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Spectrophotometric determination of Lead ions in solutions samples using Chromazurol S in the Surkhandarya region

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Abstract. In this paper, the determination of lead ions in the waters of Surkhondarya was studied by the spectrophotometric method using Chromazurol S. The formation of a complex between lead and Chromazurol S in the ion exchange phase was studied. This formed phase allowed spectrophotometric determination of lead in drinking water. According to the obtained results, the detection sensitivity was 0.02 μg/ml, and this was proposed as one of the effective methods for determining lead in Surkhondarya waters.

Keywords: Surkhondarya, lead ions, Chromazurol S, complex compounds.

Introduction.

Lead is one of the most toxic metals; its concentration has to be obligatorily controlled in assessing the quality of drinking water, food products and raw mate rials, pharmaceuticals, etc. [1]. The threshold limit value for the lead concentration in water is 0.03 mg/L, while for various food products this value is in the range from 0.05 to 10.0 mg/kg of product [2]. Lead is widespread in natural substances, and almost everyone is exposed to this toxic heavy metal in different ways, either in the workplace or in homes[3].

Spectrophotometric methods are one of the greenest analytical methods because of the consumption of low solvent volumes and instrumental hazards [4]. On the other hand, according to the 12 principles of green analytical chemistry (GAC), a decrease in time and cost in the simultaneous determination of analytes can be an effective step toward the goals of GAC [5].

Chromazurol S is used in the spectrophotometric determination of trace amounts of Al, In, Pd, Be, Cd, Cu, Fe, Hf, Zr, Mg, Sc, Th, Ti, U(VI), Y [4, 12, 13]. It has not been used for the determination of lead[6].

Simple and sensitive spectrophotometric and spectrofluorimetric methods have been developed for the determination of hydrochlorothiazide (I), indapamide (II) and xipamide(III) based on ternary complex formation with eosin and lead (II) in the presence of methylcellulose as surfactant[7].

Lead (+2) was selectively adsorbed on a solid phase extraction (SPE) gel (molecular recognition technology, MRT), quantitatively extracted, and spectrophotometrically determined as the Pb(II)-PAR (4-(2-pyridylazo)-resorcinol) complex. The linear range was 0.01 to 0.75 mg L^{-1} and the detection limit was 6.4 μg L^{-1}. The MRT-SPE allows selective Pb(II) extraction from complex ion-rich matrices, which is difficult with other techniques. Interference from common matrix ions such as Fe^{2+}, Ni^{2+}, Cu^{2+} or Co^{2+} is minimized[8].

This work investigates the applicability of the anionic complex $[NBu_4]_2[Zn(1,3\text{-dithiol-2-tione-4,5-dithiolate})_2]$ on the simultaneous spectrophotometric determination of transition metals. Principal Component Regression was used in the data analysis. This method was used to determine Hg^{2+}, Cu^{2+} and Pb^{2+} in water solution, at t=0, 20 and 60 min of reaction[9].

2. Experimental part

Materials. A 0.1 M stock solution of Pb(NO3)2 **was prepared by dissolution of a weighted portion of chemically pure** Pb(NO3)2 **in 0.1** HNO3 **and was standardized by**

chelatometric titration. Chroma zurol S (*CAZ*) was of analytical grade; it was prepared by dissolving a precisely weighted portion of the sub stance in water. Water was purified according to. Aqueous solutions of the reagents used were chemi cally pure or analytical grade. All working solutions were prepared by diluting the stock solutions before the experiment.

Methods. Absorption spectra of the solution were recorded on SF46 and SPECORD UV VIS spectro photometers. The transmittance of ionexchanger granules in water was measured on KFK3 in a 0.1 cm cuvette at the optimal wavelength (λopt), using the ion exchanger as the reference. The acidity of the solu tions was controlled with an I160 potentiometer with a glass electrode.

Synthesis of ABHPT. Preparation of the modified ionexchanger for work.

The AB17 × 8 anion exchanger (AB) with 0.30 mm grains were prepared for work according to the recom mendations of [16, 17]: 10 g of AB were soaked in sat urated solution of NaCl and left for a day. Then, the adsorbent was separated, and it was washed firstly with 0.5 M HCl until its negative reaction to iron (III) and then with water until its neutral reaction. The prepared ABCl was modified with aqueous solutions of CAZ. To do so, ~0.1 g of the colorant was dissolved in 150 mL of water, and then 10 g of airdry ABCl was introduced. Solid phase was separated by filtration, washed with water, and dried. The obtained solid phase reagent was in the form of transparent pink granules, which transmitted up to 50% of the light at 520 nm and could be kept for a long time in water in carefully closed dark glass containers.

Procedure for the adsorption of lead onto the modified anionexchanger. A 50.0mL portion of the solution of a lead salt was introduced into 150mL measuring beakers, and the required acidity was created using HNO_3 and NaOH; 0.3 g of a modified adsorbent were introduced, after which the solution was agitated with a magnetic stirrer and subjected to ultrasonic treatment (US).

The dependence of lead adsorption on the acidity of the medium was studied at constant concentrations of the lead salt and CAZ and different values of pH. A series of 2.0×10^{-5} M $Pb(NO_3)_2$ solutions with a volume of 50 mL and predetermined pH values was prepared. Then, the experiment was performed according to the procedure for the adsorption of lead onto modified anion exchanger.

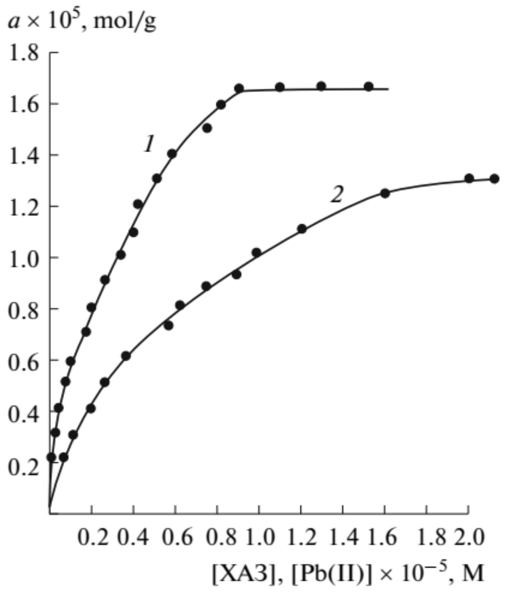

Fig. 1. Adsorption isotherms of CAZ onto AB (*1*), Pb (II) onto CAZ-AB (*2*). *1*—pH 6, $\tau_{agitation} = 30$ min, US—2 s; *2*—pH 5, $m_s = 0.3$ g, $V = 50$ mL, $\tau_{agitation} = 20$ min, $\tau_{equilibration} = 1$ hour, $l = 0.1$ cm, reference sample—AB.

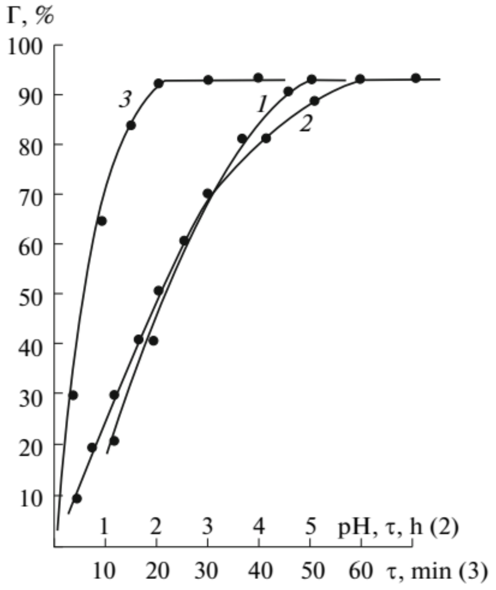

Fig. 2. Dependence of Pb (II) adsorption on pH (*1*) and τ (*2, 3*). *1*—$m_s = 0.3$ g, $V = 50$ mL, 600 nm, $\tau_{agitation} = 20$ min, $\tau_{equilibration} = 1$ hour, $l = 0.1$ cm, reference sample – AB; *2*—$m_s = 0.3$ g, $V = 50$ mL, 600 nm, pH 5, $l = 0.1$ cm, reference sample—AB; *3*—$m_s = 0.3$ g, $V = 50$ mL, 600 nm, pH 5, $\tau_{agitation} = 20$ min, US—2 s, $l = 0.1$ cm, reference sample—AB.

RESULTS AND DISCUSSION

As shown in Fig. 1 (curve 1), the isotherm of CAZ adsorption on the anionexchanger belongs to the L type. The maximal capacity of the anionexchanger towards the modifier is a max = 16.7 µmol/g; k = 9.99 ×105 L/mol. Under optimal conditions for the extraction of lead (II), CAZ, which is immobilized due to the ion exchange, can exist in the form of H2R3–AB. Assuming that the mmobilization of CAZ occurs due to the deprotonated sulfo group, its solidphase analytical form can be presented as (H2R AB)$^{2-}$.

To determine the optimal conditions for interaction between lead and solidphase CAZ, dependence of lead adsorption on the pH of the solution was investigated. The maximal recovery of lead (90%) is achieved at pH 5. For this reason, the further investigations in the solidphase system were carried out at this value of pH (Fig. 2). According to the calculations and taking into account literature data, at pH 5 lead was considered to be in the form of Pb2+. Fig. 2 presents the results of the investigation of the extraction velocity on the time for which the phases were in contact. An adsorption equilib rium is established within 1 hour. Ultrasonic treatment for 2 s allows the reduction of the time for the establishment of equilibrium down to 20 min and an increase in lead recovery up to 92%.

Taking into account the topics dis cussed above and the results of the data treatment with the slope method, the formation of a complex with a 1 : 1 ratio of the components can be assumed in the solid phase according to the following scheme:

$$Pb^{2+} + (H_2R–AB)^{2-} = (PbHR–AB)^{-} + H^{+}.$$

The investigation of the dependence of lead recovery on the volume of the solution and the mass of the adsorbent has shown that concentrating

from V **=50.0–500.0 mL is possible (**m **= 0.3 g). In this case, the distribution coefficient is** $D_{max} = 1.50 \times 104$ **mL/g. Aftersubjection to US** $D_{max} = 1.77 \times 104$ **mL/g. The average value of the conditional stability constant is** $\log\beta_{cond} = 5.49 \pm 0.04$ **at □□= 0.1.**

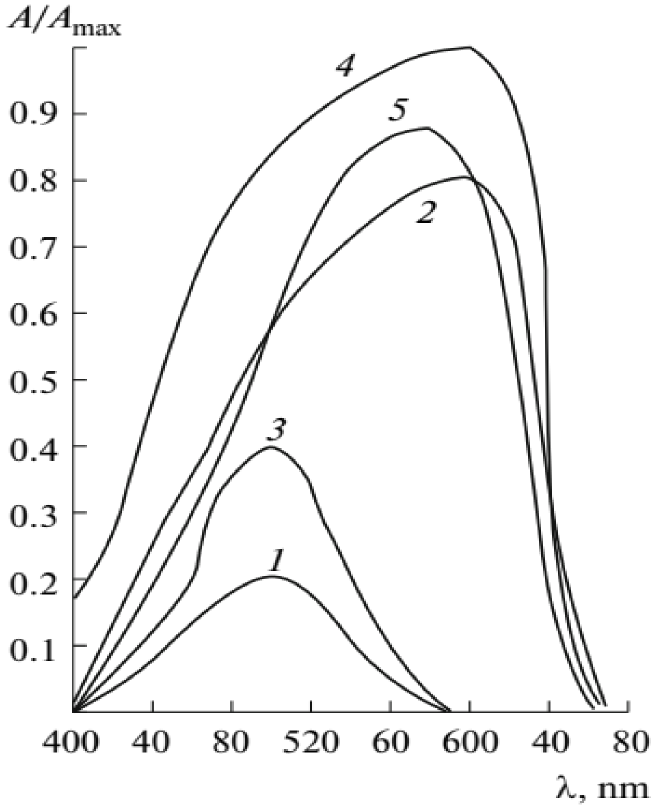

Fig. 3. Absorption spectra of CAZ-AB (*1*, 3), Pb-CAZ-AB (*2*, *4*) and Pb-CAZ (*5*). *1*—pH 6; *2*—pH 5, $m_s = 0.3$ g, $V = 50$ mL, $\tau_{agitation} = 20$ min, $\tau_{equilibration} = 1$ hour, $l = 0.1$ cm, reference sample—water; *3*—pH 6; *4*—pH 5, $m_s = 0.3$ g, $V = 50$ mL, $\tau_{agitation} = 20$ min, US—2 s, $l = 0.1$ cm, reference sample—AB; *5*—pH 5, 2×10^{-5} M Pb, 4×10^{-5} M CAZ, $l = 1$ cm, reference sample—water.

The experiment was carried out as follows. Three sam ples of water were collected; the volume of each sample being 1.0 L. In two samples, standard additions of $Pb(NO3)2$ **were introduced. Then each of the samples was divided in two 500.0 mL parts and pH 5 was created. In the first portion of water, 0.3 g of solidphase CAZ was intro duced; then it was agitated with a magnetic stirrer for 15– 20 min and subjected to US for 2 s; after which the granules were separated with a pipette, and transferred to the sec ond portion of the same sample, where similar procedures were performed. The granules were separated, put into a cuvette (*l* = 0.1 cm) and their absorbance at maximal rati εK/εR (λopt = 600 nm) was measured, using the anion exchanger as the reference. The experiment with the second sample was carried out in a similar way.**

The lead concentration was determined from the calibration curve or in the graphical variant of the standard addition method. The accuracy of the obtained result was checked by the standard addition method. The agreement between the results, obtained using the new procedure and the standard one, confirms their reliability. The results of determining lead and their statistical treatment are presented in the Table.

Determination of lead in drinking water (Kiev) using (A) **the proposed method** (A) **and** (B) **polarographic analysis after evapo ration** (P **= 0.95;** n **= 3).**

Tested solution	**Added Pb, µg**	**Found Pb, µg** (*A*)	**RSD,%**	**Found Pb, µg** (*B*)	**RSD,%**
Sample-1	**-**	**5.0 ± 0.3**	**2**	**5.0 ± 0.6**	**5**
Sample-2	**5,0**	**10 ± 1**	**4**	**10 ± 1**	**5**
Sample-3	**50,0**	**55 ± 10**	**7**	**55 ± 18**	**6**

Thus, a procedure for the solidphase spectropho tometric determination of lead using CAZ has been developed. It is characterized with satisfactory accu racy and reproducibility of the results.

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