Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Ghose, Animesh K. and Dey, Arun K.(1967) 'A Scheme for the Qualitative Analysis of Less Common Cations Using the Ring-Oven Method', Separation Science and Technology, 2: 5, 673 — 679
To link to this Article: DOI: 10.1080/01496396708049730
URL: http://dx.doi.org/10.1080/01496396708049730

SEPARATION SCIENCE, 2(5), pp. 673–679, Nov., 1967

A Scheme for the Qualitative Analysis of Less Common Cations Using the Ring-Oven Method

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Summary

The schematic separation of 14 less familiar cations, viz., gold, beryllium, thallium, cerium, titanium, zirconium, thorium, vanadium, molybdenum, tungsten, uranium, selenium, tellurium, and platinum, from mixtures has successfully been worked out by using the ring-oven method. The cations after separation are identified by spot reagents.

The work on the qualitative analysis of mixtures of less familiar cations by the ring-oven technique (*1*) is scanty. Weisz (*1*), the originator of the technique, described a scheme for the analysis of common cations that mainly employed the reactions in the classical Fresenius scheme, with the use of H_2S as a group precipitant. Subsequently, West and Mukherji (2) described a scheme for the analysis of 35 cations, including some less common ones. Their scheme, however, did not involve clear-out separations; identifications were done with selective reagents in many cases. Dey and co-workers in several publications pursued the problem of qualitative analysis by the ring-oven method. Biswas et al. (*3*) separated mixtures of 26 cations into groups on a semimicroscale in test tubes and employed the ring-oven method to separate and identify the individual members of a group. Chatterjee and Dey (*4,5*) described schemes for the analysis of common cations only in mixtures. Biswas and Dey (*6*) attempted the separation and identification of less common metal ions in ternary mixtures by the ring-oven technique.

No systematic work appears to have been done on the qualitative analysis of less familiar cations in mixtures using this method, and in this article the schematic separation of gold, beryllium, thallium, cerium, titanium, zirconium, thorium, vanadium, molybdenum, tungsten, uranium, selenium, tellurium, and platinum in mixtures will be described. The method is rapid and analysis of unknown mixtures does not require more than 75 min.

EXPERIMENTAL

Apparatus

A Weisz ring oven (National Appliance Co., Portland, Oregon) with its accessories was used, as described earlier (*3*). The same self-filling pipet (ca. 1.5 μl) was used throughout for spotting.

Metal Solutions

0.1 *M* solutions of the following were prepared using reagent-grade chemicals: $AuCl_3$, $Be(NO_3)_2 \cdot 3H_2O$, $TlCl_3$, $Ce(SO_4)_2$, $TiCl_4$, $ZrOCl_2 \cdot 8H_2O$, $Th(NO_3)_4$, NH_4VO_3, $(NH_4)_6$, $Mo_7O_{24} \cdot 4H_2O$, Na_2WO_4, $UO_2(NO_3)_2 \cdot 6H_2O$, SeO_2, K_2TeO_3, and H_2PtCl_6. Equal volumes of each of these solutions were mixed and the mixture was used for the separations.

Solvents for Washing

The following solvents were used for washing:

1. Ethyl acetate (BDH AnalaR)
2. *n*-Butanol (BDH AnalaR)
3. 20% Acetylacetone in chloroform (BDH AnalaR)
4. 30 Volumes of hydrogen peroxide (E. Merck)
5. Mixture of 60% ethanol and 3 *N* acetic acid (10:1)

Other reagents used were of reagent grade.

Detecting Reagents

The following detecting reagents were used:

Alizarin Red S: 0.1% w/v aqueous solution
Aluminon: 0.1% aluminon, 1% w/v ammonium acetate in water

Brucine: 5% w/v brucine in acetic acid
Chromotropic acid: 5% w/v aqueous chromotropic acid
8-Hydroxyquinoline solution: 2 g of 8-hydroxyquinoline (BDH), 6 ml of glacial acetic acid, 6 ml of ammonia solution (Sp. gr. 0.91), and 88 ml of water
α-Naphthylamine: 25 mg of α-naphthylamine and 22 ml of concentrated HCl in 1 liter of water
Potassium ferrocyanide: 1 *M* aqueous $K_4Fe(CN)_6$
Potassium thiocyanate: 10% w/v aqueous KCNS
Pyrogallol: 10 g of stannous chloride, 95 ml of water, and 5 ml of concentrated HCl
Stannous chloride: 5% w/v stannous chloride in 5 *N* HCl
Thiourea: 2% w/v aqueous solution
Thoron: 0.1% w/v aqueous solution

Filter Paper

Circles of Whatman No. 1 filter paper for chromatography (55 mm in diameter) were used throughout and are referred to as "paper" in the procedures that are described below.

Procedure

The solution of the mixture was spotted on the marked center of paper 1, with the help of the self-filling pipet. The spot was dried on the ring oven and was punched out (disk). The disk was placed on paper 2, 1 drop 1 *N* HNO_3 was added, and the moist disk was washed with ethyl acetate. Uranium(VI) and thallium(III) moved to ring zone A. The disk with the remaining metal ions was placed on paper 3, 1 drop 1 *N* HNO_3 was added, and the moist disk was washed 10 times with *n*-butanol to transfer gold(III), platinum(IV), selenium(IV), and vanadium(V) to ring zone B. The disk was then kept on paper 4, 1 drop 1 *N* HNO_3 was added, and this was washed 10 times with 20% acetyl acetone in chloroform to elute molybdenum(VI) and titanium(IV) to ring zone C. The disk was dried on the ring oven, placed on paper 5, and washed with hydrogen peroxide; cerium(IV) and beryllium(II) were thus transported to ring zone D. The disk containing the remaining four metal ions was placed on the paper 6 and the disk was washed 10 times with a mix-

ture of 60% ethanol and 3 *N* acetic acid. Thorium(IV), zirconium (IV), and tellurium(IV) were transferred to ring zone E. The disk now contained tungsten, which was identified as tungstic acid. The procedure is shown in Chart 1.

Chart 1. Schematic qualitative analysis.

Mixture spotted on marked circular filter paper, spot dried and punched out (disk). Disk kept on paper 2, one drop 1 *N* HNO_3 added, and moist disk washed with ethyl acetate.

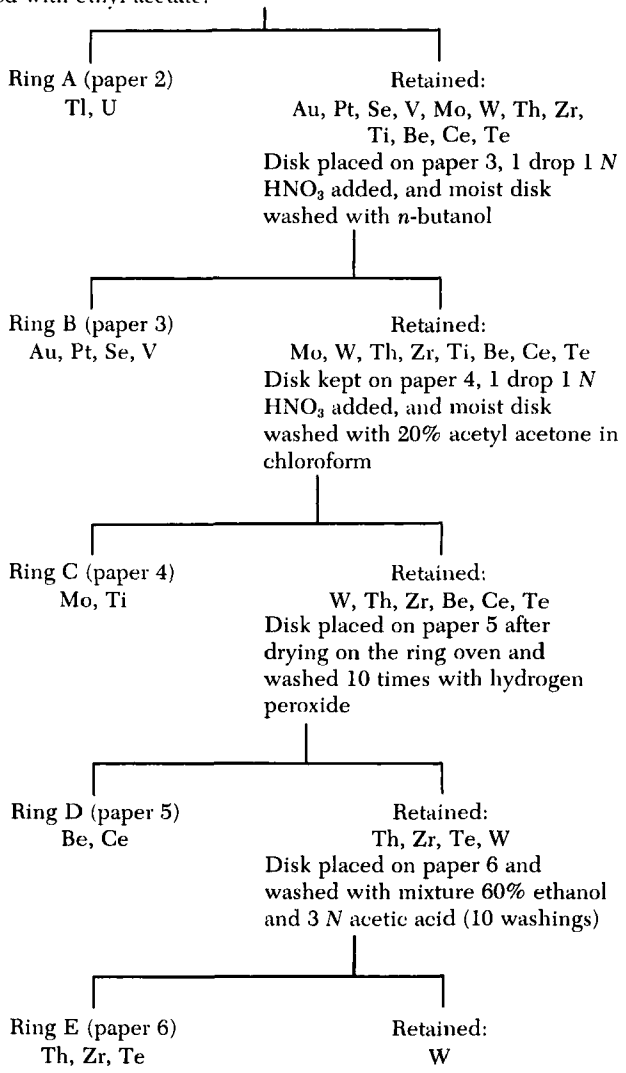

TABLE 1

Reagents for Metal Ions

Metal ion	Pretreatment	Reagent	Color	Ref.
Ring A				
U(VI)	—	$K_4Fe(CN)_6$	Dark brown	(*7*)
Tl(III)	—	Alcoholic α-naphthylamine	Violet	(*8,9*)
Ring B				
Au(III)	—	Pyrogallol + stannous chloride + conc. HCl	Red or violet	(*10,11*)
Pt(IV)	—	Freshly prepared stannous chloride	Yellow to orange	(*12*)
Se(IV)	Treated with (dil) HCl and washed	Thiourea and fumed over conc. HCl	Red	(*13*)
V(V)	—	8-hydroxyquinoline	Blue to greenish blue	(*14*)
Ring C				
Mo(VI)	Treated with 5% solution of $SnCl_2$ in 3 *N* HCl	Potassium thiocyanate	Brick red	(*15*)
Ti(IV)	—	Chromotropic acid	Red-brown, stable in presence of H_2SO_4	(*16*)
Ring D				
Be(II)	—	Aluminon + ammonium acetate	Red	(*17*)
Ce(IV)	Heated the sector on the ring oven and treated with H_2SO_4	Brucine in acetic acid	Pink	(*18*)
Ring E				
Th(IV)	—	Thoron	Crimson pink	(*19,20*)
Zr(IV)	—	Alizarin Red S + conc. HCl	Violet	(*21,22*)
Te(IV)	—	Na_2SO_3 and potassium iodide	Black	(*3*)
Disk				
W(VI)	—	Freshly prepared stannous chloride solution in 1:1 hydrochloric acid	Blue	(*23*)

Identification

The filter papers containing the metal ions in the form of a ring were cut into the required number of sectors according to the number of the metal ions present in the ring. Then each fragment was taken and tested for the particular ion under investigation with the reagents described in Table 1.

For spotting the metal solution on the filter paper, it should be mentioned that 5 drops of the solution taken with the aid of the self-filling pipet are very suitable. To keep the diameter of the spot within reasonable limits (diameter, 10 mm), it is necessary that each drop should be dried before introducing a second drop. The scheme has repeatedly been tested by the analysis of a number of artificially prepared unknowns and has been found to be entirely satisfactory for adoption in qualitative analysis. The total time required for analysis is less than 75 min.

Acknowledgment

The authors are thankful to the State Council of Scientific and Industrial Research, U.P., for financial support.

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Received by editor August 22, 1967
Submitted for publication October 2, 1967