

LETTER

A New Microdetermination of Iodide by its Catalytic Reaction

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The authors found that the orange color of ferric thiocyanate which was formed by dilute potassium thiocyanate and an excess of ferric salt in dilute nitric acid solution, faded very slowly owing to the oxidation of thiocyanate, but rapidly in the presence of minute amounts of iodide.

The catalytic action of iodide on this reaction was studied colorimetrically, and a new microdetermination of iodide was accomplished.

Iodine also catalyzed the fading of ferric thiocyanate in exactly the same way as iodide, but chloride and bromide did not. In preliminary experiments it was found that this catalytic reaction was greatly affected by the presence of small amounts of nitrite. The reaction rate depended not only on the concentrations of iodide present, but also on the concentrations of thiocyanate and nitric acid, and especially on the temperature (Fig. 1).

Therefore these factors must be kept constant for iodide determination.

The determination of iodide could be made by measuring the absorbancy in a definite period of time and using the calibration curves (Fig. 1) previously obtained under the same conditions.

Procedure and Results:

To 10 cc. of the sample solution, 1.0 cc. of mixed reagent (3×10^{-3} or 10^{-3} M in potassium thiocyanate, 3×10^{-4} M in sodium nitrite) and 2.0 cc. of ferric alum reagent (prepared by dissolving 6 g. of ferric alum in 100 cc. of 5.7N nitric acid) were added. The absorbancies were plotted against

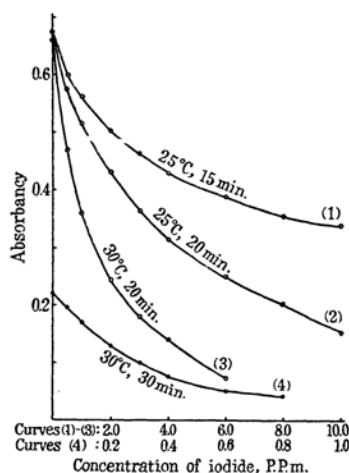


Fig. 1.—Calibration curves of iodide.

Curves (1)–(3): 3×10^{-3} M SCN⁻

Curve (4): 10^{-3} M SCN⁻

the concentrations of iodide as shown in Fig. 1.

The absorbancy was determined by a Beckman Model DU spectrophotometer at $460 m\mu$ ⁽¹⁾ using 10 mm. cell. The calibration curves shown in Fig. 1 were reproducible within $\pm 5\%$.

The determination of iodide in concentrations of 0.5–10 p.p.m. could be made under the following conditions: 3×10^{-3} M SCN⁻, 25°C., 20 minutes (Curve 2). The determination of iodide in concentrations of 0.05–0.8 p.p.m. could be made under the following conditions: 10^{-3} M SCN⁻, 30°C., 30 minutes (Curve 4).

It was also possible to determine 0.001–0.05 p.p.m. of iodide under the following conditions: 10^{-3} M SCN⁻, 45°C., 30 minutes.

By this method 0.001–10 p.p.m. of iodide could be determined in the presence of chloride and bromide.

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(1) I. Iwasaki, S. Utsumi and T. Ozawa, This Bulletin, 25, 226 (1952).