g/cm^3$ in 1000 times concentrated drinking water samples. Method is simple for performance and economical, too.

1. Introduction

There are only a few kinetic methods for micro and ultra-micro Pb(II) amounts determination in the solution, which were developed on the model systems [3]. In this work, the new kinetic method for Pb(II) traces determination was developed and then it was applied for drinking water analyse.Method is based on the reaction of alyzarine oxidation by hydrogene-peroxide which is inhibited by small amounts of Pb(II) ions.Reaction takes place in the presence of constant quantity ofCo(II) ions as catalysts, in the borate buffer. As the comparative methods, spectrophotometric and AAS method were used. The water samples from Niška Banja, Niš and Pasi Poljana aqueduct were analysed.

2. Experimental

As the oxidation of alyzarine with hydrogene-peroxide changes the violet colour of the start solution to yellow coloured oxidation product, the reaction rate was followed photometricaly on the Lange's photoelectric colourmeter with annected thermostat system. Measurements were conducted with filter with absorbtion minimum on $\lambda = 460$ nm.

Because the linear dependence which existed between the absorbance logharitam of analysed solutions and the reaction time during the first 3÷5 minutes of reaction, all kinetic results were treated by integral variant of tangent method [4].

Alyzarine solution ($2 \cdot 10^{-3}$ mol/dm^3) was prepared by measuring the solid substance and by its dissolving in 0.1 M NaOH solution.

H_2O_2 solution ($0.4 \cdot 10^{-3}$ mol/dm^3) was prepared from 30% reagent.

Pb(II) solution ($1 \cdot 10^{-4}$ g/cm^3) was prepared by measuring and drying the solid $Pb(NO_3)_2$ according the rule. Its concentration was verified complexometricaly [5].

Borate buffer was prepared of Na-tetraborate, HCl and NaOH solutions according the prescription [6].

All solutions were prepared of p.a. reagents, products of "Merck", with redestilled water, and were kept in the polyethylene vessels.

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Drinking water samples were conserved by conc. HNO_3 and were also kept in the polyethylene vessels.

Before the measurement in the four-legs flask were put the adequate volumes of reacting substances solutions, and then the redestilled water was added to the volume of 25 cm^3. The flask was thermostated for ten minutes at 298 K. Then, the flask was vigorously shaken, what started the reaction, and absorbance of the solution was measured every 15 seconds during the first 5 minutes of reaction. No masking agents were added to water samples before measurements. All samples were concentrated on the strong-cation resin IMAC C-14 before Pb(II) determination. Rinsing was conducted by adequate volume of nitric acid, and the eluated solution was then evaporeted on the water-bath till the wishing volume.

For the comparative spectrophotometric Pb(II) traces determination, the indirect method was used. It was based on the reaction with sodium-di-ethyl-di-thio carbaminate (Na-DDTK) and $CuSO_4$ [7]. As the masking agents, KCN and ammonium-citricum solution were added to the drinking water samples. Those samples were also concentrated on the strong-cation resin before measurements. The measurements were done on the UV-VIS "Carl Zeiss" spectrophotometer.

For the Pb(II) traces determination by AAS method, electro-thermal method with sample atomization in the graphite kyvete was applied [8]. Measurements were done on the atomic-absorbtion spectrometer "Perkin-Elmer", model 1100.

3. Results and discussion

The optimal conditions for indicatory reaction were found: H_2O_2 - $0.4 \cdot 10^{-3}$ mol/dm^3; alyzarine - $8 \cdot 10^{-5}$ mol/dm^3; pH of the borate buffer - 8.5; borate buffer - $6.5 \cdot 10^{-3}$ mol/dm^3; Co(II) - $1.2 \cdot 10^{-5}$ mol/dm^3.

Under those conditions the calibration curve was prepared. It can be used for Pb(II) determination in the concentration range from $8 \cdot 10^{-7}$ to $100 \cdot 10^{-7}$ g/cm^3 (Fig. 1).

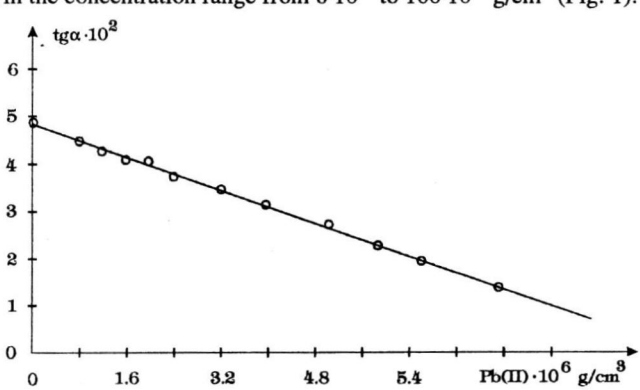

Figure 1: Dependence of tgα on the Pb(II) concentration. Initial conditions: H_2O_2 - $0.4 \cdot 10^{-3}$ mol/dm^3; alyz. - $8 \cdot 10^{-5}$ mol/dm^3; Co(II) - $1.2 \cdot 10^{-5}$ mol/dm^3; borate buffer - $6.5 \cdot 10^{-3}$ mol/dm^3; pH - 8.5; T = 298 K.

Adequate equation of the calibration curve is:

$$tg\alpha = - 0.038 \cdot c_{Pb(II)} + 0.0495, \qquad (1)$$

where is: $c_{Pb(II)}$ - concentration of Pb(II) ions in $\mu g/cm^3$.

The relationship between the reaction rates and the concentrations of the reacting components, can be described by following kinetic equations.

For the catalytic reaction:

$$- (dc_{alyz}/dt) = k_1 \cdot c_{buf} \cdot c_{alyz} \cdot c_{H_2O_2} \cdot c_{Co(II)}, \quad (2)$$

and for the catalytic-inhibitory reaction:

$$- (dc_{alyz}/dt) = k_2 \cdot c_{buf} \cdot c_{alyz} \cdot c_{H_2O_2} \cdot c_{Co(II)} \cdot c_{Pb(II)}, \quad (3)$$

where is: k_1 - constant proportional to the rate constant of the catalytic reaction, and

k_2 - constant proportional to the rate constant of the catalytic-inhibitory reaction.

The accuracy and precision investigation, showed that, for the concentration range of Pb(II) from $1.6 \cdot 10^{-6}$ to $7.6 \cdot 10^{-6}$ g/cm^3, the error varies from - 3.1 to 6.6%.

Selectivity of the method is relatively good. Only the presence of Ni(II) ang Hg(II) ions in the reaction mixture in the ratio 1:1 to the Pb(II) concentration catalyses the indicatory reaction and the presence of EDTA, in the same concentration ratio, inhibits the reaction.

After developing on the model system, method was applied for Pb(II) trace determination in the drinking water samples from Niš, Niška Banja and Pasi Poljana.

The mean values of Pb(II) concentration, received by calculating the experimental results on the real concentration in the samples, by kinetic, spectrophotometric and AAS method, respectively, are shown in Table I.

Table I: Comparative results of Pb(II) determination in drinking water, by kinetic, AAS and spectrophotometric method

Sample	Mean value of 5 measurements $x \cdot 10^9$ g/cm^3 Pb(II)			$(x_k-x_s) \cdot 100/x_s$	$(x_k-x_a) \cdot 100/x_a$
	Kinetic meth. x_k	Sp.ph.m. meth. x_s	AAS method x_a		
Niš	7.56	7.39	8.60	+ 2.30	- 12.10
Niška Banja	2.19	2.94	2.80	- 25.50	-21.80
Pasi Poljana	6.49	5.96	7.20	+8.90	-9.90

Result of Pb(II) determination in the water samples from Niš, applying a kinetic method of analyse, is for 2.3% greater than result found by using a spectrophotometric method, and for 12.1% is less than Pb(II) concentration results found by AAS method.

Mean value of Pb(II) concentration, found in the water from Niška Banja using a kinetic method of analyse, is for 25.5% greater than mean value found by spectrophotometric method, and for 21.8% is less than mean value found by AAS method of analyse.

The Pb(II) concentration, found in drinking water from Pasi Poljana, is for 8.9% greater by kinetic method than by spectrophotometric, and for 9.9% is less than by AAS method of analyse.

The analyse of those results shows that difference between results grows with the Pb(II) concentration deminution in the samples. By applying any of those methods, the greatest concentration of Pb(II) was found in the drinking water from Niš, and the least in the water from Niška Banja. All results are in the permited area for Pb(II) concentration according the Regulation of drinking water of Serbia.

So, it is conclused that this new kinetic method can be successful for drinking water investigations.

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PRIMENA NOVE KINETIČKE METODE ZA ODREĐIVANJE TRAGOVA PB(II) U PIJAĆOJ VODI

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Primenjena je nova kinetička metoda za određivanje tragova Pb(II) u pijaćoj vodi, koja je razrađena na model sistemu [1, 2]. Dobijeni rezultati ukazuju da se predložena metoda može uspešno primeniti za analizu Pb(II) od 0.8 do 10 $\mu g/cm^3$ u pijaćoj vodi, koja je koncentrovana do 10^3 puta. Metoda je jednostavna i ekonomična.