

SODIUM SALICYLATE AND 8-HYDROXYQUINOLINE IN INORGANIC MICROCHEMICAL ANALYSIS

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INTRODUCTION. In microchemical analysis very sensitive and selective or specific reactions are used to detect minute quantities of a substance to confirm its presence or absence. Such tests can be used to test the purity of chemicals, to aid in testing technical materials, and as confirmatory tests to supplement the regular scheme of analysis. The very small amount of material required and the high sensitivity obtained make these tests very useful.

There are many different forms of microchemical tests each with its definite use. These are a spot test, slide test, fiber test, test-tube test, and a bead test. These names are derived from the medium on which the test is carried out, or in the case of a spot test, the appearance of the test after completion.

When reporting the sensitivity of a test, both the identification limit and the concentration limit must be reported as it obviously makes a difference whether a gamma (10^{-6} gm) of a substance can be detected in 0.001 ml or in 1 liter. The LI (limit of identification) is the minimum quantity of a substance which can just be revealed by a given reaction and the LC (limit of concentration) is the concentration at which the minimum quantity can just be revealed.

The LI is established by actual experiment using progressive dilutions of a solution under test in conjunction with a standardized procedure and the LC by using the proportion 1: (volume of the solution used in the test times 10^6) over (gammas of the material in the given volume).

Reactions which are commonly used are those incorporating an organic reagent. The reaction products which may be formed are a normal salt, a complex compound, or an oxidation or reduction product of the reagent.

This research consisted of investigating the use of sodium salicylate for the detection of iron, both alone and in the presence of copper; and the use of 8-hydroxyquinoline in the detection of iron and uranium.

SYNOPSIS OF EXPERIMENTS

DETECTION OF THE FERRIC ION WITH SODIUM SALICYLATE

Chemical principle: When the ferric ion reacts with sodium salicylate a reddish brown inner complex compound is formed.

Procedure: A drop (0.01 ml) of the test solution which is neutral or slightly acid with acetic acid is allowed to drop on a specially impregnated filter paper. A positive reaction is indicated by a reddish brown fleck. Since the intensity varies with the concentration, it is possible to use the test colorimetrically. This test will indicate as little as 0.156 gamma in a dilution of 1:64,000.

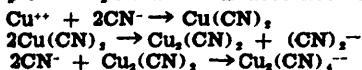
The reagent paper is made by soaking Whatman No. 40 acid washed filter paper in a 0.25 M aqueous solution of sodium salicylate and then allowing it to dry in the air. The pH is important because excess mineral acids or bases destroy the color.

Limitation: This test can not be used for the detection of iron in the presence of a large amount of copper; however the method for masking the copper follows immediately.

DETECTION OF THE FERRIC ION IN THE PRESENCE OF COPPER

Chemical principle: When a solution of cupric ion is treated with a cyanide the following reaction takes place, which ultimately results in a soluble,

colorless, complex compound which does not affect sodium salicylate:



The cupric ion reacts with the CN^- ion to form cupric cyanide which in turn decomposes into cuprous cyanide which dissolves in the excess CN^- present to form a complex soluble cyanide. Acetic acid must be present to counteract the effect of the HCN on the color.

Procedure: 0.05 ml of the sodium salicylate reagent and an equal volume of a saturated aqueous solution of sodium acetate (saturated at 20°C , 46.5 gms per 100 ml) are mixed on a spot plate; 0.05 ml of the test solution which was made acid with acetic acid is now added. The green precipitate of copper is now dissolved in a minimum of 0.5 M potassium cyanide. The color of the reaction is now easily seen. It is advisable to run a blank containing only copper along with the main test.

DETECTION OF THE FERRIC ION WITH 8-HYDROXYQUINOLINE

Chemical principle: The ferric ion reacts with 8-hydroxyquinoline to form a gray to black reaction product.

Procedure: The test solution is allowed to fall on the prepared reagent paper made by soaking filter papers in a saturated alcoholic solution of 8-hydroxyquinoline and allowing them to dry. LI: 0.078 gamma, LC: 1:128,000.

Limitations: This test can not be used in its present form as a reagent for iron if many interfering ions are present in comparatively great amounts because the reagent reacts with a great many ions and masking reactions have not yet been developed; however 8-hydroxyquinolin shows promise as being an excellent reagent.

Tests indicate that copper, zinc, uranium, cobalt, lead, aluminum and tin interfere.

DETECTION OF URANIUM WITH 8-HYDROXYQUINOLINE

Chemical principle: 8-hydroxyquinoline reacts with the uranyl ion to give an orange yellow fleck of uranyl 8-hydroxyquinolinolate.

Procedure: The procedure is the same as for the detection of iron with this reagent. The presence of uranium is indicated by an orange red fleck. LI: 0.312 gamma, LC: 1:32,000. Those limitations listed above must be observed for this test.

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