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Quantitative Electrochromatography of Uranium and Platinum on Papers Impregnated with Thorium and Antimony Based Cation Exchanger

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Electrochromatography of 32 metal ions have been studied on papers impregnated with thorium antimonate cation exchanger in aq. organic acids, aq. nitric acid as well as in EDTA buffers. On the basis of differential migration which depends on the ion exchange properties of thorium antimonate and nature of complexes formed with the electrolytes, some useful qualitative and quantitative separations of synthetic mixtures of metal ions have been achieved. The effect of some other physical parameter has also been discussed. Quantitative separation of platinum and uranium has been developed.

INTRODUCTION

Synthetic inorganic ion exchangers have great importance in the field of separation science[1–3]. Papers impregnated with antimonate, tungstate and phospho-antimonate based on stannic[4], titanium[5], cerium[6] and zirconium[7,8] has been successfully utilised in our laboratory for various qualitative and quantitative separations of metal ions. Thorium antimonate[9] has also been successfully utilised in thin layer chromatography. All these results have given us impetus to extend our work on electrochromatographic studies of metal ions on thorium antimonate impregnated papers. The present work was, therefore, undertaken to study the electrochromatographic behaviour of metal ions on papers impregnated with thorium antimonate in aqueous nitric acid, aqueous organic acids and buffer mixtures of EDTA as background electrolytes. The present study deals with the following aspects:

The effect of pH of aqueous nitric acid and organic acids on the electrophoretic mobility have been reviewed and also the variation of average mobility of metal ions with the ionisation constant of the acids used and with other parameters such as atomic numbers and charge on the metal ions has been studied. On the basis of the differential mobility of the metal ions a large number of separations of important binary, ternary and quaternary mixtures of inorganic ions have been achieved. Besides a rapid quantitative separation uranium as UO_2 (II) (25 μg to 100 μg) and Pt(IV) (25 μg to 100 μg) from binary, ternary and synthetic mixtures containing several cations has been obtained in EDTA with buffers on Th(IV) antimonate impregnated papers.

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EXPERIMENTAL

Electrophoretic studies were performed on Whatman No. 1 (chromatographic) paper strips of 46.4×2.75 cm. A horizontal type electrophoretic apparatus run on an electronically regulated power supply unit (Systronic Ltd., India) was used. Spectrophotometric studies were made using a Bausch and Lomb spectronic-20 calorimeter.

Preparation of Ion Exchange Papers

The thorium antimonate paper was prepared by dipping the paper strips in a solution of 0.1M $SbCl_5$ in 4M HCl dried for 15 minutes at room temperature, again dipped in 0.1M thorium nitrate solution in 0.1M HNO_3. The excess of the reagent was allowed to drain off and the strips were washed with deionized water twice to remove the excess reagent and finally dried at room temperature. These paper strips were used for electrochromatographic studies.

Test Solutions and Detection Reagent

The test solutions were generally 10^{-1} M metal chloride or nitrate and these were prepared as described earlier[10]. Standard spot test reagents were used for detection[11].

Background Electrolytes

The following background electrolyte solution were used in these studies

1. 10^{-1} M HNO_3, 2. 10^{-3} M HNO_3. 3. 10^{-1} M citric acid, 4. 10^{-1} M tartaric acid, 5. 10^{-1} M succinic acid, 6. 10^{-1} M oxalic acid, 7. 10^{-1} M acetic acid, 8. 10^{-1} M lactic acid, 9. 10^{-1} M formic acid, 10. 10^{-1} M EDTA+Buffer (HCl+KCl) pH 1 (1 : 1). 11. 10^{-1} M EDTA+Buffer (HCl+KCl) pH 2 (1 : 1).

Procedure

For the qualitative studies on electrophoretic migration the earlier method[10] was followed. For the quantitative work, the platinum (25 μg to 100 μg) and uranium solutions (25 μg to 100 μg) along with the other cations to be separated were applied on the strips with the help of Lambda pipette. The strips were electro-chromatographed in one of the electrolyte No. 10 or 11, for 4 hrs under potential of 150 V. After the required time the pilot strips were examined for the spots of platinum, uranium and other cations. The remaining strips were then cut according to where spots had appeared on the pilot strips and platinum and uranium were eluted with 40 ml of 10% HCl in the cold by using 20 ml twice. The strips were then washed with 20 ml of DMW. The solution was evaporated

to small volumes (2–3 ml) and the paper strips was oxidised with an oxidising mixture of $HClO_4 : HNO_3 : H_2SO_4$ in the ratio of 3 : 1 : 4. The solution was filtered off evaporated to dryness, extracted with deionised water.

A blank was run in a similar way following the entire procedure with the unspotted paper strips. Thorium antimonate ion exchanger on the strips which had remained undissolved was filtered off. Platinum and uranium were determined spectrophotometrically with the stannous chloride and hydrogen peroxide respectively[12–13].

RESULTS AND DISCUSSION

Electrochromatographic studies on the paper impregnated with thorium (IV) antimonate have shown very interesting results such as electrolyte No. 1 and 2 are simple aqueous nitric acid systems which shows the effect of change in pH on the migration of metal ions under similar experimental conditions for both systems. Electrolytes No. 3 to 9 are weak organic acids which indicate the variation of the ionization constant of the acids on the electrochromatographic mobility of metal ions on Th(IV) antimonate papers under similar experimental conditions for voltage and time.

Electrolyte No. 10 and 11 are the mixtures of EDTA + HCl — KCl buffer of pH 1 and 2 respectively. These electrolytes were found to be more suitable for the separation of several metal ions.

The effect of change of pH upon electrochromatographic mobility (M)

$$= \frac{\text{Distance moved (cm)} \times \text{length of the paper strips}}{\text{Voltage (V)} \times \text{Time (T)}}$$

has been tested in aqueous nitric acid systems of different concentrations. It is obvious from the results that the mobility of almost all the metal ions decreased with the increase in pH. This is probably due to the fact that when nitric acid concentration is high the number of H^+ ions competing for the exchange sites is high, which lowers the adsorption and enhances the mobility of metal ions. However, with the decrease in H^+ ions concentration of the number of H^+ ion competing (with applied metal ions on the paper) for exchange sites decreases causing decrease in mobility. The plain papers also behave as described above while the mobility of metal ions on impregnated papers was lower compared to plain papers. Besides, Bi^{3+}, Fe^{3+}, W^{6+}, Se^{4+} do not show any appreciable difference in movement on both types of papers.

The background electrolytes No. 3 to 9 are weak organic acids. It is observed that these weak organic acid systems produce high mobility of metal ions as compared with the aqueous acids. An increase in the mobility have shown by the metal ions from acetic acid to the tartaric acid with the exception of lactic acid and the citric acid in which it is mini-

mum. Ionisation constant of these acids is also in the same manner as in the above case of mobility *i.e.* acetic acid (1.8×10^{-5}) < succinic acid (6.4×10^{-5}) < lactic acid (13.87×10^{-5}) < formic acid (17.7×10^{-5}) < citric acid (87.0×10^{-5}) < tartaric acid (96.0×10^{-5}) < oxalic acid (6500×10^{-5}). Probably due to the high ionisation constant of oxalic acid, the average mobility of metal ions is found to be decreased. The increase in mobility with the ionization constant of acids is probably due to the increase in the number of available H^+ ions competing with the metal ions in the exchange processes. It is also inferred from Fig. 1 that the average mobility of the metal ions is positive in formic acid, citric acid, tartaric acid and oxalic acid perhaps due to the formation of anionic complexes by a majority of metal ions.

When its variation with the atomic numbers is studied, it is observed that Mi (=mobility of metal ions on plain paper—mobility of metal ions on impregnated papers) increased with increase in the atomic numbers.

Fig. 1 and 2 show that the average mobility of the metal ions varies with their charge in different electrolytic solutions. In general, the average mobility on thorium antimonate papers is found to be decreased with the increase in charge on the metal ions. However, at higher charge the mobility is found to become constant.

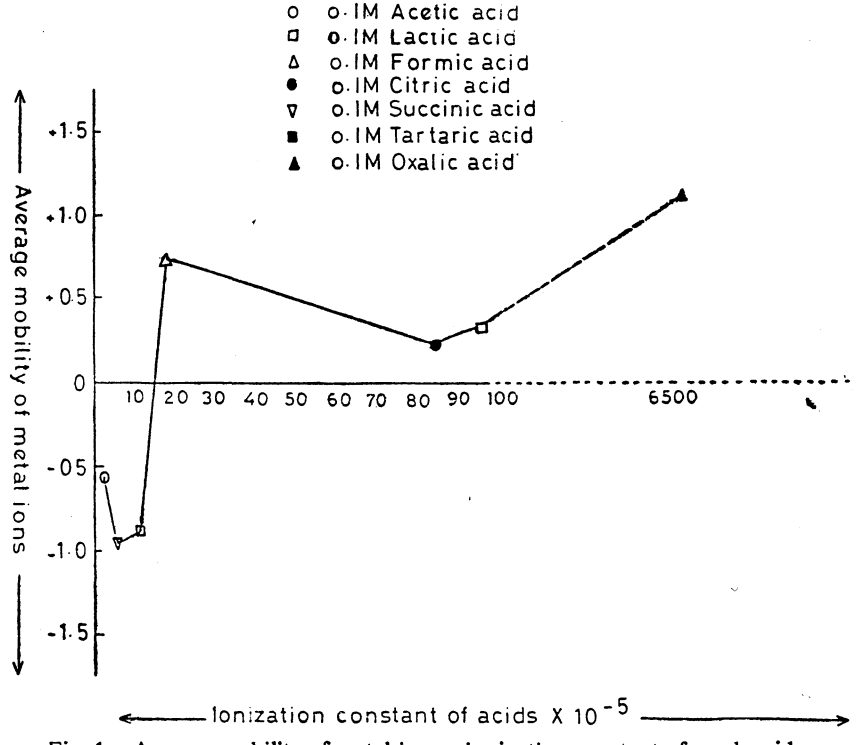

Fig. 1. Average mobility of metal ion vs ionization constant of weak acids

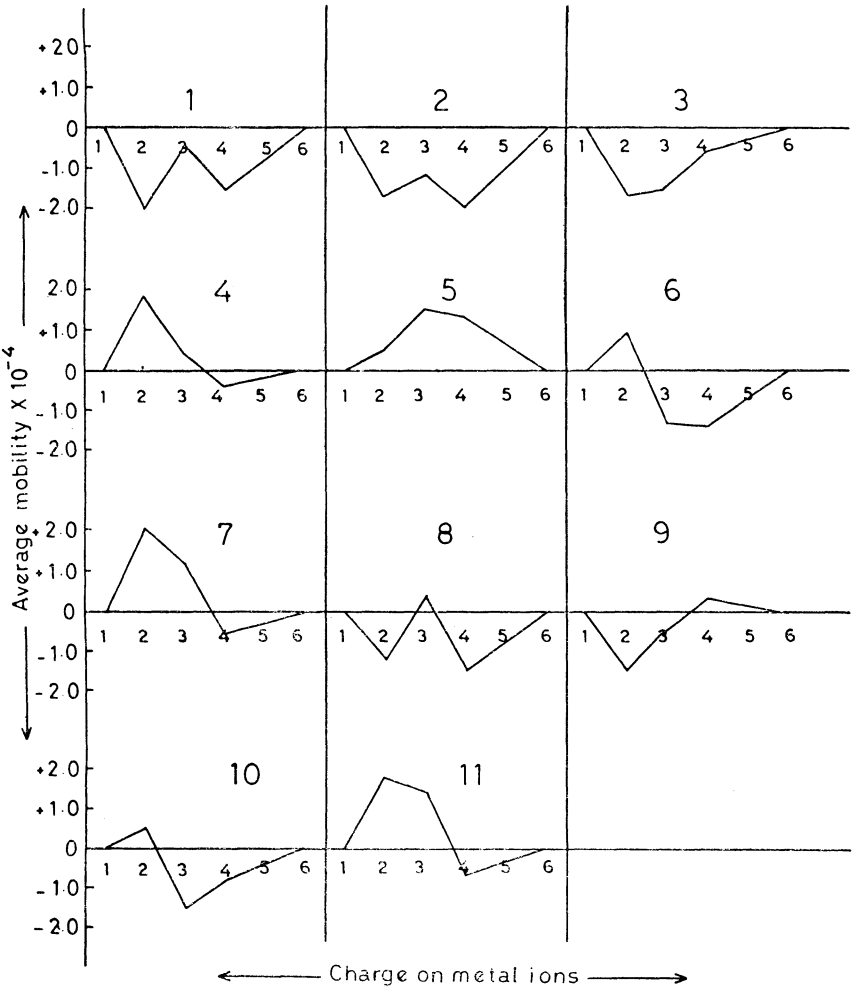

Fig. 2. Average mobility of metal ions vs charge on metal ions

The utility of thorium(IV) antimonate papers in the electrochromatography is demonstrated by achieving a large number of useful separations (Table 1). Several important and analytically difficult quaternary and binary separations of metal ions have been achieved. To mention a few Se^{4+}–Pt^{4+}; V^{2+}–Co^{2+}; Se^{4+}–Pt^{4+}–UO_2^{2+}–Co^{2+}; Bi^{3+}–Zn^{2+}–Co^{2+}–Pd^{2+}; Fe^{3+}–Co^{2+}–Pt^{4+}; Zr^{4+}–Nd^{3+}–Pr^{3+}; Cd^{2+}–Pd^{2+}–Pb^{2+}; Bi^{3+}–UO_2^{2+}–Pt^{4+}; Ni^{2+}–UO_2^{2+}–Pt^{4+}; Cr^{3+}–Fe^{3+}–Pt^{4+}; Gd^{3+}–Ho^{3+}; Dy^{3+}–Nd^{3+} etc. are the separation of analytical interest.

The unique feature of these studies has been the quantitative separation of UO_2(II) and Pt(IV) in EDTA + Buffer solutions in only 4 hrs under a potential of 150V. The separation of UO_2(II) and Pt(IV) from

TABLE 1

SEPARATIONS ACHIEVED ON THORIUM ANTIMONATE PAPERS

Background electrolyte	Electrical potential applied and time	Metal ions separations
10^{-1}M HNO_3	100 volts, 3 hours	$Bi^{3+}(0.0)$-$Co^{2+}(-5.5)$-$Fe^{3+}(+3.5)$
		$Bi^{3+}(0.0)$-$Ni^{2+}(-5.0)$-$Fe^{3+}(+3.5)$
		$Cd^{2+}(-2.0)$-$Pd^{2+}(-9.0)$-$Fe^{3+}(+3.5)$
		$Cd^{2+}(-2.0)$-$Pb^{2+}(-9.0)$-$UO_2^{2+}(+3.3)$
		$Pd^{2+}(-4.5)$-$Pb^{2+}(-9.0)$—$VO^{2+}(+3.1)$
		$Bi^{3+}(-0.0)$-$Cu^{2+}(-3.7)$-$Pb^{2+}(-9.1)$
10^{-3}M HNO_3	100 volts, 3 hours	$Sm^{3+}(-3.7)$-$Dy^{3+}(+2.8)$
		$Y^{4+}(-4.0)$-$Dy^{3+}(+2.8)$
		$Cr^{3+}(-1.2)$-$VO^{2+}(-11.2)$-$Al^{3+}(+3.2)$
		$Zn^{2+}(-1.6)$-$VO^{2+}(-11.2)$-$Al^{3+}(+3.5)$
		$Fe^{3+}(-2.1)$-$Pt^{4+}(-10.8)$-$Al^{3+}(+3.6)$
10^{-1}M citric acid	200 volts, 4 hours	$Zn^{2+}(+2.6)$-$Pt^{4+}(-5.7)$
		$Cu^{2+}(+3.8)$-$Pt^{4+}(-5.7)$
		$Fe^{3+}(0.0)$-$Co^{2+}(+4.1)$-$Pt^{4+}(-5.7)$
		$Mo^{6+}(0.0)$-$Cd^{2+}(+4.2)$-$Pt^{4+}(-5.7)$
		$Hg^{2+}(+0.7)$-$Co^{2+}(+4.1)$-$Pt^{4+}(-5.9)$
		$Bi^{3+}(0.0)$-$Ni^{2+}(+4.5)$-$Pt^{4+}(-6.0)$
		$Bi^{9+}(0.0)$-$Cd^{2+}(+4.7)$-$Pt^{4+}(-6.0)$
10^{-1}M tartaric acid	200 volts, 4 hours	$UO_2^{2+}(-0.6)$-$Al^{3+}(+4.7)$
		$Cr^{3+}(+1.8)$-$Pt^{4+}(-4.9)$
		$Ca^{2+}(-2.2)$-$La^{3+}(+3.9)$
		$Ca^{2+}(-2.2)$-$Gd^{3+}(+4.1)$
		$Ce^{3+}(-0.6)$-$Gd^{3+}(+4.1)$
		$UO_2^{2+}(-0.6)$-$Co^{2+}(+4.2)$-$Pt^{4+}(-4.9)$
		$Fe^{3+}(0.0)$-$Co^{2+}(+4.2)$-$Pt^{4+}(-4.9)$
		$Bi^{3+}(0.0)$-$Co^{2+}(+4.2)$-$Pt^{4+}(-4.9)$
		$Pb^{2+}(0.0)$-$Al^{3+}(+4.7)$-$Pt^{4+}(-4.7)$
		$Fe^{3+}(0.0)$-$Al^{3+}(+4.7)$-$Pt^{4+}(-4.9)$
10^{-1}M succinic acid	200 volts, 4 hours	$Sm^{3+}(+4.2)$-$Pr^{3+}(-2.1)$
		$Sm^{3+}(+4.2)$-$Ho^{2+}(-2.5)$
		$Sm^{3+}(+4.2)$-$Ca^{2+}(-2.8)$
10^{-1}M oxalic acid	200 volts, 4 hours	$Mo^{6+}(0.0)$-$Pt^{4+}(-5.8)$-$Co^{2+}(+8.1)$
		$Hg^{2+}(-1.6)$-$Pd^{2+}(-5.9)$-$Co^{2+}(+8.0)$
		$Bi^{3+}(-0.5)$-$Pd^{2+}(-5.9)$-$Co^{2+}(+8.0)$
		$Se^{4+}(0.0)$-$Pt^{4+}(-5.8)$-$VO^{2+}(+3.7)$-$Co^{2+}(+8.1)$
		$Se^{4+}(0.0)$-$Pt^{4+}(-5.8)$-$UO_2^{2+}(+4.0)$-$Co^{2+}(+8.1$
		$Bi^{3+}(-0.5)$-$Zn^{2+}(+3.9)$-$Co^{2+}(+8.0)$-$Pd^{2+}(-6.0$

TABLE 1 (Contd.)

Background electrolyte	Electrical potential applied and time	Metal ions separations
10^{-1}M acetic acid	200 volts, 4 hours	$Pb^{2+}(+1.9)$-$Hg^{2+}(-4.2)$
		$Pb^{2+}(+1.9)$-$Bi^{3+}(-4.1)$
		$Ni^{2+}(+2.0)$-$Pt^{4+}(-5.2)$
		$Gd^{3+}(+3.2)$-$Ho^{3+}(-2.0)$
		$Zr^{4+}(0.0)$-$Nd^{3+}(+3.5)$-$Pr^{3+}(-4.2)$
		$Zr^{4+}(0.0)$-$Gd^{3+}(+3.7)$-$Pr^{3+}(-4.2)$
		$Zr^{4+}(0.0)$-$Ce^{3+}(+3.5)$-$Pr^{3+}(-4.3)$
10^{-1}M lactic acid	200 volts, 4 hours	$Ca^{2+}(-4.2)$-$Ce^{3+}(+1.5)$
		$Ca^{2+}(-4.2)$-$Zr^{4+}(0.0)$
		$Cd^{2+}(0.0)$-$Co^{2+}(-4.7)$-$Pd^{2+}(+4.2)$
		$Cd^{2+}(0.0)$-$Co^{2+}(-4.7)$-$Bi^{3+}(+2.2)$
		$Se^{4+}(0.0)$-$Al^{3+}(-4.1)$-$Pt^{4+}(+4.2)$
10^{-1}M formic acid	200 volts, 4 hours	$Dy^{3+}(+1.0)$-$Nd^{3+}(-3.4)$
		$UO_2^{2+}(0.0)$-$Pd^{2+}(-5.5)$-$Pb^{2+}(+3.8)$
		$Fe^{3+}(-0.6)Pd^{2+}(-5.5)$-$Pb^{2+}(+3.8)$
		$Cd^{2+}(0.0)$-$Pd^{2+}(-5.5)$-$Pb^{2+}(+3.8)$
		$Bi^{3+}(-1.1)$-$Pt^{4+}(-5.8)$-$Zn^{2+}(+2.7)$
		$VO^{2+}(0.0)$-$Pt^{4+}(-5.8)$-$Zn^{2+}(+2.7)$
		$UO_2^{2+}(0.0)$-$Pd^{2+}(-5.5)$-$Zn^{2+}(+2.7)$
10^{-1}M EDTA + buffer solution of pH 1 (1 : 1)	200 volts, 4 hours	$Bi^{3+}(-0.8)$-$UO_2^{2+}(+4.9)$-$Pt^{4+}(-8.5)$
		$Bi^{3+}(-0.8)$-$Fe^{3+}(+3.3)$-$Pt^{4+}(-8.5)$
		$Pb^{2+}(+1.1)$-$UO_2^{2+}(+4.9)$-$Pt^{4+}(-8.5)$
		$Se^{4+}(0.0)$-$Fe^{3+}(+3.2)$-$Pt^{4+}(-8.7)$
		$W^{6+}(0.0)$-$Hg^{2+}(-4.1)$-$UO_2^{2+}(+4.7)$
		$W^{6+}(0.0)$-$UO_2^{2+}(+4.7)$-$Pt^{4+}(-8.8)$
		$Se^{4+}(0.0)$-$Hg^{2+}(-4.0)$-$Pt^{4+}(-8.7)$
		$Ni^{2+}(-1.9)$-$UO_2^{2+}(+4.6)$-$Pt^{4+}(-8.6)$
		$Zn^{2+}(+0.9)$-$UO_2^{2+}(+4.7)$-$Pt^{4+}(-8.8)$
10^{-1}M EDTA + buffer solution of pH 2 (1 : 1)	200 volts, 4 hours	$VO^{2+}(+1.0)$-$Fe^{3+}(+3.5)$
		$Cu^{2+}(0.0)$-$Fe^{3+}(+3.5)$
		$Cr^{3+}(-1.2)$-$Fe^{3+}(+3.2)$-$Pt^{4+}(-9.1)$
		$VO^{2+}(+1.0)$-$UO_2^{2+}(+5.3)$-$Pt^{4+}(-9.0)$
		$Pb^{2+}(-1.7)$-$UO_2^{2+}(+5.2)$-$Pt^{4+}(-9.2)$
		$Ni^{2+}(-1.9)$-$UO_2^{2+}(+5.5)$-$Pt^{4+}(-9.5)$
		$Co^{2+}(-2.1)$-$UO_2^{2+}(+5.4)$-$Pt^{4+}(-9.5)$
		$Pd^{2+}(+1.2)$-$UO_2^{2+}(+5.4)$-$Pt^{6+}(-9.4)$
		$Ni^{2+}(-1.9)$-$Fe^{3+}(+3.4)$-$Pt^{4+}(-9.4)$
		$Cu^{2+}(0.0)$-$UO_2^{2+}(+5.4)$-$Pt^{4+}(-9.4)$

several metal ions in the electrolytes mentioned above is probably due to the formation of EDTA complexes at low pH. The Tables 2–5 summa-

rise the results of the quantitative work. Apart from the binary and ternary separations reported in Tables 2 and 3, UO_2(II) and Pt(IV) were quantitatively separated from synthetic mixtures containing Hg(II), Bi(III), Cu(II), Co(II), Ni(II), Pb(II), Pd(II), Zn(II), Fe(III), Se(IV), W(VI), VO(II), Cr(III) etc. These mixtures were prepared by mixing 1 ml of 10^{-1} M each of the above mentioned cations alongwith 25 μg to 100 μg of Pt(IV) and UO_2(II) respectively.

TABLE 2

QUANTITATIVE SEPARATION OF Pt^{4+} FROM BINARY AND TERNARY MIXTURES

(Amount of Pt^{4+} applied on each strip 25 μg)

Sample No.	Mixture	Amount of Pt^{4+} found (μg)	Percentage error
1.	Co^{2+}–Pt^{4+}	24	+4
2.	Ni^{2+}–Pt^{4+}	23	−8
3.	Pd^{2+}–Pt^{4+}	21.5	−14
4.	W^{6+}–Pt^{4+}	23.5	+6
5.	Cu^{2+}–UO_2^{2+}-Pt^{4+}	25	0
6.	Bi^{3+}–Fe^{3+}–Pt^{4+}	21.5	−14
7.	Cr^{3+}–Fe^{3+}–Pt^{4+}	26	+4
8.	Se^{4+}–Hg^{2+}–Pt^{4+}	25.5	+2
9.	Pb^{2}–UO_2^{2+}–Pt^{4+}	25.5	+2
10.	VO^{2+}–UO_2^{2+}–Pt^{4+}	24.5	−2

TABLE 3

QUANTITATIVE SEPARATION OF Pt^{4+} FROM SYNTHETIC MIXTURES

Composition of mixtures	Amount of Pt^{4+} applied (μg)	Amount of Pt^{4+} found (μg)	Percentage error
1 ml each of 10^{-1}M solution of Hg^{2+}, Bi^{3+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Pb^{2+}, Pd^{2+}, Zn^{2+}, Fe^{3+}, Se^{4+}, UO_2^{2+} and W^{6+} mixed with 25 μg to 100 μg Pt^{4+}	25	23.0	−8.0
	25	24.0	−4.0
	50	48	−4.0
	50	51	+2.0
	75	75.0	0.0
	75	73	−2.67
	90	92.0	+2.22
	90	86.0	−4.44
	100	98	−2.0
	100	103	+3.0

TABLE 4

QUANTITATIVE SEPARATION OF URANIUM AS UO_2^{2+} FROM BINARY AND TERNARY MIXTURES
(Amount of UO_2^{2+} applied on each strips 75 μg)

Sample No.	Mixture	Amount of UO_2^{2+} found (μg)	Percentage error
1.	Cr^{3+}–UO_2^{2+}	78	+4.0
2.	VO^{2+}–UO_2^{2+}	79	+5.3
3.	Zn^{2+}–UO_2^{2+}	80	+6.7
4.	W^{6+}–Hg^{2+}–UO_2^{2+}	71	−5.3
5.	Pb^{2+}–Ni^{2+}–UO_2^{2+}	75	0.00
6.	Ni^{2+}–Fe^{3+}–UO_2^{2+}	69	−8.0
7.	Co^{2+}–Fe^{3+}–UO_2^{2+}	70	−6.7
8.	Bi^{3+}–Pt^{4+}–UO_2^{2+}	76	+1.3
9.	Se^{4+}–Pt^{4+}–UO_2^{2+}	75	0.0
10.	Pd^{2+}–Pt^{4+}–UO_2^{2+}	72	−4.0

TABLE 5

QUANTITATIVE SEPARATION OF URANIUM AS UO_2^{2+} FROM SYNTHETIC MIXTURES

Composition of mixtures	Amount of UO_2^{2+} applied (μg)	Amount of UO_2^{2+} found (μg)	Percentage error
1 ml each of 10^{-1}M solutions of Hg^{2+}, Bi^{3+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Pb^{2+} Pd^{2+}, Zn^{2+}, Fe^{3+}, VO^{2+}, Se^{4+}, Pt^{4+} and W^{6+} mixed with 25 μg to 100 μg uranyl	25	25	0.0
	25	26	+4.0
	50	48	−4.0
	50	51	+2.0
	50	59	−2.0
	75	70	−6.7
	75	75	0.0
	75	78	+4.0
	100	102	+2.0
	100	94	−6.0
	100	98	−2.0

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