INTERNATIONAL STANDARD

ISO 12891-4

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Retrieval and analysis of surgical implants —

Part 4: Analysis of retrieved ceramic surgical implants

Retrait et analyse des implants chirurgicaux —

Partie 4: Analyse des implants chirurgicaux en céramique retirés

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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 3.

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 part of ISO 12891 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12891-4 was prepared by Technical Committee ISO/TC 150, *Implants for surgery.*

ISO 12891 consists of the following parts, under the general title *Retrieval and analysis of surgical implants*:

— *Part 1: Retrieval and handling*

— *Part 2: Analysis of retrieved metallic surgical implants*

— *Part 3: Analysis of retrieved polymeric surgical implants*

— *Part 4: Analysis of retrieved ceramic surgical implants*

Annexes A and B of this part of ISO 12891 are for information only.

Introduction

The investigation of retrieved surgical implants and adjacent tissues can be of diagnostic value in the event of clinical complications, can deepen the knowledge about clinical implant performance and interactions between implants and the body, and can provide information on implant performance and safety, thus furthering the development of biocompatible implant materials and implants with improved functional longevity.

This part of ISO 12891 offers guidelines for the analysis of retrieved ceramic surgical implants to ensure they are not damaged, to indicate typical investigation techniques, and to allow comparisons between investigation results from different sources. These guidelines may also serve for the documentation of clinical investigations. They may also be useful for retrieval and analysis studies in animals. Other parts of ISO 12891 describe detailed procedures for the retrieval and handling of implants manufactured from materials other than ceramics, as well as methods of analysis (see the foreword).

ISO 12891-1 gives general guidelines on retrieval and handling and applies to this part ISO 12891 as well as to the other parts, which are related to the analysis of different categories of material. Informative annexes B and C of ISO 12891-1:1998 include examples of protocols for reporting data concerning the retrieval process. These protocols are not repeated in the other parts of ISO 12891; they may be reduced or expanded depending on the retrieved surgical implant, the presence of any attached or accompanying biological material, and the purpose of the retrieval and analysis.

Retrieval and analysis of surgical implants —

Part 4:
Analysis of retrieved ceramic surgical implants

1 Scope

The various parts of ISO 12891 give recommendations for the retrieval, handling and analysis of surgical implants and associated specimens which are removed from patients routinely, during revision surgery or post-mortem. The aim is to provide guidance in preventing damage to the specimens which could obscure the investigation results, and in gathering data at the proper time and under the proper circumstances to validate the study. Part 1 deals with retrieval and handling. Parts 2 to 4 concern the analysis of implants of specific materials, including protocols for reporting the data collected. For particular investigation programmes, additional, more specific, protocols may be required. If special analytical techniques are employed, the appropriate procedures should be specified.

This part of ISO 12891 provides guidance on the analysis of retrieved ceramic surgical implants. For general information on ceramics, see the bibliography. The investigation is divided into three stages that are increasingly destructive. The stage and type of investigation should be chosen as a function of the type of implant and the purpose of the investigation.

This part of ISO 12891 concerns "dense", "stable" ceramics such as alumina and tetragonal zirconia. However, where suitable, sections of this part of ISO 12891 may be also applied to less stable ceramics like hydroxyapatite or bio-accessible ceramics like tricalcium phosphate. In clause 6, some provisions are made for analyses of such types of ceramic.

This part of ISO 12891 should be used in accordance with national regulations or legal requirements concerning the handling and analysis of retrieved implants and tissues and associated biological material.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 12891. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this part of ISO 12891 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 12891-1:1998, *Retrieval and analysis of surgical implants — Part 1: Retrieval and handling.*

3 Term and definition

For the purposes of this part of ISO 12891, the following term and definition apply.

3.1
ceramic surgical implant
surgical implant consisting of ceramic material intended to be inserted into the body by surgical techniques

NOTE 1 It is also referred to simply as an "implant".

NOTE 2 The term may also be used for a component of a compound implant.

4 Retrieval, handling and packaging

Procedures for retrieval, handling, packaging and protection of the personnel involved are found in ISO 12891-1.

NOTE As a precautionary measure, removed implants should be sterilized by an appropriate means that does not adversely affect the implant or the planned investigation. Suitable methods can be found in ISO 12891-1.

5 Analysis of the implant interfaces

5.1 Implant/tissue interface

A significant part of the information associated with a retrieved surgical implant is often at the implant/tissue interface. Attention should be given to particles in the peri-implant tissue. Where possible, analyses of the chemistry and nature of the byproducts of degradation of the implant and a study of the cellular response to the implant should be considered.

In cases where implant surfaces are structured to the effect that tissue ingrowth is invited, the study of the implant tissue interface is of particular interest, and the findings should be recorded. One may even detect microscopic adherent tissue residues on surfaces where no attached tissue has been retrieved intentionally together with the implant.

Since the appearance of the tissue may vary rapidly with the distance from the implant surface, it is important that the tissue is analysed in its context with the implant (see also the relevant sections of ISO 12891-1:1998, such as 4.5).

5.2 Implant/implant interfaces

Where ceramic implants articulate or are in contact with other implant components, the condition of the contacting surface areas of the implant may be of particular interest. Their study should be considered in the context of the opposing surface, and that part of ISO 12891 which concerns the material of this opposite component may be consulted.

6 Analysis of the implant

6.1 General

This clause describes implant examinations to be considered when a retrieved implant is under investigation. The analyses of the implant are separated into three stages, with the degree of investigation increasing from stage I to stage III. The implant investigation may include macroscopic and microscopic examinations, chemical analyses and the determination of physical and mechanical properties.

6.2 Standard form for various stages of the investigation

A standard form indicating the information to be recorded at each stage of the investigation is given in annex A. This form is a framework. Sections of the form that do not apply to a particular implant analysis can be neglected. The form may also be expanded or modified.

A standard form for recording the minimum clinical information, as well as additional clinical material, is given in annex B of ISO 12891-1:1998.

Analyse each component of an implant separately, if possible and necessary. Consider other relevant parts of ISO 12891 if materials other than ceramics are involved. Because of the complexity of analyses of the variety of ceramic materials that may be used for implants, and because of the large number of potential analyses and tests suggested in this part of ISO 12891, the investigation is divided into different stages. The examinations selected will depend upon the reasons for removal and examination of the implant, and possible restrictions in destructive testing. Perform a minimum number of examinations for routine removals where the implant is not suspected to have malfunctioned, more examinations for implants suspected of having a functional impairment, and extensive examinations for implants removed because of their poor performance or behaviour or because of a malfunction.

6.3 Stage I investigation (macroscopic examination — non-destructive)

6.3.1 Identification/photography

Markings found on the implant such as logos, article numbers, lot numbers, dimensions, etc., should be recorded (see annex A). Photographic documentation of relevant findings should be made where useful.

6.3.2 Visual examination

Check the implant surface by suitable techniques to ascertain the mode of failure, destruction or surface alteration, if any such appears. In no event should any surface of a failed implant be destructively evaluated at this stage.

6.3.3 Low-power optical examination

Perform an overall examination under a low-power optical stereo-microscope. Record the findings as per annex A.

6.3.4 Further evaluation

If at the conclusion of stage I further investigation is required to clarify any observations made, to evaluate other characteristics or to evaluate the mode of failure of the implant, it should be carried out subsequently in stage II.

6.4 Stage II investigation (microscopic examination — mostly non-destructive)

6.4.1 General

Stage II should be carried out after stage I, if deemed necessary, to evaluate further the characteristics and/or failure mode of the implant. The primary aim of this stage of the investigation is to assess the mode of failure and the deterioration of the implant in the most non-destructive manner possible (see annex A).

6.4.2 Microscopic examination

Use standard optical-microscope or electron-microscope examination techniques suitable for the material under investigation.

When scanning electron microscopy is used, special preparation techniques may be required to ensure the necessary conductivity.

6.4.3 Fractographic examination

If the implant is fractured, analysis of the fracture surface by suitable techniques may help to ascertain the mode of fracture or to detect defects in the material. In general, destructive evaluation of the fracture surface should be avoided. If the implant has suffered mechanical failure, it is important to remember that it may be classified as legal evidence.

6.4.4 Surface condition

Where worn and unworn areas of the surfaces of retrieved ceramic implants are of interest, surface roughness tests may be carried out in addition to the morphological assessment (see annex A, items 3 and 4, and annex B, clause B.2).

6.5 Stage III investigation (material investigation — mostly destructive)

6.5.1 General

If further investigation is necessary, the tests listed under stage III in annex A should be carried out as deemed necessary to characterize further the implant and its history. Suitable methods are listed in annex B.

6.5.2 Material composition

6.5.2.1 It may be sufficient in the study of a given retrieved implant to verify by simple means that the type of ceramic material corresponds to the information provided by the manufacturer. If more details are required or the nature of the ceramic material is unknown, appropriate techniques should be used to determine the required physical and chemical properties. If suitable ceramic implant material standards exist, they should be quoted to identify the implant material (see annex B).

6.5.2.2 Recognized analytical methods should be used to determine the chemical composition of the ceramic implant. A distinction should be made between screening techniques, such as X-ray fluorescence analysis and EDX analysis, and highly quantitative and specific techniques such as atomic absorption spectroscopy and spectrophotometric analysis.

6.5.2.3 Where of interest, the degree of crystallinity and the atomic structure can be determined by X-ray diffraction techniques.

6.5.3 Microstructure

6.5.3.1 If required, assess microstructural features using standard ceramic preparation and investigation techniques suitable for the material under examination. Optical-microscope and electron-microscope methods are suitable.

6.5.3.2 Determine the grain size using suitable methods.

6.5.3.3 Check for inclusions and voids and potential defects. For the determination of inclusions, electro-optical micro-analysis may be employed (e.g. EDX analysis in a scanning electron microscope).

6.5.3.4 If a porous ceramic material is under investigation, the average pore size may be determined.

6.5.3.5 The position of the area or portion of the implant that has been analysed should be identified in relation to the complete implant and reported with the results. The method of analysis used should be clearly identified.

6.5.4 Mechanical properties

6.5.4.1 The types of measurement carried out at this stage will be dependent upon the implant and its application. For the assessment of mechanical properties, list 8 in annex A should be checked (see annex B for suggested methods).

6.5.4.2 Determine the density and hardness in accordance with applicable material standards (see annex B for suggested methods). Because of the high hardness of the dense, stable ceramics used in implants, the Knoop hardness test is recommended.

6.5.4.3 Where required, determine the tensile, flexural, compressive, etc., properties in accordance with applicable material specifications if possible, and such other tests as are appropriate to the specimen, which may be fabricated from the implant where dimensions allow. Deviation from the specimen dimensions specified in

standard methods may be made necessary by the shape and size of the implant under investigation. This should be taken account of when evaluating the test results.

6.6 Surface-treated or coated implants

6.6.1 If the ceramic implant has undergone surface treatment or has a surface coating, the following tests should be considered:

6.6.2 Examine the coating for structural integrity. In particular, note the occurrence of any surface regions which have become altered, such as by delamination, coating loss or other changes.

6.6.3 Note the location of any fragments or debris and examine any relationship to tissues, when accessible.

6.6.4 Where appropriate, carry out specific tests to evaluate the surface treatment, coating or substrate (e.g. the chemical, microstructural and mechanical characteristics).

6.6.5 Where appropriate and accessible, analyse the tissues associated with the implant and any fragments or debris.

6.7 Biodegradable ceramic implants

6.7.1 If the implant was manufactured from intentionally biodegradable ceramics, test procedures described in this part of ISO 12891 may still be used. However, the interpretation of the results should allow for the time-dependent physical and chemical changes to be expected with such ceramics.

6.7.2 Examine the implant for structural integrity. In particular, note the occurrence of any surface regions which have become altered, such as by delamination, loss, cracking or other changes. Describe your findings in detail.

6.7.3 Note the location of any fragments or debris and examine any relationship to tissues, when accessible.

6.7.4 Where appropriate and accessible, analyse the tissues associated with the implant and any fragments or debris.

7 Implant performance

To evaluate the clinical performance of the implant under investigation, in particular in the case of failure or deterioration, the implant application, the physiological conditions, the clinical history and the implant loading all have to be considered.

Annex A
(informative)

Standard form for the analysis of retrieved ceramic surgical implants

Report No. _____

Date _____

Examination of retrieved implant

This report is for component No._____of a total of_____components.

Condition: intact □ broken □ cracked □ damaged □ other □

NOTE This form contains guidelines as to how the implant analyses can be organized and the results presented. Sections which are not applicable may be deleted or marked as not applicable. Individual forms based on this example may be created. Where required, additional observations and findings should be included.

Stage I investigation (non-destructive)

1. Implant type (manufacturer and model if available)

Identification marks _____ Dimensions _____

2. Type of material (be as specific as possible)

3. Macroscopic examination, visually and with a low-power microscope (assess with YES, NO, DOUBT or NOT APPLICABLE — specify further if required)

	Location	Estimated degree
a)	wear (describe appearance) _____	_____
b)	discoloration_____	_____
c)	material transfer_____	_____
d)	scratching or pitting_____	_____
e)	fragmentation_____	_____
f)	major cracks_____	_____
g)	surface cracks/crazing_____	_____
h)	chipping_____	_____

i) surface erosion_____ _____

j) mechanical damage_____ _____

k) macro-porosity_____ _____

l) tissue attachment_____ _____

m) other signs of degradation_____ _____

Stage II investigation (mostly non-destructive)

Further assessment of surface and of potential defects at higher magnifications (optical and electron microscopy).

4. Wear and deterioration (If YES, identify and describe location and state method of examination)

a) adhesive wear_____

b) abrasive wear_____

c) material transfer_____

d) wear + degradation_____

e) wear + fatigue_____

f) multi-component wear_____

g) cracking/crazing_____

h) deterioration, dissolution (describe in detail)_____

5. Mechanical failure (if YES, identify mode and location and state method of identification)

a) static overload_____

b) shear_____

c) bending_____

d) torsion_____

e) impact_____

f) fatigue_____

g) fatigue combined with_____

h) stress-cracking_____

i) deterioration + cracking_____

j) combination of above (identify)_____

k) other_____

l) unable to identify_____

Stage III investigation (destructive)

6. Type of material (indicate method of analysis — see annex B)

a) chemical composition_____

7. Microstructure and defects (microscopic examination: indicate specimen location and orientation, and preparation technique)

a) grain size_____

b) inclusions_____

c) grain boundary constituents_____

d) homogeneity_____

e) different phases_____

f) micro-porosity (%)_____

g) other flaws_____

h) internal cracking _____

1) single crack_____

2) multiple cracks_____

3) crack origin_____

4) grain boundary cracks_____

i) other features_____

8. Assessment of mechanical properties (N/A = not applicable)

a) hardness (indicate method and location)_____

b) density_____

c) open porosity_____

d) closed porosity_____

e) tensile test (indicate specimen size and orientation, gauge length)_____

f) flexural strength_____

g) compressive strength_____

h) bending test_____

i) other types of test_____

9. Coatings

a) coating material_____

b) coating condition_____

c) estimated fraction of coating missing_____

d) shear strength_____

e) tensile strength_____

Annex B
(informative)

Test methods for the evaluation of ceramic materials

B.1 Chemical properties

ISO standards

Various ISO standards are available for the chemical analysis of alumina:

ISO 6474, *Implants for surgery — Ceramic materials based on high purity alumina.*

ISO 13356, *Implants for surgery — Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP).*

Other standards

ASTM C 560, *Standard Test Methods for Chemical Analysis of Graphite.*

NF S 94-065, *Materials for surgical implants — Determination of arsenic, mercury, cadium and lead on coatings based on phosphate of calcium.*

NF S 94-066, *Materials for surgical implants — Quantitative determination of the Ca/P ratio of calcium phosphate.*

NF S 94-067, *Materials for surgical implants — Qualitative and quantitative determination of the foreign phases present in calcium phosphate based powders, deposits and ceramics.*

B.2 Physical properties

ISO standards

ISO 5017, *Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity.*

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

Other standards

ASTM E 112, *Standard Test Methods for Determining Average Grain Size.*

NF S 94-068, *Materials for surgical implants — Determination of the crystallinity and apparent size of the apatite crystallites of hydroxyapatite based powders, deposits and ceramics.*

JCPDS, Sheets 4-077, 9-169, 9-432: *X-ray diffraction standards for calcium oxide, tri-calcium orthophosphate, and hydroxyapatite, respectively.*

JFCA EC4-001, *Testing method for porosity and specific weight of bioceramics* (1994).

JFCA EC4-002, *Testing method for grain size of bioceramics* (1994).

JFCA EC4-003, *Testing method of poresimetry for bioceramics* (1994).

JFCA EC4-004, *Testing method of surface for bioceramics* (1994).

JFCA EC4-006, *Testing method for crystal structure analysis of bioceramics* (1994).

JFCA EC4-001, *Testing method of solubility for bioceramics* (1994).

B.3 Mechanical properties

ISO standards

Flexural strength (no suitable standard currently available)

Elastic modulus (no suitable standard currently available)

Fracture toughness (no suitable standard currently available)

ISO 9385, *Glass and glass-ceramics — Knoop hardness test.*

ISO 4545, *Metallic materials — Hardness test — Knoop test.*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method.*

ISO 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method (scales A,B,C,D,E,F,G,H,K,N,T).*

Other standards

ASTM F 1044, *Standard Test Method for Shear Testing of Porous Metal Coatings.*

ASTM F 1147, *Standard Test Method for Tension Testing of Calcium Phosphate and Metal Coatings.*

JFCA EC4-005, *Testing method for Vickers hardness of bioceramics.*

JIS R 1610, *Testing method for Vickers hardness of high performance ceramics* (1991).

JFCA EC4-007, *Testing method for flexural strength of bioceramics* (1994).

JIS R 1601, *Testing method for flexural strength of high performance ceramics.*

JFCA EC4-008, *Testing method for compressive strength of bioceramics* (1994).

JIS R 1608, *Testing method for compressive strength of high performance ceramics* (1990).

JFCA EC4-009, *Testing method for elastic modulus of bioceramics* (1994).

JIS R 1602, *Testing method for elastic modulus of high performance ceramics* (1986).

JFCA EC4-010, *Testing method for fracture toughness of bioceramics* (1994).

JIS R 1607, *Testing method for fracture toughness of high performance ceramics* (1990).

B.4 General information

ASTM E 860, *Standard Practice for Examining and Testing Items that are or may become involved in Products Liability Litigation.*

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