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I S O T A CH O P H O R E S I S

APPLICATION NOTE No. 8

DETERMINATION of HEAVY METALS in WATER

MAIN FEATURES:

It was designed the ITP procedure for determination of heavy metals in drinking, surface and waste water on ppb level. It is necessary simple pre-treatment of the sample. This pre-treatment means to concetrate the sample several times. For this purpose it is very advantageous the application of the sorbents with chemical binding chelate groups. Chelate sorbent Spheron Thiol was used for sorption of Cd, Ni, Zn, Pb in the sample of drinking water by inserting method.

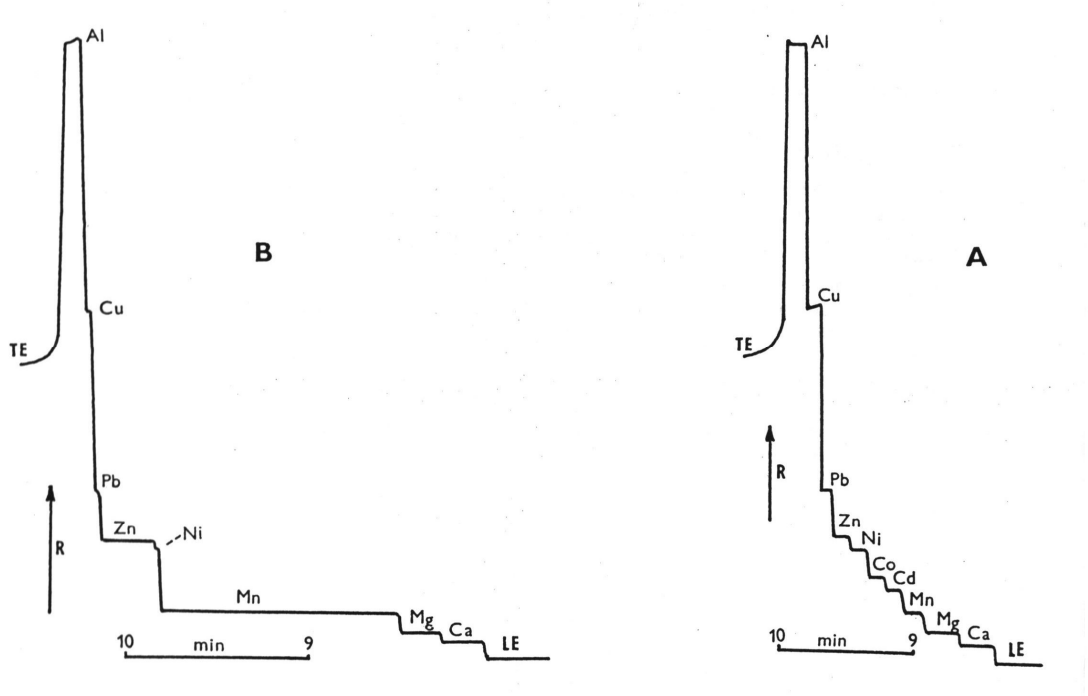

Fig. 1 : Analysis of metal ions from analytical columnal column :
A – model mixture (concentration cca 5.10^{-5} mol/l resp. 3-20 ppm)
B – drinking water after 100-times concentrating (determined concentrations in μl/l resp. ppb: Mn=50; Ni=3; Zn=22; Pb=12; Cu=3; Al=4)

Conditions : leading electrolyte (LE):
2.10^{-2} M CH_3COOH + $1{,}75.10^{-2}$ M α–hydroxyisobutiric acid (HIBA) + 10% PEG (polyethylenglycol) + 0,1% HEC (hydroxyethylcellulose), pH=4,8
terminating electrolyte (TE):
10^{-2} M CH_3COOH

preseparation column=90mm, I_1 = 350 μA, analytical column=90mm,I_2 = 75 μA, V=30 μl

Procedure for concentrating ions

To prohibit contamination from laboratory vessels it is necessary to put them in HNO (10%) for several minutes. No use glass, vessel from PE, PTFE. Solutions of electrolytes and standards and eluent solutions are recommended in vessels from PP or Si-glass.

Separated scheme for using solid phase extraction

1. treatment of sorbent 2. treatment of sample 3. sorption of sample by treatment sorbent 4. separation of sorbent from solution 5. removement of macrocomponents (Ca, Mg) by washing 0,1 M $CH_3COOHNa$, pH=7,0 (30 ml) 6. washing sorbent by deionizing water 30 ml 7. desorption of analyte by 1M HNO_3 (8 ml) 8. drying eluat by hot air (hair dryer) 9. dissolvation of evaporation residue in defined volume of 10 M HNO_3 10. analyse by ITP method.

Treatment of sorbent

For concentrating two methods can be used : a – static (inserting) or b – dynamic. For above-mentioned type of sorbent and sample the better yielding was achieved by static sorption. In the case you can use separation microcolumns Spheron Thiolom 1000, which is delivered by Institute of Polymers (Bratislava). The filling amount is 0,5 g. Working form of sorbent was achieved by succesive washing : methanol, deionizing water, HNO_3 1M, deiozing water up to neutral reaction, buffer solution CH_3 COOHNa 1M, pH=7. There were used 2 column volumes of reagent /the same as volume of sorbent) and 10 column volumes of deionizing water.

Treatment of drinking water

It was used 400 ml of city water piping, which was filtered through microfilter nylon Separon TM (producer Tessek Ltd. Praha).

Concentrating of heavy metals in water

To filtered sample of water there was inserted 0,5g treated sorbent and was added buffer solution 1M $CH_3COOHNa$, pH=7 (50ml) and solution together with sorbent stood 12 hours, occasionally to shake. Sorbent was separated from sample of water by pouring through the original microcolumn with fritted glass, After separation of all solution the sorbent was washed by 1M $CH_3COOHNa$, ph=7 (30ml), deionizing water (30ml). Desorption was done by 1M HNO_3 (8ml). Eluate was dryed and evaporation residue was dissolved in 1 ml of HNO_3 10M. All volume was fulfilled into syringe and by means of injection valve was dosed into ITP analyser. Conditions are described below the Fig.1. All pre-treatment excluding 12 hours staying takes approximately 1 hour.

Literature:

1. M.Hutta, D.Kaniansky, V.Zelenská, E.Šimuničová, V.Madajová, A.Šišková : Solid phase extraction for sample preparation in capillary ITP, Radioanal. Nucl. Chem. , 1992