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# Nuclear energy — Determination of nitrogen content in $UO_2$, $(U,Gd)O_2$ and $(U,Pu)O_2$ sintered pellets — Inert gas extraction and conductivity detection method

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This British Standard is the UK implementation of ISO 12799:2015.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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# Nuclear energy — Determination of nitrogen content in $UO_2$, $(U,Gd)O_2$ and $(U,Pu)O_2$ sintered pellets — Inert gas extraction and conductivity detection method

*Énergie nucléaire — Dosage de la teneur en azote des pastilles frittées d'$UO_2$, $(U,Gd)O_2$ et $(U,Pu)O_2$ — Méthode d'extraction par gaz inerte et méthode de mesurage de la conductivité*

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# Foreword

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The committee responsible for this document is ISO/TC 85, *Nuclear Energy*, Subcommittee SC 5, *Fuel Technology*.

# Nuclear energy — Determination of nitrogen content in $UO_2$, $(U,Gd)O_2$ and $(U,Pu)O_2$ sintered pellets — Inert gas extraction and conductivity detection method

## 1 Scope

This International Standard describes a procedure for measuring the nitrogen content of $UO_2$, $(U,Gd)O_2$, and $(U,Pu)O_2$ pellets. Nitrogen in nuclear fuel may be present either as elemental nitrogen or chemically combined in the form of nitrogenous compounds. The technique described herein serves to determine the total content of nitrogen excluding those compounds whose decomposition temperature is above 2 200 °C (most notably Pu and U nitrides).

## 2 Principle

For determining the nitrogen content of $UO_2$, $(U,Gd)O_2$, or $(U,Pu)O_2$ pellets, an analyser is employed which operates according to the carrier gas principle, using helium as the carrier gas, the nitrogen content being determined in a thermal conductivity cell.

The weighed samples to be analysed are heated in a degassed high purity graphite crucible at a temperature of more than 1 770 °C in a helium atmosphere. This high temperature destroys the majority of the nitrogen bearing compounds and causes nitrogen to be released along with other gaseous components like CO, $CO_2$, and $H_2$. The released gases are then swept by the carrier gas through oxidation and absorption columns to trap interfering species. The nitrogen passes through without being retained, and its quantity is subsequently measured in the thermal conductivity cell.

## 3 Interferences

The samples will not be heated to temperatures above 2 200 °C since this would cause a reaction to take place between the $UO_2$, $(U,Pu)O_2$, or $(U,Gd)O_2$ pellet and the graphite, resulting in large quantities of $CO_2$ gas being released. These large quantities of gas can be not completely trapped and would lead to errors in conductivity measurement.

## 4 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade. The reagents and materials below serve as examples to be used according to manufacturer’s recommendation.

### 4.1 Helium.

Use helium as carrier gas with a purity of a volume fraction ≥99,995 %.

### 4.2 Nitrogen.

If nitrogen is used as calibration gas it will be of a purity ≥99,998 % in volume fraction.

### 4.3 Reference material

A certified reference material (CRM) can be used as calibration media with certified nitrogen content (e.g. metal standards).

NOTE Reference materials are available for example from National Institute of Standard and Technology (NIST) or Commission d'Établissement des Méthodes d'Analyse (CETAMA).

### 4.4 Copper(II) oxide.

CuO purifies He/$N_2$; converting $H_2$ to $H_2O$ and CO to $CO_2$.

### 4.5 Absorption media for $CO_2$.

Ascarite (sodium hydroxide over clay) or equivalent absorbs the $CO_2$.

### 4.6 Absorption media for $H_2O$.

Anhydrone [$Mg(ClO_4)_2$] traps any $H_2O$ formed. A molecular sieve can also be used.

### 4.7 Flux/melting accelerator.

Tin, copper, or nickel granules are used to flux the melting of the sample.

## 5 Apparatus

**5.1 Nitrogen analyser**, apparatus fitted with an appropriate furnace with controllable temperature and with the analytical cell, suitably adapted for handling plutonium bearing samples where relevant.

**5.2 Graphite crucibles**, suitable for the appropriate sample types.

The impurity content should not exceed 2 µg/g.

**5.3 Balance**, with an accuracy of ≤1 mg.

## 6 Sampling

### 6.1 Sampling procedure

When sampling the fuel pellets, ensure that pellets are not contaminated; use metal forceps or pincers for sampling pellets.

### 6.2 Sample preparation

The fuel pellets are crushed in a percussion mortar. The fragments used for the analysis should be larger than 1 mm. The desired fragment size could be obtained by passing the crushed pellet through an appropriate sieve. The size of the resulting sample should be 1,0 g to 1,5 g.

## 7 Procedure

Prior to use, the nitrogen analyser will be checked to ensure that it is leak tight. Checking involves visual inspection of key parts that are prone to leak such as furnace O-ring and gas supply tubes. The thermocouple will be regularly checked (calibration).

## 7.1 Calibration

### 7.1.1 Blank test

To verify the functional capability of the analyser, blank values are determined to obtain information on the condition of the upstream gas purification columns and on the leak tightness of the analyser. Blank values exceeding the analyser detection limit require adequate measures of correction.

### 7.1.2 Calibration of the analyser

The nitrogen analyser shall be calibrated before sample measurement. There are two possibilities of calibration:

- calibration with gas;
- calibration with certified standards.

In case of calibration with gas, depending on the type of analyser, it can be performed by the analyser manufacturer or/and the final user. A well-defined volume of the calibration gas (4.2), which is corrected on standard conditions, is injected and analysed. This calibration is performed three times.

In the case of calibration with certified standards, they are weighed to the nearest 1mg accuracy.

### 7.1.3 Check of the calibration using metal standard

To check the calibration of the equipment, standard (4.3) having certified nitrogen content is weighed to the nearest 1 mg and analysed. The quantity of the released nitrogen is measured. The result may differ from the certified values by not more than the total uncertainty of the analysis process.

If it is not the case the calibration should be repeated.

## 7.2 Determination

a) Prior to loading of the sample in the graphite crucible, heat the latter first to around 2 200 °C for degassing.

b) Weigh the sample to the nearest 1 mg.

c) Insert sample and flux/melting accelerator (4.7) into the analyser.

d) Purge with carrier gas.

e) Fill the crucible with the sample and heat it up to ≥1 770 °C.

f) Measure the nitrogen content.

## 8 Calculation

Calculate the nitrogen mass fraction (µg/g sample) from the following formula:

$$w_{\mathrm{N2}} = \frac{m_{\mathrm{N2}}}{m}$$

where

$w_{\mathrm{N2}}$ is the nitrogen mass fraction in µg/g sample;

$m_{\mathrm{N2}}$ is the nitrogen mass in µg;

$m$ is the mass of sample in g;

## 9 Precision

For samples containing one gram to two grams and having a nitrogen content of 10 µg/g, a repeatability standard deviation of 20 % (relative) can be expected.

For stainless steel standards having a nitrogen content of 10 µg/g and 2 µg/g, standard deviations of 15 % and 66 % (relative), respectively, have been achieved

## 10 Test report

The test report shall include the following information:

a) identification of sample;

b) the method used by reference to this International Standard (i.e. ISO 12799);

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

d) any deviations from the procedure;

e) any unusual features observed;

f) location and date of the test;

g) detection limit.

# Bibliography

[1] ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

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Price based on 5 pages

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