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Nuclear energy — Determination of total hydrogen content in PuO_2 and UO_2 powders and 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 totale en hydrogène de poudres de PuO_2 et UO_2, et de pastilles frittées d'UO_2, $(U,Gd)O_2$ et $(U,Pu)O_2$ — Méthode d'extraction par gaz inerte et méthode de mesure de la conductivité

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

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Nuclear energy — Determination of total hydrogen content in PuO_2 and UO_2 powders and 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 total hydrogen content of UO_2 and PuO_2 powders (up to 2 000 µg/g oxide) and of UO_2 and $(U,Gd)O_2$ and $(U,Pu)O_2$ pellets (up to 10 µg/g oxide). The total hydrogen content results from adsorbed water, water of crystallization, hydrocarbon, and other hydrogenated compounds which can exist as impurities in the fuel.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Principle

The total hydrogen content is determined using a hydrogen analyser. The hydrogen analyser is based on the carrier gas method using argon or nitrogen as carrier gas. The samples to be analysed are heated up to a temperature of more than 1 770 °C in a graphite crucible. At that temperature, all volatile components are removed. The hydrogen containing compounds are cracked and released as hydrogen, oxygen, nitrogen, and carbon monoxide. The released gas is purified in the carrier gas stream, for example by oxidation and adsorption columns. The hydrogen is separated by chromatographic means and analysed in a thermal conductivity cell.

4 Interference

The temperature of >1 770 °C should be reached in a short time, within approximately 5 s; if not, the decomposition of the released water to hydrogen and oxygen might not be complete.

At temperatures of more than 2 200 °C, carbon dioxide is released because of a reduction of UO_2 by graphite according to the reaction below; carbon dioxide will interfere with the thermal conductivity measurement.

$$UO_2 + C \rightarrow UO_{2-x} + x/2\ CO_2$$

5 Reagents and materials

Use reagents of recognized analytical grade. The reagents and materials below serve as examples to be used according to manufacturer recommendation.

5.1 Carrier gas. Use argon with a purity of a volume fraction ≥99,995 % or nitrogen with a purity of a volume fraction ≥99,998 %.

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