WAPD-CTA(GLA)-146
Revised

THE DETERMINATION OF NIOBIUM IN URANIUM-NIOBIUM ALLOYS

T. C. Bryson
G. W. Goward
M. D. Hartmann

Analytical Chemistry Section

April 24, 1956

NOTE

This document is an interim memorandum prepared primarily for internal reference and does not represent a final expression of the opinion of Westinghouse. When this memorandum is distributed externally, it is with the express understanding that Westinghouse makes no representation as to completeness, accuracy, or usability of information contained therein.

WESTINGHOUSE - BETTIS PLANT
P.O. Box 1468
Pittsburgh 30, Pennsylvania

DISCLAIMER

THE DETERMINATION OF NIOBIUM IN URANIUM-NIOBIUM ALLOYS

1. Method

(a). Principle

Niobium is determined as the peroxy complex in concentrated sulfuric acid by photometric measurement at 360 millimicrons. Uranium interference is cancelled by using a solution of the sample for reference in the photometric measurement.

(b). Scope

The method covers the determination of niobium in uranium niobium alloys made from pure melting stock. Molybdenum interferes and must be removed if present. Zirconium does not interfere. The calibration curve covers a concentration range from 0.2 to 5.0 mg.

2. Apparatus

(a). Beckman Model B spectrophotometer or similar photometric instrument

(b). Platinum beakers, 250 ml.

3. Reagents

(a). Hydrofluoric Acid (48%)

(b). Nitric Acid (Spec. Gr. 1.42)

(c). Sulfuric Acid (Spec. Gr. 1.84)

(d). Sulfuric Acid (1:1)

(e). Hydrogen Peroxide (30%)

(f). Niobium Standard Solution (0.2 mg./ml.) - Dissolve exactly 0.100 gm. of pure niobium metal in a platinum beaker in 10 ml. of nitric acid by the dropwise addition of hydrofluoric acid (48%). Add 30 ml. of sulfuric acid (1:1) and take to heavy fumes. Rinse the sides of the beaker with water and refume. Transfer to a 500 ml. volumetric flask and add 140 ml. of sulfuric acid (1:1). Dilute to the mark with water.

4. Procedure

(a). Calibration Curve

Pipette 5, 10, 15, 20, and 25 ml. of the standard niobium solution into 125 ml. Erlenmeyer flasks. Fume by hand over an open flame until copious fumes pour from the neck of the flasks. Transfer the solutions to 50 ml. volumetric flasks which have been rinsed with concentrated sulfuric acid. Dilute to volume with concentrated sulfuric acid. Pipette 25.0 ml. to dry beakers and add 0.50 ml. of H_2O_2 (30%) to each. Stir well. Using the remaining solutions as reference in each case, determine the optical density of the standard solutions at 360 mu in 1 cm. cells. Plot optical density against mg. of niobium in 50 ml. of sulfuric acid.

(b). Analysis of Samples

Dissolve a 1.00 gm. sample in 10 to 20 ml. of concentrated nitric acid by the dropwise addition of hydrofluoric acid. Apply heat if necessary. Add 30 ml. of sulfuric (1:1) and take to fumes. Fume vigorously in an open beaker. Cool, rinse down the sides of the beaker with water and refume. Cool and transfer to a 500 ml. volumetric flask. Add 140 ml. of sulfuric acid (1:1) and dilute to the mark with water. Pipette a suitable aliquot into a 125 ml. Erlenmeyer flask.(Note 1)

Note 1:

% Nb in Alloy	Aliquot
0-5	25 ml.
5-10	15 ml.
10-15	10 ml.

Evaporate to fumes and fume by hand over an open flame until copious fumes pour from the neck of the flask. Transfer to a 50 ml. volumetric flask which has been rinsed with concentrated sulfuric acid. Dilute to the mark with concentrated sulfuric acid. Transfer 25 ml. to a dry beaker and add 0.50 ml. of hydrogen peroxide (30%). Stir well. Using the remainder of the solution for reference, determine the optical density of the sample solution at 360 mu in 1 cm. cells. Determine the mg. of niobium in the solution by reference to the previously prepared calibration curve.

5. Calculations

$$\% \text{ Nb} = \frac{A(50)}{BC}$$

A = Mg. of Nb found in aliquot taken

B = Aliquot taken for color development

C = Sample weight, gms.

T. C. Bryson
G. W. Goward
M. D. Hartmann

4-24-56
eap