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Spot test for potassium on paper chromatograms

Sodium tetraphenylboron has been shown to be a sensitive reagent for the detection and determination of potassium. The properties and uses of this compound are the subject of a comprehensive review and an extensive bibliography[1,2]. Potassium tetraphenylboron is quite insoluble, with a solubility product of about $2 \cdot 10^{-8}$, but its white color limits its use in the detection of potassium on filter paper chromatograms. However, a sharp color contrast can be produced by treatment of the precipitate with mercuric chloride solution in the presence of bromophenol blue indicator. Mercuric chloride reacts with tetraphenylboron salts to produce hydrochloric acid and boric acid. This test can be used to detect amounts of potassium as small as a few $\mu g/cm^2$ of paper.

Ammonium ion will also be precipitated by tetraphenylboron and can be detected by this method. If ammonium ion interferes with the potassium test, precipitation in a solution made alkaline with NaOH will prevent precipitation of moderate amounts of ammonium[3]. Cesium, rubidium, thallium, and silver ions also precipitate with the reagent and interfere with the test.

Test reagents

(1) A 2% solution of sodium tetraphenylboron in water. (2) A solution of bromophenol blue (0.1%) and mercuric chloride (2.5%) in 80% ethanol. Adjust carefully to the highest acidity which will still produce a blue color when the mixture is sprayed or brushed on dry filter paper.

Procedure

(1) Dip the chromatogram in the tetraphenylboron solution; (2) wash the paper with a stream of water to remove excess reagent; (3) dry the paper, using low heat if desired; (4) brush the dry paper lightly with a soft brush moistened with the mercuric chloride reagent, or spray lightly in a well ventilated hood. (The spray is irritating and toxic.)

Experimental

A 5-μl capillary tube was used to spot solutions of KCl and NH_4Cl on inch-wide strips of Whatman No. 1 paper. Amounts varying from 0.025 to 5.0 μmoles of ion were applied to an area of 1 cm^2 of paper. The paper was then dipped in 2% sodium tetraphenylboron solution. About 200 cm^2 of the paper was then washed uniformly with a stream of water from a wash bottle delivering about 50 ml/min. A total of about 50 ml of wash water was used for each strip, care being taken to wash alternately both sides of the paper. The strip was then dried for a few minutes on a watch glass on a hot plate at a temperature low enough to prevent spattering of water droplets. The mercuric chloride–bromophenol blue solution was then brushed lightly over the paper, producing a blue background with bright yellow spots of the same size as the original spots. For amounts as small as 0.025 μmole, the yellow color appeared slowly on drying and this amount appears to be near the lower limit of detection.

Prevention of ammonium ion precipitation was tested by using a sodium tetra-

phenylboron solution made alkaline by addition of NaOH (3 vol. of the 2% reagent mixed with 1 vol. of 6 *M* NaOH). Strips containing 0.5 μmole or less of ammonium ion produced negative tests when washed and tested as before. Spots containing 0.05 μmole of potassium ion on the same strips produced positive tests. When the amount of ammonium ion is as large as several μmoles/cm^2, a precipitate will form even in alkaline solution. This may be removed by washing with a stream of 1 *M* NaOH solution, followed by the wash with water, but this treatment also removes smaller quantities of potassium ion (0.05 μmole/cm^2).

In testing for small amounts of ions it is necessary to limit the amount of wash water. Washing a sample of 0.025 μmole of potassium with 150 ml of water, instead of 50 ml, usually resulted in a negative test. The minimum amount of water required depends on the area of the paper, efficiency of washing, etc., and should be determined for each operator by running a blank.

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