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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*



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Foreword

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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.

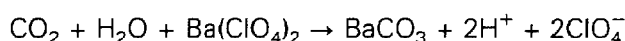
2 Principle

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



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.

4.10 SRM steels¹⁾, of certified reference material grade, chips, certified for carbon, containing 15 ppm to 500 ppm carbon.

4.11 Barium perchlorate $[\text{Ba}(\text{ClO}_4)_2]$.

4.12 2-Propanol ($\text{CH}_3\text{CHOHCH}_3$).

4.13 Absorption solution

Dissolve 200 g of barium perchlorate (4.11) in water. Add 1,0 ml of 2-propanol (4.12), dilute to 1,0 l and mix.

4.14 Sodium hydroxide, solution $[c(\text{NaOH}) = 0,10 \text{ mol/l}]$, free from carbon dioxide, standardized against potassium hydrogen phthalate.

4.15 Sodium hydroxide, solution $[c(\text{NaOH}) = 0,02 \text{ mol/l}]$, free from carbon dioxide. Prepare fresh daily by accurate dilution of the sodium hydroxide solution 4.14.

NOTE 1 Use this sodium hydroxide solution (4.15) for the titration of less than 50 µg of carbon, otherwise use the 0,10 mol/l solution (4.14).

5 Apparatus

Typical dimensions are given for the apparatus; an alternative system giving similar performances may however be used.

5.1 High-frequency induction furnace, 1,5 kVA to 2,5 kVA, 1 MHz to 20 MHz, with silica combustion tube of inner diameter 30 mm (see figure 1). The induction coil surrounding the combustion tube should be 40 mm to 60 mm high and have 4 to 5 turns.

The crucible containing the test portion and, if necessary, an accelerator is supported on an alumina pedestal and positioned within the induction coil to ensure effective coupling.

This furnace gives a temperature of 1 100 °C; an alternative giving a higher temperature may be used.

5.2 Oxygen purification and flow control system (see figure 2)

5.2.1 Oxygen purification system (see figure 3), consisting of a stainless steel tube, of inner diameter 30 mm and length 200 mm, filled with copper oxide (4.3) and heated at 650 °C. The stainless steel tube is connected to a glass tube, of inner diameter 30 mm and length 300 mm, filled with approximately 100 mm of soda lime (4.4) and approximately 200 mm of molecular sieve (4.5). The layers are separated by layers of quartz wool.

5.2.2 Pressure regulators 0 kPa to 200 kPa, manometers 0 kPa to 200 kPa, flow regulators and flow meters 0 ml/min to 100 ml/min

All connections of the gas delivery system should be made using stainless steel tubing of inner diameter 3 mm. The connections between the furnace and the measuring system should preferably be made of stainless steel of inner diameter approximately 1 mm, except for a short piece of PTFE tubing of inner diameter approximately 0,4 mm which is used as the gas inlet to the absorption vessel.

5.2.3 Two-litre buffer vessel, to supply additional oxygen during the combustion of steels.

5.2.4 Six-way valve, to supply an oxygen backflush to the combustion tube when it is open. This flush prevents absorption of carbon dioxide from the air in the combustion tube.

1) Standard Reference Steels are available from:

NBS (National Bureau of Standards), Washington, D.C., USA;

BCR (Bureau communautaire de référence), Brussels, Belgium;

BAS (Bureau of Analysed Samples), Middlesbrough, United Kingdom;

BAM (Bundesanstalt für Materialprüfung), Berlin, Germany;

IRSID (Institut de recherches de la sidérurgie française), Saint-Germain-en-Laye, France.