INTERNATIONAL STANDARD

**ISO 16429**

First edition
2004-07-01

# Implants for surgery — Measurements of open-circuit potential to assess corrosion behaviour of metallic implantable materials and medical devices over extended time periods

*Implants chirurgicaux — Mesurages sur de longues périodes du potentiel en circuit ouvert pour l'évaluation du comportement à la corrosion des matériaux métalliques et dispositifs médicaux implantables*

Reference number
ISO 16429:2004(E)

© ISO 2004

--````,`-`-`,,`,,`,`,,`---

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2004

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

## Contents

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

# 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 16429 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

# Introduction

This International Standard was developed because, in contrast to polarization measurements which are well described in ASTM G5 [6] and literature, there is no standard available on typical open-circuit potential measurements over extended time periods.

Relating to corrosion behaviour of implant materials and surgical implant devices, the long-term electrochemical behaviour in the body environment is of interest.

Metal surfaces undergo spontaneous changes at their interface with an electrolytic environment to reach a state of equilibrium. Depending on the conditions, the corresponding physico-chemical and electrochemical reactions can be highly active and corrosive, or very sluggish and passive. For passivating metals such as those usually used for surgical implants, the formation and stability of the passive film is an important prerequisite for corrosion resistance of these materials, under given conditions.

With the measurement of the open-circuit potential over a longer time period, the spontaneous reaction to the environment (electrolyte) in the form of passivation or activation, the formation of a steady state potential and its stability can be assessed. Regarding surgical implant materials and devices, the measurements of these properties is of interest because they help to characterize implant material systems and to optimize processing, surface treatments and properties. Furthermore, measurements of the long-term open-circuit potential in combination with mechanical loading give information on the effect of mechanical, dynamic conditions on the electrochemical potential, passivity and corrosion behaviour.

This International Standard specifies conditions for the measurement of the open-circuit potential over extended periods of time. Isotonic 0,9 % NaCl (see 3.5) is used as the electrolyte (testing solution). This solution is related to body fluid in that it contains approximately the same concentration of Cl ions, and Cl ions are the most likely species in this solution to cause metal corrosion due to their aggressivity. For more stringent testing conditions, solutions with higher concentrations of Cl ions are given in Annex A.

--`````,`-`-`,,`,,`,`,,`---

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS Not for Resale

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

**INTERNATIONAL STANDARD** **ISO 16429:2004(E)**

# Implants for surgery — Measurements of open-circuit potential to assess corrosion behaviour of metallic implantable materials and medical devices over extended time periods

## 1 Scope

This International Standard specifies a test method for measurements over extended time periods of the open-circuit potential of implant materials and surgically implantable devices immersed in a test environment related to body fluid, using a standard corrosion test cell to study the electrochemical corrosion properties of the devices.

This method of monitoring the open-circuit potential can also be combined with mechanical static or dynamic loading tests.

This International Standard is applicable in particular to metallic materials which form passive layers with protective properties against corrosion, as typical for surgical implant materials.

This test method is intended for the investigation of single metallic materials or alloys. It is not applicable to dissimilar material combinations, which require particular considerations in measuring and interpreting the results.

## 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*

ISO 16428, *Implants for surgery — Test solution and environmental conditions for static and dynamic corrosion and wear tests on implantable materials and medical devices*

## 3 Terms and definitions

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

**3.1**
**corrosion potential**
electrode potential of a metal in a given corrosion system

**3.2**
**corrosion system**
system consisting of one or more metals and those parts of the environment (including specimen, electrolyte, electrodes) that influence corrosion

**3.3**
**environmental test conditions**
conditions under which a sample (specimen) is tested, including temperature, aerating elements, pH, and identification, volume and exchange rate of the contacting fluid

© ISO 2004 – All rights reserved

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

### 3.4
**free corrosion potential**

corrosion potential in the absence of net (external) electrical current flowing to or from the metal surface

[ISO 8044]

### 3.5
**isotonic NaCl solution**

aqueous solution of NaCl (0,9 % mass fraction) which has the same surface tension as living tissues

NOTE In surgical applications it prevents the collapsing of tissues.

### 3.6
**open-circuit potential**

potential of an electrode measured with respect to a reference electrode or another electrode when no current flows to or from it

[ASTM G 15]

cf. **free corrosion potential** (3.4).

### 3.7
**passive layer**

surface layer on a metal that forms as result of the reaction with the environment, or spontaneously under given conditions, and has the capacity to protect the metal against corrosion

NOTE Passive layers are usually adherent and of submicroscopical thickness (see ISO 8044).

### 3.8
**reference electrode**

electrode having a stable and reproducible potential that is used as a reference in the measurement of electrode potentials

### 3.9
**working electrode**

test or working electrode in an electrochemical cell; the test specimen in the context of this standard

## 4 Significance and application

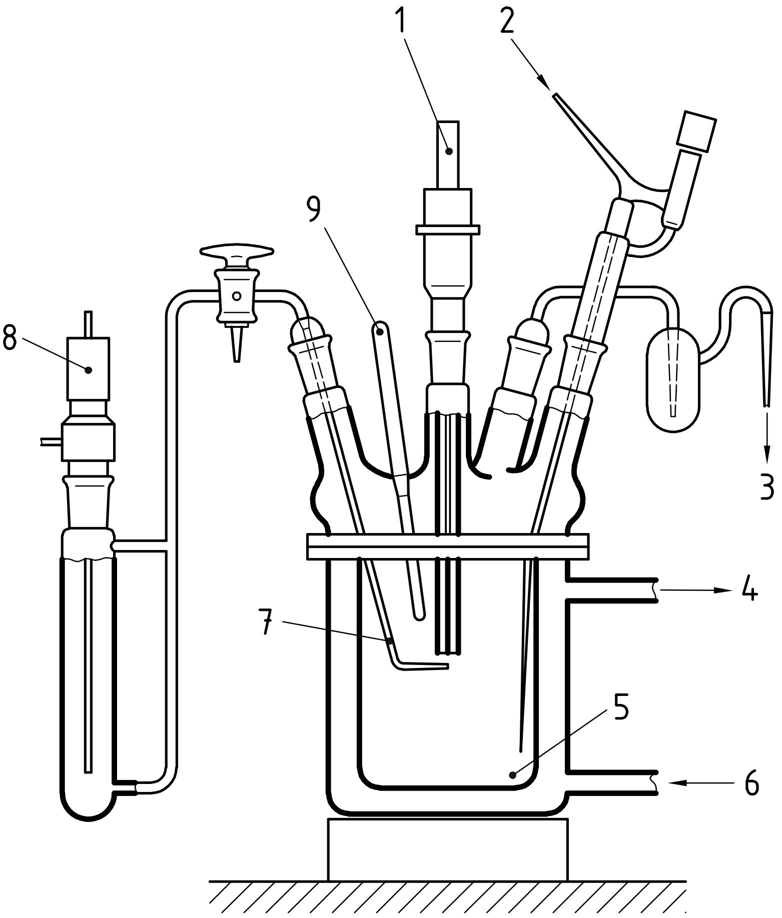
### 4.1 Principle

In this electrochemical test method, the test specimens (samples) are immersed in an isotonic test solution of NaCl[9], which relates to the physiological environment of the body as described in ISO 16428. Other test solutions and the other environmental conditions are also described in ISO 16428. For more stringent test conditions, more acidic test solutions (lower pH) are suggested in Annex A.

The tests are carried out in an electrochemical test cell similar to that described in the ASTM G 5[6]. The test cell provides access for the electrodes and other necessary equipment (Figure 1).

While the specimen is exposed to the environment for a specified period of time, the open-circuit potential is recorded continuously as a function of time. The specimen functions as the working electrode and its behaviour is measured against a reference electrode (Figure 2).

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

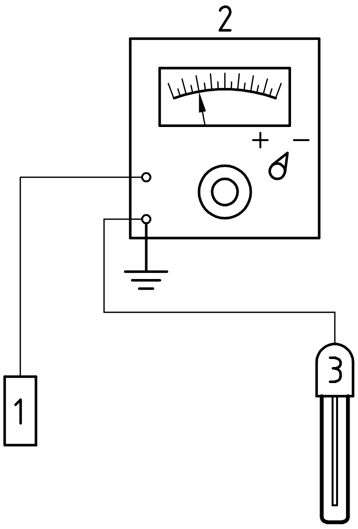


**Key**

| | |
|---|---|
| 1 | working electrode (test specimen) |
| 2 | gas inlet |
| 3 | gas outlet |
| 4 | water outlet |
| 5 | test solution (electrolyte) |
| 6 | water inlet |
| 7 | Luggin probe |
| 8 | reference electrode |
| 9 | thermometer |

**Figure 1 — Example of electrochemical test cell[3]**

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale
--`,``,`-`-`,,`,,`,`,,`---



**Key**

| | |
|---|---|
| 1 | working electrode (test specimen) |
| 2 | potential-measuring instrument (electrometer) |
| 3 | reference electrode |

**Figure 2 — Example of electrical circuit measuring open-circuit potential**

## 4.2 Significance of the measurements

The open-circuit potential reflects (electrochemical) reactions which take place at the surface of the specimen (working electrode) in contact with the testing solution. Depending on the conditions, usually 1 h or 2 h after immersion of the specimen, an equilibrium of the specimen surface with the testing solution (electrolyte) will be reached, and the potential becomes steadier. During the initial phase, with passivating materials the potential increases, usually in the direction of (more) positive values, indicating passivation or increasing passivation in the testing solution with increasing corrosion resistance. A decrease in the potential indicates an activation of the surface with decrease or loss in passivity, and thus susceptibility to corrosion. The value of the steady-state potential, shifts in the potential, and stability or instability of the potential are indicators of processes taking place within the passive film and of the corrosion behaviour of the material under investigation.

NOTE The Cl ions are the aggressive species in the isotonic NaCl testing environment. An increase in passivation in this solution indicates a high initial level of corrosion resistance of the corresponding materials.

This test method is very sensitive, and all steps shall be carried out with caution and understanding of the processes. In particular, the condition of the specimen surface is critical, and preparation procedures require careful attention and reproducibility.

## 4.3 Application

This electrochemical test method is suitable to study, on an extended time scale, the passivation and corrosion behaviour of materials and surgical implant devices as a function of their metallurgical properties, processing history, and surface condition.

Such open-circuit potential measurements, when combined with mechanical tests, can also give information on the mechanical stability of passive films.

This test method can be used for screening of materials, for comparison of materials and surface treatments, for optimization of corrosion resistance, or for the study and characterization of material systems. For extended evaluation, this test method can be used in conjunction with other electrochemical corrosion tests.

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

© ISO 2004 – All rights reserved

NOTE Terms such as corrosion system (3.2) or corrosion potential (3.1) do not mean that visible corrosion has to occur, but rather they are scientific definitions related to corrosion mechanisms and their assessment.

## 5 Apparatus

### 5.1 Electrochemical test cell

The electrochemical test cell consists of a suitable glass vessel, in principle similar to that shown in ASTM G 5[6]. An example of a test cell (flat bottom version) is shown in Figure 1. If the system is not intended to be also used for potentiostatic/potentiodynamic measurements, the counter-electrode (auxiliary electrode) may be removed.

NOTE Borosilicate glass has been proven useful for the glass vessels (ISO 3585[2]). If open-circuit potential measurements are combined with mechanical tests, or for other reasons, the vessel of the test cell can be of a different design and need not consist of glass (ISO 16428).

**5.1.1 Working electrode**, i.e. the test specimen.

**5.1.2 Reference electrode**.

A saturated calomel electrode (SCE) or other electrode may be used. In the latter case, the measurement results shall, however, also be reported in values relating to SCE using accepted conversion values[5] for easy comparison with published data.

**5.1.3 Luggin probe with salt bridge** connecting to the reference electrode.

The distance between the tip of the probe and the specimen surface (working electrode) should be 2 mm or equal to the diameter of the probe tip, whichever is greater.

**5.1.4 Gas inlet** and **gas outlet**.

**5.1.5 Thermometer**, to check the temperature of the testing solution.

**5.2 Potential-measuring instrument (electrometer or corresponding datalogger)** suitable for long-term recording, with impedance $> 10^{11}\,\Omega$ and sensitivity to detect changes of 1 mV.

The measurement range shall cover $-1$ V to $+1{,}5$ V, or more if required by the tested system.

**5.3 pH meter** with a sensitivity of ± 0,1.

**5.4 Thermostatic system** of suitable means (water bath, heating collar, etc.) to stabilize the temperature of the testing solution at $(37 \pm 1)$ °C.

NOTE In testing with a potentiostatic measurement system, platinum is suitable as material for the counter-electrode (auxiliary electrode)[6].

## 6 Specimen preparation

### 6.1 Type of specimen

Within a comparable investigation series, material specimens should have similar form and size (for example rods, cubes, rectangular bars, sheet coupons). If implantable devices are tested, they may be used as they are. If the device needs to be sectioned, the difference between the sectioned and the original surfaces, which may affect the measurements, shall be considered. Covering of the sectioned surfaces with an “inert”, adherent non-conductive substance may be a useful technique; however, it shall be ensured that this does not initiate local corrosion such as pitting or crevice corrosion.

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS Not for Resale

### 6.2 Surface preparation

The preparation of the specimen surface is a critical part of the test, and reproducibility of the procedure is an important requirement. The surface preparation for an open-circuit potential study depends on the purpose of the study, and shall be well specified regarding any special surface treatment (physical, mechanical, thermal, etc.), as well as any grinding/polishing/cleaning/probable drying/probable sterilization/storage and the time elapse between specimen preparation and measurement. Prior to immersion in the test solution, the specimens shall be free of grease and other secondary contamination. This shall be ensured by cleaning in absolute ethanol and rinsing in purified water in accordance with ISO 3696.

If the passivating behaviour of a material is to be observed without the influence of certain surface treatments (such as electropolishing, sterilization, etc.), the specimen surface shall be prepared immediately prior to starting the test by water-grinding with 240- and 600-grit SiC paper, water-rinsing, degreasing in absolute ethanol, and finally rinsing in purified water in accordance with ISO 3696.

The different steps of the surface preparation shall be reported.

### 6.3 Specimen mounting

In general the specimen mounting shall be such that the electrical connection is reliable, that it is outside the electrolyte, and that the specimen mounting does not cause erroneous measurements or local attack such as pitting or crevice corrosion.

Specimens may be mounted with a screw thread to a small rod that has undergone the same surface treatment as the specimen, or the specimen itself may be only partially immersed in the testing solution and a reliable electrical connection mounted on the section of the specimen which is outside the test solution. Within a comparative study, the portions of the specimens immersed in the solution should be the same.

## 7 Environmental conditions

### 7.1 General

In general, the environmental conditions shall be in accordance with ISO 16428.

### 7.2 Testing solution

Isotonic NaCl solution in accordance with ISO 16428 is used as testing solution.

When applied with the necessary technical precautions, a phosphate-buffered sodium chloride solution may also be used (see ISO 16428 and ASTM F 2129[4]).

If more severe conditions are desired to study corrosion behaviour, more acidic NaCl solutions may be used by adding analytical quality hydrochloric acid to establish defined pH values (see Annex A). Alternatively, solutions with multiple quantities of 0,9 % NaCl (A.2) may be applied.

There may be reasons to use other types of testing solutions. Such solutions, as well as any test solution different from the isotonic 0,9 % NaCl solution, shall be reported. Additional solutions are included in ISO 16428 and ASTM F 2129[4].

### 7.3 Testing temperature

The testing solution shall be kept thermostatically stable at $(37 \pm 1)$ °C during the testing period.

--`````,`-`-`,,`,,`,`,,`---

© ISO 2004 – All rights reserved

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

### 7.4 pH value

The pH value shall be recorded at the beginning of the test, at the end of the test, and during the test with a frequency appropriate for the tested system.

### 7.5 Aeration

During testing the solution shall be continuously flushed with gas. Care shall be taken that the gas bubbles do not interfere with the specimen surface and cause disturbance of the measurements.

The testing results may depend on the presence of oxygen in the solution. The solution is purged

a) with pure oxygen to allow for passivation;

or

b) with pure nitrogen (oxygen-free) to simulate depletion of oxygen; in this case the solution is flushed with nitrogen for 0,5 h before the test starts.

For comparison purposes, tests may be carried out with both gases and/or air.

Nitrogen purges require a sealed test cell. To reach steady-state conditions under nitrogen purge, more than 30 min are likely to be required.

### 7.6 Volume of testing solution

For comparable tests, the volume of the test solution shall be the same. In general the test cell shall be filled with 500 ml to 1 000 ml of solution. The ratio of volume testing solution to surface area shall be at least 10 ml/cm$^2$ [5].

## 8 Test procedure

### 8.1 Set-up of test

Fill the test cell with the test solution, and fill the salt bridge with test solution. When the temperature of the solution is stabilized, flush with the selected gas. Then measure the pH. Mount the test specimen, immerse it in the test solution and electrically connect it. Adjust the tip of the salt bridge about 2 mm opposite the specimen surface. Then start recording the open-circuit potential.

### 8.2 Duration of test

Carry out the test for as long as required to obtain data on the stability of the electrode potential of the test system under the given conditions. For a first screening, the open-circuit potential should be recorded for 3 days.

If the open-circuit potential measurements are combined with mechanical testing, the latter will determine the duration of recording.

### 8.3 Observations

Note any irregularities during the testing, as well as discoloration or degradation of the test solution.

After termination of the test, examine the test specimen optically under a low-power microscope for any corrosive surface changes and local attack.

Interpret the test results, based on electrochemical experience and professional skill.

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

## 9 Test report

The test report shall include the following information:

a) purpose of the test;

b) specimen material and its condition;

c) design and dimensions of specimens;

d) for implantable devices, the device designation and production lot number;

e) surface preparation;

f) description of the test cell, specimen mounting, and recording equipment;

g) description of the environmental conditions applied, including

    1) testing solution,

    2) testing temperature,

    3) pH values,

    4) aeration conditions,

    5) volume of testing solution.

h) duration of test;

i) any special conditions or observations;

j) test results (curves of open-circuit potential vs. time, expressed relative to the reference electrode, and where applicable, also converted to SCE values);

k) result of optical examination of specimen(s).

© ISO 2004 – All rights reserved

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

# Annex A
(informative)

# Additional test solutions

## A.1 General

For stringent testing conditions, the open-circuit potential may be measured in testing solutions which contain increased concentrations of Cl ions in order to challenge the stability of the potential and the passivity conditions. Solutions with higher Cl-ion concentrations may be prepared corresponding to either A.1 or A.2.

For comparative testing, one may apply stepwise test solutions with increasing Cl-ion concentrations.

## A.2 NaCl solutions with decreased pH

The isotonic 0,9 % (mass fraction) NaCl solution has a pH of 6,8 to 7,4[9]. To such solution, analytical quality HCl can be added to prepare solutions with the following pH values:

a) pH 6;

b) pH 5;

c) pH 4;

d) pH 3;

e) pH 2.

## A.3 Solutions with increased concentrations of NaCl

Solutions with multiple concentrations of analytical grade 0,9 % (mass fraction) NaCl can be prepared with purified water in accordance with ISO 3696 to give the following concentrations:

a) 1,8 % NaCl;

b) 2,7 % NaCl;

c) 3,6 % NaCl;

d) 4,5 % NaCl.

--`````,`-`-`,,`,,`,`,,`---

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

# Bibliography

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

[2] ISO 3585, *Borosilicate glass 3.3 — Properties*

[3] ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

[4] ASTM F 2129, *Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices*

[5] ASTM G 3, *Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing*

[6] ASTM G 5, *Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements*

[7] ASTM G 15, *Standard Terminology Relating to Corrosion and Corrosion Testing*

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

[9] Pharmacopoeia Helv. VIII, 1997, CH 196 with European Pharmacopoeia (Ph. Eur. 3, 1997) *Isotonic sodium chloride solution 9 g/l infundible* (Natrii chloridi solutio infundibilis *9 g/l*)

© ISO 2004 – All rights reserved

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS Not for Resale

**ICS 11.040.40**

Price based on 10 pages

© ISO 2004 – All rights reserved

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale