
**(U, Pu)O₂ Powders and sintered
pellets — Determination of chlorine
and fluorine**

*Poudres et pastilles frittées (U,Pu)O₂ — Détermination du chlore et
du fluor*





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Foreword

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The committee responsible for this document is ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear fuel cycle*.

(U, Pu)O₂ Powders and sintered pellets — Determination of chlorine and fluorine

1 Scope

This International Standard describes a method for determining chlorine and fluorine in mixed (U,Pu) O₂ powders and sintered pellets. It is applicable for the analysis of samples containing 5 µg.g⁻¹ to 50 µg.g⁻¹ of chlorine and 2 µg.g⁻¹ to 50 µg.g⁻¹ of fluorine.

For UO₂ powder and sintered pellets, refer to ISO 22875.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 22875, *Nuclear energy — Determination of chlorine and fluorine in uranium dioxide powder and sintered pellets*

ISO 9892, *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 pyrohydrolyzed at 850 °C – 1 000 °C in a tubular furnace with steam or moist oxygen. Chlorine and fluorine are trapped as halogenated acids and entrained in an aqueous solution. Chloride and fluoride ions are measured using selective electrodes or another appropriate method, for instance, ionic chromatography.

4 Reagents

Use only reagents of recognized analytical grade.

4.1 Demineralised water, complying with at least grade 1 in accordance with ISO 3696 is recommended.

4.2 Anhydrous sodium chloride (NaCl), analytical grade.

4.3 Anhydrous sodium fluoride (NaF), analytical grade.

4.4 Concentrated sodium hydroxide solution, $w(\text{NaOH}) = 32 \%$, relative density approximately 1,35 g.ml⁻¹.

Concentrated sodium hydroxide solution (4.4) is used to prepare sodium hydroxide solutions (4.4.1, 4.4.2, and 4.4.3). Concentrations of solutions (4.4.1, 4.4.2, and 4.4.3) are examples of reference solutions that are acceptable for use.

4.4.1 Sodium hydroxide solution, $c(\text{NaOH}) = 5 \text{ mol.l}^{-1}$.

This reagent can also be prepared by using solid sodium hydroxide in various ways.

For example, the procedure can be as follows.

Pour 460 ml of concentrated sodium hydroxide solution (4.4) into 1 000 ml volumetric flask, dilute to the mark using demineralised water (4.1), and mix.

This reagent can also be prepared by dissolving solid sodium hydroxide (4.5) in solution using demineralised water (4.1).

4.4.2 Sodium hydroxide solution, $c(\text{NaOH}) = 0,5 \text{ mol.l}^{-1}$.

Pour 46 ml of concentrated sodium hydroxide solution (4.4) into 1 000 ml volumetric flask, dilute to the mark using demineralised water (4.1), and mix.

4.4.3 Sodium hydroxide solution, $c(\text{NaOH}) = 0,05 \text{ mol.l}^{-1}$.

Pour 100 ml of $0,5 \text{ mol.l}^{-1}$ sodium hydroxide (4.4.2) into 1 000 ml volumetric flask, dilute to the mark using demineralised water (4.1), and mix.

4.4.4 Sodium hydroxide solution, $c(\text{NaOH}) = 10^{-4} \text{ mol.l}^{-1}$ (or pH 10).

The concentration of solution (4.4.4) is an example of a receiver solution that can be used for a measurement by chromatography.

4.5 Anhydrous sodium hydroxide (NaOH), granules.

4.6 Glacial acetic acid, (CH_3COOH), relative density approximately $1,06 \text{ g.ml}^{-1}$.

4.7 Sodium acetate, (CH_3COONa) analytical grade.

4.8 Buffer solution.

Using the following buffer solution is recommended in order to ensure that the absorbing solution remains alkaline. An example of the procedure to prepare the buffer solution is as follows.

Pour 15 ml of acetic acid (4.6) into 1 000 ml polyethylene volumetric flask and dissolve 50 g of sodium acetate (4.7). Adjust the solution volume to about 900 ml with demineralised water (4.1). Adjust the pH to 5,3 with the 5 mol.l^{-1} sodium hydroxide solution (4.4.1), for example, then dilute to the mark using demineralised water (4.1) and mix.

pH adjustment can be performed with a different sodium hydroxide solution concentration.

Another reagent such as NaOH solution with phenolphthalein can also be used.

4.9 Chloride reference solution.

4.9.1 Primary chloride reference solution, $c(\text{Cl}^-) = 0,5 \text{ g.l}^{-1}$.

Dissolve 166 mg of dry anhydrous sodium chloride (4.2) in demineralised water (4.1). Pour into a 200 ml volumetric flask, then dilute to the mark using demineralised water (4.1) and mix.

Primary chloride reference solution (4.9.1) is used to prepare chloride reference solutions (4.9.2, 4.9.3, 4.9.4, 4.9.5, and 4.9.6). Concentrations of solutions (4.9.2, 4.9.3, 4.9.4, and 4.9.5) are examples of reference solutions that can be used for calibration of selective electrodes. Exact concentrations of calibration solutions should be determined by the user based upon the expected concentration of analyte in the NaOH buffer solution.