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STANDARD 9891

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Determination of carbon content in uranium dioxide powder and sintered pellets — High-frequency induction furnace combustion — Titrimetric/coulometric/infrared absorption methods

Détermination de la teneur en carbone dans la poudre et les pastilles frittées de dioxyde d'uranium — Combustion dans un four électrique à induction — Méthodes par titrimétrie/coulométrie/absorptiométrie infrarouge

Reference number
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Foreword

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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.

International Standard ISO 9891 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

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Determination of carbon content in uranium dioxide powder and sintered pellets — High-frequency induction furnace combustion — Titrimetric/coulometric/infrared absorption methods

1 Scope

This International Standard specifies titrimetric/calometric/infrared absorption methods for determining the carbon content in uranium dioxide powder and sintered pellets, the test sample being heated in an induction furnace.

It is applicable to the determination of 5 µg to 500 µg of carbon in uranium dioxide powder and pellets. Interference from sulfur and halogens is prevented by the use of appropriate traps.

2 Principle

A portion of the test sample is heated at a temperature of at least 1 100 °C to 1 200 °C in a quartz-encapsulated tungsten crucible or in a platinum crucible, in an oxygen atmosphere. The evolved oxidation products are passed over a purification trap, filled with manganese dioxide and silver permanganate catalyst. Manganese dioxide absorbs nitrogen oxides. Silver permanganate catalyst will oxidise carbon monoxide to carbon dioxide and absorb sulfur oxides and halogens.

The carbon dioxide is trapped in an absorption solution of barium perchlorate, adjusted to pH = 10,0. Absorption of carbon dioxide causes a decrease of the pH. The initial pH is restored continuously by the addition of hydroxyl ions either by potentiostatic titrimetry or by coulometry.

Alternatively, the carbon dioxide may be determined by absorption of infrared radiation and integration of the signal obtained.

3 Reaction

$$CO_2 + H_2O + Ba(ClO_4)_2 \rightarrow BaCO_3 + 2H^+ + 2ClO_4^-$$

4 Reagents and materials

During the analyses, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Oxygen, of commercial grade, better than 99,9 % (*V/V*) purity.

4.2 Carbon dioxide, commercial grade, better than 99,9 % (*V/V*) purity.

4.3 Copper oxide, 1 mm to 2 mm granules.

4.4 Soda lime, of the self indicating type, 1 mm to 2 mm granules.

4.5 Molecular sieve 4A, 1,6 mm (1/16 in) pellets, preheated at 300 °C.

4.6 Silver permanganate catalyst (in Europe available as Korbl's combustion catalyst), for element analysis.

4.7 Manganese dioxide, activated, combustion analysis grade, 0,5 mm to 1,5 mm granules.

4.8 Accelerators, tin metal, powder form, low in carbon, or tungsten granules.

4.9 Moist hydrogen reduced iron, chips, containing less than 5 ppm of carbon.