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Simultaneous determination of vanadium(IV) and vanadium(V) by flow injection analysis using kinetic spectrophotometry with Xylenol Orange

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ABSTRACT

A flow injection method of analysis has been developed for the simultaneous determination of V(IV) and V(V) using the difference in the rate of complex formation between vanadium in two different oxidation states and Xylenol Orange in acidic media. The proposed method used a spectrophotometric detector equipped with two flow cells aligned with the same optical path to yield two successive peaks per each sample injection. V(IV) and V(V) were determined by solving simultaneous equations in two unknowns, the concentrations of V(IV) and V(V), obtained from measurements of the two peak heights. The detection limit calculated as 3σ of noise signals was 0.01 μg/mL for both V(IV) and V(V). The relative standard deviations for V(IV) and V(V) at the 1 μg/mL level were 1.6 and 2.4%, respectively. The sample throughput was found to be about 19 h^{-1}. The proposed method was successfully applied to the simultaneous determination of V(IV) and V(V) in synthetic scrubbing solutions used in desulphurization with reasonable accuracy.

1. Introduction

Vanadium is an important element in environmental and biological studies. Vanadium can exist in oxidation states from II to V in aqueous solution. However, most analytical methods have concentrated on its determination in the states IV and V, as these are the most common forms encountered in inorganic and biological systems. The toxicity of vanadium is dependent on its oxidation state, with V(V) being more toxic than V(IV) [1,2]. Therefore, it is essential to differentiate V(V) from V(IV) in environmental and biological samples for a better understanding of the toxicity of vanadium.

A little information is available about the simultaneous determination of V(IV) and V(V). Jen and Yang developed a liquid chromatographic method for the simultaneous determination of V(IV) and V(V) by reversed-phase ion-pair liquid chromatography using a C_8 column and an eluent containing acetonitrile, ethylenediaminetetraacetic acid (EDTA) and tetrabutylammonium ion [3]. The separation of V(IV) and V(V) as EDTA complexes was also carried out using a C_8 column and an eluent containing ammonium acetate, tetrabutylammonium hydroxide, ammonium diphosphate and EDTA [4]. Chromatographic separation between V(IV) and V(V) in river and lake waters was performed with a mixture of ethylenediamine and tartaric acid as the eluent and a separation column packed with strongly acidic cation exchange resin [5].

Recently, capillary electrophoresis (CE) was applied to the simultaneous determination of V(IV) and V(V) as negatively charged complexes with EDTA [6] or diethylenetriaminepentaacetic acid (DTPA) [7]. Despite advantages with respect to simplicity and selectivity, the pre-column complexation methods do not meet all requirements for routine analysis, because of the decomposition of V(IV) and V(V) chelates during CE separation. Kitazumi et al. [8] developed a following capillary electrophoretic method in order to solve the above problem. The method is based on the pre-column formation of the $[P(V^{IV}Mo_{11})O_{40}]^{5-}$ and $[P(V^{V}Mo_{11})O_{40}]^{4-}$ complexes, which were then separated from each other by capillary electrophoresis with direct UV detection. An automated method for determining V(IV) and V(V) in natural waters was developed by combining a selective column extraction using two kinds of chelating resins and an air-segmented continuous flow analyzer (ASCFA) coupled with catalytic detection with Bindschelder's green leuco base-$KBrO_3$ system. V(V) in a sample solution adjusted at pH 2.2 to 3.8 was selectively adsorbed on an acetylacetone-immobilized resin column and V(IV) in the eluent was then adsorbed on an 8-quinolinol-immobilized resin column. V(V) and V(IV) adsorbed on the columns were successively eluted with dilute hydrochloric acid and the eluent was introduced into the ASCFA [9]. Although the proposed method gives excellent sensitivity and reproducibility, it requires various reagent solutions and the whole analysis system is somewhat tedious to use.

The present work was undertaken to develop a kinetic flow injection method for the simultaneous determination of V(IV) and V(V) based on the difference in the rate of complex formation of

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V(IV) and V(V) with Xylenol Orange (XO) in acidic media. To the best of our knowledge, the complexation kinetics of V(IV/V) and XO was only used in spectrophotometric determination of V(IV) in the presence of V(V) by the H-point standard addition method [10] and has not been considered previously for the simultaneous determination of V(IV) and V(V) by flow injection analysis.

2. Experimental

2.1. Reagents

All reagents used were of analytical reagent grade. Distilled and deionized water was further purified via passage through a Milli-Q Ultrapure water purification system (Nihon Millipore, Tokyo).

Vanadium(IV) stock solution: 0.9937 g of $VOSO_4 \cdot nH_2O$ (Kanto Chemicals, Tokyo) was dissolved in 4 mL of 0.5 M H_2SO_4 and an appropriate amount of ascorbic acid to give the final concentration of 1 μM, followed by dilution to 200 mL with water. The vanadium(IV) solution thus prepared was standardized by back titration with thorium standard solution against Xylenol Orange, yielding 0.970 mg V(IV)/mL.

Vanadium(V) stock solution: 0.4592 g of NH_4VO_3 (Kanto Chemicals, Tokyo) was dissolved in water and diluted to 200 mL with water. The vanadium(V) solution thus prepared was standardized as above after addition of ascorbic acid to reduce V(V) to V(IV), yielding 0.999 mg V(V)/mL.

The other metal stock solutions were standard solutions for atomic absorption spectrometry (Wako Pure Chemical Industries, Tokyo).

XO stock solution: 0.7166 g of XO (3,3′-bis[*N*,*N*-di(carboxymethyl)aminomethyl]-*o*-cresolsulfonphthalein, disodium salt) (Dojin Chemical Laboratories, Kumamoto, Japan) was dissolved in water and diluted to 200 mL with water. The XO solution was used as a chromogenic reagent solution at 0.5 mM after 10 fold dilution with water.

Formate buffer solution (pH 3.5): 8 mL of 98% formic acid (Wako Pure Chemical Industries, Tokyo) and 6.47 g of sodium formate (Kanto Chemicals, Tokyo) were dissolved in water and diluted to 100 mL with water. The formate buffer solution prepared thus was diluted 10 times with water before use.

Citrate (pH lower than 2.8) and acetate (pH higher than 3.9) buffer solutions were also used to examine the effect of pH on the heights of the second peaks of V(IV) and V(V).

2.2. Instrumentation

A Toa-Denpa Industry HM-7E pH meter (Toa-Denpa Industries, Tokyo) was used to measure pH of solutions. Absorption spectra of vanadium–XO complexes were measured by a Hitachi U-3210 spectrophotometer (Hitachi, Tokyo) in preliminary experiments.

Fig. 1 shows the schematic diagram of the FIA system used for the simultaneous determination of V(IV) and V(V). The flow system was assembled from 0.5 mm bore PTFE tubing. The carrier (0.02 M H_2SO_4) and Xylenol Orange solutions were delivered by GL Sciences PU611 HPLC pumps (GL Sciences Inc., Tokyo) and the formate buffer solution was delivered by a GL Sciences PUD-16 non-pulsation pump (GL Sciences Inc., Tokyo). The absorbance of the vanadium complexes was monitored by a Soma S-3250 spectrophotometric detector (Soma Optics, Tokyo) and recorded by a Hitachi 561 recorder (Hitachi, Tokyo).

2.3. General procedure for simultaneous determination of V(IV) and V(V)

A 90-μL volume of the 0.02 M H_2SO_4 solution containing V(IV) and V(V) was injected into the carrier stream (0.02 M H_2SO_4, 1.1 mL/min) and merged with the 0.5 mM XO solution (1.3 mL/min). Vanadium(IV) and V(V) reacted with XO in the first reaction coil(RC1) (0.5 mm × 1 m) and the absorbance (A_1) of the formed V(IV/V)-XO complexes was measured at 530 nm in one of the two flow cells aligned with the same optical path of a spectrophotometric detector. The sample zone was then merged with the 0.2 M formate buffer solution (pH 3.5, 0.7 mL/min) and passed through the second reaction coil (RC2) (0.5 mm × 17 m), followed by the measurement of the absorbance (A_2) of V(IV/V)–XO complexes at 530 nm in another flow cell. Hence, two peaks were obtained per injection as shown in Fig. 2 and the concentrations of V(IV) and V(V), namely, [V(IV)] and [V(V)], were calculated by solving the following simultaneous equations.

$$A_1 = (X_{1,\mathrm{IV}}[\mathrm{V(IV)}] + Y_{1,\mathrm{IV}}) + (X_{1,\mathrm{V}}[\mathrm{V(V)}] + Y_{1,\mathrm{V}}) \quad (1)$$

$$A_2 = (X_{2,\mathrm{IV}}[\mathrm{V(IV)}] + Y_{2,\mathrm{IV}}) + (X_{2,\mathrm{V}}[\mathrm{V(V)}] + Y_{2,\mathrm{V}}) \quad (2)$$

Here, the constants, $X_{1,\mathrm{IV}}$, $X_{1,\mathrm{V}}$, $X_{2,\mathrm{IV}}$, $X_{2,\mathrm{V}}$, $Y_{1,\mathrm{IV}}$, $Y_{1,\mathrm{V}}$, $Y_{2,\mathrm{IV}}$ and $Y_{2,\mathrm{V}}$, were obtainable by each calibration graph constructed for V(IV) and V(V), respectively. The complex formation reaction of V(V) with XO under acidic conditions was much faster than that of V(IV) under the same conditions [10,11]. Actually, peak heights were recorded and used to calculate the concentrations of V(IV) and V(V).

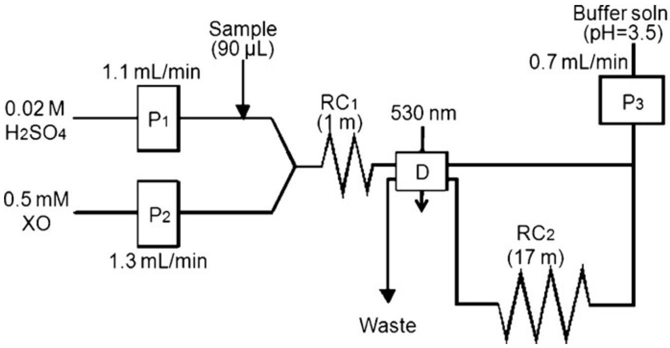

Fig. 1. Flow injection system used for simultaneous determination of V(IV) and V(V). P1, P2 and P3: plunger pumps; RC1 and RC2: reaction coils; D: spectrophotometric detector equipped with two flow cells aligned with the same optical path.

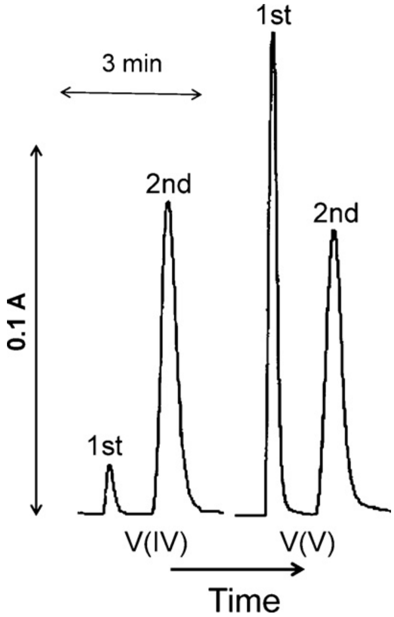

Fig. 2. FI profiles for V(IV) and V(V). V(IV): 3.5 μg/ml; (V): 5.0 μg/mL.

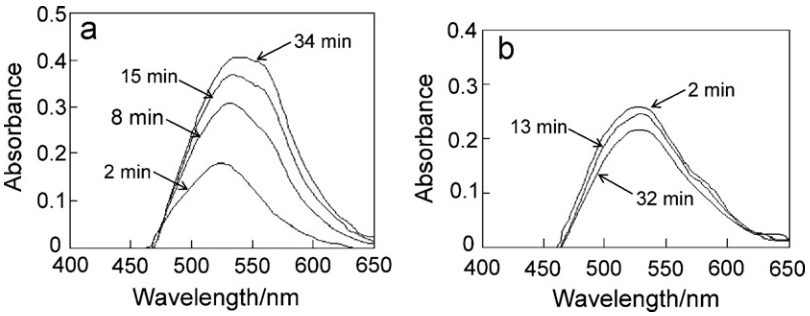

Fig. 3. Variation of visible absorption spectra of V(IV)–XO and V(V)–XO complexes as a function of elapsed time after the preparation of sample solutions. (a) V(IV)–XO, (b) V(V)–XO. V(IV), V(V): 1 μg/mL, XO: 2.5×10^{-4} M, pH: 1.5.

2.4. *Analysis of the synthetic scrubbing solutions in the desulphurization*

A sample solution was prepared according to the method used by De Haan and Vermaire [12]. About 2.4 g of $NaHCO_3$ and 1.0 g of NaSCN were dissolved in 30 mL of water and appropriate amounts of V(IV) and V(V) stock solutions were added. The solution was transferred into a 50-mL volumetric flask and diluted to the mark with water. A 90-μL aliquot of the sample solution prepared thus was introduced into the flow injection analysis system for the simultaneous determination of V(IV) and V(V) as described in Section 2.3.

3. Results and discussion

3.1. *Complexation kinetics of V(IV/V) with XO*

The time variation of absorption spectra for V(IV/V)–XO complexes at pH 1.5 was measured in preliminary experiments. As show in Fig. 3, the V(V)–XO complexes were formed rapidly in agreement with the results by Budevsky and Pribil [13]. On the other hand, the V(IV) reacted rather slowly with XO to form complexes and their absorbance increased with time, showing the progress of the complex formation. These results are in accord with those by Safavi et al. [10] and Otomo [11] and suggest the possibility of the simultaneous kinetic determination of V(IV) and V(V) with XO.

3.2. *Optimization of experimental conditions of FIA*

3.2.1. *Wavelength for absorbance measurement*

Preliminary batchwise experiments showed that the absorption maximum wavelength of V(IV)–XO complexes shifted gradually from 530 nm to ca. 560 nm with the elapse of time, while that of V(V)–XO complexes remained almost constant at 540 nm (see Fig. 3). Based on these observations, the absorbance of V(IV)/V(V)–XO complexes were monitored at 530 nm.

3.2.2. *Reaction coil length*

The effect of the length of RC1 was examined over the range from 50 to 300 cm keeping the length of RC2 at 300 cm (Fig. 4). Both first and second peak heights for V(V) were practically constant over the coil length tested. The first peak height for V(IV) increased slightly with increasing length of RC1, while the second peak height for V(IV) showed the opposite tendency. Therefore, the length of RC1 was fixed at 100 cm.

A PTFE tubing of 17 m was used as RC2 to ensure the separation between the first and second peaks and accelerate the reaction of V(IV) with XO.

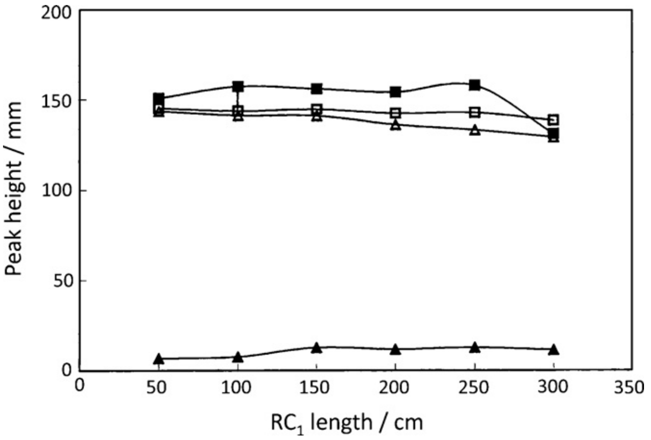

Fig. 4. Effect of the length of RC1. V(IV), V(V): 1.2 μg/mL, carrier: 0.02 M H_2SO_4, XO concentration: 5×10^{-4} M, the length of RC_2: 300 cm. (■) First peak of V(V), (□) second peak of V(V), (▲) first peak of V(IV), (△) second peak of V(IV).

3.2.3. *Concentration of carrier solution (sulfuric acid)*

The rate of complex formation between V(IV) and XO is slower than V(V) complex formation under acidic conditions (pH less than 2) and increases markedly between pH 2 and 3 [10,11]. Accordingly, 0.02 M sulfuric acid was used as a carrier solution to keep the slow rate of V(IV)–XO complex formation and detect a large difference in the first peak heights for V(IV) and V(V).

3.2.4. *Effect of pH of buffer solution*

The effect of pH of a buffer solution added to the sample solution after measuring the first peak height was examined over the pH range of 2.8–4.7 and the results obtained are illustrated in Fig. 5. The second peak heights for V(IV) were almost constant at the pH around 3 and then gradually decreased with an increase in pH. Therefore, formate buffer solution of pH 3.5 was merged with the sample solution to adjust its pH at 3.2 after the measurement of the first peak heights.

3.2.5. *Effect of XO concentration*

Increasing concentration from 0.05 mM to 0.3 mM of XO in the chromogenic reagent solution resulted in the increase of all peak heights for V(IV) and V(V) as shown in Fig. 6. The second peak heights for both V(IV) and V(V), however, increase only slightly in the XO concentration region of higher than 0.3 mM, and 0.5 mM XO solution was hereinafter used for the chromogenic reagent solution.

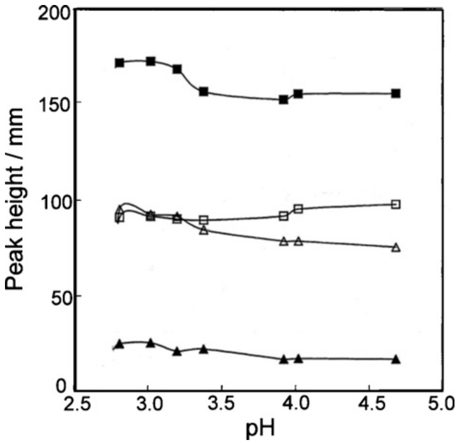

Fig. 5. Effect of pH of the buffer solution. V(IV), V(V): 1.2 μg/mL, carrier: 0.02 M H_2SO_4, XO concentration: 5×10^{-4} M. (■) First peak of V(V), (□) second peak of V(V), (▲) first peak of V(IV), (△) second peak of V(IV).

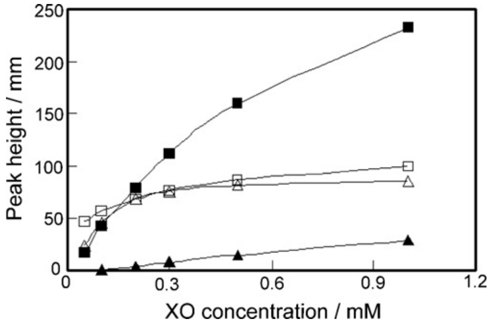

Fig. 6. Effect of XO concentration. V(IV), V(V): 1.2 μg/mL, carrier: 0.02 M H_2SO_4, pH of buffer solution: 3.5. (■) First peak of V(V), (□) second peak of V(V), (▲) first peak of V(IV), (△) second peak of V(IV).

3.2.6. *Effect of flow rates of carrier, chromogenic reagent and buffer solutions*

The effect of flow rates of carrier, XO and buffer solutions were examined over 0.5–1.3, 0.5–1.6, and 0.5–1.3 mL/min, respectively and the results obtained are summarized in Fig. 7. The first peak heights for V(IV) and V(V) slightly decreased with increase in the flow rate of the carrier solution. The second peak height for V(V) remained almost constant regardless of the flow rate of the carrier solution while the second peak height for V(IV) increased slightly with increase in the carrier flow rate and reached a plateau at 1 mL/min. Therefore, the flow rate of the carrier solution was fixed at 1.1 mL/min.

The increasing flow rate of XO solution gave a slight decrease in the first peak heights for both V(IV) and V(V). On the other hand, the second peak heights for V(IV) and V(V) showed a little increase with the flow rate of XO solution. The flow rate of XO solution was adjusted at 1.3 mL/min as the compromise between the decrease tendency of the first peak heights and the increase tendency of the second peak heights as described above.

The flow rate of the buffer solution hardly affected the first and second peak heights for both V(IV) and V(V), so that it was fixed at 0.7 mL/min.

3.3. *Effect of diverse ions*

The effect of diverse ions was examined and their tolerable concentrations are summarized in Table 1. Fe(III), Ti(IV), Ga(III), In(III), Al(III), Bi(III) and Th(IV) interfered seriously with the determination of V(IV)/V(V). Organic complexing reagents, *e.g.*, polycarboxylic acids and aminopolycarboxylic acids, cannot be used here as masking agents because those reagents mask V(IV) and V(V), too. Therefore, masking of the interfering metal ions was attempted by adding 0.016% sodium fluoride in the chromogenic reagent solution. The results are collected in Table 2, showing that fluoride is effective to mask Al(III) and Th(IV), permitting the determination of V(IV) and V(V) with relative errors of 7% and 11% at the most in the presence of 4 μg/mL Al(III) and 5 μg/mL Th(IV), respectively.

3.4. *Performance of the FIA system*

The detection limit calculated as 3σ of noise signals was 0.01 μg/mL for both V(IV) and V(V). The linear calibration graphs for both the first and second peaks were obtained in the range 0.3–5 μg/mL for V(IV) and 0.4–5 μg/mL for (V), respectively. The relative standard deviations for V(IV) and V(V) at the 1 μg/mL level were 1.6 and 2.4%, respectively. The sample throughput was found to be about 19 h^{-1}.

Examples of the calibration graphs obtained under the optimized experimental conditions are as follows:

$$H_1 = 12.9\,[\mathrm{V(IV)}] + 133.8\,[\mathrm{V(V)}] - 11.3 \quad (3)$$

$$H_2 = 74.6[\mathrm{V(IV)}] + 67.6\,[\mathrm{V(V)}] + 3.4 \quad (4)$$

where H_1 and H_2 are the first peak height (in mm) and the second peak height (in mm), respectively, and [V(IV)] and [V(V)] are the concentrations (in μg/mL) of V(IV) and V(V), respectively.

4. Simultaneous determination of V(IV) and V(V) in admixture

V(IV) and V(V) in admixture (0.02 M sulfuric acid solution) were determined simultaneously by the general procedure given in Section 2.3 and the results obtained are given in Table 3. Each admixture of V(IV) and V(V) was analyzed once so that the paired *t*-test [14] was used to check if the measured concentrations were statistically significantly different from the added concentrations. The calculated *t*-values were 1.31 and 2.21 for V(IV) and V(V),

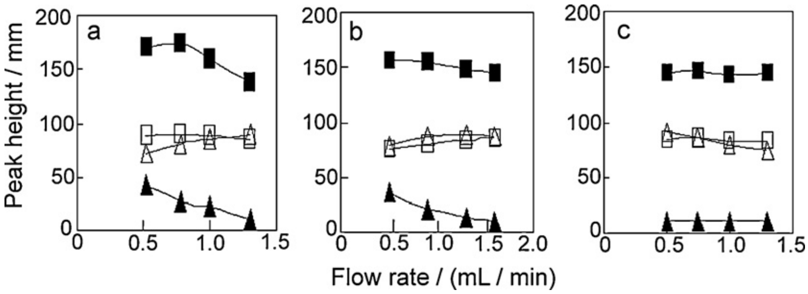

Fig. 7. Effect of flow rates of carrier, XO and buffer solutions. (a) Carrier solution, (b) XO solution, (c) buffer solution. V(IV), V(V): 1.2 μg/mL, carrier: 0.02 M H_2SO_4; XO concentration: 5×10^{-4} M; pH of buffer solution: 3.5. (■) First peak of V(V), (□) second peak of V(V), (▲) first peak of V(IV), (△) second peak of V(IV).

Table 1
Tolerable concentrations of diverse ions.[a]

Ion	Concentration, μg/mL	Ion	Concentration, mol/L
Fe(III)	0.1	$S_2O_3^{2-}$, citrate, oxalate	2×10^{-5}
Ti(IV)	0.2	Tartrate	0.001
Ga(III), In(III)	0.3	PO_4^{3-}	0.01
Al(III), Bi(III), Th(IV)	0.5	SO_4^{2-}	0.03
Cu(II), Mo(VI), Pd(II)	1	Cl^-	0.1
Co(II), Ni(II), U(VI), Zn(II)	2	NO_3^-	0.2
Ca(II), Mg(II), W(VI)	4		
Cd(II), Se(IV)	20		
Mn(II)	40		
Sb(III), Sr(II)	100		

[a] The highest concentration giving relative errors less than ±10% to each one of the V ions in the simultaneous determination of 1 μg/mL each of V(IV) and V(V).

Table 2
Masking with fluoride.[a]

Cation	Added, μg/mL	V(IV)		V(V)	
		Added, μg/mL	Found, μg/mL	Added, μg/mL	Found, μg/mL
Al(III)	4	1.00	1.07	1.00	0.94
Bi(III)	5	1.00	0.44	1.00	2.29
Fe(III)	1	1.00	1.02	1.00	1.79
Ga(III)	3	1.00	2.20	1.00	1.25
In(III)	3	1.00	0.77	1.00	2.09
Th(IV)	5	1.00	1.11	1.00	1.04
Ti(IV)	2	1.00	1.20	1.00	1.13

[a] 0.016% NaF added to XO solution.

Table 3
Simultaneous determination of V(IV) and V(V) in admixtures.[a]

Run		Added, μg/mL	Found, μg/mL	Run		Added, μg/mL	Found, μg/mL
1	V(IV)	0.56	0.47	4	V(IV)	1.4	1.4
	V(V)	0.80	0.83		V(V)	2.0	2.1
2	V(IV)	0.56	0.60	5	V(IV)	1.4	1.4
	V(V)	1.2	1.2		V(V)	3.0	3.1
3	V(IV)	0.84	0.84	6	V(IV)	2.1	2.2
	V(V)	0.80	0.79		V(V)	2.0	2.1

[a] Prepared as 0.02 M sulfuric acid solution.

The calculated *t*-values for V(IV) and V(V) were 1.31 and 2.21, respectively. The tabulated *t*-value for five degrees of freedom at 95% confidence level is 2.51.

Table 4
Simultaneous determination of V(IV) and V(V) in synthetic solution used in the removal process for hydrogen sulfide.[a]

Added V(IV), μg/mL	V(V), μg/mL	Found[b] V(IV), μg/mL	V(V), μg/mL
0.0	2.0	0.03 ± 0.08 (t = 0.649)[c]	2.04 ± 0.07 (t = 0.989)
0.50	1.5	0.58 ± 0.04 (t = 3.46)	1.50 ± 0.04 (t = 0)
1.0	1.0	1.08 ± 0.04 (t = 3.46)	0.99 ± 0.06 (t = 0.288)
1.5	0.50	1.56 ± 0.08 (t = 1.30)	0.53 ± 0.03 (t = 1.73)
2.0	0.0	2.02 ± 0.07 (t = 0.494)	0.11 ± 0.02 (t = 9.52)

[a] 0.25 M in NaSCN and 0.57 M in $NaHCO_3$ [12].
[b] Av. ± S.D. (n = 3).
[c] Calculated *t*-values. The tabulated value of *t* at the 95% confidence level is 4.303 for two degrees of freedom.

respectively. The tabulated *t*-value for five degrees of freedom at 95% confidence level is 2.51 so that there is no significant difference between the measured concentrations and the added concentrations at this confidence level.

4.1. Simultaneous determination of V(IV) and V(V) in synthetic scrubbing solutions in the desulphurization

The proposed method was applied to the simultaneous determination of V(IV) and V(V) in synthetic scrubbing solutions in the desulphurization, which were prepared according to the literature [12], and the results obtained are summarized in Table 4. In order to check if the measured concentrations are statistically significantly different from the added concentrations, the *t*-test was utilized regarding the added concentrations as “true” concentrations [15]. The *t*-values calculated for V(IV) and V(V) in each run are included in Table 4. The tabulated *t*-value for two degrees of freedom at 95% confidence level is 4.303. Therefore, there is no significant difference between the added concentrations and the measured concentrations at this confidence level except for V(V) in Run 6, and this indicates that the presence of $NaHCO_3$ and NaSCN as matrix components give no interference.

5. Conclusion

A simple FIA method was developed for the simultaneous determination of V(IV) and V(V) using the difference in the rate of their complex formation with XO in acidic media. The proposed flow injection method of analysis does not require any special reagents and equipment, and skilled techniques. The present method was easily applied to the analysis of synthetic scrubbing solutions used in desulphurization with a reasonable accuracy.

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