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THE DETERMINATION OF NIOBIUM IN URANIUM NIOBIUM ALLOYS (1)

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For The Atomic Energy Commission

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NIOBIUM (1)

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1. Method

(a). Principle

The alloy is dissolved in hydrofluoric and nitric acids and the fluoride removed by fuming with sulfuric acid. The niobium is hydrolyzed in an acid-sulfur dioxide medium and converted to niobic oxide by ignition at 900°C.

(b). Scope

This method covers the determination of niobium in uranium-niobium alloys having a niobium content of 8 to 12 percent.

Any metal which hydrolyzes readily in dilute acid solution interferes.

2. Apparatus

- (a). Platinum beakers (250 ml. capacity).
- (b). Platinum crucibles (30 ml.).

3. Reagents

- (a). Nitric Acid (Sp. Gr. 1.42).
- (b). Hydrofluoric Acid (48%).
- (c). Sulfuric Acid (1:1).

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- (1) Adapted from Kriege, O. H., "The Gravimetric Determination of Niobium in Uranium-Niobium Alloys", Report LA-2049 (1956).
In this laboratory uranium-niobium alloys would not dissolve completely in the hydrochloric acid-peroxide mixture recommended by Kriege. Complete solution is obtained in a nitric-hydrofluoric acid mixture.

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- (d). Niobium Metal (99.9 percent pure).
- (e). Uranium Metal (99.9 percent pure).
- (f). Sulfurous Acid (saturated solution) - Pass SO_2 from an SO_2 cylinder into distilled water for 15-20 minutes.

4. Procedure (Note 1)

(a). Sample

From the total sample available obtain a representative sample containing 30 to 60 mg. of niobium. Transfer to a 250 ml. platinum beaker and dissolve the alloy in 10 ml. of HNO_3 with the dropwise addition of hydrofluoric acid. Add 10 ml. of 1:1 H_2SO_4 and take to fumes of SO_3 . Rinse the sides of the beaker and reflux. Cool and transfer the sample to a 400 ml. beaker with a saturated solution of SO_2 . Dilute to 200 ml. with a saturated solution of SO_2 and boil gently for 10 minutes. Add paper pulp, stir well and let stand 15 minutes. Filter the warm solution on a #40 Whatman 11 cm. filter paper. Wash the precipitate with 200 ml. of a hot saturated solution of SO_2 . Ignite to constant weight in a platinum crucible at 900°C and weigh as Nb_2O_5 .

(b). Control Standard

With every set of samples analyzed, carry through a control sample as follows. Prepare a synthetic alloy by weighing 30 to 60 mg. of niobium metal and an amount of uranium metal to give the same composition as the alloy being analyzed. Dissolve the mixture and analyze the sample in exactly the same manner as the alloy samples are analyzed. The results obtained on such standards serve to establish the precision and accuracy of the method as well as to maintain a control of the analytical procedure.

Note 1 - This analysis must be performed in a well ventilated hood.

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Calculations

$$\% \text{Nb} = \frac{A (69.594)}{B}$$

where: A = grams of niobic oxide

B = grams of sample

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