

# Colorimetric Determination of Ruthenium with $p$ -Nitrosodimethylaniline

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The growth in importance of the chemistry of ruthenium has brought with it demands for methods of determining very minute amounts of the metal. From several organic compounds tested for color formation with ruthenium salts,  $p$ -nitrosodimethylaniline has been selected for more detailed study. This reagent in aqueous solution will develop a bottle-green color when warmed with a solution of ruthenium chloride or bromide buffered to pH 4.1. Maximum absorption occurs at 610 m $\mu$  when this solution is compared with a reagent blank. Ruthenium may be separated from the other platinum metals by conventional distillation procedures and then determined in the distillate. The recommended procedure is simple, requires little of the operator's time, and is more sensitive than other methods in the literature.

THE colorimetric determination of ruthenium has been the subject of several investigations in recent years. Brockmeyer and Singer (1) applied 3-hydroxyquinoline-8-carboxylic acid; Ayres and Young (2) and DeFord (3) independently studied thiores as a colorimetric reagent; Ayres and Young (3) also examined rubeanic acid (dithio-carboxide) for this purpose. Sandell (4) discussed these and other methods. Marshall and Rickard (5) described a procedure in which the color of potassium rubeinate was made the basis of a colorimetric determination but this was applicable to higher concentrations than any of the foregoing methods.

In this paper  $p$ -nitrosodimethylaniline in aqueous solution is recommended as a colorimetric reagent of high sensitivity for ruthenium. This substance has already been successfully applied by Yoc and Overholser (6) to the colorimetric determination of palladium. Ogburn (8) reported that various nitroso compounds, including  $p$ -nitrosodimethylaniline, gave no visible reaction with ruthenium salts. However, the reaction described in this paper requires heating to develop the color, and Ogburn may have overlooked this.

## APPARATUS AND SOLUTIONS

**Optical Instruments.** Absorption measurements were made mainly with a Klett-Summerson photoelectric colorimeter. The variation of absorbancy with wave length was examined with a Beckman Model DU spectrophotometer. In order to establish a relationship between the logarithmic scale of the Klett instrument and conventional absorbancy units, some comparisons were made with the same solution in 2-cm. rectangular cells in the Klett colorimeter, in 1-cm. rectangular cells in a Lumetron Model 402 EF colorimeter, and with the same filter in each instrument. The filter used was a Lumetron monochromatic No. M 610, having maximum transmittance at 610 m $\mu$ . It was found that, under these conditions, 519 Klett units equaled unit absorbancy.

Most of the measurements were made with the solutions contained in cylindrical Klett tubes, and these were carefully matched in the following manner. The absorption of samples of a large batch of green chromium(III) chloride solution was measured five times in each of a dozen Klett tubes. The entire sixty measurements were made in random order. The concentration of this solution was selected to give an absorption of about 200 Klett units when the 610 m $\mu$  filter was used. The results were examined by conventional statistical methods to compare the variance between tubes with that within tubes. On the basis of this examination it was possible to select sets of tubes for the following experiments for which the variance of measurements from tube to tube was not significantly greater (to a probability of 5%) than that of measurements within any one tube.

\* Present address, Canadian Industries, Ltd., McMasterville, Quebec, Canada.

**Standard Ruthenium Solution.** A stock solution of "ruthenium chloride" was prepared by distillation of ruthenium tetroxide followed by refluxing in 6  $M$  hydrochloric acid, according to the procedure of Rogers, Beamish, and Russell (9). The stock solution was standardized by the thionalide method described by these authors.

The thionalide precipitation continues to be successfully applied to the determination of ruthenium in this laboratory. Recent statements by Flagg (6) that results on semimicro quantities tend to be low by as much as 10% have never, so far as the authors are aware, been supported by experimental evidence. Such statements cannot be reconciled with the observations of Thiers, Graydon, and Beamish (11), made with the aid of radioactive ruthenium, that, within the prescribed acidity range, precipitation of 8  $\mu$ g. of ruthenium was complete to less than 0.6 microgram.

Solutions of concentration suitable for colorimetric work were prepared by volumetric dilution from the stock solution. Two such solutions, which were used for many of the results in this paper, contained 44.6 and 4.46 micrograms of the metal per ml.

**Buffer Solutions.** Buffer solutions containing acetic acid and sodium acetate were made up in concentrated form, so that 1 ml., when diluted to 25 ml. gave the desired stabilized pH. The buffer in the recommended procedure was 4  $M$  in acetic acid and 1  $M$  in sodium acetate, and this gave a pH of 4.1 when diluted 1 to 25. Measurements of pH were all made with a Beckman Model G meter.

**Color Reagent.**  $p$ -Nitrosodimethylaniline (D.P.I. No. 188)

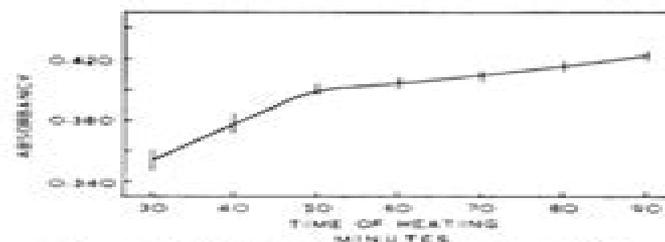


Figure 1. Effect of Time of Heating on Color Intensity

0.89  $\mu$ g. of ruthenium  
Klett-Summerson colorimeter with tube cells. Vertical lines represent ranges of results.

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