Isoquinoline in Chemical Microscopy: A Reagent for Zinc and Cadmium

HAROLD F. SCHAEFFER, Valparaiso University

As pointed out by Spakowski and Freiser (1), when a solution containing isoquinoline and a thiocyanate is added to solutions of certain divalent cations, precipitates corresponding to the formula $M(C_9H_7N)_2(CNS)_2$ are formed. Although these two workers applied the reagent in the quantitative precipitation of zinc and copper, they did not investigate its possibilities in the field of chemical microscopy. We have now found that the isoquinoline-thiocyanate reagent can be employed in the microchemical detection of zinc or cadmium ions because the resulting metallo-organic compounds separate in the form of microscopic crystals.

Since isoquinoline is only slightly soluble in water, it is used in the form of the hydrochloride. For use in chemical microscopy the reagent is prepared by dissolving 13 g. isoquinoline in 75 ml. of N-hydrochloric acid, adding 19 g. of ammonium thiocyanate, and diluting to one liter with distilled water. Such a solution will contain approximately 0.25 mole of ammonium thiocyanate and 0.1 mole of isoquinoline hydrochloride per liter. The isoquinoline obtained by recrystallizing E. K. Co. *practical* grade has been found satisfactory for this purpose.

In performing a test a small drop of reagent is allowed to flow into

Figure 1. Crystals formed by the presence of zinc ions. (Scale for Fig. 1 should be labeled 0.4 mm.)

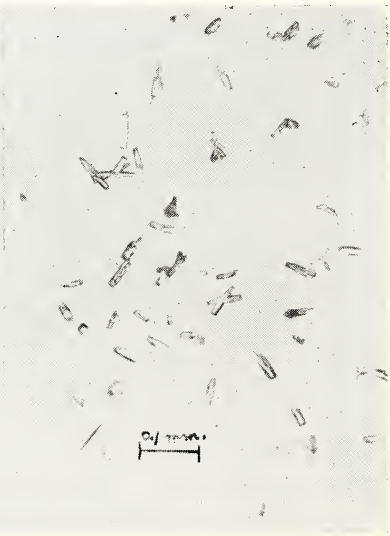

Figure 2. Crystals formed by the presence of cadmium ions.

a small drop of test solution on a slide. Characteristic crystals (Fig. 1) are formed with dilute solutions of various zinc salts, including the chloride, nitrate, acetate, sulfate, etc. With zinc acetate, for example, good tests have been obtained on solutions containing less than one part zinc in 10,000. This makes it possible to detect one gamma in a microdrop of sample. The composition of the crystals is represented by the formula $Zn(C_9H_7N)_2(CNS)_2$.

When treated with the same isoquinoline-thiocyanate reagent, cadmium yields characteristic crystals somewhat smaller than those formed by zinc salts (Fig. 2). The test appears less sensitive for cadmium, although good tests are obtained on solutions containing 1 part cadmium in one or two thousand parts of solution. The complex compounds formed by both zinc and cadmium yield anisotropic crystals. Common cations which may interfere with the tests include silver, mercuric, nickel, cobalt, cupric, and trivalent bismuth and antimony. In the presence of ferric ion the test for cadmium or zinc may be obscured by the formation of practically opaque cubic crystals and a red solution. However, if the iron is present in very small amounts (1 part in 10,000) the tests will not be obscured.

Literature Cited

1. SPAKOWSKI, A. E. and H. FREISER. 1949. Isoquinoline as a reagent in inorganic analysis. Anal. Chem. **21**:986.