# SLOVENSKI STANDARD
# oSIST prEN 16866:2015

**01-september-2015**

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**Kovinske in druge anorganske prevleke - Istočasno določevanje debeline in potenciala elektrode posameznih plasti v večplastnih nikljevih depozitih (preskus STEP)**

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

Metallische und andere anorganische Überzüge - Schichtpotentialmessung von galvanischen Mehrfach-Nickelschichtsystemen (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)

**Ta slovenski standard je istoveten z:** **prEN 16866**

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**ICS:**

| | | |
|---|---|---|
| 25.220.40 | Kovinske prevleke | Metallic coatings |

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

**DRAFT**
**prEN 16866**

May 2015

ICS 25.220.40

English Version

# 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)

Metallische und andere anorganische Überzüge - Schichtpotentialmessung von galvanischen Mehrfach-Nickelschichtsystemen (STEP-Test)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 262.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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# Foreword

This document (prEN 16866:2015) has been prepared by Technical Committee CEN/TC 262 “Metallic and other inorganic coatings”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

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# Introduction

STEP test is an abbreviation for the term "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 and, provided the suitable instruments be applied, to document them as well.

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 whose 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, so far no uniform and generally acknowledged potential difference values are 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, so far, 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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# 1 Scope

This European Standard applies to the measurement of the thickness of the individual nickel layers in electroplated multilayer nickel coatings and to the measurement of 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 European Standard.

# 2 Normative references

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

EN ISO 2177, *Metallic coatings - Measurement of coating thickness - Coulometric method by anodic dissolution (ISO 2177)*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

# 3 Test equipment

## 3.1 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.

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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 some 0,1 ml per second.

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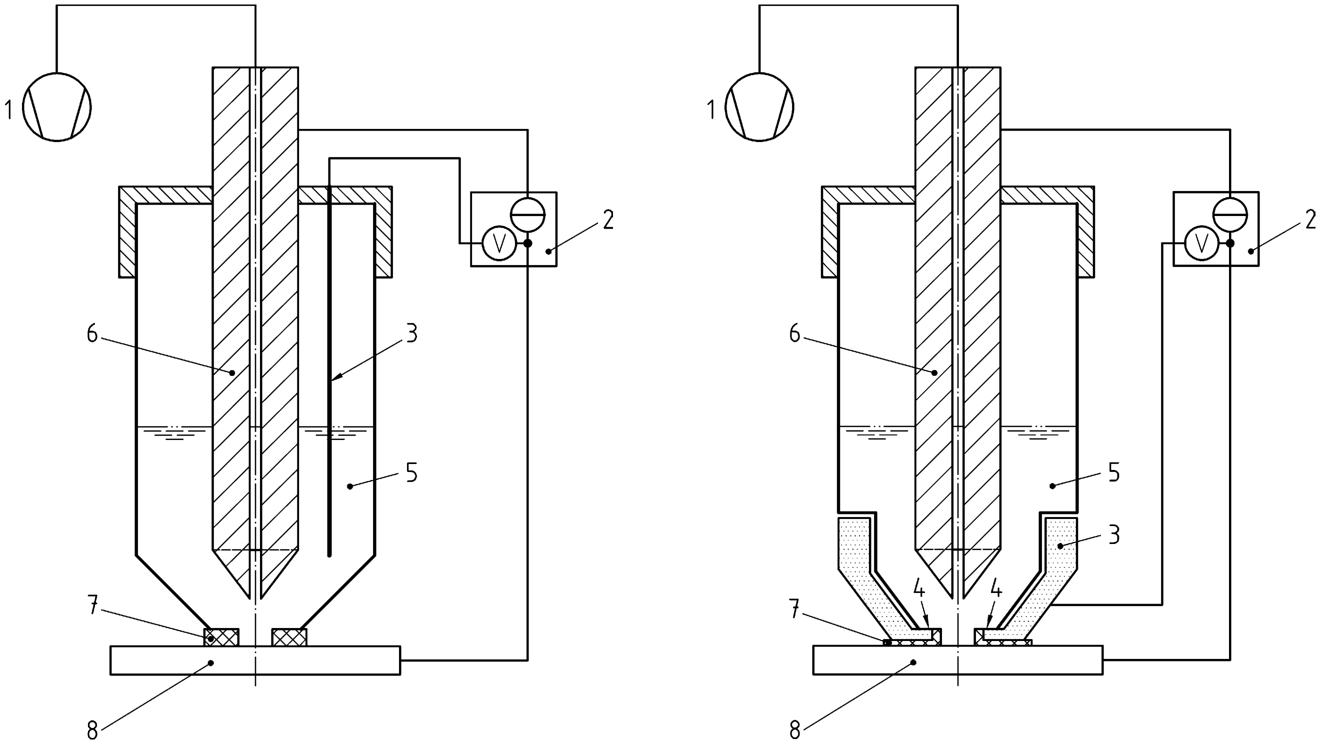

**a)** **Measuring cell with silver wire, coated with AgCl, used as the reference electrode**

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

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**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**

## 3.2 Composition of the test solution

| | |
|---|---|
| Nickel(II)-chloride hexahydrate ($NiCl_2 \cdot 6\ H_2O$) | 300 g/l |
| Sodium chloride (NaCl) | 50 g/l |
| Boric acid ($H_3BO_3$) | 25 g/l |
| pH value | 3,0 |

Water of grade 3 in accordance with EN 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.

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## 4 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.

## 5 Sampling

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

## 6 Factors influencing measurement accuracy

### 6.1 Electrolyte

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

### 6.2 Conditioning

New reference electrodes or ones which had 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. 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.

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### 6.3 Ni deposits

As a result of repeated measurements, Ni is gradually deposited on the surface of the cathode. From a certain thickness on, 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).

### 6.4 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 4).

### 6.5 Contact pressure

The contact pressure exerted on the test specimen by the measuring cell shall be sufficiently high to enable a leakage-free sealing between the cell and the sample surface. Too high a pressure, on the other hand, can lead to a reduction of the active dissolution surface (increase of the sealing surface with regard to a calibration carried out beforehand), thereby causing measurement errors. When placing the measuring cell, the relevant manufacturer's provisions shall be met.

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## 6.6 Electrical contact

Good contacts shall be ensured at the electrical connections of the installation. It is particularly a lack of cleanliness or the occurrence of corrosion at the contact surfaces that leads to increased contact resistances, which, in turn, can result in very noisy courses of measurement.

## 6.7 Complete dissolution

For a correct thickness measurement, the last Ni layer shall have been completely dissolved from the basis material once the measurement is complete. This shall be checked visually following the removal of the measuring cell (with optical means, if appropriate). If there are still any Ni residues detectable, meaning that the dissolution process is incomplete, the measurement shall be rejected.

# 7 Procedure

## 7.1 General

Nickel can be deposited from both plain baths (in the form of dull or semi-bright layers) and from additive-containing baths with different properties regarding brightness, levelling, ductility, hardness, internal stress, porosity, abrasion resistance, etc. The additives change the structure of the precipitation; they are partly incorporated as such and partly in the form of their decomposition products.

Structure and composition manifest themselves by a certain activity of the nickel precipitation that can be determined on the basis of stationary electrochemical measurements.

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In the STEP test, the layer thicknesses are determined by measuring the dissolution time, with the layer thicknesses being calculated in accordance with EN ISO 2177. The dissolution potentials of the different types of nickel coating are obtained by measuring the voltage between the reference electrode and the anode (test specimen).

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NOTE 1 In both measurements, a uniform dissolution rate is achieved by a pre-defined constant dissolution current.

NOTE 2 With most commercially available instruments, the thickness of the dissolved layer is automatically calculated from the dissolution time, and the measured potential curves are represented as a function of the layer thickness [µm] in order to facilitate the evaluation.

## 7.2 Measurement

The work piece previously de-chromed and activated by the appropriate means (see Clause 4 or 6.2 and 6.4, respectively) is positioned in accordance with Figure 1 a) or Figure 1 b) below the measuring cell and pressed against the gasket from below, in a liquid-tight manner (consider 6.5). Following filling of the measuring cell with the test solution (see 3.2) and, if required, adjustment of the conditioned reference electrode, the measurement is performed in accordance with the instrument manufacturer's provisions.

The instrument's measurement accuracy shall be checked in regular intervals by means of a calibration sample. The manufacturer instruction should be considered.

The calibration sample provides both calibration values for the individual layer thicknesses of a multilayer nickel system and the potential difference between the individual nickel layers. For control purposes the total thickness of all nickel layers is stated (measured, e.g. by applying the X-ray spectrometric method in accordance with EN ISO 3497).

## 7.3 Evaluation

Figures 2 and 4 show typical developments of layer thickness/potential curves of multilayer nickel systems, in Figure 3 the coating structure corresponding to the course of measurement given in Figure 2 is represented schematically.