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NOTE

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Abstract

A scheme has been developed for the qualitative separation of the six platinum metals from a drop of mixture by using the ring-oven technique. Osmium (VIII), palladium(II), ruthenium(III), rhodium(III), iridium(III), and platinum(IV) were transferred one by one to the ring zone by washing, respectively, with acetic anhydride, acetone, dimethylformamide–benzene mixture (1 : 3 v/v), methanol, dimethylformamide, and nitric acid (0.5 *N*). The rings obtained were developed by Alizarin Red S (Os); 1-(2-pyridylazo)-2-napthol (Pd); acidic solution of rubeanic acid (Ru); stannous chloride–potassium iodide (Rh and Pt); and benzidine in acetic acid after fuming the paper with bromine (Ir). The time required for the separation is not more than 45 min.

The separation of platinum metals from binary and ternary mixtures has been attempted by various workers using paper chromatography. Majumdar and Chakravartty (*1*) separated quinary mixtures of the platinum metals. The separation of six platinum metals was achieved in these laboratories by a combination of paper chromatography and ring-

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oven methods (*2*), and also by the paper electrophoretic method (*3*). However, both procedures involve two stages for the separation.

An effort has been made to develop a separation procedure for the platinum metals in a spot of solution by the ring-oven technique (4).

Selective solvents for transporting the metal ions to the ring zone were chosen by trial and error experiments. The mixture containing the platinum metals was taken in a single spot on a filter paper circle, and the spot was washed with the chosen solvents so as to transport these metal ions to the ring zones one by one. After separation, the rings of metal ions were developed by suitable chromogenic reagents. It was observed that Alizarin Red S (ARS) serves as a better developing reagent for osmium than the conventionally used thiourea when present singly.

EXPERIMENTAL

Apparatus

A Weisz ring oven (National Appliance Co., Portland, Oregon) was used with a suitable power unit yielding 25 V from 220 V/50 cycles a.c. mains. A self-filling pipet ($\sim 1.5\ \mu l$) was employed in each case.

Filter Paper

Circles of Whatman No. 1 filter paper (55 mm diam) were employed and are referred to as "paper" in the following account.

Solvents for Washing

The following solvents were used for washing the spot:

(1) Acetic anhydride (BDH AnalaR)
(2) Acetone (BDH AnalaR)
(3) Dimethylformamide (Riedel)
(4) Benzene (BDH AnalaR)
(5) Methanol (BDH AnalaR)
(6) Nitric acid (BDH AnalaR)

Metal Solutions

Stock solutions (0.05 *M*) of palladous chloride, chloroplatinic acid, iridium trichloride, rhodium trichloride, ruthenium trichloride, and osmium tetraoxide in HCl (all obtained from Johnson, Matthey and Co.,

London) were prepared by dissolving in water and determining the metal contents (*4*). Mixtures of these were prepared by mixing equal volumes of each and diluting as necessary.

Detecting Reagents

The following detecting reagents were used:

Potassium iodide: 2% w/v in water.

Stannous chloride: solution was prepared by dissolving 10 g of hydrated $SnCl_2$ in 100 ml of 6 *N* HCl and adding 1 g of granular tin.

Rubeanic acid: 1% w/v in ethanol.

1-(2-*Pyridylazo*)-2-*napthol* (PAN): 0.1% w/v in ethanol.

Alizarin Red S (ARS): BDH Indicator solution.

Benzidine: 0.1% w/v in acetic acid (1: 10).

Procedure

The solution of the mixture was spotted on the marked center of Paper I 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 II and was washed with acetic anhydride (5–6 times). Osmium(VIII) moved to Ring Zone A. The disk containing the remaining metal ions was kept on Paper III and was washed with acetone (10 times) to transfer palladium(II) to Ring Zone B. The disk was then kept on Paper IV and this was washed with a mixture of dimethylformamide–benzene (1: 3 v/v) (6 times) to elute ruthenium(III) to Ring Zone C. The disk was then placed on Paper V and washed with methanol (5–6 times), rhodium(III) was thus transported to Ring Zone D. The disk containing the remaining two metal ions (iridium and platinum) was placed on Paper VI and

TABLE 1

Metal ions	Pretreatment	Reagent	Color
Ru(III)	—	Rubeanic acid	Blue
Rh(III)	—	KI + $SnCl_2$	Orange to maroon
Pd(II)	—	1-(2-pyridylazo)-2-napthol	Green
Os(VIII)	—	Alizarin Red S	Red
Ir(III)	Fumed over bromine	Benzidine	Blue
Pt(IV)	—	KI + $SnCl_2$	Yellow to brownish yellow

The mixture was spotted, dried, and the disk punched out. The disk was kept on Paper II and washed with acetic anhydride (5–6 times).

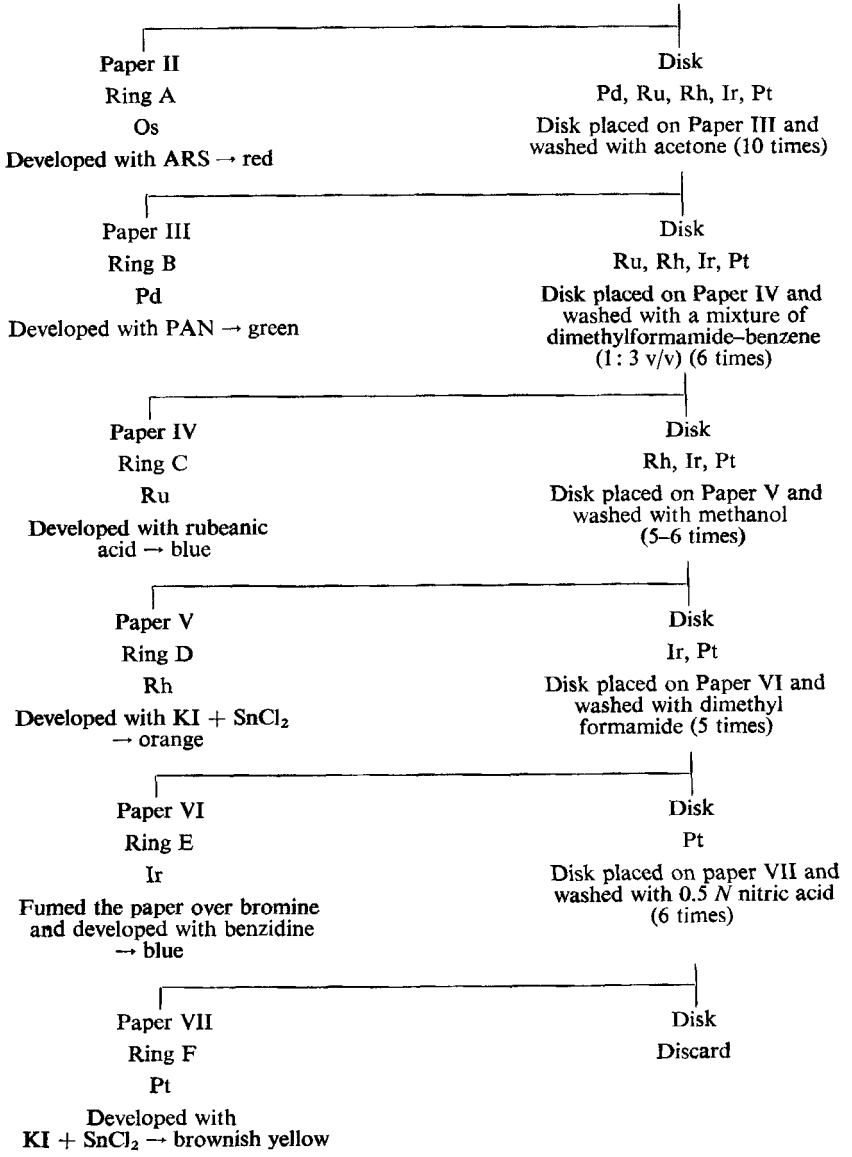

CHART 1. Schematic qualitative analysis.

washed with dimethylformamide (5 times). Iridium(III) was thus transferred to Ring Zone E, and the disk containing only platinum(IV) was placed on Paper VII and washed with 0.5 *N* nitric acid (6 times) to Ring Zone F. The disk was then rejected. Papers II, III, IV, V, VI, and VII containing osmium, palladium, ruthenium, rhodium, iridium, and platinum, respectively, were developed in the ring zone with the reagents described in Table 1.

RESULTS

The scheme was repeatedly tested with a number of artificially prepared unknowns and has been found to be entirely satisfactory for adoption in qualitative analysis. The scheme of separation is summarized in Chart 1.

It has been found that the detection of the ions is possible when they are present in the following amounts in a single drop: Ru 0.76 μg; Rh 0.77 μg; Pd 0.79 μg; Os 1.42 μg; Ir 1.42 μg; and Pt 1.46 μg.

The total time required for analysis is less than 45 min.

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