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Nuclear fuel technology — Uranium dioxide powder and pellets — Determination of uranium and oxygen/uranium ratio by gravimetric method with impurity correction

Technologie du combustible nucléaire — Dioxyde d'uranium en poudres et en pastilles — Détermination de la teneur en uranium et du rapport oxygène/uranium en utilisant la méthode gravimétrique avec correction des impuretés

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Foreword

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ISO 12795 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology.*

INTERNATIONAL STANDARD **ISO 12795:2004(E)**

Nuclear fuel technology — Uranium dioxide powder and pellets — Determination of uranium and oxygen/uranium ratio by gravimetric method with impurity correction

1 Scope

This International Standard specifies a method of determining the mass fraction of uranium and the oxygen-to-uranium atomic ratio in hyperstoichiometric uranium dioxide (UO_{2+X}) powders and pellets.

This International Standard is used for the determination of the U mass fraction and the O/U-ratio of nuclear grade uranium dioxide. The precision and accuracy stated in 8.2 are valid, if the total amount of impurities in the sample does not exceed the figures given in 8.2.

An impurity correction in the U mass fraction and the O/U-ratio determination should be performed, if the amount of total impurities in oxide form exceeds 1 500 µg per gram of sample. Lower impurity levels influence the O/U-ratio by less than 0,000 5 and can be neglected. The non-volatile impurities shall be determined by an appropriate technique, and the correction applied. If the total content of the non-volatile impurities in oxide form is greater than 1 500 µg per gram of sample, the overall precision of the method depends on the accuracy of these impurity measurements.

If no impurity correction is made and if the enrichment of the sample is not considered as described in 8.11, the precision and accuracy figures will be poorer than in 8.2.

2 Principle

For stoichiometric uranium dioxide, the oxygen to uranium ratio is expressed by the atomic (O/U) ratio and is therefore 2,000.

In this method, a weighed sample of uranium dioxide powder or pellet is oxidised by heating in air under closely controlled temperature conditions. The final product is cooled down to room temperature and weighed.

Under the conditions described, a stoichiometric U_3O_8 is produced. This shall be verified experimentally by a precise chemical uranium determination method on the final product of the ignition (Bibliography [1], [2], [3]) or using an appropriate reference material. The verification applies to the material analysed.

This does not mean that the U_3O_8 from every determination of O/U-ratio has to be analysed chemically. If the muffle furnace temperatures are checked on a regular basis, the verification is only required when there is a change in the type of sample material analysed.

3 Reactions

$$3\ UO_{2+X} + (1 - 1{,}5\ X)\ O_2 \rightarrow U_3O_8$$

4 Reagents

4.1 Dilute nitric acid (HNO_3), 1:1 solution, mix equal volumes of concentrated nitric acid [$c(HNO_3) = 14$ mol/l, $\rho(HNO_3) = 1{,}40$ g/ml] and deionised water (see ISO 3696).

5 Apparatus

5.1 **Muffle furnace 1**, controlled up to 550 °C.

5.2 **Muffle furnace 2**, controlled up to 950 °C.

5.3 **Platinum crucibles**, of capacity 10 cm^3 to 20 cm^3.

5.4 **Desiccator**, with desiccant.

5.5 **Analytical balance**, capable of weighing to the nearest 0,1 mg or better.

6 Sampling and samples

A representative laboratory sample shall be taken from the batch to be analysed. Precautions shall be taken to prevent oxidation of the sample during the sampling procedure, if necessary. These precautions may be purging with nitrogen gas during sampling, storing the sample under nitrogen in a gastight sample bottle, etc. These precautions shall be established by the analytical laboratory. They primarily depend on the reactivity of the uranium dioxide in air and the accuracy required in the U- and O/U-determination.

7 Procedures

7.1 Calibration

7.1.1 Temperature profile

The temperature profile of the muffle furnaces shall be checked periodically using thermocouples.

7.1.2 Reference samples

The performance of this International Standard shall be verified with reference samples of known U mass fraction (e.g. CBNM EC standard No. 110).

7.2 Determination

7.2.1 Cleaning the crucible

Ignite an empty crucible in air in the muffle furnace (5.2) at about 900 °C.

7.2.2 Cooling the crucible

Remove the empty crucible from the furnace and place it in a desiccator. The cooling time shall not exceed 45 min. The hot crucibles should be handled with tongs.

7.2.3 Weighing the crucible

Weigh the empty crucible, to the nearest 0,5 mg or better.

7.2.4 Sample mass

Add at least 8 g of the powder or at least 5 g of pellet sample and weigh to the nearest 0,5 mg or better. Record the net mass m_1.

7.2.5 Pre-ignition

For pellet samples, ignite the crucible plus sample in air in the muffle furnace (5.1) at 500 °C ± 50 °C at least for 2 h (for powder samples omit this step, a pre-ignition is not required).

7.2.6 Ignition

Ignite the crucible plus sample in air in the muffle furnace (5.2) at 900 °C ± 50 °C for 3 h.

7.2.7 Cooling the sample

Remove the crucible from the muffle furnace and place it in a desiccator. The cooling time shall not exceed 20 min.

7.2.8 Final mass

Weigh the crucible to the nearest 0,5 mg or better. Repeat 7.2.6 and 7.2.7, until constant weight of sample and crucible is attained within the precision of the measurement. Record the net mass m_2.

7.2.9 Disposal

Dispose of the U_3O_8 in an appropriate container.

7.2.10 Cleaning

Clean crucibles by heating in dilute nitric acid (4.1) near boiling, until the U_3O_8 residue has dissolved. Wash thoroughly with deionised water.

8 Expression of results

8.1 Method of calculation and formulae

Calculate the mass fraction of uranium and the O/U-ratio with impurity correction.

8.1.1 Mass fraction of uranium

$$w_{\mathrm{U}} = 100 F_{\mathrm{S}} \left(\frac{m_2 - m_2 w_{\mathrm{I}}}{m_1} \right) - C$$

where

w_{U} is the mass fraction of uranium, in percent;

m_1 is the original sample mass, in grams;

m_2 is the sample mass, in grams, after oxidation;

w_{I} is the mass fraction of all non-volatile impurities in oxide form in U_3O_8;

F_{S} is the stoichiometric factor for the conversion of grams of U_3O_8 to grams of U. F_{S} has to be calculated for each enrichment as described in 8.1.1.1 and verified by a precise U determination method. For natural uranium, F_{S} is 0,848 0;

C is the correction for the mass fraction of non-volatile element impurities, which are not analysed, in percent. A typical mass fraction value of C is 0,003 % of the uranium content. This figure has to be established for each type of UO_2-powder and pellet fabrication process.

8.1.1.1 Calculation of the stoichiometric factor, F_S

$$F_S = \frac{3A_U}{3A_U + 8A_O}$$

where

A_U is the relative atomic mass of uranium;

A_O is the relative atomic mass of oxygen = 15,999 4;

A_U is calculated according to the formula:

$$A_U = \frac{100}{\sum_N \frac{w_{U\text{-}N}}{A_{U\text{-}N}}}$$

where

$w_{U\text{-}N}$ is the mass fraction, in percent, of the isotope N;

$A_{U\text{-}N}$ is the relative atomic mass of the isotope N;

N = uranium isotopes (234, 235, 236, 238).

Isotope N	Relative atomic mass $A_{U\text{-}N}$
234	234,040 9
235	235,043 9
236	236,045 6
238	238,050 8

Uranium isotopes other than ^{235}U and ^{238}U are neglected in non-reprocessed uranium fuel, because they do not contribute significantly to the relative atomic mass of U.

8.1.1.2 Calculation of impurity mass fraction

$$w_I = \Sigma\, c_i \times g_i \times 10^{-6}$$

where

w_I is the impurity mass fraction, in percent;

c_i is the concentration of element i, in micrograms per gram of U_3O_8;

g_i is the gravimetric conversion factor in Table 1.

8.1.2 O/U-ratio

$$\text{O/U} = \frac{(100{,}000 - w_{\text{U}} - w_{\text{V}} - w_{\text{H}_2\text{O}} - w_{\text{M}})A_{\text{U}}}{A_{\text{O}} \times w_{\text{U}}}$$

where

w_{U} is as defined in 8.1.1;

A_{U} and A_{O} are as defined in 8.1.1.1;

w_{V} is the mass fraction of volatile compounds, in percent;

$w_{\text{H}_2\text{O}}$ is the mass fraction of moisture in the initial sample, in percent;

w_{M} is the mass fraction of the sum of the metallic impurities in oxide form, in percent.

Table 1 — Gravimetric conversion factors for non-volatile impurities

Impurity	Probable form of impurity	Conversion factor g_i
Ag	Ag	1,00
Al	Al_2O_3	1,89
Am	AmO_2	1,13
B	B_2O_3	3,22
Ba	BaO	1,12
Be	BeO	2,78
Bi	Bi_2O_3	1,11
Ca	CaO	1,40
Cd	Cd	1,00
Co	CoO	1,27
Cr	Cr_2O_3	1,46
Cu	Cu	1,00
Fe	Fe_3O_4	1,38
K	K_2O	1,21
Mg	MgO	1,66
Mn	Mn_3O_4	1,39
Na	Na_2O	1,35
Ni	Ni_2O_3	1,40
P	P_2O_5	2,29
Pb	PbO	1,07
Rare earth elements	M_2O_3	1,16
Sb	Sb_2O_3	1,20
Si	SiO_2	2,14
Sn	SnO	1,13
Ta	Ta_2O_5	1,22
Th	ThO_2	1,14
Ti	TiO_2	1,67
V	V_2O_5	1,78
W	WO_3	1,26
Zn	ZnO	1,24
Zr	ZrO_2	1,35

8.2 Precision and accuracy

8.2.1 Uranium

For UO_2-powder with a maximum impurity level of up to 4 000 µg of impurities per gram of ignited material. and for UO_2-pellets with a maximum impurity level of up to 300 µg of impurities per gram of ignited material, the precision is ± 0,02 % in mass fraction of U absolute, the accuracy is ± 0,03 % in mass fraction of U absolute, both figures are valid at 2 sigma level.

8.2.2 O/U-ratio

For UO_2-powders with O/U-ratios of 2,06 to 2,15, the precision was found to be ± 0,005 U absolute at 2 sigma level. The accuracy is $\leqslant \pm 0{,}008$ absolute at 2 sigma level.

For UO_2-pellets with total non-volatile impurities $\leqslant$ 300 µg of impurities per gram of ignited material, the precision was found to be ± 0,002 6 absolute and the accuracy was determined to be ± 0,003 absolute at 2 sigma level.

9 Test report

The test report shall include the following information:

a) identification of sample;

b) the method used by reference to this International Standard;

c) the results and the form in which they are expressed;

d) any unusual features noted during the test;

e) any operations not included in this International Standard, or regarded as optional.

Bibliography

[1] Vita, O.A., Trivisonno, C.F., Walker, C.R. *An Improved Titrimetric Method for the Precise Determination of Uranium*, Goodyear Atomic Corp., Portsmouth, Ohio 1962 (GAT-471)

[2] Petit, G.S. and Kleinberger, C.A. *Preparation of Stoichiometric U308*, USAEC Document K-1424, AERDB, 1960

[3] ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

[4] ISO 9989, *Determination of uranium in uranium dioxide powder and pellets — Iron (II) sulfate reduction/potassium dichromate oxidation titrimetric method*

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