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# First derivative spectrophotometric determination of uranium(VI) and vanadium(V) in natural and saline waters and some synthetic matrices using PAR and cetylpyridinium chloride

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## Abstract

A first derivative spectrophotometric method has been developed for the simultaneous determination of uranium(VI) and vanadium(V) using 4-(2'-pyridylazo)resorcinol (PAR) in the presence of cetylpyridinium chloride (CPC) and EDTA. As a result of the addition of CPC, the first derivative spectra of vanadium showed two minima at 575 and 600 nm, the latter being found to be more useful in its determination. Calibration graphs based on first derivative measurements of uranium and vanadium at 563 and 600 nm were found to be linear in the range of 0.4–4.0 and 4.0–16  $\mu\text{g ml}^{-1}$  with a  $3\sigma$  detection limit of 0.25 and 3.0  $\mu\text{g ml}^{-1}$ , respectively. The determination of 2  $\mu\text{g ml}^{-1}$  uranium and 4.0  $\mu\text{g ml}^{-1}$  vanadium in spiked river water samples gave quantitative recovery and relative standard deviations ( $n = 3$ ) of 0.6 and 0.8%, respectively. The method has been applied to the determination of uranium and vanadium in some synthetic matrices, river and saline water samples. © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** Uranium(VI); Vanadium(V); Spectrophotometry

## 1. Introduction

Uranium and its compounds are of great interest especially during the processing of uranium ores, and the reprocessing of nuclear fuel. Therefore the development of reliable analytical methods for the determination of uranium is always in demand. Spectrophotometry represents an attractive, common technique adequate for solving many analytical problems. And many spectrophotometric methods using different reagents have been proposed for the determination of uranium. Among

the common reagents used for uranium determination are Chrome Azurol S [1], chlorophosphonazo [2], 5-(2-carboxyphenyl)azo-8-quinolinol [3] *p*-carboxychlorophosphonazo [4], 4-(2-thiazolylazo)-6-chlororesorcinol [5] and arsenazoIII [6]. These methods are not completely free from interference and the addition of masking agents or the use of a pre-separation process is always needed to overcome interferences. The thiocyanate-triphenylmethane dyes method [7] is quite sensitive but also lacks selectivity. Pyridylazo compounds such as 4-(2'-pyridylazo)resorcinol (PAR) are widely used as analytical reagents for the sensitive spectrophotometric determination of uranium [8–12], despite also being non-selective. The addition of surfactants has been used to increase the sensitivity [13–16]. The extraction of

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U(VI)-PAR using tridodecylethylammonium bromide has been reported [17]. Using PAR in the presence of masking agents such as EDTA [12], fluoride [12], CDTA [11], or sulphosalicylic acid [11,12], most interfering ions except vanadium can be masked. Pure vanadium is one of the important elements that accompanies uranium in ores (such as carnotite [18]), was the principal interfering element in the spectrophotometric determination of uranium [12,19,20]. However, the V-PAR chelate has been proposed as the basis of a sensitive spectrophotometric method for the determination of vanadium at 550 nm, where CDTA has been used to mask most interfering metal ions; only U, Nb and Ti interfere [21]. Babko et al. [22] have shown that the complex formed at pH 5–6 is anionic and is extractable by chloroform in the presence of organic cations such as tetraphenylarsonium, tetraphenylphosphonium [23], nitron and zephiramine [24]. Thus, the extractive spectrophotometric determination of vanadium in natural water and aluminum alloys using PAR and iodinitrotetrazolium chloride has been reported [25].

Few data have been reported for the simultaneous determination of uranium and vanadium in their binary mixtures [26,27], and no reports about using PAR for such a determination. Derivative spectrophotometry [28–30], a powerful tool for overcoming interference due to the spectral overlap, offers the distinct advantage of increasing selectivity and sensitivity. The present study describes a first derivative spectrophotometric method for the simultaneous determination of uranium and vanadium using PAR as a reagent.

## 2. Experimental

### 2.1. Apparatus

A Shimadzu UV/VIS spectrophotometer, Model 2401 (Kyoto, Japan) was used for measuring the absorbance and recording the normal and derivative spectra. First derivative spectra were recorded using  $\Delta\lambda = 16$  nm and a scaling factor of 20. An Orion model 330 pH meter was used for pH measurements.

### 2.2. Reagents and materials

All chemicals used were of analytical grade and doubly distilled water was always used. Vanadium(V)

oxide, PAR, sodium acetate and acetic acid were obtained from Merck. Uranyl acetate, cetyltrimethylammonium bromide (CTMAB) and cetylpyridinium chloride monohydrate (CPC) were obtained from BDH. A uranium stock solution containing  $1000 \mu\text{g ml}^{-1}$  of U(VI) was prepared by dissolving 0.1782 g of uranyl acetate in 1% nitric acid, and diluting to 100 ml. A stock solution of vanadium was prepared by dissolving 0.1789 g of vanadium(V) oxide in 1 ml of concentrated sulfuric acid, then making up to volume with water in a 100 ml calibrated flask to obtain a  $1000 \mu\text{g ml}^{-1}$  V(V) solution. The 0.01 M solutions of PAR, CPC and acetate buffer, pH 6.0 and 1% EDTA were prepared in water.

### 2.3. Analytical procedure

Transfer an aliquot of the sample solution containing appropriate amounts of U(VI) and V(V) into a 25 ml measuring flask, and add 10 ml of buffer and 1 ml of EDTA solution. Add 2.5 ml of PAR solution and 2 ml of CPC solution. Complete to the mark with water and measure the spectra from 450–650 nm against the reagent blank (1 cm cells). Record the first derivative spectra using  $\Delta\lambda = 16$  nm and a scaling factor of 20. Measure the distance  $h_1$  and  $h_2$  for uranium and vanadium at 563 and 600 nm, respectively, in the derivative spectra (see below), and calculate the concentration of each element from preconstructed calibration graphs.

## 3. Results and discussion

### 3.1. Absorption spectra

Due to the anionic nature [17,22,31] of the U(VI)-PAR and V(V)-PAR complexes, cationic surfactants like CPC and CTMAB were examined for their possible effect on the absorbance of both complexes. In the presence of either CPC or CTMAB the absorbance increased, but CPC showed a more distinctive effect. The zero-order absorption spectra of uranium(VI) and vanadium(V) as well as a mixture of the two ions, in solutions containing PAR, CPC and EDTA, are shown in Fig. 1. The wavelength of maximum absorbance ( $\lambda_{\text{max}}$ ) was found to be 551 and 563 nm for the U(VI)-PAR-CPC and V(V)-PAR-CPC,

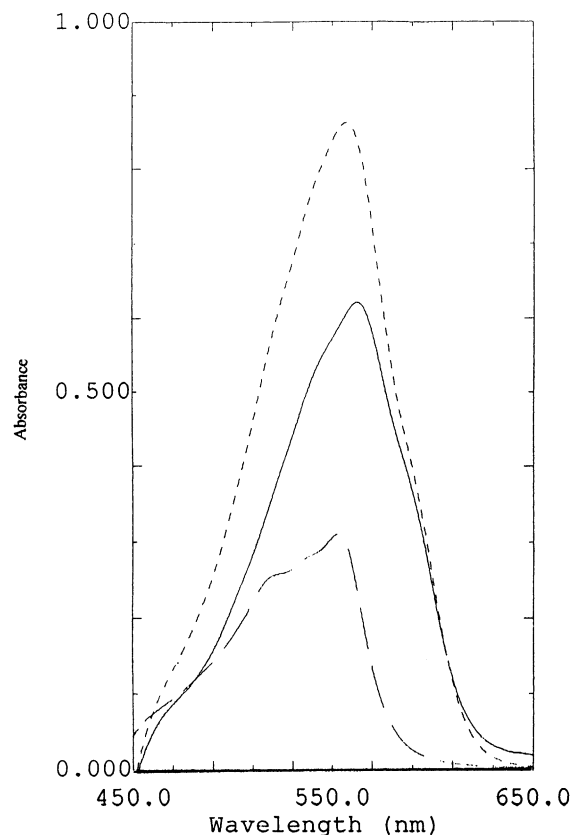


Fig. 1. Zero-order absorption spectra of  $2.0 \mu\text{g ml}^{-1}$  U(VI) (curve, - - -),  $8.0 \mu\text{g ml}^{-1}$  V(V) (curve, —) and a mixture of  $2 \mu\text{g ml}^{-1}$  U(VI) and  $4.0 \mu\text{g ml}^{-1}$  V(V) (curve, ···), using PAR and CPC at pH 6.0.

respectively, compared with 530 and 550 nm for U(VI)-PAR and V(V)-PAR systems [31]. This indicates a bathochromic shift as a result of the formation of a ternary metal ion complex with PAR and CPC with improved optical properties. These results are in agreement with data reported earlier for U(VI) [8] and V(V) [32,33]. At the same time the spectra of U and V in the mixture overlap significantly, rendering the simultaneous determination of the two ions using zero order spectra quite difficult.

### 3.2. Effect of pH

The effect of variation of pH on the absorbance of PAR-U(VI)-CPC and PAR-V(V)-CPC has been examined. It has been found that the absorbance of  $2 \mu\text{g ml}^{-1}$  U(VI) in the presence of  $8 \times 10^{-4}$  M CPC

and  $1 \times 10^{-4}$  M PAR and 1 ml of 1% EDTA has its maximum value over the pH range 6–9. At the same time the absorbance of  $8 \mu\text{g ml}^{-1}$  V(V) under the same conditions was found to have its maximum value at pH 6.0. These values are comparable with pH 7–8 for U-PAR and pH 6.5 for V-PAR [27]. An Acetate buffer of pH 6.0 was chosen for carrying out the forthcoming measurements.

### 3.3. Effect of PAR Concentration

The effect of PAR concentration on the absorbance of  $2 \mu\text{g ml}^{-1}$  U and  $8 \mu\text{g ml}^{-1}$  V using  $8 \times 10^{-4}$  M CPC at pH 6.0 was examined. The maximum absorbance was obtained at  $1 \times 10^{-4}$  M PAR in both instances and remained almost constant with increasing concentration up to  $2.5 \times 10^{-4}$  M.

### 3.4. The effect of surfactant concentration

On studying the effect of CPC concentration on the absorbance of  $2 \mu\text{g ml}^{-1}$  U(VI) and  $8 \mu\text{g ml}^{-1}$  V(V), it was found that the absorbance reaches to its maximum value at  $8 \times 10^{-4}$  M CPC in both instances, therefore, this concentration was used in all subsequent measurements. It is worth mentioning that in the presence of CPC, not only is the absorbance of the uranium and vanadium complexes increased, but also the trough of V is split into two troughs in the first-order derivative spectrum as shown in Fig. 2.

### 3.5. Effect of diverse ions

The effect of the presence of diverse ions on the determination of uranium(VI) with the PAR in the presence of CPC has been investigated. Table 1 shows the tolerable amounts of the examined ions in the determination of  $4 \mu\text{g ml}^{-1}$  U(VI). It is clear that in the presence of 1 ml of 1% EDTA solution all interfering ions are masked except vanadium(V) and titanium(III), the latter must be absent in the determination of uranium and vanadium.

### 3.6. Effect of EDTA on the absorbance of uranium and vanadium

The PAR is known to form colored complexes with many metal ions that contribute to the absorbance in

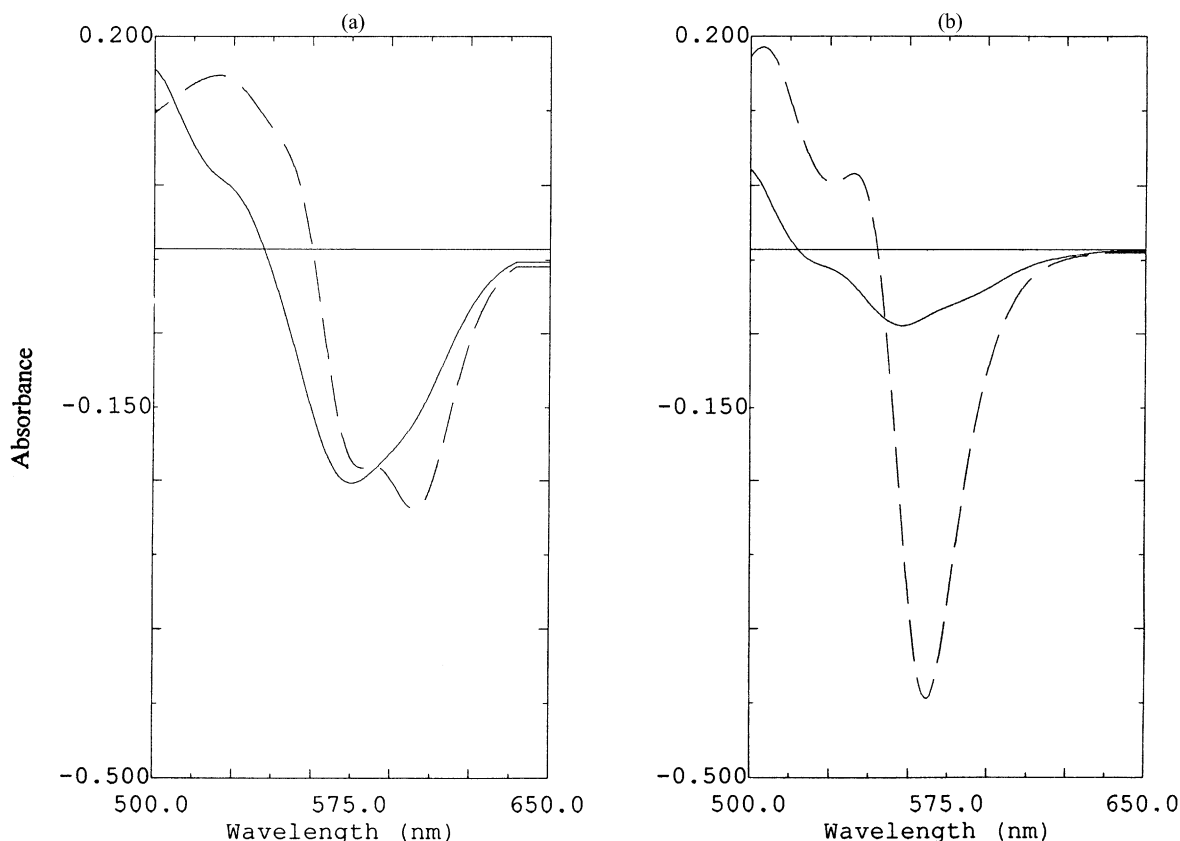


Fig. 2. Effect of  $8 \times 10^{-4}$  M CPC on the derivative signal of (a)  $10 \mu\text{g ml}^{-1}$  V(V) and (b)  $2 \mu\text{g ml}^{-1}$  U(VI), (curve, - - -) with and (curve, —) without CPC.

the region of maximum absorbance of uranium and vanadium. Therefore, a masking reagent is usually used to increase the selectivity of PAR toward the analyte. Among the most common reagents used for this

Table 1

The effect of various ions on the determination of  $4 \mu\text{g ml}^{-1}$  U(VI) with PAR in the presence of CPC

Ion	Ion/U(VI) ratio	Error (%)
Ti(III), V(V)	1	>100
Zr(IV), $\text{Fe}^{2+}$ , Ti(IV), $\text{Cr}^{3+}$	1	>5
$\text{Cd}^{2+}$ , Sb(III), $\text{Th}^{4+}$ , $\text{Pb}^{2+}$	10	<2
$\text{Ti}^{+}$ , $\text{Ca}^{2+}$ , $\text{Co}^{2+}$ , $\text{Ni}^{2+}$ , $\text{Cu}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Mg}^{2+}$ , $\text{K}^{+}$ , $\text{Mn}^{2+}$	20	<2
$\text{Cl}^{-}$ , $\text{SO}_4^{2-}$ , $\text{PO}_4^{3-}$ , $\text{SiO}_3^{2-}$ , $\text{I}^{-}$ , $\text{CO}_3^{2-}$	20	<2

purpose are EDTA and CDTA. While some authors reported that CDTA can mask V(V) in the determination of U(VI) [11], others have reported that it still interferes [12]. Moreover, the determination of vanadium with PAR in the presence of CDTA as a masking agent has been reported [21]. In the same time, EDTA has been reported to decrease the signal of both uranium and vanadium more than CDTA [11,12,21]. However, when investigating the effect of both reagents on the PAR-CPC system, (results shown in Fig. 3), it was found that 4 ml of 1% EDTA solution has decreased the uranium signal to 91.0% of its nominal value, while the vanadium signal was decreased to 4.5% of its value. On the other hand 4 ml of 1% CDTA reduced the signal of uranium and vanadium to 35.0 and 3.5% of their nominal values, respectively. It is also worth mentioning that the amount of CDTA used to mask

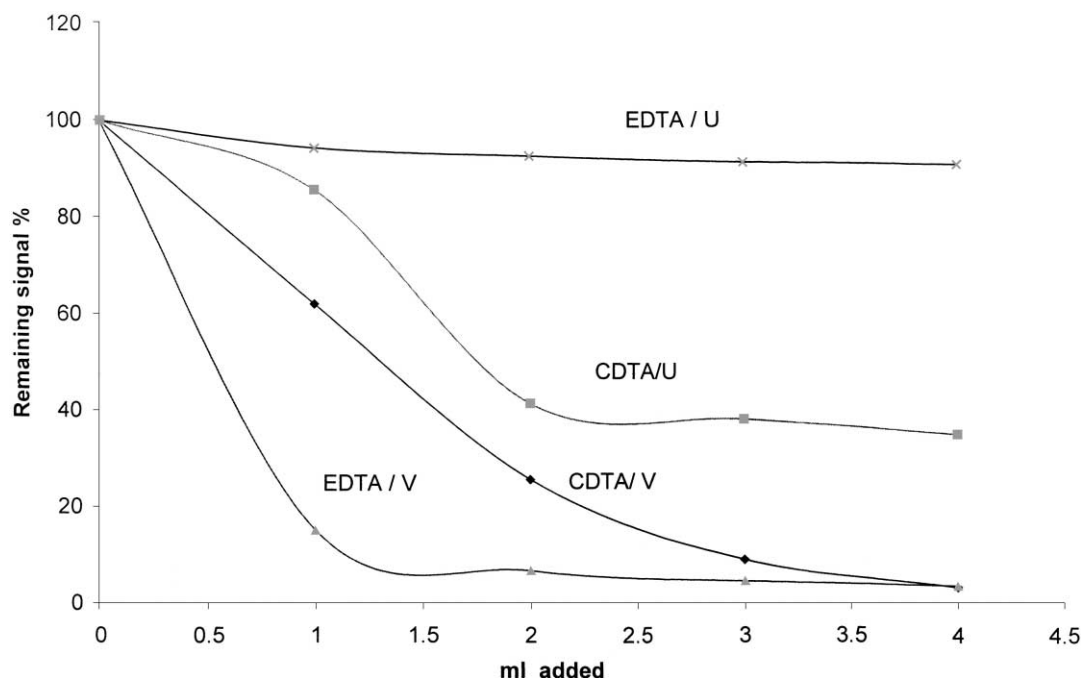


Fig. 3. The effect of addition of different volumes of 1% solution of EDTA and CDTA on the signal of  $4 \mu\text{g ml}^{-1}$  U(VI) and  $10 \mu\text{g ml}^{-1}$  V(V).

the interference in the determination of vanadium [21] and uranium [12] was 5 ml of an 0.05 M solution and 2 ml of a 5% CDTA solution, respectively. The recommended amount of CDTA in both reports is almost equivalent to 10 ml of 1% EDTA solution, which is quite large. Therefore, we decided to use EDTA. Thus, by addition of 1 ml of 1% EDTA solution the signal for uranium was very slightly decreased, while that of vanadium, which has been decreased to 18% of its value, remained measurable as shown in Fig. 4.

### 3.7. Derivative spectra

The first-order derivative spectra of U(VI)-PAR-CPC and V(V)-PAR-CPC were found to have zero-crossing points at 551 and 563 nm, respectively, as shown in Fig. 2. At the same time the first-order derivative spectra of two sets of solutions, each set containing 10 ml of acetate buffer of pH 6.0, 1 ml of EDTA, 2.5 ml of PAR, and 2 ml of CPC solution, were recorded in the range 450–650 nm. The first set contained increasing concentrations of uranium (0.0, 0.5, 1.0, 1.5, 2.0,

2.5, 3.0, and  $4.0 \mu\text{g ml}^{-1}$ ) and a fixed concentration of vanadium ( $10 \mu\text{g ml}^{-1}$ ) and the other contained increasing concentrations of vanadium (0.0, 4.0, 6.0, 8.0, 10.0, 12.0, 14.0, 16.0 and  $18.0 \mu\text{g ml}^{-1}$ ) and a fixed concentration of uranium ( $2.0 \mu\text{g ml}^{-1}$ ). The first derivative spectra of the first set of solutions were found to exhibit an isodifferential point at 551 nm and two troughs at 563 and 600 nm as shown in Fig. 5a. The amplitude ( $h_1$ ) of the trough at 563 nm (zero crossing point of vanadium) was found to be proportional to the concentration of U(VI). For the derivative spectra of the second set of solutions exhibited an isodifferential point at 563 nm and two troughs at 575 and 600 nm as shown in Figs. 4b and 5. The concentration of vanadium was found to be proportional to the signal at 551 nm (zero crossing point of uranium). Also, the concentration of vanadium correlates linearly with the amplitude of the trough at 600 nm ( $h_2$ ). However, a calibration graph based on  $h_2$  was found to be more accurate ( $\varepsilon = 2.8 \times 10^3 \text{ l mol}^{-1} \text{ cm}^{-1}$ ) compared to that constructed using measurements at 551 nm, the zero crossing wavelength of uranium

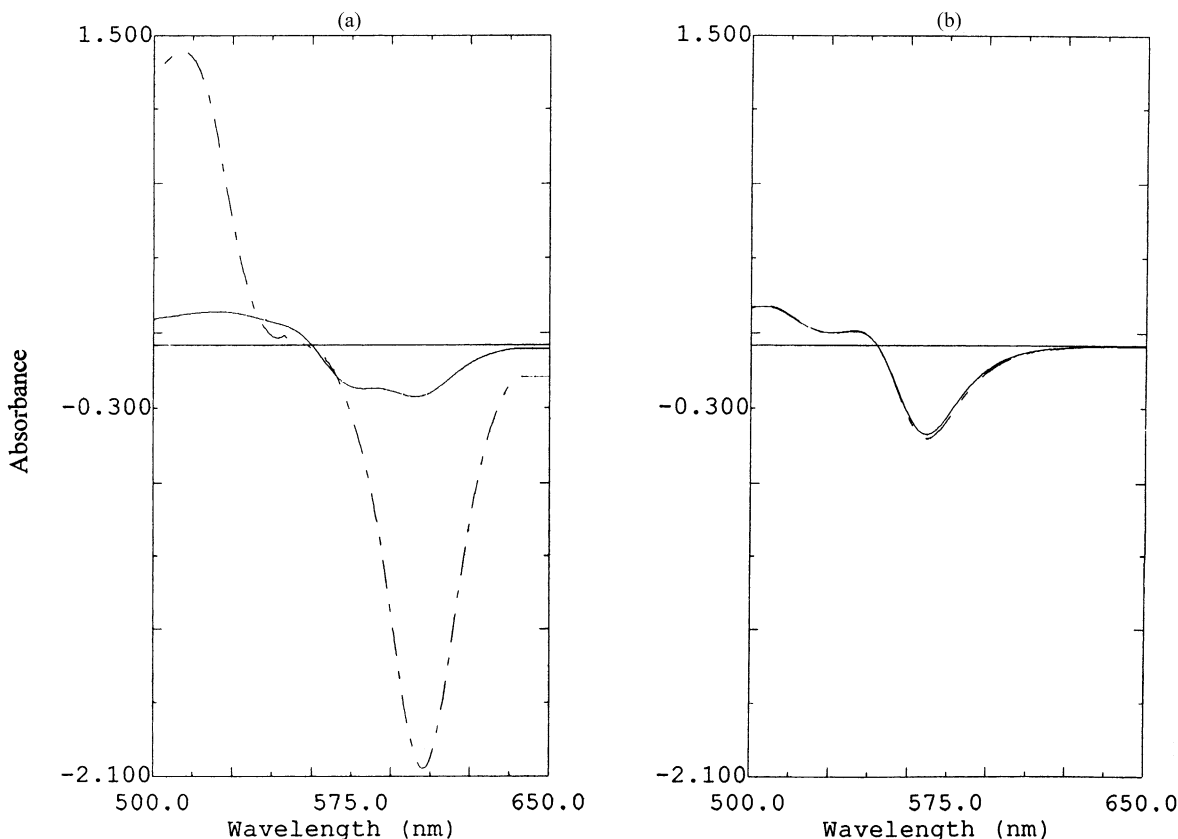


Fig. 4. The effect of addition of 1 ml of 1% EDTA solution on the derivative signal of (a)  $10 \mu\text{g ml}^{-1}$  V(V) and (b)  $2 \mu\text{g ml}^{-1}$  of U(VI), (curve, —) with and (curve, - - -) without EDTA.

( $\epsilon = 6.2 \times 10^2 \text{ l mol}^{-1} \text{ cm}^{-1}$ ). The trough at 600 nm appeared as a result of the addition of CPC to the reaction system as mentioned before. Calibration graphs were prepared by plotting the distances  $h_1$  and  $h_2$ , the amplitude of the troughs at 563 and 600 nm in the first-order spectra, against the concentration of uranium and vanadium, respectively. The concentrations of uranium and vanadium in their binary solutions were determined by measuring  $h_1$  and  $h_2$  in the first-order derivative spectra of the solutions, and comparing the values obtained with the preconstructed calibration graphs for U(VI) and V(V).

### 3.8. Calibration graphs

The calibration graph for uranium was found to be linear over the range  $0.4\text{--}4.0 \mu\text{g ml}^{-1}$  ( $r =$

$0.998$ ,  $n = 6$ ) with a  $3\sigma$  detection limit of  $0.25 \mu\text{g ml}^{-1}$ . The calibration graph for vanadium was found to be linear over the range  $4\text{--}18 \mu\text{g ml}^{-1}$  ( $r = 0.965$ ,  $n = 6$ ) with a detection limit of  $3 \mu\text{g ml}^{-1}$ . The calibration graphs furnished a molar absorptivity of  $2.7 \times 10^4$  and  $2.8 \times 10^3 \text{ l mol}^{-1} \text{ cm}^{-1}$  for uranium and vanadium, respectively.

### 3.9. Simultaneous determination of uranium and vanadium

To investigate the versatility of the method, a series of five test solutions containing different known amounts of uranium and vanadium were prepared and analyzed by the proposed analytical procedure. The results in Table 2 show quantitative recovery of both elements.

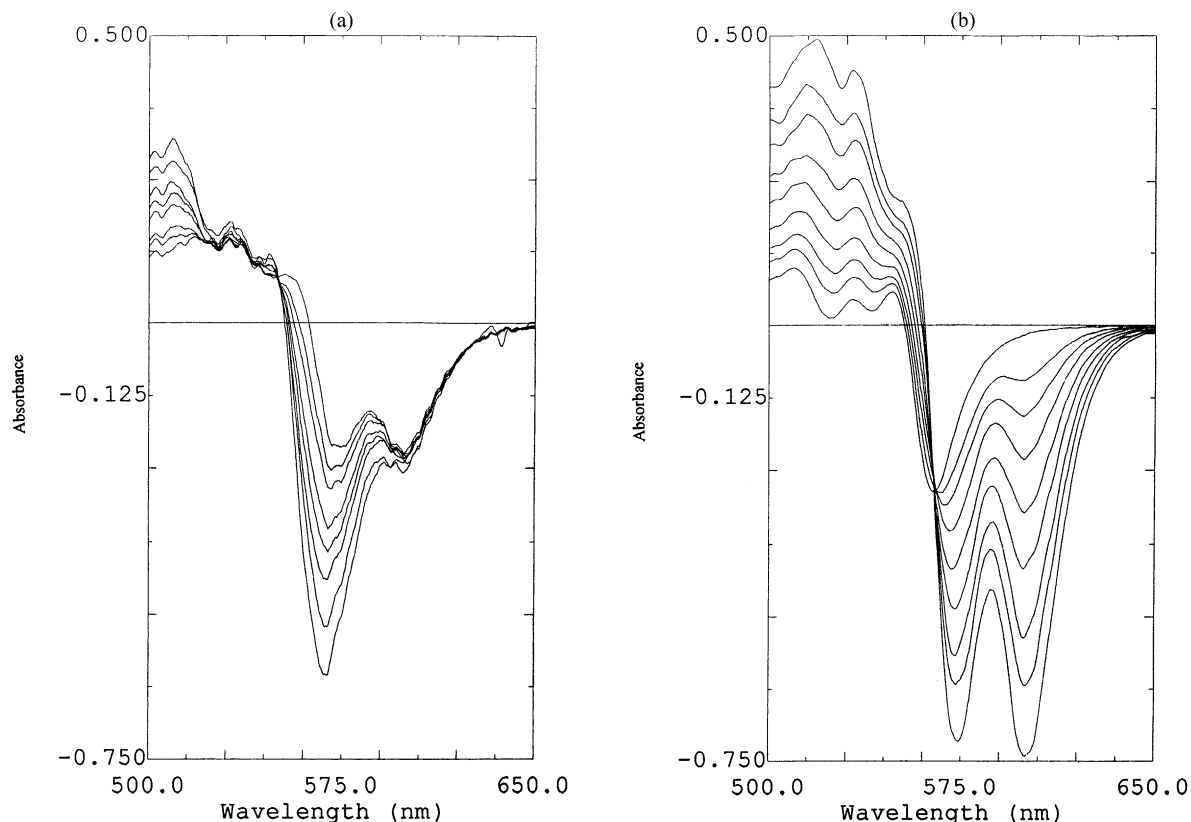


Fig. 5. The first derivative spectra of two sets of solutions, each containing PAR, CPC, and EDTA at pH 6.0: (a) containing  $10 \mu\text{g ml}^{-1}$  vanadium and different concentrations of uranium (0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, and  $4.0 \mu\text{g ml}^{-1}$ ). (b) Containing  $2.0 \mu\text{g ml}^{-1}$  uranium and different concentrations of vanadium (0.0, 4.0, 6.0, 8.0, 10.0, 12.0, 14.0, 16.0 and  $18.0 \mu\text{g ml}^{-1}$ ).

### 3.10. Determination of uranium and vanadium in spiked river water samples

The performance of the described method was tested in real water matrices using River Nile water

samples spiked with uranium and vanadium. The results in Table 3 show a 99.2% average recovery of uranium and a relative standard deviation (R.S.D.) of 0.6 at  $2.0 \mu\text{g ml}^{-1}$ , and a 97.5% average recovery of vanadium and R.S.D. of 0.55 at  $8 \mu\text{g ml}^{-1}$  (both  $n = 6$ ).

Table 2  
Simultaneous determination of U(VI) and V(V) in their mixtures<sup>a</sup>

Sample no.	U(VI) ( $\mu\text{g ml}^{-1}$ )				V(V) ( $\mu\text{g ml}^{-1}$ )			
	Added	Found	<i>R</i> (%)	R.S.D. (%)	Added	Found	<i>R</i> (%)	R.S.D. (%)
1	0.5	0.51	102.0	0.60	7.0	7.07	101.0	0.40
2	1.0	0.99	99.0	0.50	4.0	3.93	97.5	0.41
3	2.0	2.05	102.0	0.60	5.0	4.90	98.0	0.80
4	1.0	0.97	97.0	0.70	9.0	8.95	99.0	1.20
5	2.0	1.98	99.0	0.55	12.0	12.10	100.8	0.57

<sup>a</sup> Each result is the average of three determinations; *R*: recovery.



Table 3  
Simultaneous determination of U(VI) and V(V) in spiked River Nile water<sup>a</sup>

Sample no.	U(VI) ( $\mu\text{g ml}^{-1}$ )				V(V) ( $\mu\text{g ml}^{-1}$ )			
	Added	Found	R (%)	R.S.D. (%)	Added	Found	R (%)	R.S.D. (%)
1	2.0	2.04	102.0	0.50	8.0	8.08	101.0	0.55
2	1.0	1.02	101.5	0.65	10.0	10.1	101.0	0.60
3	4.0	3.92	98.0	0.40	4.0	4.08	102.0	0.80
4	2.0	1.94	97.0	0.80	6.0	5.97	99.5	0.70
5	3.0	2.92	97.5	0.75	12.0	11.7	97.5	0.90

<sup>a</sup> Each result is the average of three determinations.

Table 4  
Simultaneous determination of uranium and vanadium in spiked saline water<sup>a</sup>

Sample no.	U(VI) ( $\mu\text{g ml}^{-1}$ )				V(V) ( $\mu\text{g ml}^{-1}$ )			
	Added	Found	R (%)	R.S.D. (%)	Added	Found	R (%)	R.S.D. (%)
1	2.0	1.82	91.0	0.90	8.0	7.76	97.0	0.80
2	1.0	0.865	86.5	0.40	10.0	9.89	98.9	1.10
3	4.0	2.92	73.0	0.45	4.0	4.12	103.0	0.95
4	2.0	1.72	86.0	0.55	6.0	6.10	101.0	0.65
5	3.0	2.58	86.0	0.70	12.0	1.83	98.6	0.85

<sup>a</sup> Each result is the average of three determinations.

Table 5  
Determination of uranium and vanadium in some synthetic matrices<sup>a</sup>

Sample no.	Ion present ( $\mu\text{g ml}^{-1}$ )	Found uranium (vanadium) ( $\mu\text{g ml}^{-1}$ )	Recovery (%), uranium (vanadium)	R.S.D. (%)
1	U(VI) (1.0), Ti(IV) (2.0), Ca <sup>2+</sup> (50), Th <sup>4+</sup> (10.0), Fe <sup>3+</sup> (5.0), V(V) (4.0)	0.995 (3.92)	99.5 (98.0)	0.7 (0.6)
2	U(VI) (2.0), Cu <sup>2+</sup> (40), PO <sub>4</sub> <sup>3-</sup> (40), Fe <sup>3+</sup> (5.0), Ca <sup>2+</sup> (50), CO <sub>3</sub> <sup>2-</sup> (40), V(V) (5.0)	2.02 (4.91)	101.0 (98.3)	0.5 (0.7)
3	U(VI) (4.0), Cu <sup>2+</sup> (40), PO <sub>4</sub> <sup>3-</sup> (40), Zr <sup>4+</sup> (20), Mg <sup>2+</sup> (20), Ni <sup>2+</sup> (20), CO <sub>3</sub> <sup>2-</sup> (40), V(V) (4.0)	3.92 (3.90)	98.2 (97.5)	0.7 (0.8)
4	U(VI) (2.0), K <sup>+</sup> (100), Zr <sup>4+</sup> (20), Cu <sup>2+</sup> (40), Mg <sup>2+</sup> (20), V(V) (4.0)	1.94 (9.8)	97.0 (98.0)	0.6 (0.7)

<sup>a</sup> Each result is the average of three determinations.

### 3.11. Determination of uranium and vanadium in spiked saline water samples

The results obtained for saline water (3 M NaCl) spiked with uranium and vanadium are given in Table 4. The average recovery of uranium was found to be 90.2% with a R.S.D. of 0.45% at  $4 \mu\text{g ml}^{-1}$ , while vanadium gave a 99.7% average recovery and a R.S.D. of 0.65 at  $6 \mu\text{g ml}^{-1}$  ( $n = 6$ ).

### 3.12. Simultaneous determination of uranium and vanadium in some synthetic matrices

The proposed method has been investigated for simultaneous determination of uranium and vanadium in some synthetic matrices resembling those obtained from dissolution of some ores. The results in Table 5 show a 98.9 and 97.9% average recovery for uranium and vanadium, respectively.

## 4. Conclusion

The simultaneous determination of U(VI) and V(V) using PAR and CPC in the presence of EDTA has been achieved using first-order derivative spectrophotometry. Ti(III) interferes and must be absent. The method has been successfully applied for the determination of uranium and vanadium added to different matrices.

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