INTERNATIONAL STANDARD

ISO 22875

First edition
2008-01-15

Nuclear energy — Determination of chlorine and fluorine in uranium dioxide powder and sintered pellets

Énergie nucléaire — Détermination du chlore et du fluor dans les poudres de dioxyde d'uranium et les pastilles frittées

Reference number
ISO 22875:2008(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22875 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

Introduction

This International Standard describes a method for determining the chlorine and fluorine concentrations in uranium dioxide and in sintered fuel pellets by pyrohydrolysis of samples, followed either by liquid ion-exchange chromatography or by selective electrode measurement of chlorine and fluorine ions.

Many ion-exchange chromatography systems and ion-selective electrode measurement systems are available; the equipment and operating procedure are, therefore, not described in detail.

Nuclear energy — Determination of chlorine and fluorine in uranium dioxide powder and sintered pellets

1 Scope

This International Standard describes a method for determining chlorine and fluorine in uranium dioxide powder and sintered pellets. It is applicable for the analysis of samples with a mass fraction of chlorine from 5 µg/g to 500 µg/g and with a mass fraction of fluorine from 2 µg/g to 500 µg/g.

2 Normative references

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 9892:1992, *Uranium metal, uranium dioxide powder and pellets, and uranyl nitrate solutions — Determination of fluorine content — Fluoride ion selective electrode method*

3 Principle

The samples are pyrohydrolysed at 900 °C to 1 000 °C in a tubular furnace with wet air or steam heated to the same temperature. Chlorine and fluorine are trapped as halogenated acids and entrained in an aqueous solution.

Two measurement methods may be used to measure the chlorine and fluorine ions:

a) liquid ion-exchange chromatography;

b) selective electrode measurement.

4 Reagents

Use reagents of recognized analytical grade.

4.1 Water, complying with at least grade 1 in accordance with ISO 3696.

4.2 Anhydrous sodium chloride (NaCl).

4.3 Anhydrous sodium fluoride (NaF).

4.4 Sodium carbonate (Na_2CO_3).

4.5 Anhydrous sodium bicarbonate ($NaHCO_3$).

4.6 Glacial acetic acid (CH_3COOH), $\rho(CH_3COOH) = 1{,}06$ g/ml.

4.7 Potassium acetate (CH_3COOK).

4.8 Concentrated eluant solution, $c(Na_2CO_3)$ = 0,018 mol/l and $c(NaHCO_3)$ = 0,017 mol/l.

Dissolve 1,908 g of Na_2CO_3 (4.4) and 1,428 g of $NaHCO_3$ (4.5) in water (4.1). Pour into a 1 l volumetric flask. Dilute to 1 l with water (4.1). Homogenize.

4.9 Standard eluant solution, Add 100 ml of concentrated eluant solution (4.8) to a 1 l volumetric flask. Dilute to 1 l with water (4.1). Homogenize.

4.10 Make-up eluant solution, $c(Na_2CO_3)$ = 0,09 mol/l and $c(NaHCO_3)$ = 0,085 mol/l.

Dissolve 9,540 g of Na_2CO_3 (4.4) and 7,140 g of $NaHCO_3$ (4.5) in water (4.1) Pour into a 1 l volumetric flask. Dilute to 1 l with water (4.1). Homogenize.

4.11 Buffer solution, $c(CH_3COOH)$ = 0,005 mol/l and $c(CH_3COOK)$ = 0,005 mol/l.

Pour 250 µl of acetic acid (4.6) and 0,50 g of potassium acetate (4.7) into a 1 l polyethylene volumetric flask. Dilute to 1 l with water (4.1). Homogenize.

The concentration of the buffer solution can alternatively be chosen between 0,001 mol/l and 0,1 mol/l.

4.12 Chloride reference solution, $\rho(Cl)$ = 1 g/l. Dissolve 1,648 g of dry anhydrous sodium chloride (4.2) in water (4.1). Pour into a 1 l volumetric flask. Dilute to 1 l with water (4.1). Homogenize.

To achieve dry sodium salt, heat at 120 °C for 4 h just before use and keep in exicator.

4.13 Chloride reference solution, $\rho(Cl)$ = 0,1 g/l.

Pipette 10 ml reference solution (4.12) into a 100 ml volumetric flask. Dilute to 100 ml with water (4.1). Homogenize.

4.14 Chloride reference solution, $\rho(Cl)$ = 0,01 g/l.

Pipette 10 ml reference solution (4.13) into a 100 ml volumetric flask. Dilute to 100 ml with water (4.1) Homogenize.

Solutions may be stored for two months.

4.15 Fluoride reference solution, $\rho(F)$ = 1 g/l.

Dissolve 2,210 ± 0,001 g of dry anhydrous sodium fluoride (4.3) in water (4.1). Pour into a 1 l volumetric flask. Dilute to 1 l with water (4.1). Homogenize.

To achieve dry sodium salt, heat at 120 °C for 4 h just before use and keep in a desiccator.

4.16 Fluoride reference solution, $\rho(F)$ = 0,1 g/l.

Pipette 10 ml reference solution (4.15) into a 100 ml volumetric flask. Dilute to 100 ml with water (4.1). Homogenize.

4.17 Fluoride reference solution, $\rho(F)$ = 0,01 g/l.

Pipette 10 ml reference solution (4.16) into a 100 ml flask. Dilute to 100 ml with water (4.1). Homogenize.

Solutions may be stored for two months.

4.18 Chloride and fluoride calibration standard solutions for chromatography, $\rho(Cl)$ = 0,2 mg/l; $\rho(Cl)$ = 0,5 mg/l; $\rho(Cl)$ = 1,0 mg/l; $\rho(F)$ = 0,2 mg/l; $\rho(F)$ = 0,5 mg/l; $\rho(F)$ = 1,0 mg/l.

Into three 100 ml volumetric flasks, pipette quantities (2 ml, 5 ml and 10 ml respectively) of the 0,01 g/l chloride reference solution (4.14) and the 0,01 g/l fluoride reference solution (4.17). Add 2 ml of concentrated eluant solution (4.8) to each flask. Dilute to 100 ml with water (4.1). Homogenize.

These solutions now contain 0,2 mg/l, 0,5 mg/l and 1,0 mg/l, respectively, of chloride and fluoride ions.

Prepare the calibration solutions fresh on the day of use.

4.19 Chloride calibration standard solutions for ion analysis, $\rho(\mathrm{Cl}) = 0{,}5$ mg/l; $\rho(\mathrm{Cl}) = 1{,}0$ mg/l; $\rho(\mathrm{Cl}) = 2{,}0$ mg/l.

Into three 100 ml volumetric flasks, pipette quantities (5 ml, 10 ml and 20 ml) of the 0,01 g/l chloride reference solution (4.14). Add 20 ml of buffer solution (4.11). Dilute to 100 ml with water (4.1). Homogenize.

These solutions now contain 0,5 mg/l, 1,0 mg/l and 2,0 mg/l, respectively, of chloride ions.

Prepare the calibration solutions fresh on the day of use.

4.20 Fluoride calibration standard solutions for ion analysis, $\rho(\mathrm{F}) = 0{,}5$ mg/l; $\rho(\mathrm{F}) = 1{,}0$ mg/l; $\rho(\mathrm{F}) = 2{,}0$ mg/l.

Pipette 5 ml, 10 ml and 20 ml of the 0,01 g/l fluoride reference solution (4.17) into three 100 ml volumetric flasks. Add 20 ml of buffer solution (4.11). Dilute to 100 ml with water (4.1). Homogenize.

These solutions now contain 0,5 mg/l, 1,0 mg/l and 2,0 mg/l, respectively, of fluoride ions.

Prepare the calibration solutions fresh on the day of use.

5 Apparatus

5.1 Standard laboratory equipment.

5.2 Pyrohydrolysis apparatus.

5.2.1 Tubular furnace, equipped with a calibrated temperature regulator.

5.2.2 Tube with steam heater and condenser.

The tube (Inconel[1)], platinum or quartz) in the furnace is 400 mm long and 20 mm in diameter.

The diameter of the junction tube is 5 mm.

In the case of a pyrohydrolysis device with steam heating, the junction tube is wound around the tube inside the furnace and is connected to this tube before the closing system.

In this case, the steam at the exit of the steam generator is heated to the temperature of the furnace. The extractions of chlorine and fluorine ions are more effective.

5.2.3 Steam generator, consisting of a reservoir for water (4.1) and provisions for heating and temperature regulation to adjust the flow rate of the steam.

5.2.4 Combustion boats, of Inconel, platinum, ceramic or quartz.

5.3 Flasks, 50 ml, 100 ml, 200 ml, 250 ml and 1 000 ml, of any material that can be verified not to create Cl and F contamination.

5.4 Balance, capable of reading to the nearest 0,1 mg.

---

1) Inconel is an example of a suitable product available commercially. This information is given for the convenience of users of ISO 22875 and does not constitute an endorsement by ISO of this product.