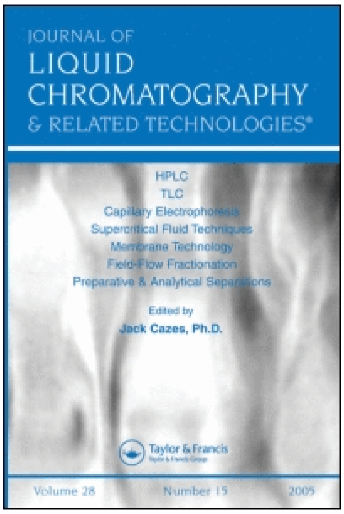

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QUANTITATIVE SEPARATION OF Zr^{4+} from La^{3+} and Ce^{3+}; W^{6+} FROM Cr^{3+}, Mo^{6+} and VO^{2+} BY THIN LAYER CHROMATOGRAPHY.

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ABSTRACT

Methods have been developed for the quantitative separation of Zr^{4+} from La^{3+} and Ce^{3+} using 0.1M ammonium oxalate solution as eluant. W^{6+} can also be separated quantitatively from Cr^{3+}, Mo^{6+} and VO^{2+} using DMSO-1MHCl (1:9). Both the methods are fast and quantitative. The separation takes about 40-50 min. The average error is about 3%.

INTRODUCTION

The thin layer chromatography of metal ions on silica gel layers has been studied during the last ten years (1-11). Very few references are available for the quantitative separation of metal ions using TLC (4,5) which offers a simple, rapid and versatile method for the separation of metal ions. However, it has been used more in organic analysis than in inorganic one.

In this article, we present a method for the separation of Zr^{4+} from La^{3+} and Ce^{3+}. This separation has been achieved using a simple solvent i.e., 0.1M ammonium oxalate solution. W^{6+} has been separated from Cr^{3+}, Mo^{6+} and VO^{2+} using DMSO-1M HCl (1:9) as the developing system.

MATERIALS AND METHODS

Apparatus

Thin layer chromatography apparatus (Toshniwal, India) for the preparation of silica gel-G layers on 20x3 cm. glass

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plates was used. These plates were developed in glass jars (20x5cm). Spectrophotometric studies were performed on a Spekol colorimeter (made in Germany).

Reagents

Silica gel-G, dimethyl sulphoxide (DMSO), sodium tungstate and zirconium oxychloride were all from B.D.H., England. Other chemicals were of AnalaR grade.

Test solutions and Detectors

The test solutions were generally 0.1M in metal chloride, nitrate or sulphate. 0.1M solutionsof sodium tungstate and zirconium oxychloride were used. All the cation solutions were made in 5% citric acid solution. Conventional spot test reagents were used for detection purposes (12).

Preparation of Silica gel-G layers

The slurry used was prepared by mixing silica gel-G with demineralised water in the ratio of 1:3 with constant shaking for 5 min. The slurry was coated on clean glass plates with the help of an applicator to give a layer of ~ 0.15 mm thickness for qualitative studies and ~ 0.50 mm thickness for quantitative studies. The plates were first dried at room temperature and then in an electric oven at 100 ± 5°C for about 2 hr. These plates were then stored in an oven at room temperature until used.

Procedure

The metal ion solution was spotted with the help of a lambda pipette in the form of a streak. The development was performed in the chosen solvent systems. The solvent was allowed to ascend 13 cm from the starting line on the plates in all cases. A pilot plate was run simultaneously in order to locate the exact position of the spot on the plate. The area of Zr^{4+} and W^{6+} was scratched and was eluted with 1:1 HCl and 0.5M NaOH solutions respectively. The final volume of the filtrate in case of Zr^{4+} was reduced to about 1 ml. by evaporation and then was determined spectrophotometrically using alizarin at 560 mμ (13). In case of W^{6+} the final volume of the filtrate was reduced to about 10 ml and was determined spectrophotometrically by thiocyanate method (14).

RESULTS

The quantitative separation of Zr^{4+} from La^{3+} and Ce^{3+} in 0.1M ammonium oxalate is summarized in table 1. Table 2 shows the quantitative separation of W^{6+} from Cr^{3+}, Mo^{6+} and VO^{2+} in DMSO-1M HCl (1:9).

TABLE 1

Quantitative Separation Of Zr^{4+} From La^{3+} and Ce^{3+}

Zr^{4+} taken (μg)	Interfering ions added (μg)	Zr^{4+} found (μg)	Error (%)
20	La^{3+} (52.9)	19.25	-3.75
80	La^{3+} (26.4)	78.75	-1.56
20	Ce^{3+} (52.5)	20.00	-
80	Ce^{3+} (26.2)	77.50	-3.12
20	(La^{3+}+Ce^{3+}) (26.4+26.2)	19.25	-3.75
80	(La^{3+}+Ce^{3+}) (26.4+26.2)	76.00	-5.00

DISCUSSION

0.1M ammonium oxalate solution is an interesting solvent. It separates Zr^{4+} (R_f = 0.10) from La^{3+} (R_f = 0.83) and Ce^{3+} (R_f = 0.84). Zr^{4+} is probably precipitated as zirconium oxalate while La^{3+} moves almost to the solvent front. When 0.1M ammonium oxalate solution is added to the La^{3+} solution in test tube, a white precipitate is obtained. This points to the fact that the reaction in the test tube may be quite different from the reaction on the plate. The results in table 1 show that Zr^{4+} can be separated quantitatively from La^{3+} in the ratio of 1:2.5 and 2.5:1 and from Ce^{3+} in varying ratios. Zr^{4+} can also be separated from a mixture of La^{3+} and Ce^{3+} by using this solvent.

W^{6+} is quantitatively separated from Mo^{6+}, Cr^{3+} and VO^{2+} using DMSO-1M HCl (1:9). In excess HCl, W^{6+} is probably precipitated

TABLE 2

Quantitative Separation Of W^{6+} From Cr^{3+}, Mo^{6+} And VO^{2+}

W^{6+} taken (μg)	Interfering ions added (μg)	W^{6+} found (μg)	Error (%)
480	Cr^{3+} (80)	460	-4.16
200	Cr^{3+} (80)	190	-5.00
480	Mo^{6+} (123)	468	-2.56
200	Mo^{6+} (123)	182	-4.03
480	VO^{2+} (175)	471	-1.87
200	VO^{2+} (175)	182	-4.03

as tungstic acid while Mo^{6+}, Cr^{3+} and VO^{2+} move almost to the solvent front. The results are given in table 2. The methods are precise and accurate and it is possible to separate small amounts of Cr^{3+}, Mo^{6+} and VO^{2+} from a very large amount of W^{6+}.

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