

Simultaneous determination of trace amounts of thorium and zirconium using spectrophotometric partial least-squares calibration method

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A new, attractive and applicable method, based on spectrophotometric partial least-squares procedure, was proposed for simultaneous determination of thorium and zirconium using SPADNS as a colour reagent. Absorbance measurements were made in the interval of 541–620 nm with 1.0 nm steps in buffered solutions at pH 3.5. The linear dependences were obtained in the ranges of 0.5–11.5 and 1.5–14.5 $\mu\text{g}\cdot\text{ml}^{-1}$ for Th^{4+} and Zr^{4+} ions, respectively. The limits of detection were determined 0.4 and 1.2 $\mu\text{g}\cdot\text{ml}^{-1}$ for thorium and zirconium, respectively. The standard deviation ($n = 3$) and recovery percentage of 10 samples in the prediction set were obtained in the range 0.22–0.38 $\mu\text{g}\cdot\text{ml}^{-1}$ and 91.3–109.2, respectively. The proposed method was used for simultaneous determination of mentioned ions in spiked real water samples and wastewater of AENTC. The satisfactory results showed that the method was applicable to the analysis of samples with similar matrix.

Key words: thorium, zirconium, spectrophotometric determination, SPADNS.

INTRODUCTION

For a number of reasons, spectral measurement is one of the powerful methods in quantitative analysis of chemical mixtures. It is relatively easy to generate good data in short time by proper using of spectroscopy. However, getting useful results from a set of spectral data is not always straightforward. Determining the amounts of the components of a mixture can often be problematic without a prior separation steps because of the overlap of spectral response. For this reason, the analysts have increasingly turned to chemometrics in dealing with spectral data [1].

Quantitative spectrophotometry has been greatly improved by the use of variety of multivariate statistical methods such as classical least square (CLS), inverse least squares (ILS), principal component regression (PCR), and partial least squares (PLS). Multivariate calibrations are effective in spectrophotometric analysis because the simultaneous inclusion of multiple spectral intensities can greatly improve the precision and applicability. The widespread use of these methods is due to the proliferation of commercial software for laboratory computers and detectors, capable of recording full spectra very rapidly [2–4].

Partial least-squares modelling is a powerful multivariate analysis of spectroscopic data. PLS is

capable of being a full spectrum method such as principal component regression and classical least squares. It has also characteristics and the advantages of inverse least-squares method, which is limited in the number of spectral frequencies that can be included in the analysis. The use of PLS method in chemical analysis was pioneered by Wold and co-workers [5]. A particularly detailed study of multivariate calibration by PLS was carried out for spectrophotometric determination of metals [6–11].

Heavy metal ions represent a major environmental problem and their detection and monitoring in waste water outlets, rivers, reservoirs or sources of drinking water is necessary [12]. Thorium is a naturally occurring element that has a number of industrial and medical applications. It is present in very small quantities in virtually all rocks, soils, waters, plants and animals. Where high concentrations occur in rock, thorium can be mined and refined, producing waste products such as mill tailings. If not properly controlled, wind and water can introduce the tailings into the wide environment [13]. Zirconium is used in a wide range of applications including nuclear caps, catalytic converters, surgical appliances, metallurgical furnaces, superconductors, ceramics, lamp filaments, anti-corrosive alloys and photographic purposes. Hence, nanogram level determinations of zirconium are critically important [14, 15].

However, there are few reports on the determination of zirconium and thorium simultaneously. Although strong claims are made for the specificity

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and sensitivity of NAA, ICP-AES and ICP-MS, some interference causes a problem using this method [16]. Therefore, spectrophotometric methods for thorium and rare earth continue to be of interest [17–20].

UV-Vis spectrophotometric techniques, due to both the simplicity and rapidness of the method, are the most widely used analytical methods in multi-component analysis [21–24]. However, the simultaneous determination of thorium and zirconium ions by the use of the traditional spectrophotometry technique is difficult because, the absorption region and the superimposed curves are not suitable for quantitative evaluation. There is a serious problem in most complex samples spectral overlap because the information for each component obtained from the overlapping spectra is very limited and the condition number of the absorbance coefficient matrix is too large to give satisfactory results. In most cases of analysis of complex samples, physical separation and non-standard instrumentation are required. Spectrophotometric techniques as well as chemometrics methods have been recently used to solve these problems [9, 10, 25–27].

In this work, a spectrophotometric method based on using partial least-squares multivariate calibration is proposed for simultaneous determination of Th(IV) and Zr(IV) ions in synthetic and real samples. The method is based on the reaction between these ions with SPADNS as chromogenic reagent at pH 3.5.

EXPERIMENTAL

Reagent

All of the chemicals used were analytical reagent grade of purity. Doubly distilled water was used throughout. Standard solutions of Th(IV) and Zr(IV) ($1000 \mu\text{g}\cdot\text{ml}^{-1}$) were made using $\text{Th}(\text{NO}_3)_4\cdot 6\text{H}_2\text{O}$ in 0.1 M HNO_3 and $\text{ZrOCl}_2\cdot 8\text{H}_2\text{O}$ in 2.0 M HCl , respectively (Merck Co.). The diluted Th(IV) and Zr(IV) solutions were prepared with dilution of stock solutions. A 1.5×10^{-3} M solution of SPADNS as sodium salt was prepared and used daily. Adjusting the pH values of the working solution was carried out using acetic acid and sodium acetate solutions. The foreign ions were introduced using their suitable salts having analytical grade of purity.

Apparatus

A portable 100 UV-Vis scanning spectrophotometer was used to record the absorbance spectra of Th–SPADNS and Zr–SPADNS complexes with a 1.0 cm path length quartz cell. The cell temperature

is controlled by circulating water around the cell by thermostat. The slit width was set at 2 nm and a fast scan speed at 600 nm/min was used. The spectra were recorded between 400 and 700 nm at 1 nm scanning intervals. A Metrohm 691 pH meter equipped with combined glass electrode was used for measurement of the pH. The computations were performed on a Pentium IV computer. All the programs in the computing process were written in MATLAB[®] for Windows.

Procedure

Individual calibration. In order to obtain the calibration curve for each element as an analyte, 1.0 ml of 1.5×10^{-3} M SPADNS as reagent, 2.0 ml of buffer with pH 3.5 and appropriate amounts of the metal ion solution were added to 10 ml volumetric flask and made up to the mark with doubly distilled water. The concentrations of Th(IV) and Zr(IV) ions were 0.50–11.50 and 1.50–14.50 $\mu\text{g}\cdot\text{ml}^{-1}$, respectively, that means the proposed method is a valuable method for simultaneous determination of Th^{4+} and Zr^{4+} in a sample. The absorbances were measured at 580 nm, against a reagent blank for thorium and zirconium ions, respectively.

PLS calibration. To a series of 10 ml volumetric flasks, 1.0 ml of 1.5×10^{-3} M SPADNS as reagent and 2.0 ml of buffer with pH 3.5 were added. The appropriate amounts of each metal ion containing 5.0–115.0 μg of Th(IV) and 15.0–145.0 μg of Zr(IV) were also added and the solutions were made up to the mark with distilled water. These solutions were used for preparation of data sets of calibration and prediction in PLS multivariate calibration method. Excess concentration of SPADNS has been applied to ensure quantitative formation of the complexes in the whole range of calibration. The absorbance of the solutions was measured in the range of 561–640 nm with 1.0 nm steps and therefore 80 experimental points (λ) per spectrum were obtained.

RESULTS AND DISCUSSION

SPADNS or (4,5-dihydroxy-3-(*p*-sulphophenyl-azo)-2,7-naphthalene disulphonic acid, trisodium salt) can be used as an indicator for determination of zirconium and thorium [28]. The complexes stoichiometry of Th(IV)–SPADNS and Zr(IV)–SPADNS is 1:1 and 2:1, respectively. Both normal absorption spectra of Th–SPADNS and Zr–SPADNS complexes show maximum absorption (λ_{max}) value at 580 nm; whereas the free ligand has λ_{max} at 510 nm (Fig. 1).

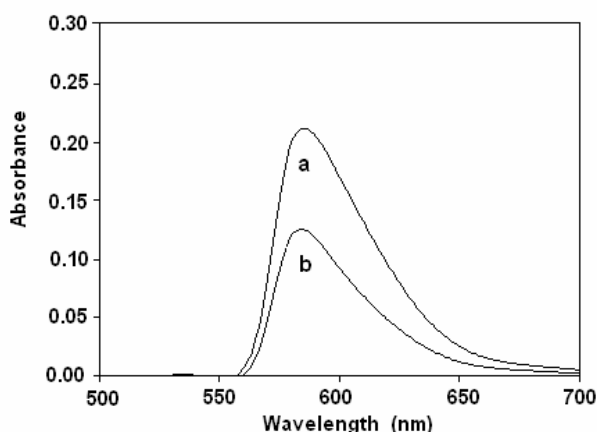


Fig. 1. Absorbance spectra of (a) Th-SPADNS and (b) Zr-SPADNS against reagent blank. Conditions: $C_{\text{SPADNS}} 1.5 \times 10^{-4} \text{ M}$, $C_M 2 \mu\text{g}\cdot\text{ml}^{-1}$, pH 3.5 and $t 25^\circ\text{C}$.

Therefore, a calibration curve was obtained with absorbance measurement of standard solutions of metal complex versus concentration of each metal ion. As it is seen in Fig. 1, since there is overlapping of obtained spectra in the amplitude 560–640 nm, the presence of each metal ion is interfered in measurement of the other ion. Thus, multivariate calibration such as partial-least squares method can be used to determine two analytes in a mixture sample.

Assay conditions, such as pH of the solutions, were investigated for optimization. The effect of pH on the spectrum of each complex was studied separately in the pH range of 2.0–5.0. The absorbance of each complex solution at a constant concentration of metal ion was measured at its λ_{max} and as a function of pH. In the case of both formed complexes, the absorbance of solutions increased up to pH 3.5, then diminished in the pH above 3.5 for Th-SPADNS, whereas, the absorbance of Zr-SPADNS decreased in the pH above 4.5. Therefore, the pH value of 3.5 of acetate buffer was selected as an optimum pH for simultaneous determination of Th^{4+} and Zr^{4+} ions. This pH was achieved via addition of 2.0 ml of buffer solution (pH 3.5) per 10 ml of final solution. Excess concentration of SPADNS ($1.5 \times 10^{-4} \text{ M}$) has been applied to ensure quantitative formation of the complexes in the whole range of calibration.

Single component calibration. In order to find the linear range of concentration of each metal ion, single component calibration was performed for each analyte. In a 10 ml volumetric flask, 1.0-ml SPADNS $1.5 \times 10^{-3} \text{ M}$, 2.0 ml acetate buffer 3.5 and different volumes of $100 \mu\text{g}\cdot\text{ml}^{-1}$ solution of thorium(IV) ion were added and diluted to the mark with distilled water. The absorbance of the thorium(IV) ion standard solutions was measured at

λ_{max} of Th-SPADNS complex (580 nm). The same procedure was followed for zirconium and the absorbance of the solutions was monitored at 580 nm. The linear dependences were obtained in the ranges of 0.50–11.50 and 1.50–14.50 $\mu\text{g}\cdot\text{ml}^{-1}$ for Th^{4+} and Zr^{4+} ions, respectively. The R^2 values for thorium and zirconium calibration curves were obtained to be 0.9987 and 0.9981, respectively. The limits of detection were also determined 0.4 and 1.2 $\mu\text{g}\cdot\text{ml}^{-1}$ for thorium and zirconium, respectively.

Two components calibration. The calibration set contains 30 standard solutions. The compositions of the calibration mixtures were selected, randomly, in the amplitude of calibration curve of each analyte. For preparation of each solution, different volumes of two analyte solutions ($25 \mu\text{g}\cdot\text{ml}^{-1}$) were added to 1.0 ml of SPADNS $1.5 \times 10^{-3} \text{ M}$ and 2.0 ml of acetate buffer 3.5 in a 10 ml volumetric flasks. The concentration of each cation was in the linear range of the cation in single component calibration. The calibration matrix used for the analysis is presented in Table 1. After 5 min, the absorption spectra of the prepared solutions were recorded.

Table 1. Concentration of the components ($\mu\text{g}\cdot\text{ml}^{-1}$) in the calibration set.

No.	Th(IV)	Zr(IV)	No.	Th(IV)	Zr(IV)	No.	Th(IV)	Zr(IV)
1	0.50	1.50	11	10.50	4.00	21	5.50	5.50
2	1.50	3.00	12	11.50	6.00	22	7.50	7.50
3	2.50	4.50	13	1.00	12.00	23	9.50	9.50
4	3.50	6.00	14	3.00	10.00	24	11.50	11.50
5	4.50	7.50	15	5.00	8.00	25	11.50	14.50
6	5.50	9.00	16	7.00	6.00	26	8.50	10.00
7	6.50	10.50	17	9.00	4.00	27	6.50	8.00
8	7.50	12.00	18	11.00	2.00	28	4.50	6.00
9	8.50	13.50	19	1.50	1.50	29	2.50	4.00
10	9.50	2.00	20	3.50	3.50	30	1.50	2.00

Selection of the number of factors. The selection of the number of the factors in the PLS algorithm is very important to achieve the best prediction. The number of factors was estimated in the PLS1 by cross-section validation method, leaving out one sample at a time and plotting the prediction residual sum of squares (PRESS) versus the number of factors for each individual component [29–31]. Predictive residual error sum of squares (PRESS) was computed using the following equation:

$$\text{PRESS} = \sum (C_{\text{true},i} - C_{\text{pred},i})^2$$

Where, $C_{\text{true},i}$ is the known concentration for i^{th} sample and $C_{\text{pred},i}$ represents the concentration predicted by the model. The PLS calibrations were performed based on the 29 spectra out of 30 calibration spectra. The components in the sample left out during calibration were predicted using this

calibration. This process was repeated 30 times and in each step of calibration, one sample was left out. The prediction residual sum of squares was calculated by comparing the predicted concentration of components in each sample with known concentration of components in standard solutions. To optimize the number of factors, the F-statistic with F-ratio probability of 0.75 was used. The optimum number of factors was selected for the first PRESS values the F-ratio probability, which drops down below 0.75. The Fig. 2 shows, the PRESS obtained by optimizing the calibration matrix of the absorbance data with PLS. The optimal number of factors for thorium and zirconium was obtained to be 5 and 6, respectively.

Statistical parameters. To evaluate prediction ability of a multivariate calibration model, the root mean square error of prediction (*RMSEP*), the square of the correlation coefficient (R^2) and relative standard error of prediction (*RSEP*) can be used.

$$RMSEP = \sqrt{\sum_{i=1}^n (C_{pred} - C_{true})^2 / n}$$

$$R^2 = \frac{\sum_{i=1}^n (C_{pred} - \bar{C}_{true})^2}{\sum_{i=1}^n (C_{true} - \bar{C}_{true})^2}$$

$$RSEP(\%) = 100 \times \sqrt{\sum_{i=1}^n (C_{pred} - C_{true})^2 / \sum_{i=1}^n (C_{true})^2}$$

where C_{pred} is the predicted concentration in the sample, C_{true} is the true value of the concentration in the sample, \bar{C}_{true} is the mean true value of the concentration in the sample and n is the number of samples in the prediction test.

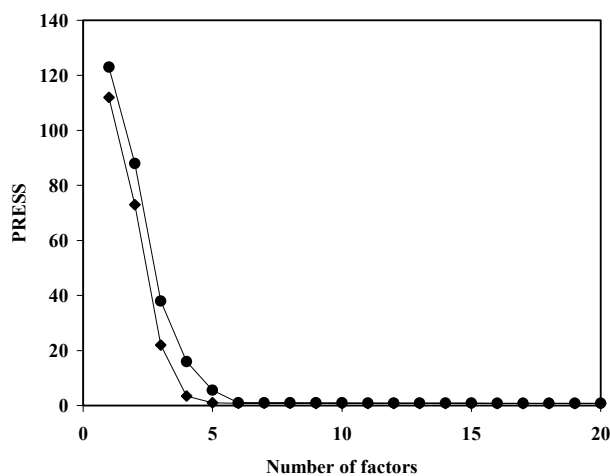


Fig. 2. Plot of PRESS versus number of factors: (♦) Th(IV) and (●) Zr(IV).

Simultaneous determination of thorium and zirconium was made on the 10 synthetic test samples by the proposed method. Figs. 3 and 4 show plots of predicted concentrations versus true concentrations using the optimized model. Predicted results and their recovery percentage are given in Table 2.

Table 2. Concentration of the components ($\mu\text{g}\cdot\text{mL}^{-1}$) in the prediction set, predicted values and recovery percent.

No	True value		Predicted value ^a		Recovery, %	
	Th(IV)	Zr(IV)	Th(IV)	Zr(IV)	Th(IV)	Zr(IV)
1	0.50	1.50	0.52 ± 0.25	1.37 ± 0.25	104.0	91.3
2	1.50	3.00	1.53 ± 0.27	2.96 ± 0.24	102.0	98.7
3	1.50	10.00	1.60 ± 0.22	10.92 ± 0.28	107.0	109.2
4	4.00	3.00	3.86 ± 0.28	3.06 ± 0.31	96.5	102.0
5	4.00	12.00	4.16 ± 0.25	11.96 ± 0.25	104.0	99.7
6	10.00	2.50	10.28 ± 0.38	2.33 ± 0.27	102.8	93.2
7	10.00	10.00	9.78 ± 0.25	10.10 ± 0.25	97.8	101.0
8	5.00	1.50	5.36 ± 0.26	1.60 ± 0.30	107.2	106.7
9	5.00	8.00	5.42 ± 0.33	7.77 ± 0.28	108.4	97.1
10	11.50	14.50	11.28 ± 0.25	14.24 ± 0.35	98.00	98.2

^a Mean ± S.D. (n = 3)

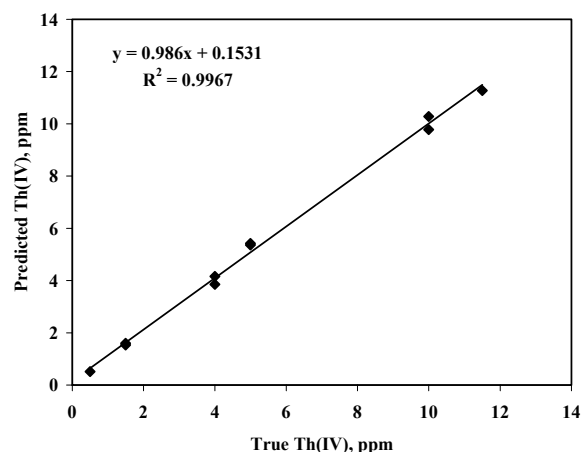


Fig. 3. Plots of predicted concentration versus true concentration for Th(IV) ion in the prediction set.

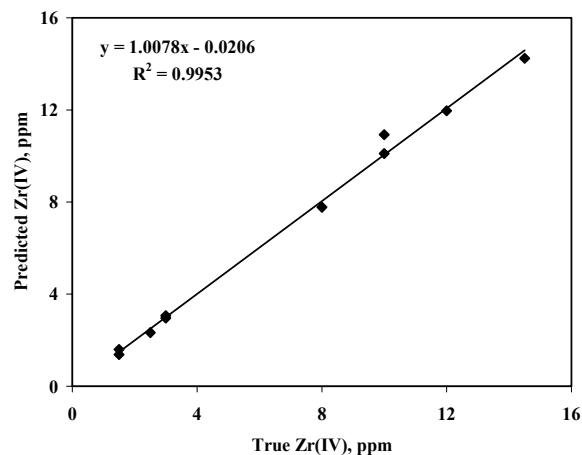


Fig. 4. Plots of predicted concentration versus true concentration for Zr(IV) ion in the prediction set.

Table 3. Statistical parameters of the test matrix.

Analyte	No. of factors	RMSEP	R ²	RSEP, %
Th(IV)	5	0.0448	0.9967	2.18
Zr(IV)	6	0.0535	0.9953	1.18

Table 4. Tolerance limits for the diverse ions in the determination of Th(IV) and Zr(IV) ions.

Interfere ion	Tolerance limit (W _{Ion} /W _M)
Li ⁺ , Ag ⁺ , K ⁺ , NH ₄ ⁺ , Mg ²⁺ , Sr ²⁺ , Ba ²⁺ , Ca ²⁺ , HCO ₃ ⁻ , I ⁻ , Br ⁻ , Cl ⁻ , F ⁻ , NO ₃ ⁻ , NO ₂ ⁻ , S ₂ O ₈ ²⁻ , SO ₄ ²⁻ , SCN ⁻ , S ₂ O ₃ ²⁻ , IO ₃ ⁻ , ClO ₃ ⁻	1000
Hg ²⁺ , Ti(IV), Mo(VI), Cr(VI), Mn(VII)	500
Cr ³⁺ , Sn ⁴⁺ , Sn ²⁺ , Ti(III), V(IV), V(V)	100
Mn ²⁺ , Co ²⁺ , Ni ²⁺ , Zn ²⁺ , Cu ²⁺ , Cd ²⁺ , Pb ²⁺ , Bi ³⁺ , HPO ₄ ²⁻ , H ₂ PO ₄ ⁻	50
Fe ²⁺ , PO ₄ ³⁻	10
F ⁻ , Fe ³⁺ , Al ³⁺	1

The standard deviation and recovery percentage of 10 samples in the prediction set were obtained in the range 0.22–0.38 µg·ml⁻¹ and 91.3–109.2, respectively. Also, the values of RMSEP, R², RSEP (%) and number of factors according to the obtained results for prediction test are summarized in Table 3.

Effect of foreign ions. The interference by several cations and anions on the determination of two components (5 µg·ml⁻¹) has been investigated by multivariate calibration method. The tolerance limit for each foreign ion is obtained when its presence at tested weight ratio produced a variation in concentration of analytes lower than 5%. The results (Table 4) indicate that the most of the cations and anions did not show any significant spectral interference at weight ratio greater than 1000. The most interfering ions are F⁻, Fe³⁺ and Al³⁺ ions.

Application of the method. The proposed method was successfully applied to determination of thorium and zirconium in several real water spiked samples and wastewater of AENTC after dilution (Table 5). Thorium and zirconium were not detected prior to spiking samples using AAS (atomic absorption spectroscopy) method. As it is seen from Table 5, good recovery and low standard deviation was obtained by the PLS method using absorbance data. Therefore, this proposed method can be used for the analysis of samples of similar matrix, effectively.

CONCLUSION

Thorium(IV) and zirconium(IV) have chemical properties which can interfere with each other in their determination. For example, in spectrophotometric methods using colour reagents for complex formation with these elements, a high spectral overlapping can be observed between the absorption spectra of these components. Thus, there are reliable

Table 5. Analysis of real samples.

Sample	Spiked, µg/ml ^a		Found, µg/ml ^b		Recovery, %	
	Th(IV)	Zr(IV)	Th(IV)	Zr(IV)	Th(IV)	Zr(IV)
Tap water	5.0	5.0	5.2 ± 0.2 (5.1 ± 0.2) ^c	4.8 ± 0.3 (4.7 ± 0.1)	104.0	96.0
River water	5.0	5.0	4.9 ± 0.3 (4.9 ± 0.2)	5.3 ± 0.1 (4.9 ± 0.3)	98.0	106.0
Spring water	5.0	5.0	4.8 ± 0.3 (4.9 ± 0.2)	4.6 ± 0.2 (4.7 ± 0.3)	96.0	92.0
Waste water of AENTC ^d			240 ± 6 (245 ± 5)	195 ± 4 (198 ± 4)		

^a Thorium and zirconium were not detected prior to spiking samples using AAS method; ^b Proposed method and mean ± S.D. (n = 3); ^c AAS; ^d The actual amounts of Th and Zr are 250 and 200 µg/ml, respectively.

methods for simultaneously determination of these elements in the real samples. However, the partial least-squares (PLS) method shows two advantages in this work: (i) it is applicable to analysis of two analyte in a sample; (ii) there is not need of pre-treatment in complex samples.

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ЕДНОВРЕМЕННО СПЕКТРОФОТОМЕТРИЧНО ОПРЕДЕЛЯНЕ НА СЛЕДИ ОТ ТОРИЙ И ЦИРКОНИЙ ЧРЕЗ ЧАСТИЧЕН МЕТОД НА НАЙ-МАЛКИТЕ КВАДРАТИ

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(Резюме)

Предложен е нов спектрофотометричен метод, основаващ се на процедурата на най-малките квадрати, за едновременно определяне на торий и цирконий с реагент SPADNS. Абсорбционните измервания са проведени в интервала 541–620 nm при стъпка 1.0 nm в буферен разтвор с рН 3.5. Получени са линейни зависимости в областите 0.5–11.5 и 1.5–14.5 $\mu\text{g}\cdot\text{ml}^{-1}$ съответно за Th^{4+} и Zr^{4+} . Границите на откриване са 0.4 и 1.2 $\mu\text{g}\cdot\text{ml}^{-1}$, стандартното отклонение ($n = 3$) е 0.22–0.38 $\mu\text{g}\cdot\text{ml}^{-1}$ и процентният добив от 10 образци е 91.3–109.2% съответно за торий и цирконий. Предлагаият метод е използван за едновременно определяне на посочените йони в дотирани реални проби от вода, както и в отпадни води. Задоволителните резултати показват, че методът е приложим за анализ на образци с подобен състав.