Determination of the minimum sample mass of $U_3Si_2$ to be used as candidate reference material for chemical analyses of total Uranium and total Silicide

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Objectives and Methodology

The objective is to perform a preliminary test with three mass sub samples with 0.5, 0.8 and 1.2g of U₃Si₂ to elucidate the minimum sample size to be used for repeatable candidate reference material here prepared.

**Determination of the total U by Potentiometric Titration method Davies & Gray**

- Dissolution sample in
- Filtration for removed the silicide
- Dry the aliquot
- Potentiometric Titration with potassium dichromate

**Determination of the total Si for method Gravimetric**

- Dry the filter paper with silicide
- First calcinations
- Weighing the crucible
- Solubilization by hydrofluoric acid
- Dry the residue
- Second calcinations
- Weighing the residue
Summary of Results

![Graphs of Relative Standard Deviation vs Range Mass for Total U and Total Si](image)

**FIG. 1. STANDARD DEVIATION IN RELATIVE WEIGHT FUNCTION FOR THE STUDY OF HOMOGENEITY IN THE VIAL TOTAL U DAVIES & GRAY TEST AND TOTAL SI ALL BY GRAVIMETRIC ASSAY**

It was observed that the average value of the deviation was equal to $0.16 \pm 0.04\%$ (total U) representing a variation of 0.05 to 0.28\% over the weight, and $0.35 \pm 0.7\%$ (total Si), ranging from 0.08 to 0.84\%.

For the masses 0.5 g, 0.8 and 1.2 g was found that the average value of the elements did not show statistically significant differences, however the individual results for the standard deviation higher dispersion. Thus 1.2 g mass selected as the minimum weight of the sample, since individual values are mutually compatible.