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Colorimetric Determination of Nickel in Uranium

Metal or Oxides

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A Colorimetric Method for Determination of Nickel in
Uranium Metal or Oxides*

R. I. Martens

Abstract

The colorimetric method of Murray and Ashley for the determination of nickel has been adapted to the determination of the element in uranium materials. A prior separation of uranium as the tetroxide is performed.

The red colored complex that oxidized nickel forms with dimethylglyoxime has been used for some time for the determination of small amounts of nickel. The first use as a method of determination was reported in 1926 by Rollet.(1) Later Jones applied the method to the determination of nickel in steel after a separation of iron by precipitation with ammonia in the presence of cyanide.(2) Murray and Ashley used the method without a separation by keeping the iron in solution with citric acid and measuring the absorption of the wine-red nickel dimethylglyoxime complex at 5300 Å. The alkaline iron citrate has practically no absorption above 5000 Å. (3) Uranium left in solution after the uranium tetroxide separation would be precipitated by ammonium hydroxide but it also forms a soluble complex with citric acid. It is colored a light yellow and interferes if the determination of transmittance is made at 420 millimicrons the wavelength of maximum absorption for the nickel complex. However, determinations of the transmittance of uranium citrate solutions showed practically 100% transmittance at 520 millimicrons, while the absorption of the nickel complex is still great at this wavelength. No difficulties

* Compiled by R. W. Bane

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were encountered due to interferences of other elements in the analysis of the relatively pure uranium metal or oxides. The presence of elements such as copper or manganese would be undesirable. Saturated bromine water is used as the oxidant. Other oxidants such as lead peroxide, ammonium persulfate and hypochlorite have been employed successfully elsewhere.

Procedure

A sample of uranium metal or oxide weighing 5 to 10 grams is dissolved in nitric acid (1-1). The peroxide separation is carried out as described in Chapter XIV, Analytical Chemistry of the Manhattan Project. The filtrate is evaporated to dryness and the residue taken up in a little water. It may be necessary to add a few drops of nitric acid and heat to effect complete solution of the residue. The entire solution or an aliquot of the same is transferred to a 50 ml volumetric flask. The following reagents are added in the order given and the flask shaken after each addition: 5 ml of 10% citric acid reagent, 2.5 ml of saturated bromine water, 2.5 ml of ammonium hydroxide (1:1), 1.5 ml of a 1% alcoholic solution of dimethylglyoxime. The solution is diluted to 50 ml with distilled water and the transmittance determined at 520 millimicrons. The readings should be completed within ten minutes since there is a tendency for the color to fade. The reference solution is prepared by taking the same amounts of citric acid, bromine water, ammonium hydroxide and dimethylglyoxime as the sample and diluting to 50 ml with water. The concentration of the nickel is read from

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a concentration - transmittance curve, Figure I. This curve was prepared from measurements on nickel sulfate solutions to which citric acid plus all reagents were added. The nickel content of the standard solution was determined gravimetrically with dimethylglyoxime. The sensitivity of the method can be increased by making the final dilution 25 ml using half quantities of the reagents, and/or employing longer light path cells.

Note: Haight has demonstrated that nickel can be determined in the presence of uranium by use of a reference of the same composition as the sample but without the dimethylglyoxime.(4) However, the peroxide separation can be used to advantage in the concentration of the nickel when very small amounts are present.

References

- (1) Rollet, A. P., Compt. rend., 183, 212 (1926).
- (2) Jones, B., Analyst 54, 585 (1929).
- (3) Murray, W. M. and Ashley, S.E.Q., Ind. Eng. Chem., Anal. Cd. 10, 1 (1938).
- (4) Haight G. P., Jr.; Princeton University: A-1033, Section 2-D May 31, (1944).

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FIG. 1. CONCENTRATION - TRANSMITTANCE CURVE, NICKEL-DIMETHYLGlyoxime.