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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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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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.