
**Nuclear fuel technology — Guidelines
for ceramographic preparation of UO₂
sintered pellets for microstructure
examination**

*Technologie du combustible nucléaire — Lignes directrices pour la
préparation céramographique de pastilles UO₂ frittées pour l'examen
de la microstructure*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This second edition cancels and replaces the first edition (ISO 16793:2005), which has been technically revised.

Nuclear fuel technology — Guidelines for ceramographic preparation of UO_2 sintered pellets for microstructure examination

1 Scope

This document describes the ceramographic preparation of uranium dioxide (UO_2) sintered pellets for qualitative and quantitative microstructure examinations.

These examinations can be carried out before and after thermal or chemical etching.

They enable

- observations of fissures, inter- or intra-granular pores and inclusions, and
- measurement of pore and grain size and measurement of pore and grain size distributions.

The measurement of average grain size can be carried out using a classical counting method as described in ISO 2624 or ASTM E112[3], i.e. intercept procedure, comparison with standard grids or reference photographs.

The measurement of pore-size distributions is usually carried out by an automatic image analyser. If the grain-size distributions are also measured with an image analyser, it is recommended that thermal etching be used to reveal the grain structure uniformly throughout the whole sample.

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 Principle

Ceramographic preparation of UO_2 sintered pellets is carried out in two main stages.

- a) Sample polishing: the sample can either be mounted in resin, or held in an appropriate mechanical gripping device (an example is shown in [Figure 1](#)).
- b) Sample etching: the microstructure can be revealed by chemical or thermal etching. Thermal etching cannot be done on a mounted sample.

5 Procedures

5.1 General

Ceramographic samples can be prepared using three different procedures: the sample can be unmounted (5.2), mounted without subsequent dismounting (5.3), or mounted with subsequent dismounting (5.4).

5.2 Sample preparation without mounting

Prepare samples using the following stages:

- Sectioning (8.1),
- Grinding (8.3);
- Polishing (8.4);
- Chemical (9.2) or thermal (9.3) etching.

5.3 Sample preparation with mounting, not needing subsequent dismounting

Prepare samples using the following stages:

- Sectioning (8.1);
- Mounting with any suitable resin (8.2);
- Grinding (8.3);
- Polishing (8.4);
- Chemical etching (9.2).

5.4 Sample preparation with mounting, needing subsequent dismounting

Prepare samples using the following stages:

- Sectioning (8.1);
- Mounting with polyester or acrylic resin (8.2);
- Grinding (8.3);
- Polishing (8.4);
- Dismounting (8.5);
- Chemical (9.2) or thermal (9.3) etching.

6 Apparatus

6.1 Metallographic sectioning machine, with a diamond wheel and a water-inlet system.

6.2 Manual polishing machine, or automatic polishing machine with a force system capable of maintaining a constant pressure on the samples, recommended between $0,8 \times 10^5$ Pa and $1,0 \times 10^5$ Pa (see Figure 2).

6.3 Ultrasonic cleaning bath.

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