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**Metallic and other inorganic  
coatings — Simultaneous thickness  
and electrode potential determination  
of individual layers in multilayer  
nickel deposits (STEP test)**

*Revêtements métalliques et autres revêtements inorganiques —  
Détermination simultanée de l'épaisseur et du potentiel d'électrode  
de couches individuelles dans des dépôts de nickel multicouches  
(essai STEP)*

[ISO 16866:2020](https://standards.iso.org/standards/catalog/standards/sist/38fb50c2-b8a1-4673-8c7e-c8fd5256e0d/iso-16866-2020)

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CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

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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](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by the European Committee for Standardization (CEN) (as EN 16866:2017) and was adopted, without modification, by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

The abbreviated term “STEP” represents “Simultaneous Thickness and Electrode Potential determination”.

The STEP test can be used to measure, in one single operating step, the parameters (thickness of the individual nickel layers and the potential differences among them) relevant for the course of corrosion in a multilayer nickel system. Provided suitable instruments are applied, it can also be used to document them.

The test is a modification of the well-known coulometric method for the measurement of the coating thickness. This method takes advantage of the fact that, following the anodic dissolution of a nickel coating, a potential jump takes place of which the magnitude can be measured against a reference electrode.

Although, nowadays, the STEP test has been incorporated into a number of company standards, particularly in the automobile industry, there are currently no uniform and generally acknowledged potential difference values available. At present, values between 80 mV and 150 mV are assumed for double nickel layers, with the semi-bright nickel layer always being nobler than the bright one.

Likewise, no obligatory numerical values are available, currently, regarding the potential difference between bright nickel layers and existing special nickel layers (e.g. in the case of micro-porous chromium plating). According to the current practical experience, the potential difference is larger than approximately 20 mV, with the bright nickel layer always having to be less noble than the special nickel layer.

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# Metallic and other inorganic coatings — Simultaneous thickness and electrode potential determination of individual layers in multilayer nickel deposits (STEP test)

## 1 Scope

This document specifies a method for measuring the thickness of the individual nickel layers in electroplated multilayer nickel coatings and measuring the potential differences between the individual nickel layers in electroplated multilayer nickel coatings.

The measurement of coatings or layer systems other than electroplated multilayer nickel coatings is outside the scope of this document.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution*

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

ISO 16866:2020

## 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/>

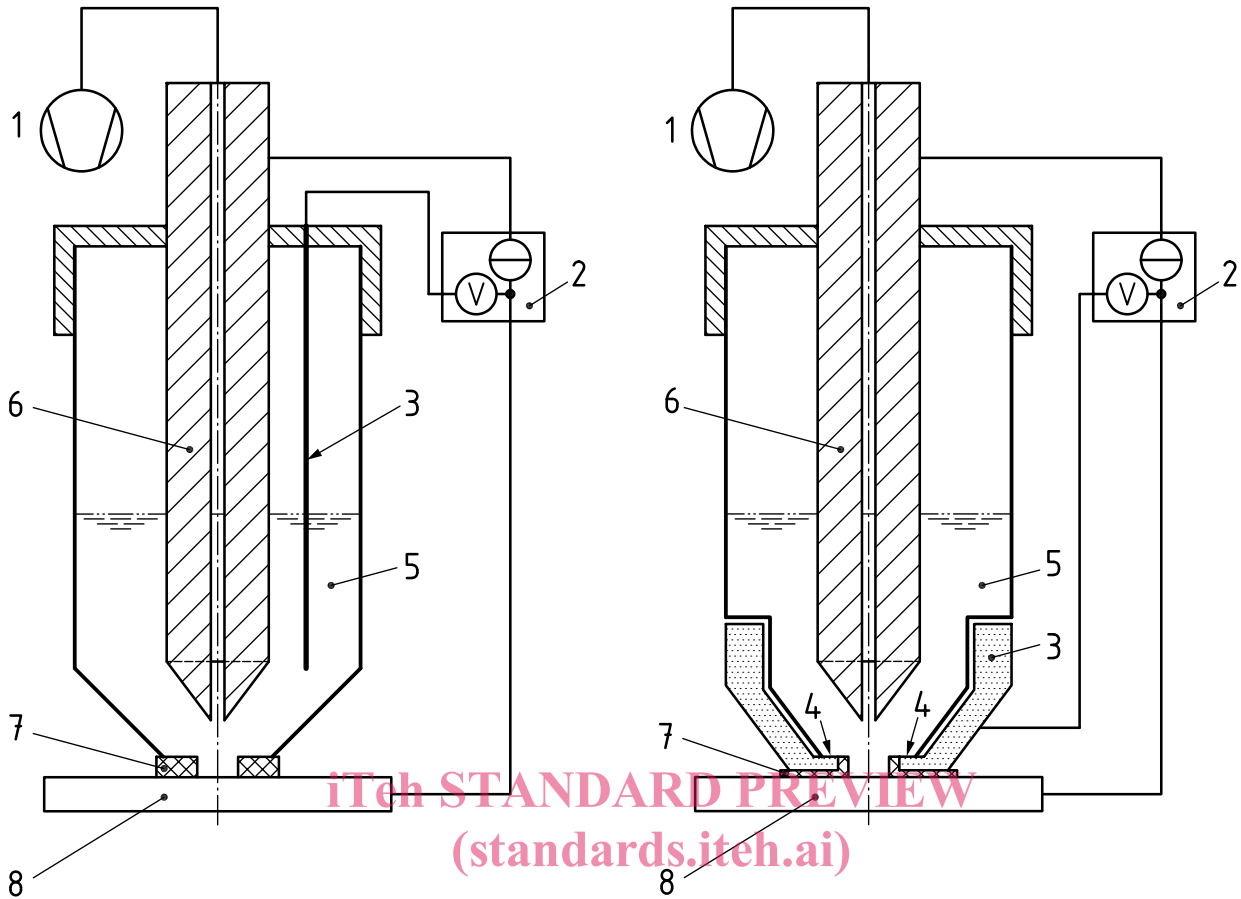
## 4 Test equipment

### 4.1 Construction of the measuring cell

[Figure 1](#) shows two examples of the typical schematic construction of a measuring cell used for the simultaneous determination of layer thicknesses and potential differences in multilayer nickel systems. The cells differ with regard to the implementation of the reference electrode. In [Figure 1 a\)](#), the reference electrode is a silver wire coated with silver chloride and positioned at the edge of the cell; in [Figure 1 b\)](#), it is a silver ring coated with silver chloride and positioned at the bottom of the cell. With regard to measurement uncertainty, both variants provide the same result for the measurement of the potential difference and (following calibration) the measurement of the layer thickness, independent of the concrete implementation of the reference electrode.

NOTE 1 The silver ring used as the reference electrode in [Figure 1 b\)](#) is of advantage insofar as the adjustment of the silver wire, which would otherwise be required, becomes unnecessary, leading to results that are more exact and more reproducible.

NOTE 2 The circulated volume of electrolyte solution is typically around 0,1 ml per s.



**a) Measuring cell with silver wire coated with AgCl, used as the reference electrode**      **b) Measuring cell with silver ring, coated with AgCl in the electrolyte-wetted area, used as the reference electrode**

**Key**

- 1 pump
- 2 measuring instrument (with constant-current source and voltmeter)
- 3 reference electrode
- 4 electrode surface wetted with electrolyte
- 5 dissolving electrolyte
- 6 counter electrode (cathode)
- 7 gasket
- 8 working electrode (anode, measurement object with nickel layer system)

**Figure 1 — Typical schematic constructions of the measuring cell**

**4.2 Composition of the test solution**

Nickel(II)-chloride hexahydrate (NiCl <sub>2</sub> · 6 H <sub>2</sub> O)	300 g/l
Sodium chloride (NaCl)	50 g/l
Boric acid (H <sub>3</sub> BO <sub>3</sub> )	25 g/l
pH value	3,0

Water of grade 3 in accordance with ISO 3696.



The pH value should be complied with as closely as possible. If required, it is adjusted by means of diluted hydrochloric acid or sodium hydroxide solution.

## 5 Requirements

The geometry of the parts to be tested shall be such that the measuring cell can be attached to the surface in a liquid-tight way, at the respective points to be measured.

Prior to this, the surface to be measured shall be appropriately de-chromed and activated (e.g. in a 1:1 dilution of chemically pure, concentrated hydrochloric acid). When selecting the de-chroming and activating procedures, it shall be ensured that the nickel coating is not visibly damaged by these procedures.

## 6 Sampling

Sampling from the ongoing production of parts should be performed in accordance with ISO 2859-1 and ISO 2859-2. The tolerable margin of error is specified by the client.

## 7 Factors influencing measurement accuracy

### 7.1 Electrolyte

Each measurement shall be carried out using a fresh electrolyte. Used electrolytes can result in falsified measurement results.

### 7.2 Conditioning

New reference electrodes or ones which have not been in use for an extended period of time shall be conditioned (creation of a silver chloride layer at the electrode surface) prior to use. The lack of or inadequate conditioning results in falsified or atypical courses of measurement that cannot be correctly interpreted. Any conditioning and potentially required cleaning are carried out in accordance with the instrument manufacturer's information.

**NOTE** The conditioning quality is checked on the basis of comparative measurements using reference standards of known potential differences and layer thicknesses.

### 7.3 Ni deposits

As a result of repeated measurements, Ni is gradually deposited on the surface of the cathode. From a certain thickness onwards, these Ni deposits can become dissolved from the cathode and move around in the electrolyte in the form of "flocs", leading to very noisy measurement curves and a faulty evaluation. It is, therefore, necessary to remove any Ni deposits early enough from the surface of the cathode (this is done mechanically or, in the case of persistent deposits, chemically).

### 7.4 Surface cleanliness

In order to achieve a uniform dissolution process across the whole measurement area, the surface of the test specimen shall be free from contaminations and oxide layers (see also [Clause 5](#)).

### 7.5 Measurement area and contact pressure

Depending on the size and shape of the test specimen, different gaskets defining the measurement area can be selected. The measurement area influences the measurement uncertainty. Usually larger measurement areas result in smaller uncertainties.