

# CONCENTRATION OF HEAVY METALS WITH USE OF DIANTIPYRILMETHANE

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## INTRODUCTION

The problem of group concentration of heavy metals continues to remain actual in liaison with necessity of lowering of a limit of detection, simplicity and speed of the analysis, extension of a circle of definite of elements. Recently greatest distribution was received by methods of atomic - emission spectrometry with inductive - connects by plasma [1-4]. The concentration of elements for want of it is carried conducted in main with use of chelating sorbents or extraction by new reactants and solvents. Many extragents having high extraction ability in relation to large group of metals at the same time are known. One from such is the diantipyrimethane (DAM), which allows to extract up to 20 elements simultaneously, using thiocyanate solutions [5].

In the present work the optimization of conditions of extraction of large group of metals of the DAM from water solutions is conducted at the presence of thiocyanate of an ammonium with reference to their consequent atomic - emission determination.

## EXPERIMENTAL

**Material and methods.** The extraction ability of the DAM in relation to 20 elements was studied: Zn (II), Ag (I), Mn (II), Ti (IV), Sb (III), Mo (VI), V (V), Au (III), Bi (III), Co (II), In (III), Cr (III), Cd (II), Tl (III), Pb (II), Cu (II), Sn (II), B (III), As (III), Ni (II). Analytical line elements and their concentration in the working solution are preset based on experiments, and the data [6]. Initial extraction conditions set according to [7].

The spectra were recorded on a DFS-452 spectrograph with a general purpose generator UGE-4. Exposure time - 35 at AC 8 A. Carbon electrodes. Each point was performed at least three parallel determinations.

**The experimental procedure.** In a separating funnel pipetted working solution was added a solution of ammonium thiocyanate, sulfuric acid is the desired concentration, the volume adjusted with distilled water to 20 ml and extracted into the organic phase elements within 5-15 minutes. After settling, the chloroform extract was filtered into a porcelain crucible with the sample of coal powder 0.05 g solvent was evaporated in an oven at 100 °C. After

calcination, the crucible in a muffle at 450-500 °C their contents placed in the crater of the carbon electrodes and the spectra were recorded in the above conditions.

## RESULTS AND DISCUSSION

In order to optimize the extraction conditions of elements "thiocyanate series" [5] the diantipyrilmethane, the dependence degree of extraction of the concentration of the reagent, ammonium thiocyanate, sulfuric acid, the contact time of the phases, the number of extractions, the nature of the solvent. Were used as solvents 1,2-dichloroethane, chloroform, and the chloroform-butanol mixture. Found that increased extraction elements are observed when using a mixture of  $\text{CHCl}_3:\text{C}_4\text{H}_9\text{OH}=7:3$ . Thus the concentration of ammonium thiocyanate can be varied within 0.25-1.0 mol/l, and  $\text{C}(\text{H}_2\text{SO}_4) - 0.5-2.0$  mol/l.

When a single extraction under the above conditions in chloroform-butanol mixture was thoroughly extracted Co, Bi, Mo, Ti, Cd, Zn, Sn and Ag. The lowest extraction degree ( $E=43\%$ ) is observed at a thallium(III) (except for arsenic, which practically is not extracted), that the chloride - ion is connected, obviously, to absence in a water phase. This factor also affects the extraction of lead.

Elements with the highest degree of oxidation extracted much better, so solutions pre-treated by boiling with the ammonium persulfate. To increase the degree of extraction of thallium (III) was introduced into the extraction system hydrochloric acid (0.3 mol/l). Nickel more fully extracted from weakly acid solutions [7], so its quantitative extraction (and all the other elements), we suggested the following conditions, mol/l:  $\text{C}(\text{NH}_4\text{SCN}) = 0.5$ ;  $\text{C}(\text{H}_2\text{SO}_4) = 0.5$ ;  $\text{C}(\text{HCl}) = 0.3$  – with preprocessing of test  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ . In this case, double extraction of 0.05 mol/l solution of the DAM in a chloroform-butanol mixture (7:3) provides maximum extractability of the test elements (88% (Mn, Cr, Ni) — 99%), except arsenic.

The influence of macro components of water (Ca, Mg, Si, Al, Na, K, Fe) on a degree of distribution of microelements was investigated. Is established, that  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+} < 0.2$  mol/l,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Si}^{4+}$ ,  $\text{Al}^{3+} < 0.06$  mol/l do not hinder the analysis. The interfering effect of  $\text{Fe}^{3+}$  (up to 1 mg/20 ml) was removed ascorbic acid (0.2 g/20 ml), which also contributes to a more complete extraction of copper. To eliminate the effect contained in natural waters, humic and fulvic acid sample was boiled in the presence of  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ . Found that the processing of samples of ammonium persulfate is most effective when samples evaporation to 30 ml.

Method extraction-spectral determination elements 19 in water were developed based on the results obtained.

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### Abstract of the article

#### L. Toropov « CONCENTRATION OF HEAVY METALS WITH USE OF DIANTIPYRILMETHANE»

The conditions of group concentration 20 elements by diantipyrilmethane from thiocyanate solutions for their consequent atomic - emission determination are investigated. Is established, that in a chloroform-butanol mixture (7:3) 19 elements (except for As) quantitatively are extracted. The allowable contents of hindering macro components of water (Ca, Mg, Si, Al, Na, K and Fe) are determined. The optimum conditions of preprocessing of tests by persulfate of an ammonium are found. Because of conducted researches the highly effective technique of group extraction-spectral determination of elements in natural and industrial waters is developed.

Keywords: heavy metals, extraction, diantipyrilmethane, spectral determination