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Determination of carbon content of UO_2, $(U, Gd)O_2$ and $(U, Pu)O_2$ powders and sintered pellets — Combustion in a high-frequency induction furnace — Infrared absorption spectrometry

Détermination de la teneur en carbone des poudres et des pastilles frittées d'UO_2, $(U, Gd)O_2$ et $(U, Pu)O_2$ — Combustion dans un four à induction haute fréquence — Spectrométrie d'absorption infrarouge

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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Determination of carbon content of UO_2, (U, Gd)O_2 and (U, Pu)O_2 powders and sintered pellets — Combustion in a high-frequency induction furnace — Infrared absorption spectrometry

1 Scope

This International Standard describes a method for determining the carbon content in UO_2, (U,Gd)O_2 and (U,Pu)O_2 powder and sintered pellets by combustion in an induction furnace and infrared absorption spectroscopy measurement.

It is applicable for determining 10 µg/g to 500 µg/g of carbon in UO_2, (U,Gd)O_2 and (U,Pu)O_2 powder and pellets.

2 Principle

The sample is heated to a temperature above 1 500 °C in an induction furnace, under pure oxygen atmosphere, to convert any carbon compounds to carbon dioxide gas. The resulting carbon dioxide gas is filtered and dried before measurement using infrared spectroscopy to measure the carbon dioxide signal at 2 350 cm^{-1}. The result is converted into the carbon content of the material analysed.

3 Reagents and materials

3.1 Oxygen (O_2), with a volume fraction of 99,999 % purity grade.

3.2 Sodium hydroxide (NaOH), of analytical grade.

3.3 Magnesium perchlorate $(MgClO_4)_2$, of analytical grade.

3.4 Tungsten (W).

The results in this International Standard were achieved through the use of tungsten as the catalyst. The use of the other materials, such as granular tin, iron, copper, and copper oxide, would need to be validated on the equipment.

3.5 Alumina crucibles, suitable for use in an induction furnace.

3.6 Aluminium foil, used to protect alumina crucibles from pollution after calcining.

3.7 Reference materials.

The following are appropriate reference materials:

— “OPERA” RM (uranium with certified C content) from CETAMA;

— EURONORM-CRM 295-1;

— SRM Steel.

4 Apparatus

4.1 Muffle furnace, capable of maintaining a temperature of (1 200 ± 50) °C.

4.2 Desiccator, of glass; containing silica gel.

4.3 Analytical balance, with an accuracy of at least ± 0,1 mg.

4.4 Crusher, made of bronze or stainless steel.

4.5 Carbon analyser, complying with containment requirements for radioactive materials (glove-box installation). The analyser comprises the following.

4.5.1 Induction furnace, coupled with a high-frequency generator and capable of being opened to insert the crucible whilst maintaining oxygen flow to prevent outside air from entering the furnace. The temperature obtained shall not be less than 1 500 °C.

4.5.2 Tungsten catalyst (3.4), which is analytical grade (carbon-free), placed in the crucible located within the induction coil to ensure efficient coupling.

4.5.3 Oxygen gas supply system.

The oxygen pressure is reduced to a pressure between 150 kPa and 180 kPa, purified and filtered before entering the furnace. The oxygen supply shall be purified (3.1) before entering the furnace using a sodium hydroxide trap (3.2) to remove any carbon dioxide. The oxygen and CO_2 leaving the furnace shall be purified through a magnesium perchlorate trap (3.3) before passing through into the measurement cell.

4.5.4 Measuring system, comprising an infrared source, a measurement chamber, a reference chamber, and a detector, coupled with measurement, amplification and signal integration electronics.

5 Test sample

5.1 Powder

No preparation is necessary.

5.2 Pellets

The pellets shall be broken up using the crusher (4.4), taking care not to produce excessive quantities of fine particles.

6 Procedure

6.1 Crucible preparation

The alumina crucibles (3.5) shall first be calcined in a suitable muffle furnace (4.1) for 8 h at (1 000 ± 50) °C.

Remove from the furnace and place in a desiccator (4.2) to cool down in a moisture free atmosphere. After cooling, the crucibles shall be handled with tongs.

Set aside the number of crucibles necessary for one day of analyses. Store the unused crucibles in the desiccator after protecting them with aluminium foil.