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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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## Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21614 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

# 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.

### 6.2 Startup

The analyser (4.5) shall be cleaned and the sodium hydroxide (3.2) and magnesium perchlorate (3.3) reagents renewed in the traps before each analysis.

Switch the unit on and open the gas valve to allow the oxygen (3.1) to flow through the furnace.

Carry out the recommended self-test routines and settings.

Wait 10 min before proceeding with any measurements.

### 6.3 Blank verification/determination

#### 6.3.1 Blank verification

Take a calcined crucible (3.5). Add 1,5 g of tungsten (3.4) using the measurement spoon.

Place the crucible in the induction furnace (4.5.1) and perform the measurement.

The value obtained shall correspond to the acceptance criteria specified for the blank determination.

If not, readjust the blank setting (see 6.3.2).

#### 6.3.2 Blank determination

Perform three measurements as described in 6.3.1.



Based on the three determinations, adjust the analyser (4.5) zero setting and calculate the acceptance criteria for the blank verification.



### 6.4 Checking/Calibration

#### 6.4.1 Calibration check

Take a calcined crucible (3.5).

Accurately weigh out 1 g of CRM (3.7) to the nearest 0,1 g using a calibrated balance (4.3).

Add 1,5 g of tungsten (3.4) using a suitable measurement spoon.

Place the crucible in the induction furnace (4.5.1) and perform the measurement.

The value obtained shall correspond to the acceptance criteria specified for the calibration check.

If not, recalibrate the system (see 6.4.2).

#### 6.4.2 Calibration

Perform three measurements as described in 6.4.1.

Based on the three determinations, calibrate the analyser (4.5) and calculate the acceptance criteria for the calibration check.

On completion of this procedure, perform a blank verification with the analyser.

### 6.5 Analysis

Take a calcined crucible (3.5). Weigh out about 1 g of the test sample to the nearest 0,1 g using the calibrated balance.

Add 1,5 g of tungsten (3.4) using the measurement spoon.

Place the crucible in the induction furnace (4.5.1) and perform the measurement.

At the end of the series of sample measurements perform a blank verification and a calibration check.

NOTE The number of tests per sample is determined by the required measurement precision.

## 7 Reporting the results

### 7.1 Calculations.

The final result is the expression of the measured value or the mean value of the measurements performed on the sample. The carbon content as mass fraction of the oxide shall be expressed in micrograms per gram (µg/g).

If the carbon concentration is requested as mass fraction of the metal, the results shall be corrected for the stoichiometric ratio coefficient of the samples.

### 7.2 Performance.



The detection limit is 10 µg.

The accuracy is:



— approximately –2 % for a measured value of 58 µg,

— approximately –1 % for a measured value of 166 µg.

The typical coefficient of variation for the technique is

— 40 % (1 s) at 58 µg of total carbon,

— 6 % (1 s) at 166 µg of total carbon.

## 8 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 International Standard, i.e. ISO 21484:2008;

c) the method used;

d) the results of the test, including the results of the individual determinations and their mean;

e) any deviations from the procedure specified;

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

g) the date of the test.