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Nuclear fuel technology — Dissolution of plutonium dioxide-containing materials —

Part 2: Dissolution of MOX pellets and powders

Technologie du combustible nucléaire — Dissolution des matériaux contenant du dioxyde de plutonium —

Partie 2: Dissolution de pastilles et poudres de MOX (ou mélanges d'oxydes)

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Foreword

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This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

A list of all the parts in the ISO 18256 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes a method to dissolve samples consisting of MOX pellets or powders to provide suitable aliquots for subsequent analysis of elemental concentration and isotopic composition.

Nuclear fuel technology — Dissolution of plutonium dioxide-containing materials —

Part 2:
Dissolution of MOX pellets and powders

1 Scope

This document specifies the dissolution of samples consisting of MOX pellets or powders to provide suitable aliquots for subsequent analysis of elemental concentration and isotopic composition.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Summary of the method

Among the factors affecting the formation of solid solution and hence, the ease of dissolution are:

— the method of fuel fabrication (i.e. mechanically blended oxides, co-precipitated oxides, microwave denitrated oxides or sol-gel oxides);

— the degree of sintering.

Therefore, different dissolution methods are applied depending on the type of MOX sample to be dissolved.

The radiological hazard of plutonium and the need to minimize the waste shall be taken into account when choosing the mass of the sample to be dissolved. A MOX mass of 0,1 g to 10 g should be sufficient for most of the analyses. Some analysis may however require more material.

For the highest possible assay accuracy only gravimetric dissolution methods are recommended. However for a less critical assay, volumetric dissolution may be appropriate.

For grain-size powders, the sample is transferred in a pre-weighed flask or vial made of glass, high-density polyethylene (HDPE), polytetrafluoroethylene (PTFE), Nalgene™[1] or other container types, but the choice of the container has consequences.

— When using a glass dissolution vessel, the added fluoride will attack the glass (resulting in a falsification of the pre-weighed tare) but with the benefit that the fluoride is ultimately removed from the solution via release of volatile SiF_4. The resulting reduction in fluoride content of the final solution guards against the formation of insoluble plutonium fluoride (especially below 8 M), which would negatively bias the plutonium element content up to 0,1 % [or more when large quantities of hydrofluoric acid (HF) are used). The tare mass of the etched glass vessel may be re-established by careful cleaning, drying and re-weighing of the vessel.

— HDPE, PTFE or Nalgene™[1] dissolution vessels are not reactive to HF, therefore tare mass remains unaffected. However, these materials have the disadvantage that fluoride ions in the dissolution liquor promote the precipitation of insoluble plutonium fluoride when the oxidation state of plutonium is reduced from IV to III (e.g. by later volume adjustments using a low molarity acid).

An aqueous dissolving mixture comprising nitric acid (about 8 mol/l to 14 mol/l) and an effective catalytic amount of fluoride ions from either hydrofluoric acid (about 0,05 mol/l to 0,1 mol/l) or ammonium bifluoride (about 2 g/l) is added and the pre-weighted dissolution vessel is closed with a reflux cap (or watch glass). The vessel is placed on a hotplate and heat is applied until the dissolution is completed. The solution is then ready for concentration determination of plutonium or uranium per gram of solution by suitable analytical methods. The solution can be used for other analyses, e.g. plutonium or uranium isotopic abundances or impurities determination.

5 Apparatus and reagents

WARNING — Because of the radiological hazard of plutonium, it shall be handled in glove boxes or hot cells and extreme care shall be taken to prevent direct contact, inhalation or ingestion.

5.1 Apparatus

Normal laboratory equipment for a plutonium laboratory and, in particular, the following.

5.1.1 Analytical balance, with a capacity up to 1 200 g and an accuracy of 0,001 g.

5.1.2 Dissolution vessels, dissolution flask or vial with a PTFE-lined screw cap.

The volume of the flask should be adapted to the volume of solution for the appropriate concentration required for subsequent analysis. PTFE-lined screw cap is recommended for the samples which will be stored for long term.

5.1.3 Glass or **PTFE reflux cap**, or **watch glass**.

5.1.4 Reflux condenser.

5.1.5 Shielded hotplate, equipped with a temperature control unit and a fume hood.

5.1.6 PTFE coated tweezers.

5.2 Reagents

Use only reagents of recognized analytical grade.

1) Nalgene™ is the trademark of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

CAUTION — Prepare the reagents in accordance with the local laboratory safety instructions. Nitric acid and hydrofluoric acid are very corrosive and can caused painful burns. Hydrofluoric acid is particularly dangerous, therefore safety precautions and working instructions shall be available and understood.

5.2.1 Water, in accordance with grade 3 of ISO 3696 is recommended.

5.2.2 Concentrated nitric acid, $w(HNO_3)$ = 65 %, $c(HNO_3)$ = 14,4 mol/l (density $\rho_{20\,°C}$ ~1,39 g/cm^3).

5.2.3 Nitric acid, $c(HNO_3) = C_2$ mol/l prepared by dilution of concentrated nitric acid (5.2.2) with water (5.2.1).

5.2.4 Hydrofluoric acid, $w(HF)$ = 40 %, $c(HF)$ = 22 mol/l, stored in a polyethylene dropper bottle.

5.2.5 Ammonium hydrogen bifluoride, NH_4HF_2.

5.2.6 Mixed nitric-hydrofluoric acid solution, $c(HNO_3)$ = 14 mol/l or 7 mol/l to 8 mol/l, $c(HF)$ = 0,05 mol/l to 0,1 mol/l.

The following is an example of a suitable method to prepare the solution.

In a 200 ml polyethylene flask:

— add about 1 ml of hydrofluoric acid (5.2.4);

— adjust the volume of the flask with concentrated nitric acid (5.2.2) or 7 mol/l to 8 mol/l nitric acid (5.2.3);

— homogenize.

Never add concentrated HF to a plutonium nitrate solution since this may cause precipitation of plutonium fluoride which may not be re-dissolved.

5.2.7 Mixed nitric-ammonium hydrogen bifluoride solution, $c(HNO_3)$ = 14,4 mol/l, $c(NH_4HF_2)$ = 2 g/l, $c(HF)$ = 0,07 mol/l.

This reagent is prepared by dissolution of ammonium hydrogen bifluoride (5.2.5) in concentrated nitric acid (5.2.2).

6 Sample dissolution

6.1 Procedure for common plutonium containing materials

a) Calibrate the balance and check the calibration with appropriate standard weights.

b) Weigh a labelled dissolution vessel with a labelled cap, m_1.

c) Transfer the sample into the vessel. Be very careful when transferring powder or crushed pellet to avoid the presence of sample on the outside or in the neck of the vessel since material on the outside will induce a weighing error and powder in the neck of the vessel is difficult to recover.

d) Powders are also hygroscopic and therefore prone to moisture uptake. The weighing of powders should be carried out confidently and quickly with minimum exposure to the analytical cell atmosphere. It is preferable that such manipulations are done in a dry inert atmosphere

e) Close the vessel and weigh it again to obtain the gross mass, m_2.

Sample mass is the difference $m_2 - m_1$.