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Implants for surgery — Test solutions and environmental conditions for static and dynamic corrosion tests on implantable materials and medical devices

Implants chirurgicaux — Solutions d'essai et conditions environnementales pour les essais statiques et dynamiques de corrosion sur les matériaux et dispositifs médicaux implantables

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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 16428 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

Introduction

In many instances testing of medical devices and materials in a physiological environment is highly desirable for scientific purposes and development work as well as for the assessment of the performance of surgical implants and devices. The application of original physiological fluids is often difficult because of the rapid deterioration of such media.

The application of artificial media is common, but there is the disadvantage that the compositions vary widely and testing results are often not comparable.

This International Standard specifies basic reproducible environmental conditions using a test fluid of isotonic sodium chloride (NaCl) solution. This solution is appropriate because it is used for injections and irrigation in surgery and has an ion content similar to that of human body fluids. Of particular importance are the chloride (Cl^-) ions because the corrosion resistance of most metals is very sensitive to them. Correspondingly, the isotonic NaCl solution is already widely used in the testing of medical devices.

Implants for surgery — Test solutions and environmental conditions for static and dynamic corrosion tests on implantable materials and medical devices

1 Scope

This International Standard specifies standard environmental conditions for the testing of metallic materials intended for implantation, surgical implants, and medical devices. The test conditions described simulate physiological conditions in a simplified manner controlling the test solution, the temperature, the gaseous atmosphere and the proportions of sample size and volume of solution.

These environmental testing conditions can be employed where necessary in combination with various static or dynamic tests where the effect of the physiological environment is to be considered. Typical applications are corrosion fatigue tests and selected fretting and wear tests, as well as general electrochemical tests.

Typical articulating joint simulator tests and aspects particular to the dental field are not considered by this International Standard. Solutions that attempt to replicate the tribological properties of body fluids, such as those used in wear studies, are outside the scope of this International Standard.

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

3 Terms and definitions

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

3.1
corrosion fatigue testing
assessment of corrosion fatigue behaviour where cyclic loading tests are carried out in an aqueous test solution which is related to the human physiological environment

NOTE The test solution may either cause visible corrosion effects and/or acceleration of the fatigue process.

3.2
environmental testing conditions
conditions under which a sample (specimen) is tested including the testing fluid, temperature, aeration, the pH, and the volume ratio and exchange of the fluid

3.3
isotonic sodium chloride solution
aqueous solution of sodium chloride (0,9 % NaCl mass fraction) which provides the same osmotic pressure in living tissues as the physiological fluid (blood serum)

NOTE In surgical applications, it prevents the collapse of tissues and serves as an infusion solution.

3.4
Ringer's solution
isotonic aqueous solution of NaCl with additional compounds which are constituents of the human body fluids (blood serum)

NOTE See also Annex A.

3.5
static and dynamic test
mechanical set-up of the test within the context of this International Standard

NOTE This does not refer to electrochemical conditions.

4 Significance and application

4.1 Significance of test solution

The described environmental conditions are intended for applications where a testing environment for metallic medical devices or materials is required that relates to physiological conditions. The isotonic NaCl solution is used for injections [3] and for flushing and cleaning in surgery. Its ion concentration is similar to that of human body fluids. Of particular significance are the Cl^- ions because they have corrosive effects on metals, in particular on such metals and alloys which form passive films that protect against corrosion.

4.2 Application

4.2.1 General

The test conditions described are applicable for static and dynamic mechanical tests to assess the potential susceptibility to corrosion effects related to the physiological environment.

4.2.2 Testing under static conditions

The environmental conditions described are suitable for static immersion tests to study, for example, special corrosion effects such as pitting or crevice corrosion, leaching, or the performance of special surface treatments. Such tests may include uniform mechanical loading.

NOTE On highly corrosion resistant metallic implant materials, no visible surface deterioration might be detectable in purely static immersion tests. The environmental conditions described might show only effects under more stringent polarized conditions or mechanical loading and/or dynamic conditions.

4.2.3 Testing under loaded dynamic conditions

The environmental conditions described are added to mechanical test arrangements which are commonly carried out in air. They are applied, for example, in fatigue testing to assess the susceptibility to corrosion fatigue, or in selected fretting and friction tests where corrosion and wear effects are of concern.

This International Standard is not applicable to typical articulating joint simulator tests and aspects particular to the dental field nor that attempt to replicate the tribological properties of body fluids, such as those used in wear studies. In short-term handling tests (e.g. clamping), where friction or fretting are of concern, the test solution(s) (5.1) may be employed while some of the other environmental conditions (Clause 5) are neglected depending on the issue.

4.2.4 Electrochemical studies

In general, the environmental conditions described are applicable to electrochemical testing. However, depending on the type of tests some more stringent conditions may be required by certain test protocols.

5 Environmental testing conditions

5.1 Test solution

For the preparation of an isotonic aqueous 0,9 % (mass fraction) sodium chloride solution, 9 g of NaCl of analytical quality is added to purified water in accordance with ISO 3696. This results in 1 000 ml of test solution (for additional information see Reference [3]).

If a test solution other than the isotonic NaCl has been used in the testing, this shall be reported indicating some rationale.

There may be specific reasons to use a modified isotonic NaCl solution for the intended testing, for example a phosphate-buffered solution. The latter would have to be applied with technical care. There are various compositions known as “Ringer’s solutions” which contain additions that are constituents of the body fluids; in Annex A, a common composition is given under A.1. Under A.2, a modified solution with low pH is given, in case more stringent testing conditions are desired.

Additional acceptable test solutions related to the human physiological environment include those identified in ASTM F 2129:2003, Annex X2 [8].

5.2 Testing temperature

During the test, the temperature of the solution is kept stable thermostatically at (37 ± 1) °C.

5.3 pH value

The test solution has nearly a neutral pH value. During long-term testing the pH value shall be recorded on a regular basis.

Smaller shifts of the pH may be caused by air (CO_2). Significant changes of the pH value may indicate deterioration of the testing solution. This may be caused by degradation products (such as corrosion products or wear debris) from the test samples or possibly from parts of the test chamber. As soon as the test solution deteriorates, for example indicated by shifting of the pH or by discoloration, the test solution shall be exchanged and the test chamber washed out prior to the refill.

If the degradation products and/or their effects are to be investigated, the test solution may remain unchanged, but this should be recorded and an explanatory note should be included in the test report.

5.4 Aeration

For defined conditions, reproducibility, and assessment of the corrosion behaviour, the test solution shall be flushed with pure gases:

a) with pure oxygen to allow for passivation of the metal surface; or

b) with pure nitrogen to reduce the passivating effect of dissolved oxygen on the specimen surface for more stringent testing.

Depending on the purpose of the investigation, it may be necessary to carry out tests with both gases to investigate the effect on the passivity of the metal surface. For certain tests it may suffice to flush with air.

The conditions of aeration shall be reported.

5.5 Volume of test solution

The ratio of the volume of the test solution to the exposed surface area of the specimen shall be at least 10 ml/cm^2 [1].

5.6 Circulation of the solution

If the specimen undergoes cyclic loading and the test solution is purged with gas, the agitation of the solution will normally suffice. If there is suspicion that portions of the test solution stagnate at certain specimen areas, additional agitation or pumping in connection with a reservoir vessel may be indicated.

For test series which are intended for comparison, the circulation conditions of the test solution shall be the same.

5.7 Test chamber

Adjust the test chamber to the type of test carried out. The test chamber typically consists of a suitable glass or polymer vessel. For electrochemical or corrosion testing, the relevant test protocols shall be observed.

In general, the mounting of the test specimens and the chamber should be such that erroneous test results are prevented. Thus, metallic mounting components should be avoided or should be of such material and design that galvanic corrosion is avoided. Care should also be taken to avoid crevice corrosion that could be caused by certain mounting conditions or at recesses of the test specimen.

6 Test specimens

The test specimens may represent typical material samples designed for a particular type of test (see Reference [2]), or they may consist of surgical implants or their components or of other medical devices.

The preparation of the surface of the test specimens can highly influence the test results. Therefore the specimens should be free of any contamination such as grease, fingerprints or accidental uncontrolled oxidation. (See B.2 for further considerations.)

The surface preparation and cleaning procedures shall be reported. If sterilization is included, this shall be reported mentioning the method applied.

7 Evaluation and reporting

7.1 Evaluation of test results

The evaluation of test results should be subject to any particular test specifications or protocols. Aspects that may be considered are given in B.2.

7.2 Test report

In reports on tests with environmental conditions according to this International Standard, the following information shall be included:

a) reference to this International Standard (ISO 16428:2005):

b) any deviations from the procedure or any operations regarded as optional;

c) test solution;

d) test temperature;

e) pH value(s);

f) aeration;

g) volume of testing solution;

h) circulation of the test solution;

i) description of test chamber including its material and design;

j) specimen preparation.

Annex A
(informative)

Additional test solutions

A.1 Isotonic Ringer's solution

Add the following quantities of compounds of analytical quality:

— NaCl 8,36 g

— KCl 0,3 g

— $CaCl_2$ 0,15 g

to purified water in accordance with ISO 3696 to make 1 000 ml solution [4].

NOTE This solution is addressed as isotonic electrolyte solution (electrolytorum solutio composita) for infusion or irrigation and is administered when Ringer's solution is required. A synonymous term is physiological Ringer's solution [5].

A modification of this solution contains an addition of 0,1 % $NaHCO_3$ which is added before the $CaCl_2$. It reduces the lifetime of the solution and prevents that sterilization at 121 °C can be used. This composition was given originally by Ringer as a replacement for blood serum [6].

A.2 Aqueous 0,9 % NaCl solution with low pH in the acidic range

To the 0,9 % (mass fraction) NaCl solution as described in 5.1, add analytical quality HCl to give a solution of pH 2.

This solution has proven useful where more stringent testing conditions are desired.

Annex B
(informative)

Considerations for surface preparation and test evaluation

B.1 Considerations for surface preparation of specimens

When the surface preparation of test specimens is selected, and no particular testing protocol is given, the following may be considered.

The test specimens may be prepared as the corresponding implants, prior to insertion. The investigator will need to decide whether this should include sterilization or not. Most metallic implant materials oxidize during the sterilization procedure, which can enhance passivation and influence the testing results. Consistency of the procedure within comparable test series should be observed.

The effect of a certain surface finish may be the reason for a study undertaken. In this case again, the effect of sterilization should be considered.

B.2 Considerations regarding test evaluation

Depending on the type of test, the effect of the environmental conditions may be evaluated under the optical microscope or under the scanning electron microscope.

The reduction of the endurance limit in corrosion fatigue testing may be taken as a criterion in the evaluation of the environmental effects.

For the assessment of the effect of the environmental conditions in dynamic testing (e.g. fatigue), additional specimens in air under ambient conditions may be tested for comparative purposes.

In fatigue testing (or other dynamic testing) where (material) systems have shown no sensitivity to the environmental conditions, subsequent tests under ambient conditions in air may be conducted, when suitable.

Bibliography

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[2] ISO 11845:1995, *Corrosion of metals and alloys — General principles for corrosion testing*

[3] ASTM F1801:1997, *Standard Practice for Corrosion Fatigue Testing of Metallic Implant Materials*

[4] Pharmacopoeia Helv. VIII, 1997, CH 196 with European Pharmacopoeia (Ph.Eur. 4, 2001) *Isotonic sodium chloride solution 9 g/l infundible (Natrii chloridi solutio infundibilis 9 g/l)*

[5] Pharmacopoeia Helv. VIII, 1997, CH 94 with European Pharmacopoeia (Ph.Eur. 4, 2001) *Electrolyte solution (Electrolytorum solutio composita)*

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[7] *Roempps Chemie Lexikon*, Otto-Albrecht Neumueller, 8th Edition, Frankh'sche Verlagshandlung, Stuttgart

[8] ASTM F2129:2004, *Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices*

[9] JIS T 0302, *Testing method for corrosion resistance of metallic biomaterials by anodic polarization measurement*

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