

CHROM. 3339

## QUANTITATIVE SEPARATION OF TITANIUM FROM NUMEROUS METAL IONS AND OF IRON, ALUMINUM AND TITANIUM BY PAPER CHROMATOGRAPHY

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(Received November 20th, 1967)

## SUMMARY

Titanium has been quantitatively separated from numerous metal ions using formic acid-hydrochloric acid-acetone (3:5:2). The method has been found useful for the quantitative separation of about 200  $\mu\text{g}$  of titanium from a synthetic mixture of 17 metal ions including  $\text{Mo}^{6+}$ ,  $\text{W}^{6+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{UO}_2^{2+}$  and  $\text{V}^{4+}$ . Quantitative separation of  $\text{Fe}^{3+}$ - $\text{Al}^{3+}$ - $\text{Ti}^{4+}$  has been achieved using formic acid-hydrochloric acid-acetone (3:4:3). Both separations are fast and selective.

Titanium is an important metal which is used in pigments and in alloys. Its separation from numerous metal ions is therefore important. SCHEFFER has pointed out in the treatise on Analytical Chemistry that "The present application of paper chromatography is restricted to qualitative identification of titanium but this separation technique is worthy of serious consideration"<sup>1</sup>. Iron, aluminum and titanium are also difficult to separate by classical means; LACOURT therefore developed a quantitative separation of iron, aluminum and titanium<sup>2</sup>. Her separation suffers from many limitations: (i) Fe and Ti are separated only if the sample is prepared in a particular manner; (ii) the method cannot be used for varying ratios of iron, titanium and aluminum, and according to MACEK<sup>3</sup> "when the samples are applied as solutions in 10% citric acid or in acetate buffer, all three cations are separated. When the cations are dissolved in dil. HCl or in dil.  $\text{HNO}_3$ , Ti and Fe migrate as a single zone". As a result her method has not gained wide acceptance. Paper chromatography was therefore undertaken: (i) to quantitatively separate titanium from numerous metal ions; (ii) to quantitatively separate iron, aluminum and titanium. The present report summarizes the results of such a study.

## EXPERIMENTAL

*Apparatus*

Development was performed in 25 by 5.5 cm glass jars employing the ascending technique. The dimensions of the paper strip were 16 by 4 cm. Estimation was done on a Bausch and Lomb spectronic-20 colorimeter. All pH measurements were made on pH Meter Model LI-10 (ELICO, Hyderabad, India).

### Reagents

Whatman No. 1 paper was used for all quantitative work. All chemicals were of reagent grade.

Standard titanium solution (containing 40 mg Ti per ml) was prepared by diluting Analar grade titanous chloride solution with HCl and water and was standardized against potassium dichromate<sup>4</sup>.

The stock solution of ferric ammonium sulfate (containing 34.85 mg of Fe/ml) was prepared in 5% H<sub>2</sub>SO<sub>4</sub>.

The stock solution of aluminum (containing 8.537 mg Al/ml) was prepared in 1% H<sub>2</sub>SO<sub>4</sub> and was standardized against EDTA<sup>4</sup>.

4% gallium chloride and 0.1 M indium chloride were prepared in 4 M HCl. 0.1 M sodium molybdate and 1% sodium tungstate were prepared in water.

Solutions of other metal ions were prepared by taking their chlorides, nitrates or sulfates in 15% HCl, HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>, respectively.

The developer used for the separation of titanium from numerous metal ions was a mixture of formic acid–hydrochloric acid–acetone in the ratio of (3:5:2) and the developer for the separation of Fe–Al–Ti was formic acid–hydrochloric acid–acetone in the ratio of (3:4:3). Titanium, iron and aluminum were detected by 0.1% alcoholic alizarin Red-S and exposing over ammonia.

### Procedure

(a) *Quantitative separation of titanium from numerous metal ions.* The solution containing titanium with other metal ions as impurities was streaked on the line of application with the help of a  $\lambda$ -pipette. The strips were dried in air and saturated for 15 min with the vapours of the developer and the developer was allowed to ascend 12.5 cm. Blank paper was run simultaneously and detected; 15% HCl solution was streaked as a blank. Zones were cut into small pieces and were eluted thrice with 5% H<sub>2</sub>SO<sub>4</sub> by taking 10 ml each time and heating on a water bath for 1 h. Papers were washed twice by heating with 10 ml of distilled water for 15 min. The volume was reduced to 2–3 ml by evaporation on a water bath and the paper pulp was oxidized with 2 ml of a mixture of HNO<sub>3</sub> + HClO<sub>4</sub> (1:3) + 2 ml of H<sub>2</sub>SO<sub>4</sub>. The solution was evaporated to 0.5–1 ml. It was taken into a standard 50 ml flask and made up to volume with water and titanium was determined by sulphosalicylic acid<sup>6</sup> at 410 m $\mu$ .

(b) *Separation and determination of titanium, iron and aluminum.* A mixture of titanium, iron and aluminum was streaked and developed in the given developer. Elutions were made as in procedure (a). The eluting reagent for titanium and aluminum was also 5% H<sub>2</sub>SO<sub>4</sub>, while for iron it was 1% HCl. The determination of titanium, iron and aluminum was done with sulphosalicylic acid<sup>6</sup>; 1,10-phenanthroline<sup>7</sup>; and triochrome cyanine-R<sup>8</sup>, respectively.

## RESULTS

### *Determination of titanium in presence of numerous metal ions*

Titanium in presence of common impurities was studied and the quantitative separation of titanium from these ions was studied. The sample contained Ti<sup>4+</sup> and the impurities were in the ratio of (1:1).

A (1:1) synthetic mixture of titanium with 17 metal ions: Ga<sup>3+</sup>, In<sup>3+</sup>, K<sup>+</sup>, Cs<sup>+</sup>,

$\text{NH}_4^+$ ,  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{UO}_2^{2+}$  and  $\text{V}^{4+}$ , was studied and found quantitative. The results are given in Table I.

*Quantitative separation of  $\text{Ti}^{4+}$ ,  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$*

0.01 ml of a mixture of  $\text{Ti}^{4+}$ ,  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$  was applied. Zones of  $\text{Ti}^{4+}$ ,  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$  were cut out and determined by the procedure described earlier. The results are summarized in Table II.

TABLE I

DETERMINATION OF  $\text{Ti}^{4+}$  IN THE PRESENCE OF IMPURITIES  
200  $\gamma$   $\text{Ti}^{4+}$  applied.

Impurity added	$\text{Ti}^{4+}$ recovered ( $\gamma$ )	% Error
$\text{Ga}^{3+}$	210	+ 5.0
	204	+ 2.0
$\text{In}^{3+}$	204	+ 2.0
	204	+ 2.0
$\text{Mo}^{6+}$	200	$\pm$ 0.0
	204	+ 2.0
$\text{W}^{6+}$	204	+ 2.0
	204	+ 2.0
$\text{K}^+$	210	+ 5.0
	204	+ 2.0
$\text{Cs}^+$	196	- 2.0
	200	$\pm$ 0.0
$\text{NH}_4^+$	204	+ 2.0
$\text{Cu}^{2+}$	210	+ 5.0
$\text{Cd}^{2+}$	204	+ 2.0
	192	- 4.0
$\text{Fe}^{3+}$	200	$\pm$ 0.0
	204	+ 2.0
$\text{Cr}^{3+}$	192	- 4.0
	200	$\pm$ 0.0
$\text{Al}^{3+}$	196	- 2.0
	200	$\pm$ 0.0
$\text{Ni}^{2+}$	200	$\pm$ 0.0
	210	+ 5.0
$\text{Co}^{2+}$	192	- 4.0
	200	$\pm$ 0.0
$\text{Mn}^{2+}$	196	- 2.0
	204	+ 2.0
$\text{Zn}^{2+}$	200	$\pm$ 0.0
$\text{Ba}^{2+}$	196	- 2.0
$\text{Mg}^{2+}$	200	$\pm$ 0.0
	200	$\pm$ 0.0
$\text{UO}_2^{2+}$	192	- 4.0
	192	- 4.0
$\text{V}^{4+}$	204	+ 2.0
	196	- 2.0
17 metal ions	208	+ 4.0
	204	+ 2.0
	204	+ 2.0
	204	+ 2.0

TABLE II

DETERMINATION OF Fe<sup>3+</sup>, Al<sup>3+</sup> AND Ti<sup>4+</sup>

Ti <sup>4+</sup> applied ( $\gamma$ )	Ti <sup>4+</sup> recovered ( $\gamma$ )	% Error	Fe <sup>3+</sup> applied ( $\gamma$ )	Fe <sup>3+</sup> recovered ( $\gamma$ )	% Error	Al <sup>3+</sup> applied ( $\gamma$ )	Al <sup>3+</sup> recovered ( $\gamma$ )	% Error
133.3	133.3	0.0	23.2	23.1	-0.4	28.0	27.0	-3.0
133.3	133.3	0.0	23.2	23.1	-0.4	28.0	29.0	+3.0
133.3	133.3	0.0	11.46	11.5	+0.3	28.0	27.0	-3.0
133.3	133.3	0.0	11.46	11.5	+0.3	28.0	26.5	-4.5
133.3	133.3	0.0	11.46	11.5	+0.3	28.0	28.0	$\pm 0.0$

## DISCUSSION

The results show that the methods of separation are fast, selective and quantitative. Titanium is easily separated from most of the ions which are likely to be found in actual analytical work. Using paper chromatography iron, aluminum and titanium have been separated only by LACOURT<sup>2</sup>. The method of separation developed by us is however faster, more selective and is not subject to common interferences.

## ACKNOWLEDGEMENTS

The authors are grateful to A. R. KIDWAI for providing research facilities.

The financial assistance given to (J.P.R.) by C.S.I.R., India and to (F.K.) by U.G.C. is gratefully acknowledged.

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