

The Spectrophotometric Determination of Chromium with 2-Hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-naphthalein (Sodium Salt)

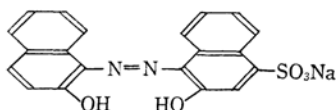
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2-Hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-naphthalein (Sunchromine blue black R) has been examined spectrophotometrically in order to determine the molybdenum,¹⁾ zirconium²⁾ and titanium³⁾ in a methanol-hydrochloric acid solution; it has also been examined as an indicator of the complexometric titration of calcium in the presence of magnesium.⁴⁾

In this paper, a new method will be described for the spectrophotometric determination of trace amounts of chromium(III). The method is based on the stable blue complex formed by the reaction between chromium ions and 2-hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-naphthalein.

The structure of the reagent is as follows:



Experimental

Reagents.—The reagent solution was prepared by dissolving 0.1 g. of Sunchromine blue black R in 100 ml. of distilled water.

The standard solution, containing 100 μ g. of chromium(III) per milliliter, was prepared by dissolving chromium alum (analytical grade) in 0.01 N sulfuric acid. The other reagents used were also of analytical grade.

The Procedure for the Determination of Chromium.—Transfer the solution containing 5 to 75 μ g. of chromium to a 50 ml. volumetric flask, and add 5 ml. of the 0.1% reagent solution. Then add 10 ml. of a borax-sodium hydroxide buffer solution (pH 10), and make up to the mark with distilled water. After ten minutes, measure the absorbance at 630 $m\mu$, using a reagent blank solution as a reference.

Results and Discussion

Absorption Spectra.—The absorption spectra of the complex are shown in Fig. 1. At pH 10, the maximum absorbance is at 630 $m\mu$.

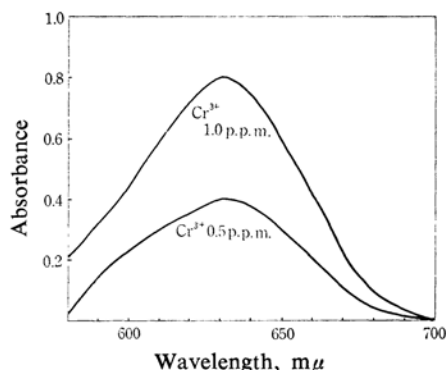


Fig. 1. Absorption spectra of the Cr-complex. pH 10, reagent 80 p. p. m., against reagent blank.

The Effect of pH.—A borax-sodium hydroxide buffer was satisfactory for the determination of chromium. The effect of pH on the color intensity of the complex is shown in Fig. 2. The complex has a maximum intensity in the pH range from 9.8 to 10.3.

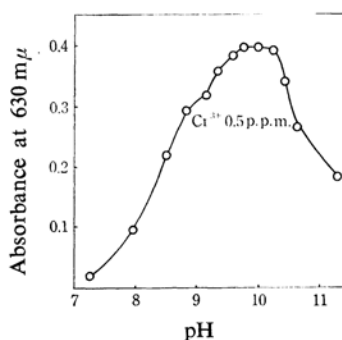


Fig. 2. Effect of pH on the Cr-complex.

Rate of Color Formation.—The color intensity reaches a maximum in ten minutes and remains constant for about an hour.

Beer's Law.—Beer's law is obeyed up to about 1.2 p. p. m. of chromium at 630 $m\mu$ (see Fig. 3).

Sensitivity.—The molar extinction coefficient is about 42000 at 630 $m\mu$, and the spectrophotometric sensitivity is 0.0013 μ g. Cr/cm² for $\log I_0/I=0.001$.

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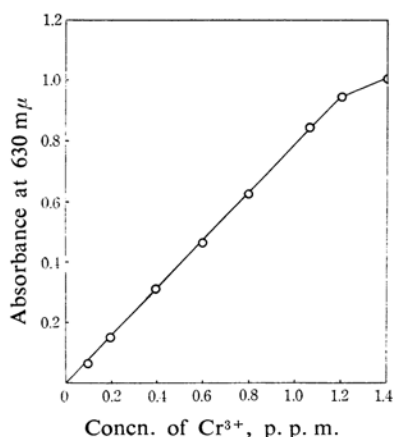


Fig. 3. Beer's law curve, pH 10.

Interfering Ions. — The effect of various cations was studied; it was found that aluminum, beryllium, iron(III), scandium, and large amounts of calcium, cobalt, zinc and titanium interfere. The presence of these interfering metal ions gives the smaller absorbance at 630 mμ. Hexavalent chromium, chloride and sulfate are without effect; 50 μg. of chromium(III) can thus be successfully determined in the presence of 100 mg. of any of these ions. The interfering metals must be removed or masked prior to the determination. Alumi-

num^{5,6}) and iron(III)⁶) can be removed by the 8-hydroxyquinoline chloroform extraction method from chromium. The acetylacetone chloroform extraction method⁷) effected a good separation of aluminum, iron(III) and beryllium from chromium.

Summary

A spectrophotometric method for the determination of traces of chromium(III) with 2-hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-naphthalein has been described. Beer's law is obeyed up to 1.2 p.p.m. of chromium. The spectrophotometric sensitivity is 0.0013 μg. Cr/cm² based on an absorbance of 0.001 unit at 630 mμ. The accuracy and precision are satisfactory. The interference of foreign ions can be avoided by 8-hydroxyquinoline chloroform extraction or by acetylacetone chloroform extraction.

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