

Spectrophotometric determination of uranium by flow injection analysis using U/TEVA.Spec™ chromatographic resin

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A flow injection analysis (FIA) system is described for the determination of uranium. The system consists of a microcolumn packed with, U/TEVA.Spec™, chromatographic resin for on-line sample separation. The eluate is mixed with 4-(2-pyridylazo) resorcinol (PAR). The colored product is continuously monitored spectrophotometrically.

Introduction

In our previous work,¹ a simple flow injection analysis (FIA) was proposed for the determination of uranium in tin tailings using 4-(2-pyridylazo)resorcinol (PAR). The procedure is suitable at relatively high uranium concentrations. Higher sensitivity and more tolerance to interfering ions can be achieved² by using also PAR as color reagent, but with more complicated FIA system with a microcolumn packed with cation exchange resin, Duolite C-225(H) for on-line separation and preconcentration.

A novel chromatographic resin, U/TEVA. Spec™, was introduced being very selective to uranium and tetravalent actinide ions. The resin consists of diamyl amylphosphonate sorbed on an inert polymeric support (Amberchrom™ CG-71 or Amberlite XAD-7).³

This paper describes the use of U/TEVA. Spec™ for the flow injection on-line separation column in combination with spectrophotometric determination of uranium using PAR color reagent. Comparison of its performance with the one of Duolite C-225(H) resin will be discussed.

Experimental

The flow system

The system shown in Fig. 1 is similar to the one described in our previous work² but it is modified. Peristaltic pump was used for propulsion of reagent streams. A column (40 mm long, 4 mm i.d.) was packed with U/TEVA.Spec™ resin (80–100 μm grain size, Eichrom, Darien, USA). The column replaced the

sample loop of the home-made injection valve.⁴ A standard and sample in 4M HNO₃ solution was passed through the column for 1 minute at a flow-rate of 3 ml·min⁻¹. After washing by passing through 3M HCl solution, the valve was switched to another position where 0.1M HCl eluent passed through for 1 minute at 2.5 ml·min⁻¹ flow-rate. The eluate was then merged with a buffer stream (0.5M triethanolamine, pH 10, 0.007% w/v CDTA) and followed by mixing with PAR stream (0.004% w/v) with a glass bead column (mixing device). The colored product was continuously monitored at 530 nm. The out-put signal was recorded by either a strip chart recorder or PC interfacing, using an in-house software (CMUFIA program).⁵ A peak height vs. concentration of uranium calibration curve was plotted.

Results and discussion

Examples of FIA peaks recorded by PC interfacing are represented in Fig. 2, yielding a linear calibration curve of $y = 425.3x + 71.4$, at least up to 2 ppm U and detection limit of 0.2 ppm U. When using a single standard solution with various loading time, i.e., different amounts of uranium loaded onto the column, a non-linear curve for plot of peak height vs. the uranium amounts obtained, not like in the case of using Duolite C-225(H) resin which yielded a linear relationship.² The relative standard deviation (RSD) was 2.5% (1.0 ppm U, $n = 11$). Sample through-put of 20–25 injections·h⁻¹ can be achieved. Table 1 summarizes the condition for U/TEVA.Spec™ in comparison with the ones of Duolite C-225(H). Study on interferences from some ions are represented in Table 2.

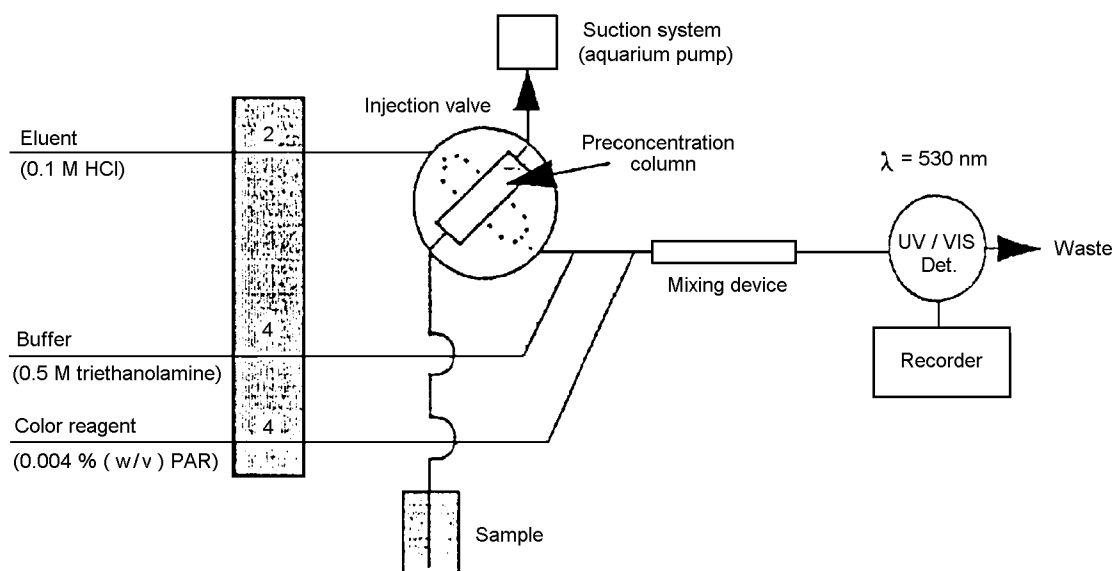


Fig. 1. The flow injection analysis system for uranium determination with on-line separation column

Table 1. Conditions used for U/TEVA.Spec™ and Duolite C-225(H)

	U/TEVA.Spec™	Duolite C-225(H) ²
Loading medium	4M HNO ₃	HNO ₃ pH 2–8
Eluent	0.1M HCl	1M HCl
Buffer solution	0.5M triethanolamine	1M triethanolamine
Masking solution	0.007% w/v CDTA	0.7% w/v CDTA
Loading flow rate	3 ml/min	2 ml/min
Eluent flow rate	2.5 ml/min	4 ml/min
Color reaction	U(VI)-PAR complexation	U(VI)-PAR complexation
Detection	VIS-spectrophotometer (λ=530 nm)	VIS-spectrophotometer (λ=530 nm)

Table 2. Tolerance limit of some interfering ions

Ion/U mass ratio causing 5% deviation in the determination of 0.5 ppm U	Ions
≥1000	Ca(II), Cr(IV), PO ₄ ³⁻ , SO ₄ ²⁻ , CO ₃ ²⁻ , Cl ⁻
200	Ti(IV), Cr(III), Zr(IV), Al(III), F ⁻ , Co(II)
100	Mn(II), Ni(II), Zn(II), Ce(III), Th(IV), Pb(II), W(VI)
50	Cu(II), Sn(IV), Sn(II), Cd(II), La(III)
10	Fe(II), Fe(III), Hg(II)

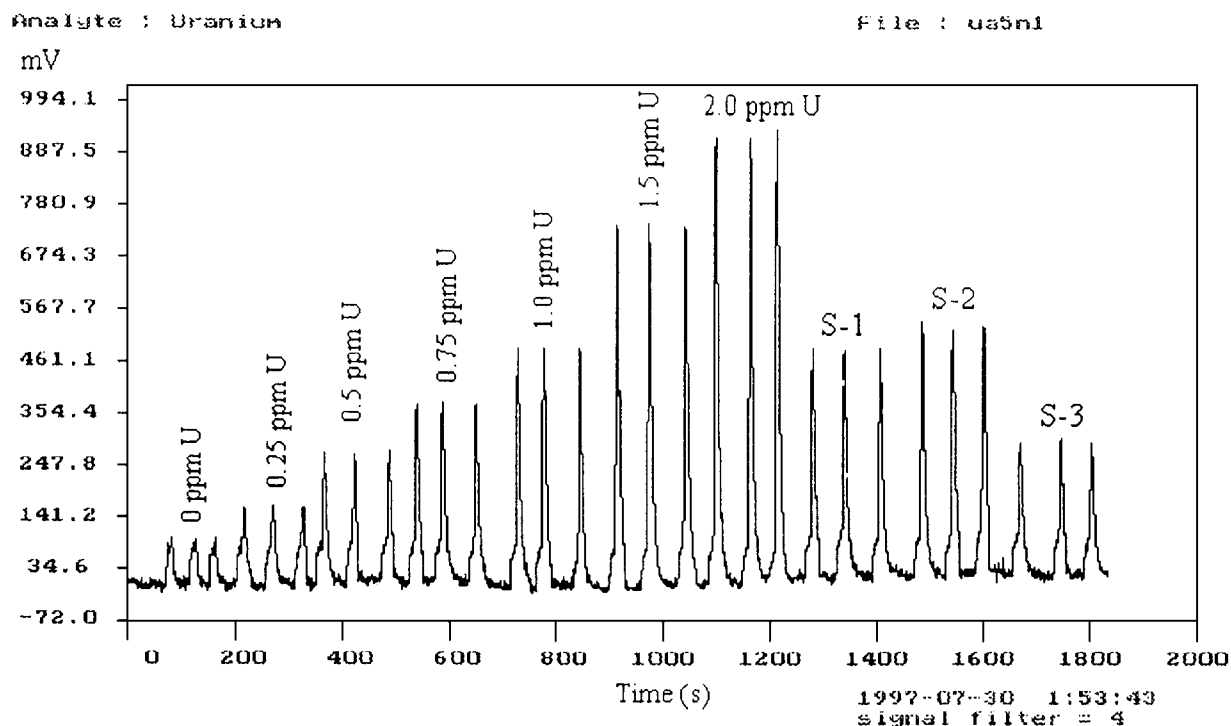


Fig. 2. Examples FIA peaks recorded using PC interface with CMUFIA program

Conclusions

On-line separation column containing a novel chromatographic resin, U/TEVA.Spec™, provides a rapid and high tolerance to interfering ions in the flow injection analysis of uranium using PAR color agent. The application to real samples containing complicate matrix such as geological samples is now under investigation.

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