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**Nuclear energy — Standard method for  
testing the long-term alpha irradiation  
stability of matrices for solidification of  
high-level radioactive waste**

*Énergie nucléaire — Méthode d'essai normalisée de la stabilité à long  
terme à l'irradiation alpha des matrices de confinement des déchets  
radioactifs de haute activité*

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Published in Switzerland

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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.

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 6962 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

This second edition cancels and replaces the first edition (ISO 6962:1982), which has been technically revised.

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## Introduction

It is generally agreed that a solid is the best form in which to store or dispose of highly radioactive waste (High Level Waste: HLW) from the first stage of a nuclear fuel reprocessing plant. This solid will usually be in the form of blocks having the mass of several hundred kilograms, cast or formed in a steel container. The solid will receive a large dose of radiation of every kind and it is important that this radiation should not significantly alter the properties of the solid for very long periods of time. Thus, proposed compositions must be tested to ensure their radiation stability.

Although the  $\beta$ -decays of the fission products will far out-number the  $\alpha$ -decays of the incorporated actinides, most of the energy of the  $\beta$  particles (electrons) is dissipated by ionization of the atoms in their path and this will only have a transient effect. On the other hand, almost all the atom displacements in the solid will be caused by the  $\alpha$ -decays, with the recoiling actinide nuclei being responsible for the great majority of these. Alpha-decays generates helium and helium atoms are a foreign body in solids. During long-term storage, helium pressure within the solids is built up to some atmospheres. Thus, it is the stability of the solid to  $\alpha$ -decays that must be tested.

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# Nuclear energy — Standard method for testing the long-term alpha irradiation stability of matrices for solidification of high-level radioactive waste

## 1 Scope

This International Standard specifies a method designed to check the long-term stability of a solid to alpha disintegration by detection of all modifications in the properties of an irradiated sample.

The material favoured hitherto is a borosilicate glass, but possible alternatives include:

- ceramics or glass-ceramics,
- other glass compositions.

## 2 Normative references

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

ISO 16797, *Nuclear energy — Soxhlet-mode chemical durability test — Application to vitrified matrixes for high-level radioactive waste*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### **radioactive waste**

any residue containing radioactive materials not currently considered useful or economically recoverable

### 3.2

#### **package**

#### **waste package**

product of conditioning that includes the waste form as well as any container(s) and internal barriers (e.g. shielding materials and liner), as prepared in accordance with requirements for handling, transportation, storage and/or disposal

### 3.3

#### **waste form**

waste in its physical and chemical form after treatment or conditioning prior to packaging and which is a component of the waste package

### 3.4

#### **container**

outer shell of a waste package

**3.5**  
**matrix**  
**waste matrix**

part of the waste form inside a waste package in which the radioactive substances are dispersed

**4 Principle**

Because most of the atom displacements are caused by the recoiling actinide nuclei, external radiation with  $\alpha$ -particles is not considered a satisfactory simulation. A satisfactory simulation however is as follows: a sample of the candidate solid is made up in a realistic manner using the proper concentrations of the fission-product elements, although these can and, for convenience, usually will be the non-active nuclides. This sample is "doped" with a short half-life  $\alpha$ -emitter so that it will receive the same number of  $\alpha$ -decays per gram in few years as the actual storage medium will receive over a much longer time.

NOTE The difference of dose rate between real waste form and doped form includes the obligation to study this aspect.

The important properties of the sample can then be examined for changes.

It should be noted that it is the detection of any changes in sample properties with radiation that is important. The Soxhlet leach test, as described in ISO 16797, will adequately detect any significant changes and so is satisfactory in this context, although it has only limited environmental significance.

**5 Test Method**

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**5.1 Calculation of the necessary dose**

The concentration of the actinides in the particular discharged fuel can be calculated using a computer program. The amount of these actinides that is or will be incorporated in the high-level waste stream of the reprocessing plant must then be ascertained. If this information is not available, it should be assumed that all the americium and curium and 0,5 % to 1 % of the plutonium left in the waste stream makes a significant contribution to the integrated radiation dose to the solid only after thousands of years. The age of the solid that is to be simulated must then be decided. It is recommended that this should be at least several thousand years (between 1 000 and 10 000 years, for instance). At short times,  $^{238}\text{Pu}$ ,  $^{244}\text{Cm}$  and  $^{241}\text{Am}$  are the most important nuclides. At long times,  $^{239}\text{Pu}$  and  $^{240}\text{Pu}$  contribute to the dose.

**5.2 Choice of nuclide to use**

Short half-lived alpha emitters, principally  $^{238}\text{Pu}$ ,  $^{241}\text{Am}$ ,  $^{242}\text{Cm}$  and  $^{244}\text{Cm}$ , should be used to dope the simulated waste form. The one chosen will often depend on availability, but the following criteria must be considered:

- a)  $^{238}\text{Pu}$  is the easiest short-lived nuclide to handle.
- b) The half-lives are
  - $^{238}\text{Pu}$  87,7 years,
  - $^{241}\text{Am}$  433 years,
  - $^{242}\text{Cm}$  163 days,
  - $^{244}\text{Cm}$  18,1 years.

Therefore, more Am and Pu must be added for a given dose-rate than when curium is used.

- c) Plutonium oxide is not very soluble in some complex matrixes; thus, the preparation of a sample might lead to undissolved plutonium oxide, and the actinides will partition unequally in different phases of the sample. Autoradiography and microscopic examination on a sample cut from the interior of the specimen should be used to check that there is no gross segregation.



Once the required dose is decided, the concentration of the chosen nuclide needed to produce it in a reasonable time can be determined. Again, this must be calculated in each case, since the isotopic purity of the actinide available will vary.

## 6 Sample composition

The composition of the test samples shall be as near as possible to that used in the industrial process. In order to make the minimum alteration to the solids chemistry, curium shall be added to the simulated waste instead of a) other actinides, and b) the rare earth elements, on an atom for atom basis. Similarly,  $^{238}\text{Pu}$  shall replace cerium or uranium first and then, if necessary, some of the rare earth elements. Undoped samples shall also be prepared for comparison purposes.

## 7 Sample preparation

The sample preparation can be checked by, for example, auto-radiography and microscopic examination. It is essential to verify the uniform distribution of the alpha-dopant in the material. Also, for non-vitreous material, the distribution of actinides in the crystal phases must be known and the dopant must distribute in the same way. Otherwise, a realistic picture of damage may not be obtained. This is true because alpha particles (which cause ionization) can penetrate into phases adjacent to the one in which the decay takes place. Recoil nuclei, which cause atomic displacements, travel very short distances (approximately 100 Å) and only result in damage to the phases in which the decay takes place.

## 8 Measurements before storage

The following measurements should be made on both doped and undoped samples as soon as possible after the specimens have been prepared:

- a) initial leach rate,
- b) density, <https://standards.iteh.ai/catalog/standards/sist/8dcf129a-4c5f-4cb0-87a9-a3ef508edc65/iso-6962-2004>
- c) optical and microscopic examination of a sample,
- d) X-ray diffraction examination,
- e) heat emission,
- f) mechanical properties (optional).

The techniques to be used are listed in Clause 10.

## 9 Storage

The specimens shall be stored at room temperature for the predetermined period; this will often be a year or more. The storage shall be in dry air or in an inert atmosphere such as argon. Optionally, a second set of specimens may be stored at some appropriate elevated temperature.

**NOTE** If waste that has been cooled for several years is to be solidified in large-diameter cylinders, then the cooling rate of the solid near the centre of the cylinder will be very slow and holding a sample at some elevated temperature may be appropriate. It seems likely, however, that most of the effects of radiation will decrease with increasing temperature, so that storing samples at the minimum temperature to be experienced by the solid is the crucial consideration.