

not normally warrant the cost of such a device, and a realistic selection of the best aid from this small field would be based on an assessment of other contenders for simplicity in construction and manipulation, for accuracy, and for compatibility with the thin-layer technique.

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Centrifugal chromatography

XIII. Centrifugal chromatography of cations*

A complete qualitative analysis of cations is usually a time-consuming operation. Nevertheless one very often needs extensive information about cations present in a given sample. In a laboratory that is arranged for inorganic analysis this is not a difficult task, because besides classical separation procedures with H_2S , other methods like flame photometry may be used. But in laboratories of a more biochemical character one always meets with difficulties in inorganic analysis. Recently POLLARD *et al.*^{1,2} have published a systematic analysis of cations via paper chromatography that is very suitable for this purpose, but it has the disadvantage common to chromatographic separations that it takes a long time. The present paper shows how the chromatographic analysis of cations can be shortened by means of a centrifugal acceleration technique.

Experimental

For centrifugal paper chromatography the apparatus with central-spot development described by PAVLÍČEK *et al.*³ was used.

Chromatographic separation was performed on Whatman No. 3 paper, the solvent system being *n*-butanol with 0.5 % benzoyl-acetone saturated with 0.1 N nitric acid.

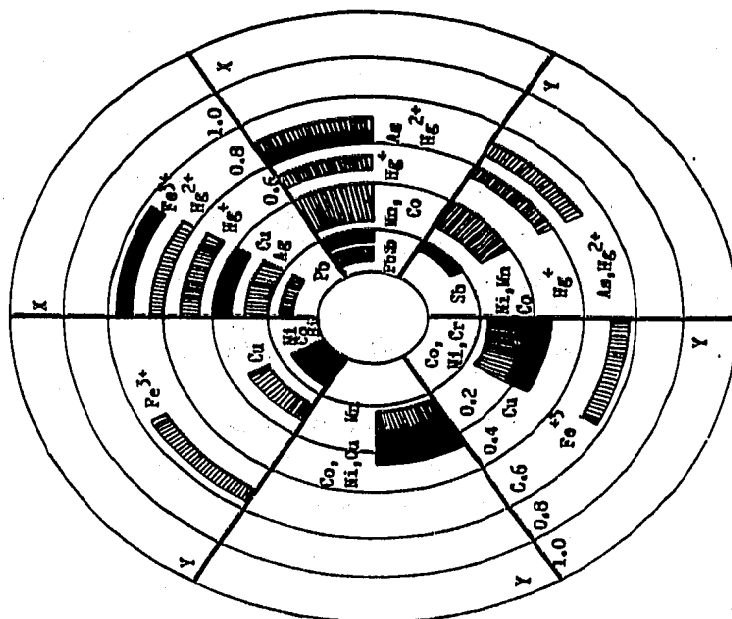
The sample for analysis (if in the solid state) was dissolved or mixed (if it is in

* For Part XII, see J. ROSMUS, M. PAVLÍČEK AND Z. DEYL, *Proc. Symp. Thin-Layer Chromatography, Rome, May, 1963*.

1. K_2CrO_4 , 1% soln. in water: Pb, Ag, Hg^+ , Hg^{2+} .
2. Concentrated ammonia: Pb, Ag, Cu, Hg^+ , Fe^{3+} .

1. 2 N HCl + $(NH_4)_2S$, dil. (1:1): Pb, Mn, Co, Ni, Ag, Cd, Hg^+ , As, Hg^{2+} .
2. 2 N NaOH + 20% H_2O_2 , then SO_2 : Ni, Hg^+ , Hg^{2+} .

1. $(NH_4)_2S$ + concentrated HCl, then 5% soln. of Na_3PO_4 in water: Ni, Mn, Co, Hg^+ , As, Hg^{2+} .
2. 5% soln. of phosphotungstic acid: Sb.



(Fig. 1a)

1. KCNS sat. soln. in H_2O + acetone (1:1): Bi, Co, Cu.
2. Ammonia + 1% soln. of dimethylglyoxime in ethanol: Ni.

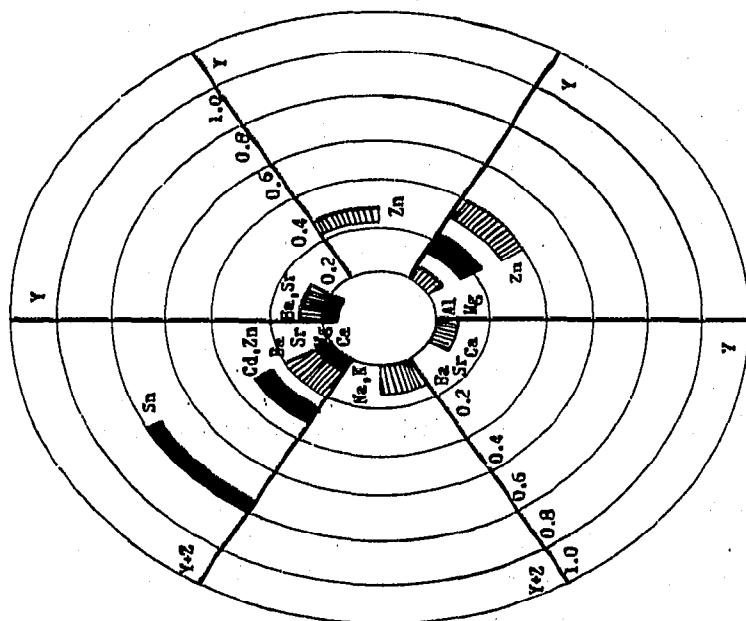
1. 2 N NaOH saturated with Br_2 + 2 N NaOH (2:1), then SO_2 , then 0.05% soln. of benzidine in acetic acid and ammonia vapours: Mn.
2. Cu salt of thio-oxamide, 0.1% soln. in methanol, then ammonia vapour: Co, Ni, Cu.

1. 2 N NaOH sat. with Br_2 , diluted with 2 N NaOH (2:1): Mn, Co, Ni, Cr, Cu, Fe^{3+} .
2. 2 N H_2SO_4 + 20% H_2O_2 (1:1): Cu, Cr.

1. 8-Hydroxyquinoline, 0.5% soln. in 60% aq. ethanol, ammonia vapours: Al, Ca, Sr, Ba, Mg, Cd, Zn, Sn.
2. Acetic acid: Al, Zn, Sn.

1. Uranylacetate + Zn-acetate, saturated soln. in 1 N acetic acid: Na.
2. Pb-Co nitrate: K.

1. Calciumacetophenone, 1% soln. in NH_4OH : Ca, Sr, Ba.



(Fig. 1b)

Fig. 1. Scheme of cation separation. X, Y and Z designate different modes of preparing the sample on the start-line (see text); texts at individual sections describe the composition of the detection agent and the detected cations respectively.

If two detecting agents are presented then the section is cut into two sub-sections and each is detected separately. Only when the second reagent is introduced by the word "then", is the whole section detected by successive spraying with both reagents.

1. NH_3 vapours + sodium rhodizonate, 0.1% soln. in water: Sr, Ba, Zn, Sn.
2. 2 N HCl + 2 N CH_3COOH (1:1): Ba.

1. $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, 1% soln. in water, soln. contains 10% of H_2SO_4 ; then: 2 g HgCl_2 + 3 g NH_4SCN in 100 ml H_2O : Zn.

1. Quinalizarin, 0.02% soln. in methanol: Al, Zn.
2. 2 N NaOH + 20% H_2O_2 (1:1): Mg.

solution) either with 2 *N* nitric acid (spotted as X), or with 2 *N* hydrochloric acid (spotted as Y).

Spots on the startline, designated as Z, were prepared by spotting the solution Y and spraying with sodium hypobromite, and after drying they were sprayed twice with 2 *N* acetic acid.

The samples were spotted on two discs of Whatman No. 3 chromatographic paper. On the first, two samples of X and four samples of Y were spotted. On the second chromatogram there were two samples designated as Z and four samples designated as Y. The system of detection and the nature of detection agents are given in Fig. 1.

One run took about 40 min in the solvent system used, and a complete analysis was finished within 1.5 h (mostly passive working time). After development the chromatograms were dried and cut into six sections, each for detection purposes.

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Cation-exchange separation of protactinium-233 from irradiated thorium

A knowledge of the analytical separation of protactinium is of technical importance in connection with the chemical processing of irradiated thorium. Several ion-exchange methods have been proposed for separating protactinium from thorium¹⁻⁶. Because of strong hydrolytic and irreversible behavior of protactinium in contact with glass and metal surfaces, most of the ion-exchange systems suffer from an appreciable loss of protactinium during the ion-exchange chromatographic process except for the system involving hydrofluoric acid as a component of eluent. We have been exploring the effective ion-exchange procedures for several elements in thiocyanate-hydrochloric acid media⁷⁻¹⁰. The present paper describes a new ion-exchange chromatographic procedure for separating carrier-free protactinium-233 from neutron-irradiated thorium with a strong acid cation exchanger in thiocyanate-hydrochloric acid solution. Because of the tendency of protactinium to form a stable complex with thiocyanate ions, the cumbersome hydrolytic property of protactinium can almost be excluded so that the rapid separation of protactinium-233 from irradiated thorium was achieved with a shorter column.

Experimental

Dowex 50 W, X-8, of "analytical grade", was used (particle size 100 to 200-mesh). Before use the resin was further purified as described before⁷. Two grams of the

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