The problem of natural waters pollution by various toxicants, like phenols or rocket fuel components, forces to use of high precision analytical methods including preconcentration. Solid phase extraction (SPE), especially performed in column mode, is the most promising technique for the preconcentration of organic substances from solutions, because it overcomes many disadvantages of liquid-liquid extraction.

For the choice of sorbents for SPE it is necessary to define strict criteria of sorption efficiency. The most integral criterion proposed in this work is the concentration efficiency (CE) determined as the sample flow rate on the preconcentration step under quantitative recovery of analyte. It was demonstrated that the maximum achievable CE has a single value for the given preconcentration factor and the recovery; therefore this criterion can be used for the comparison of different sorbents.

Mathematical modeling is most promising approach to description of SPE process and prediction of sorption efficiency. In present work using of phenomenological liquid film diffusion and solid diffusion models the relation between model parameters describing thermodynamic and kinetic properties of adsorption (distribution ratio, solid diffusion coefficient of sorbate in a sorbent phase, liquid film coefficient, dimension of the sorbent particles) and CE, as well as optimum SPE conditions (sorbent bed size and sample flow rate) has been established.

The adsorption of phenols on hexadecylsilica, styrene-divinylbenzene copolymers with various cross-linking degree and hyper cross-linked polystyrenes was investigated. The adsorption isotherms and dynamic breakthrough curves of phenol and catechol were obtained. The type of mass-transfer was determined and parameters of the model (distribution ratio, solid diffusion coefficient, liquid film coefficient) were calculated. To compare the sorbents investigated for the preconcentration of phenol and catechol, the values of maximum achievable CE were calculated. The group of hyper cross-linked polymeric sorbents possessing both high thermodynamic and kinetic parameters was found the most promising for the preconcentration of phenols.

Optimal conditions for SPE of 1,1-dimethylhydrazine derivate – 1,1-dimethylhydrazon of 4-nitrobenzaldehyde (DHB) on C16-silica were chosen. In this case choice of sorbent is not critical, because of high distribution ratio due to hydrophobic nature of this compound. The adsorption isotherms and dynamic breakthrough curves of DHB on C16-silica were obtained. To calculate optimum preconcentration conditions parameters of the model were determined. Bed sizes of 20X4 mm i.d. and flow rate 4.2 ml/min were chosen. Under these conditions the quantitative recovery of analyte from 120 ml of sample and enrichment factor of 400 were achieved.

THIN-LAYER FILTERS FOR PRECONCENTRATION OF GOLD AND PALLADIUM FROM SOLUTIONS

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Key words: sorption preconcentration, filters, noble metals, analysis of ores and alloys

One of the promising ways for determination of small amounts of noble metals in solutions of complicated composition is working out of the combined methods of analysis including preconcentration stage. Dynamic sorption preconcentration that provides high enrichment factors and does not require separation of phases is mostly effective.
For preconcentration gold and palladium from solutions of complicated composition new sorption filters prepared by impregnation of a filter paper with solution of tri-n-octylamine (TOA) and paraffin in non-polar solvents are proposed. The content of nitrogen in obtained sorbent is 0.57 mmol g\(^{-1}\). Sorption of palladium and gold from hydrochloric acid solutions on such filters is caused by formation of ionic associates including anionic chloride complexes of metals with protonated molecules of immobilized TOA.

Gold and palladium are quantitatively recovered on a filter (filtering surface diameter 23 mm, thickness 0.15 mm) from 0.5 - 1 M HCl at flow rates of 2-3 ml min\(^{-1}\). The recovery of both metals does not vary at the presence of large amounts of matrix elements – macrocomponents of ores and alloys. The possibility of quantitative recovery of gold and palladium from solutions obtained after decomposition of a nickel matter and sulfide ore was shown.

Gold and palladium were determined directly on a filter-sorbent by X-ray fluorescence using domestic instruments - scanning crystal diffraction spectrometer SPECTROSCAN-U and energy depressive PRİM-İM. The detection limits for gold and palladium are 0.3 and 0.5 fig of metal on a filter, respectively (X-ray tube voltage 40 kV, exposure time 30 s). Using scheme of the analysis of ores and breeds involving nickel sulfide fire assay, the limits of detection of gold and palladium in these materials are \(6 \times 10^{-7}\) and \(1 \times 10^{-6}\) %. The possibility of determination of palladium on filters by diffuse reflectance spectroscopy after formation of colored compounds of metal with 4-(2-pyridylazo)resorcinol was demonstrated.

NEW PRECONCENTRATION TECHNIQUE FOR X-RAY FLUORESCENT DETERMINATION OF HEAVY METALS IN WATERS

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Key words: heavy metals, preconcentration, filters, X-ray determination

For determination of heavy metals in environmental objects an application of modern multielemental instrumental methods of the analysis, in particular, X-ray fluorescent spectrometry (XRF) possessing high reproducibility and selectivity is perspective. However, the sensitivity of this method is low for direct determination of marginal amounts of toxic elements in waters and soils. Thus preconcentration of elements to be determined is required. The application of thin layer organ polymeric filters for preconcentration providing achievement of high enrichment factors (hence, large improvement in sensitivity of XRF determination of elements) is especially effective.

In the present work a new process for the recovery of Pb, Zn, Cu, Ni, Co, Fe(III) and V(IV) from solutions on thin layer cellulose filters was proposed. Metals were extracted as dialkyldithiocarbamate complexes obtained in dynamic conditions after merging of sample solution with solutions of corresponding reagents. Sodium diethyldithiocarbamate (DTC) and ammonium pyrrolidine dithiocarbamate (APDC) were utilized as reagents.

The optimal conditions for the recovery of elements were determined. All metals are quantitatively extracted from 50-100 ml of sample at pH 4-7, concentration of reagent 0.01-0.04 %, sample flow rate 3-5 ml min\(^{-1}\) and reagent flow rate 0.7-1.0 ml min\(^{-1}\). The variation in size of the mixer from 0.02 up to 2 ml has no influence on the recovery of elements. Under these optimal conditions metals are effectively extracted at the presence of potential concomitant