INTERNATIONAL STANDARD

ISO 21484

Second edition
2017-01

Nuclear Energy — Fuel technology — Determination of the O/M ratio in MOX pellets by the gravimetric method

Énergie nucléaire — Technologie du combustible — Détermination du rapport O/M dans les pastilles MOX par la méthode gravimétrique

Reference number
ISO 21484:2017(E)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection,* Subcommittee SC 5, *Nuclear installations, processes and technologies*.

This second edition cancels and replaces the first edition (ISO 21484:2008), which has been technically revised.

Nuclear Energy — Fuel technology — Determination of the O/M ratio in MOX pellets by the gravimetric method

1 Scope

This document describes a method for determining the Oxygen-to-Metal (O/M) ratio in mixed uranium-plutonium oxide $(U,Pu)O_{2 \pm X}$ pellets. The parameters given in the following paragraphs are relevant for pellets within a range of O/M ratio corresponding to 1,98 to 2,01. The method described in the document is adapted, with regard to the parameters, if the expected values of O/M ratio are outside the range.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The $(U,Pu)O_{2 \pm X}$ sample is submitted to controlled oxidation-reduction under thermodynamic conditions designed to change the O/M ratio to a value of 2,000. The initial stoichiometric deviation, X, is determined from the sample mass difference before and after heat treatment.

5 Reactions

The main reactions are as follows:

a) $(U,Pu)O_{2 \pm X} \mp {}^{X}/_{2}\ O_2 \rightarrow (U,Pu)O_{2,000}$;

b) $(U,Pu)O_{2+x} + xH_2 \rightarrow (U,Pu)O_{2,000} + xH_2O$.

6 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

6.1 Nitric acid solution, 50 % per volume nitric acid aqueous solution.

6.2 Purge gas.

6.2.1 Air, a volume fraction of 99,99 % purity grade is recommended.

6.2.2 Inert gas, such as Argon or nitrogen can be used with a recommended volume fraction of 99,99 % purity grade [O_2 < 0,000 5 % (5 ppm), H_2O < 0,000 5 % (5 ppm), N_2 < 0,000 5 % (5 ppm)].

6.2.3 Hydrogen or mixture of hydrogen and inert gas, a volume fraction of 99,99 % purity grade to which water vapour may be added to obtain an oxygen potential (ΔG_O) near –420 kJ/mol (–100 kcal/mol).

7 Apparatus

7.1 Furnace, one of the following furnaces can be used.

7.1.1 Muffle furnace, controlled up to 950 °C, having a device for measuring the temperature and having sweeping possibility with various gases.

7.1.2 Other furnaces, other furnaces, with same performance can also be used such as Infrared gold image furnace (IR lamp heating).

7.2 Crucibles, use platinum or quartz crucibles.

7.3 Analytical balance, use an analytical balance with an accuracy of at least ±0,1 mg when muffle furnace is used or at least ±0,01 mg if samples are smaller.

8 Sampling

A representative sample shall be taken from the pellet batch for analysis. If necessary, crush pellets to make smaller samples according to the furnace type or the crucible size.

If necessary, care shall be taken to avoid sample oxidation during the sampling procedure and the sample preparation. Typical precautions include performing the sampling operation under argon atmosphere, placing the sample in an argon-filled bottle, etc. The laboratory performing the analyses shall establish the procedure.

9 Procedure

9.1 Preliminary test

The balance shall be checked at regular intervals using a standard mass.

9.2 Preparing the crucibles

The crucibles shall be clean and weighed before use. An example of a cleaning procedure that could be used is as follows.

a) Clean the crucibles by heating them in nitric acid (6.1) near the boiling point.

b) Rinse thoroughly with deionized water.

c) Dry the crucibles in the furnace for 30 min at 150 °C under inert atmosphere.

d) Allow the crucibles to cool to 35 °C in the furnace under inert atmosphere.

e) Remove each crucible from the furnace and weigh it to within ±0,1 mg or better.

f) Record the crucible mass, m_0, in grams.

9.3 Weighing the sample

9.3.1 Place the sample, sampled as indicated in Clause 8, in an empty crucible.

9.3.2 Weigh the crucible containing the pellets to within ±0,1 mg or better.

9.3.3 Record the mass, m_1, in grams of the crucible containing the pellets.

9.4 Heat treatment cycles

9.4.1 General

The sample shall be heat treated in such a way that the O/M ratio is changed to exactly 2,000. The duration of the oxidation-reduction cycles and the gas flow rates in the furnace shall be optimized according to the nature of the furnace used, the number of sample pellets measured, the pellet composition, etc.

Load the furnace with the crucible containing the sample pellets. Apply the desired heat treatment cycle.

9.4.2 Example 1

9.4.2.1 Under argon sweeping, raise the temperature to 900 °C ± 30 °C and hold for 5 min.

9.4.2.2 Under air sweeping, maintain the furnace temperature for 1 min to 7 min at 900 °C ± 30 °C.

9.4.2.3 Under argon sweeping, maintain the furnace temperature for 5 min at 900 °C ± 30°C.

9.4.2.4 Under argon +5 % to 7 % H_2 sweeping, maintain the furnace temperature for 8 h to 13 h at 900 °C ± 30 °C.

9.4.2.5 Under argon +5 % to 7 % H_2 sweeping, shut off the furnace heating and allow the temperature to drop to 35 °C.

9.4.3 Example 2

9.4.3.1 Heat the samples for 16 h at 800 °C, in an atmosphere comprising a volume fraction of 4 % H_2 in argon saturated with water at room temperature.

9.4.3.2 Cool under dry argon containing a volume fraction of 4 % H_2.

9.5 Final weighing

9.5.1 Remove the crucible from the furnace.

9.5.2 Weigh the crucible to within 0,1 mg or better.

9.5.3 Record the mass, m_2, in grams of the crucible after oxidation-reduction heat treatment.

10 Calculation

10.1 Mean atomic mass of oxide after heat treatment

Calculate the mean atomic mass of the oxide after treatment, when O/M = 2,000 exactly, by using Formula (1):

$$m_a = \frac{m(\mathrm{Pu})\cdot x_{\mathrm{Pu}} + m(\mathrm{U})\cdot x_{\mathrm{U}} + m(\mathrm{Am})\cdot x_{\mathrm{Am}}}{x_{\mathrm{Pu}} + x_{\mathrm{U}} + x_{\mathrm{Am}}} + 2m(\mathrm{O}) \quad (1)$$

where

m_a is the mean atomic mass of the oxide of heavy metals;

m(Pu) is the mean atomic mass of plutonium in the oxide;

m(U) is the mean atomic mass of uranium in the oxide;

m(Am) is the mean atomic mass of americium in the oxide;

x_{Pu} is the mole fraction in percent of plutonium in the oxide;

x_U is the mole fraction in percent of uranium in the oxide;

x_{Am} is the mole fraction in percent of americium in the oxide;

m(O) is the atomic mass of oxygen (15,999 4);

$x_{Pu} + x_U + x_{Am} = 100$ %.

10.2 Calculation of O/M ratio

Calculate the O/M ratio by using Formula (2):

$$\mathrm{O/M} = 2,000 - \frac{m_a \times (m_2 - m_1)}{m(\mathrm{O}) \times (m_2 - m_0)} \quad (2)$$

where

m_0 is the mass of empty crucible, in g;

m_1 is the mass of crucible with test sample before oxidation-reduction, in g;

m_2 is the mass of crucible with test sample after oxidation-reduction heat treatment, in g;

m(O) is the atomic mass of oxygen (15,999 4);

m_a is the mean atomic mass of the oxide of heavy metals.

11 Precision

11.1 Accuracy

30 determinations on stoichiometric mixed oxide pellets containing approximately 7 % plutonium gave a mean O/M ratio of 2,000.

11.2 Precision

The standard deviation calculated from 30 determinations by two operators on a control sample of stoichiometric mixed oxide pellets containing approximately 7 % plutonium is better than 0,001.

11.3 Sensitivity

Typically for a 15 g sample a change in weight of 0,5 mg results in a change in O/M ratio of 0,001.

12 Test report

The test report shall contain the following information:

a) all information necessary for identification of the sample tested;

b) a reference to this document, i.e ISO 21484:2017;

c) the method used;

d) the results of the test, including the results of the individual determinations and their mean, calculated as specified in Clause 10;

e) any deviations from the procedure specified;

f) any unusual features (anomalies) observed during the test;

g) the date of the test.