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## Note

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### Ion-exchange paper chromatography of inorganic ions

#### XX. The behaviour of metal ions in nitrite solutions

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Sodium nitrite has been used as an eluent in only very few ion-exchange separations (*e.g.*, refs. 1 and 2) and, as far as we know, no general survey of the adsorption of metal ions on ion exchangers from sodium nitrite solutions has been published. This lack of information is probably due to the fact that nitrite cannot be used in acidic solutions and most metal ions are unstable in alkaline solutions, thus leaving only the range around pH 7 for useful experimental work.

The purpose of this paper is to indicate the limitations and possibilities of sodium nitrite solutions as eluents in ion-exchange thin-layer chromatography.

#### TECHNIQUE

The thin layers used were Polygram Ionex-25 SB-Ac (Macherey, Nagel & Co., Düren, G.F.R.), which is a strongly basic ion-exchange resin thin layer in the acetate form, and Polygram Ionex-25 SA-Na (Macherey, Nagel & Co.), which is a strongly acidic ion-exchange resin thin layer in the sodium form.

Strips 12 cm long and 5 cm wide were developed in glass reagent jars with tight-fitting plastic stoppers (16 cm high and 8 cm diameter) by standing the thin layers in about 1 cm of the eluent at the bottom of the jar.

The metal ions were detected after a development of about 6 cm with either ammonium sulphide or ammoniacal alcoholic 8-hydroxyquinoline.

#### RESULTS

Fig. 1 shows the positions of the spots when developed with 1 *N* sodium nitrite solution (A) on the sulphonic cation-exchange thin layer and (B) on the strongly basic anion-exchange thin layer.

The solutions were prepared by dissolving a few crystals of the metal salts in 1 *N* sodium nitrite solution and heating the solution for a few minutes over a flame. For Pd(II), Pt(IV), Rh(III) and Ru(III), solutions in hydrochloric acid or aqua regia were evaporated just to dryness on a steam-bath and then dissolved in 1 *N* sodium nitrite solution and heated as recommended by Blasius and Fischer<sup>3</sup>. Distinct colour changes are noted with these platinum-group metals, the nitrite solutions being either colourless or pale yellow.

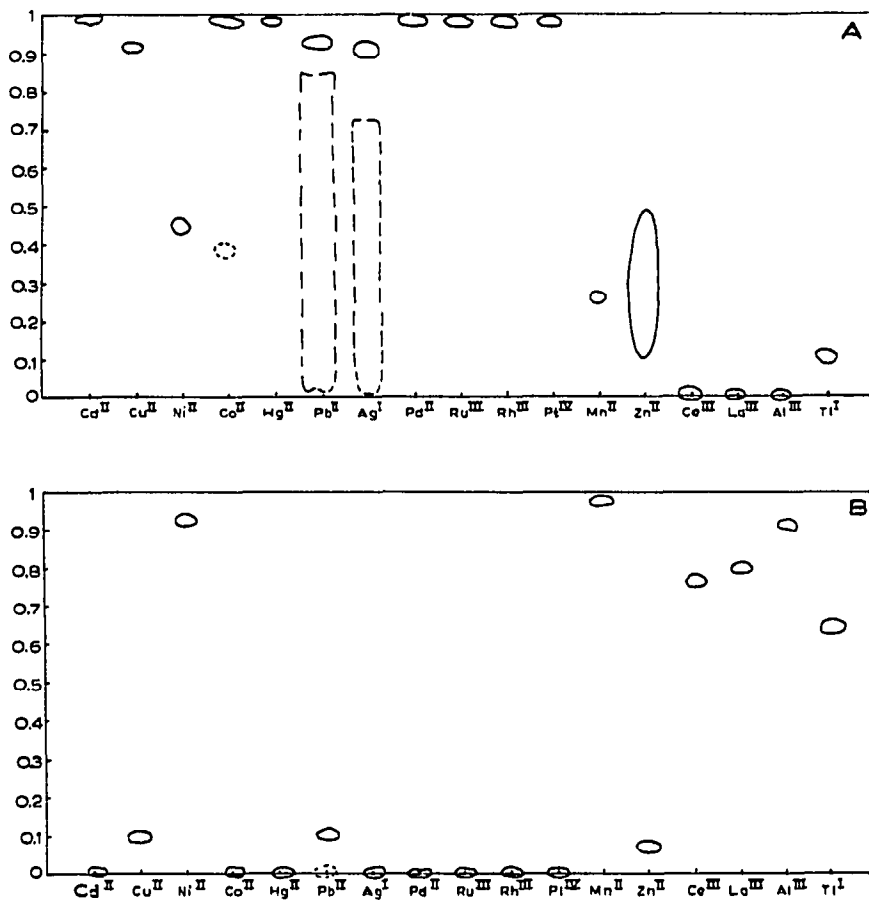


Fig. 1. Schematic representation of the movement of metal ions on ion-exchange resin thin layers developed with 1 *N* sodium nitrite solution. (A) Polygram Ionex-25 SA-Na; (B) Polygram Ionex-25 SB-Ac.

Cobaltinitrite was prepared as recommended<sup>4</sup> for the detection of Co(II) and also fresh solutions of pure sodium cobaltinitrite were chromatographed. None of these solutions gave only a single spot, minor spots being consistently observed, as already reported by Carunchio and Gerardi<sup>5</sup>. Although in this work the nitrite complex of Ru(III) yielded only a single spot, this need not be the case, as we reported earlier<sup>6</sup>, and several spots may occur in some instances.

Au(III) and Hg<sup>2+</sup> are decomposed to the metallic state when chloroauric acid (HAuCl<sub>4</sub>) and mercury(I) nitrate (Hg<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>) are dissolved in 1 *N* sodium nitrite solution. Bi(III) and Fe(III) nitrates, when dissolved in 1 *N* sodium nitrite solution, yield dense precipitates, presumably by hydrolysis to the hydroxides.

It is thus evident that the occurrence of more than one species for Co(II) and Ru(III), the precipitation of numerous ions in neutral nitrite and the formation of tails with Pb(II) and Ag(I) (hydrolysis or chloride precipitation) make the nitrite system useless as a medium for separation of numerous metal ions.

The nitrite system has, however, one useful property: Cd(II), Hg(II), Pd(II),

(RuIII), Rh(III) and Pt(IV) complexes are very strongly adsorbed on the anion exchanger and not adsorbed at all on the cation exchanger. This is unusual behaviour and we would like to suggest that the size and nature of the nitrito group as complexant make the complexes highly hydrophilic so that they are not adsorbed on the resin network of the sulphonic resin, while on the anion exchanger the very strong adsorption must be due mainly to electrostatic attraction. This unusual behaviour may be useful in the future for the concentration of some of these complexes from non-complexed or less complexed metal ions.

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