

Modification of the Orthotolidine Method for Determining Small Amounts of Iodine

W. E. THRUN, Valparaiso University

The Lange-Ward (2) method has been modified to increase reliability and sensitivity. A buffer is used which insures the optimum pH of 4.0 for the color reaction. The method is not suitable for use with a colorimeter because of constant color change. Its sensitivity is about one tenth that of the starch Iodine-iodide reagent of Gross et al. (1).

Reagents

A .6% alcoholic (95%) solution of orthotolidine or an aqueous solution prepared by dissolving one gram of orthotolidine in 100 ml. water and adding 6.5 ml. of glacial acetic acid.

A buffer solution. Dissolve 3.0 grams sodium acetate Trihydrate, 7.7 mls. of glacial acetic acid in 10 mls. of water.

A 3% solution of hydrogen peroxide.

Standard solutions of potassium iodide containing 0.2, 0.4, 0.6, 0.8 and 1 to 10 p.p.m. iodine. In the upper range intervals of 0.5 or less p.p.m. iodine may be desirable.

15 x 25 mm matched specimen or test tubes.

Procedure

Five ml. of the sample and of standard solutions are pipetted into the tubes. Four drops of the buffer solution and 0.2 ml. orthotolidine solution are added and mixed. Then 3 ml. of hydrogen peroxide solution are added and mixed again. Colors are compared 10 to 15 minutes later.

Discussion

The 0.2 p.p.m. iodine solution does not give a distinct color until it is 10 minutes old.

The aqueous solutions of orthotolidine do not become brownish and gelatinous as soon as the alcohol with the test solutions. Both reagent solutions turn somewhat brownish upon standing, but this does not affect the comparative results. In a range of 0.2 to 1.0 p.p.m. it is possible to differentiate between solutions of .2 p.p.m. differences and in the range of 1 to 5 p. p. m. between differences of 0.5 p. p. m.

Literature Cited

1. GROSS, W. G., L. K. WOOD, and J. S. MCHARGUE, 1948 *Analytical Chemistry* **20**:900-1.
2. LANGE and WARD, 1925. *J. Am. Chem. Soc.* **47**:1000-3.