INTERNATIONAL STANDARD

ISO 9892

First edition
1992-04-01

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

Métal d'uranium, poudre et pastilles frittées de dioxyde d'uranium, et solutions de nitrate d'uranyle — Détermination de la teneur en fluor — Méthode de l'électrode sélective des ions fluorure

Reference number
ISO 9892:1992(E)

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.

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 9892 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Sub-Committee SC 5, *Nuclear fuel technology.*

Annex A forms an integral part of this International Standard.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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

1 Scope

1.1 This International Standard specifies an analytical method for determining the fluorine content in uranium metal, uranium dioxide powder and pellets and solutions of uranyl nitrate.

1.2 The method can be used within the concentration range of 1 µg to 0,01 g of fluorine per gram of the sample. Impurity levels of up to 300 µg of boron and 3 000 µg of silicon, aluminium and iron in the final measured solution can be tolerated. Zirconium interferes seriously and should be absent. The applicability of the method to samples containing significant impurity levels can be confirmed by modifying the basic procedure.

2 General requirements

2.1 Principle

A weighed portion of the laboratory sample of uranium metal or uranium dioxide is dissolved in nitric acid in a closed polyethylene bottle to prevent loss of hydrogen fluoride. The nitric acid used is dosed with a known amount of fluoride to give a blank concentration which is higher than the lowest concentration of linear response of the fluoride electrode, thus ensuring that all subsequent measurements will take place within the linear response range of the electrode.

The determination is performed by a known addition procedure in which a small volume of a relatively concentrated fluoride standard solution is added to the initial solution. The result is then calculated using the basic standard addition equation, which is readily deduced from the Nernst equation (see 2.2) as follows:

$$m_i = \frac{m_a}{10^{|E_2 - E_1|/S} - 1}$$

where

m_i is the total mass, in micrograms, of fluorine in the inital solution;

m_a is the total mass, in micrograms, of fluorine in the known addition of fluoride standard solution;

$|E_2 - E_1|$ is the absolute value of the change in potential, in millivolts, which occurs on making the standard addition;

S is the electrode slope at the temperature of the determination.

Potentials are measured using a fluoride ion-selective electrode, reference electrode and digital millivoltmeter.

2.2 Use of Nernst equation

In solutions of constant ionic strength, the fluoride-ion-selective electrode responds to the fluoride ion concentration $[F^-]$ of a solution according to the Nernst equation:

$$E = E'_o - S \lg [F^-]$$

where

E is the measured potential, in millivolts;

E'_o is the standard cell potential, in millivolts;

S is the theoretical value of the Nernst slope (58,2 mV at 20 °C).

In nitric acid solutions of uranium (VI), fluoride ion is complexed by H^+ and UO_2^{2+} ions mainly as HF and UO_2F^+. Both these complexes dissociate to give a very small fraction of free fluoride ions, to which the electrode responds.

The function ϕ is defined as