

Homogeneity Study of UO₂ Pellet Density for Quality Control

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1. Introduction

A homogeneity study has been performed with various densities of UO₂ pellets as the work of a quality control. The densities of the UO₂ pellets are distributed randomly due to several factors such as the milling conditions and sintering environments, etc. After sintering, total fourteen bottles were chosen for UO₂ density and each bottle had three samples. With these bottles, the between-bottle and within-bottle homogeneity were investigated via the analysis of the variance (ANOVA).[1] From the results of ANOVA, the calculated F-value is used to determine whether the distribution is accepted or rejected from the view of a homogeneity under a certain confidence level. All the homogeneity checks followed the International Standard Guide 35.[2]

2. Methods and Results

The reference material is divided into two categories: certified reference material (CRM) and quality control material (QCM). The CRM is defined as a reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unity in which the property values are expressed, and for which certified value is accompanied by an uncertainty at a stated level of confidence. A CRM is widely used for the calibration of an apparatus, for the assessment of a measurement method, or for assigning values to materials or objects. The QCM is newly defined as a standard reference material for a quality control, which is produced in the laboratory for internal use such as an internal quality control chart, comparison of results within laboratories or between laboratories, product release testing, instrument performance check, repeatability study and check samples. But a QCM should not be used to construct traceability to calibrate extra-lab instruments.

It has been attempted to make a standard nuclear fuel with a certain standard criteria such as the pellet density, grains size, etc. Thus, a standard nuclear fuel may be considered as QCM not CRM due to a lack of a traceability. Lots of UO₂ pellet samples are made and their densities are measured by a usual method.[3] The UO₂ densities (ρ_{UO_2}) are calculated by the difference between the weights of out of water (w_o) and in the water (w_i) based on Archimedes' principle.

$$F_b / g = w_o - w_i = V_{UO_2} \times \rho_w, \quad (1)$$

$$\rho_{UO_2} = m_{UO_2} / V_{UO_2} = m_{UO_2} \times \rho_w / (w_o - w_i), \quad (2)$$

where

F_b : buoyant force, g : gravity constant,

ρ_w : density of water,

V_{UO_2}, m_{UO_2} : volume and mass of UO₂ pellet, respectively.

It is necessary to test several items to check the reference material such as a homogeneity, stability, life time, and shelf time, etc. Among the several tests for QCM, the homogeneity test on the densities of the UO₂ pellets are performed in this study. Total 14 bottles are chosen, which had 3 samples as seen in Fig. 1. One-way analysis of variance (ANOVA) test was performed to verify the homogeneity with a 95% confidence level. Table I shows the results of the ANOVA test for the UO₂ pellet densities. From the results, F-value (1.1871) is less than the rejection value(2.0889), which means that the distribution is homogeneous enough within the 95% confidence.

To maintain the within-bottle homogeneity, the within-bottle standard deviation(s_{wb}) is usually estimated using the repeatability standard deviation(s_r)

$$s_r^2 = \sum_{i=1}^p \sum_{j=1}^n (Y_{ij} - \bar{Y}_i)^2 / (n-1)p, \quad (3)$$

where

Y_{ij} : density of sample unit j from bottle i,

\bar{Y}_i : average density of bottle i

n, p : numbers of samples and bottles, respectively.

It is not always feasible to perform a homogeneity study with a measurement method which is sufficiently repeatable. If u_{bb} denotes the uncertainty due to a batch inhomogeneity to be included in the model for a certification, it is estimated as follows

$$u_{bb}^2 = s_r^2 / n \times \sqrt{2/(n-1)p}. \quad (4)$$

And the uncertainty should be noted that

$$s_{bb}^2 \leq u_{bb}^2 \leq s_{bb}^2 + s_r^2 / n, \quad (5)$$

where

$$s_{bb}^2 = \sum_{i=1}^p (\bar{Y}_i - \bar{\bar{Y}})^2 / np - s_r^2 / n, \quad (6)$$

$$\bar{\bar{Y}} = \sum_{i=1}^p \sum_{j=1}^n Y_{ij} / np. \quad (7)$$

Inequality of Eq. (5) was satisfied in this test: u_{bb}^2 is calculated as 6.839E-04, s_{bb}^2 is obtained as 2.689E-04, and $s_{bb}^2 + s_r^2 / n$ is 1.706E-03.

From the above tests, the density of the UO₂ pellet has enough homogeneity under a 95% confidence level.

3. Conclusion

The homogeneity study for the densities of the UO₂ pellet was performed based on the ISO Guide 35. It was found that the UO₂ pellets were made homogeneously from the view of the density from the ANOVA test. Additionally, the uncertainty due to an inhomogeneity is estimated and it satisfied the inequality condition. To become a QCM, the UO₂ pellet should satisfy not only the homogeneity but also the stability and other criteria. It is expected that this homogeneity test could be applied to produce stable experimental data for various engineering area.

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REFERENCES

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- [2] *Certification of Reference Materials – General and Statistical Principles*, ISO GUIDE 35, 2001.
- [3] K.H. Kang, et.al., “The Effect of the Temperature and the Density on the Oxidation Behavior of UO₂ in Air At 300-600 °C”, *Korea Nuclear Society Spring Meeting*, Cheju, May, 2005.

Figure 1. Density distribution of UO₂ pellets.

TABLE I. One-way ANOVA Results for Homogeneity Test of UO₂ Pellet Density

Factor	Process	Residue	Total Sum
Square Sum	0.06653	0.1207	0.18723
Degree of Freedom	13	28	41
Square Mean	0.005117	0.004311	
F-ratio	1.1871		
F-rejection	2.0889		

* F-ratio < F-rejection

