IS 14126 (1994): Metallic coatings - measurement of thickness - coulometric method [MTD 7: Light Metals and their Alloys]

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भारतीय मानक

धात्विक लेपन — ऐनोडी विलयन द्वारा मोटाई लेपन कूलॉमीटरी पद्धति का मापन

Indian Standard

METALLIC COATINGS — MEASUREMENT OF COATING THICKNESS — COULOMETRIC METHOD BY ANODIC DISSOLUTION

UDC 669·053:531·717 [543·432]

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Price Group 5

Metallic and Non-metallic Finishes Sectional Committee, MTD 20

NATIONAL FOREWORD

This Indian Standard is identical with ISO 2177:1985 'Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution' issued by the International Organization for Standardization (ISO), was adopted by the Bureau of Indian Standards on the recommendation of Metallic and Non-metallic Finishes Sectional Committee and approval of the Metallurgical Engineering Division Council.

The text of the ISO Standard has been approved as suitable for publication as Indian Standard; attention is drawn especially to the following :

a) Comma (,) has been used as a decimal marker, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

b) Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'Indian Standard'.

Technical Committee has reviewed the provisions of ISO 2064 to which reference is made in the text and has decided that they are acceptable for use in conjunction with this standard.

In reporting the results of test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard
METALLIC COATINGS — MEASUREMENT OF COATING THICKNESS — COULOMETRIC METHOD BY ANODIC DISSOLUTION

1 Scope and field of application

This International Standard specifies a coulometric method by anodic dissolution for measuring the thickness of metallic coatings.

Typical combinations of coatings and substrates that can be tested by this method are given in table 1. Other combinations may be tested with electrolytes in current use (see annex B), or new electrolytes may be developed for them, but, in both cases, it is necessary to verify the suitability of the complete system.

The method is also applicable to multi-layer systems, for example Cu/Ni/Cr (see also 8.6).

This method may be used to measure the thickness of coatings applied by various means, if due account is taken of special features, such as the presence of an alloy layer. In some cases, the presence and thickness of diffusion layers can also be measured. It can also be used to measure the thickness of coatings on cylindrical specimens and wires (see 8.8).

2 Reference

ISO 2064, *Metallic and other non-organic coatings — Definitions and conventions concerning the measurement of thickness.*

3 Definitions

For the purpose of this International Standard, the definitions of ISO 2064, and the following, apply.

measuring area: The area of the significant surface over which a single measurement is made.

The measuring area for this method is the area enclosed by the sealing ring of the cell.

4 Principle

Anodic dissolution of a well-defined area of the coating using a suitable electrolyte. Detection of the virtually complete dissolution of the coating by a rapid change in cell voltage. Calculation of the coating thickness from the quantity of electricity (in coulombs) used, which may in turn be calculated from

a) the time interval between the start and the end of the test, if it is conducted at constant current density;

b) the integrated quantity of electricity used in dissolving the coatings.

5 Instrumentation

5.1 Suitable instruments may be constructed from readily available components but proprietary instruments are usually used (see annex A).

5.2 Proprietary direct reading instruments are available for use with electrolytes recommended by the manufacturer. Other instruments record the quantity of electricity, in coulombs, used in dissolving the coating from the measuring area (see clause 3), usually in arbitrary units, from which the thickness is calculated using factors or tables.

With direct reading instruments, the calculation of thickness from current density is made electronically.

5.3 The performance of the instrument shall be checked using specimens of known coating thickness. If the instrument readings agree to ±5 % of the known thicknesses of the specimens, the instrument may be used without further adjustment. Otherwise, the cause of the discrepancy shall be removed. Proprietary instruments shall always, however, be calibrated in accordance with the manufacturer's instructions.

Suitable specimens of known coating thickness shall consist of the same type of coating and substrate as the specimen to be measured, and they shall have an accuracy of 5 % or better. If measuring alloy coatings, the use of correct specimens is of particular importance.

6 Electrolyte

The electrolyte shall have a known, adequate shelf-life and shall be such that:

a) there is no reaction with the coating metal in the absence of an impressed flow of current;

b) the coating dissolves anodically at an efficiency as close to 100 % as possible;

Table 1 — Typical combinations of coatings and substrates which can be tested by the coulometric method

Coating	Substrate (basis material)							
	Aluminium*	Copper and copper alloys	Nickel	Nickel-cobalt-iron alloys (such as Kovar)	Silver	Steel	Zinc	Non-metals
Cadmium	X	X	X	—	—	X	—	X
Chromium	X	X	X	—	—	X	—	X
Copper	X	Only on brass and copper-beryllium	X	—	—	X	X	X
Lead	X	X	X	X	X	X	—	X
Nickel	X	X	—	X	—	X	—	X
Nickel, autocatalytic**	X	X	X	X	—	X	—	X
Silver	X	X	X	—	—	X	—	X
Tin	X	X	X	—	—	X	—	X
Tin-lead alloys***	X	X	X	X	—	X	—	X
Zinc	X	X	X	—	—	X	—	X

* The detection of the change in the cell voltage may be difficult with some aluminium alloys.

** The coulometric method can only be used if the phosphorus or boron contents of these coatings are within certain limits.

*** The method is sensitive to alloy composition.

c) a detectable sharp change in electrode potential occurs as the coating is penetrated and an increasing area of substrate is exposed;

d) the test area exposed in the test cell is completely wetted.

The electrolyte shall be chosen in relation to the coating and substrate materials, the current density and electrolyte circulation within the test cell. Typical electrolytes that have been found satisfactory for use with one type of test apparatus for testing various electrodeposited coatings on specific substrates are described in annex B.

For proprietary instruments, the electrolytes shall normally be chosen on the recommendations of the manufacturer.

7 Factors affecting the measuring accuracy

The following factors may affect the accuracy of coating thickness measurements.

7.1 Coating thickness

Generally, the accuracy is lower than optimum for coating thicknesses greater than 50 µm and less than 0,2 µm, unless special equipment is used.

With coatings thicker than 50 µm, there may be an appreciable amount of bevelling or undercutting as the anodic dissolution proceeds. The amount of bevelling is largely dependent on the method employed for stirring the electrolyte. Undercutting may be eliminated, or reduced, by increasing the rate of dissolution, i.e. by increasing the current density used in the test.

7.2 Current variation

For instruments using the constant current and time measuring technique, current variation will cause errors. For instruments using a current-time integrator, too large a change in current may change the anode current efficiency or interfere with the end-point causing an error.

7.3 Area variation

The accuracy of the thickness measurement will be no better than the accuracy to which the measuring area is known. Area variations due to gasket wear, gasket pressure, etc., may lead to measurement errors. Much greater accuracy can be obtained if the electrolytic cells are so designed that sealing rings consistently give well-defined measuring areas.[1] In some cases it may be advantageous to measure the area after it has been deplated and compensate accordingly.

7.4 Agitation (if required)

Inadequate agitation can cause a false end-point.

7.5 Alloy layer between coating and substrate

The measurement of coating thickness by the coulometric method assumes implicitly that a sharply defined interface exists between the coating and the substrate. If an alloy layer

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[1] Error due to variations of the measuring area may, in some instances, be minimized by modifying the instrument calibration using coating thickness calibration standards. Such standards should create similar test conditions to those during actual tests, particularly if testing curved surfaces.

exists between the coating and the substrate as, for example, in the case of coatings applied by hot dipping[1], the coulometric end-point may occur at some point within the alloy layer, thus giving a high value for the thickness of the unalloyed coatings.

7.6 Purity of coatings

Materials that co-deposit with a coating metal (including alloying metals) may change the effective electrochemical equivalent of the coating metal, the anode current efficiency, and the coating density.

7.7 Condition of test surface

Oil, grease, paint, corrosion products, polishing ingredients, conversion coatings, passivity of nickel coatings, etc., may interfere with the test.

7.8 Density of coating material

Because the coulometric method measures intrinsically mass per unit area, variations in density from the normal density of the coating metal will cause corresponding variations in linear thickness measurements. Normal variations of the composition of an alloy result in small, but significant changes in alloy density and its electrochemical equivalent.

7.9 Cleanliness of the cell

Deposition of metal may take place on the cathode in some electrolytes. This deposit can alter the cell voltages or block the cell aperture. It is, therefore, essential to keep the cathode clean.

7.10 Cleanliness of electrical connections

In the case of instruments other than the constant current type, if the electrical connections are not clean, the current/potential relationship will be disturbed and false end-points obtained.

7.11 Calibration standards (if used)

Measurements made using calibration standards are subject to the additional error of the standards. If the thickness of alloy coatings is being determined, it is usually necessary to use coating standards and to test them using the same procedure.

7.12 Non-uniform dissolution

If the rate of dissolution is not uniform over the measuring area, a premature end-point may be obtained, and yield low results. Hence, one should examine the surface after the test to verify that most of the coating has dissolved. On some substrates, however, a visible, but insignificant, portion of the coating may remain.

The presence of other material in the coating, the roughness of the coating surface and interface and the presence of porosity in the coating can cause fluctuation of the cell voltage. Such fluctuations can prematurely affect the end-point.

8 Procedure

8.1 General

If commercial equipment is used, follow the manufacturer's instructions with respect to the operating procedure for measurement, the electrolytes and, if necessary, calibration (see 5.3). Appropriate attention shall be given to the factors listed in clause 7.

8.2 Pre-set voltage

If using instruments that require a pre-set voltage, it should be noted that the actual value is dependent on the particular metallic coating, current density, electrolyte concentration and temperature, and circuit resistance, for example lead-out connections. For these reasons, it is considered advisable to first perform an evaluation test.

8.3 Preparation of test surface

If necessary, clean the test surface (see 7.7) with a suitable organic solvent. It may also be necessary to activate the test surface by mechanical or chemical means, but care should then be taken to avoid removal of metal.

8.4 Cell application

Press the electrolytic cell, fitted with its flexible sealing ring, on to the coating so that a known area is exposed to the test electrolyte. If the cell body is metallic, for example stainless steel, it normally forms the cell cathode, but, otherwise, insert a suitable cathode (incorporated in some instruments as part of the electrolyte agitation mechanism).

8.5 Electrolysis

Introduce the appropriate electrolyte and make sure that no air bubbles occur on the measuring surface. If required, put the agitation mechanism into the cell. Make the electrical connections, and operate the agitator as appropriate. Continue electrolysis until dissolution of the coating is complete, as indicated by a sharp change in the anode potential or cell voltage, or by the operation of the automatic cut-out.

8.6 Undercoats

When one or more undercoats are to be measured, after measuring the top coating ensure that the top coat has first been completely removed from the whole of the measuring area. Remove the electrolyte from the cell with a suitable suction device, and thoroughly rinse out the cell with distilled or demineralized water.

Do not disturb the cell at any time during these operations. Abandon the test if the slightest displacement has occurred.

Reset the instrument controls for the next coating, introduce the appropriate electrolyte and continue the test as before.

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[1] See the footnote to clause A.1.

8.7 Examination after test

After completion of the test(s), remove the electrolyte from the cell, rinse out with water, lift off the cell and examine the specimen to ensure that the removal of the coating is complete over the area enclosed by the seal (see 7.12).

8.8 Coatings on cylindrical specimens

If the surface area is too small to use the normal cell with a flexible gasket, replace it by an electrolyte container and an appropriate holding device, using an agitator if necessary. This device shall be adjustable and shall be pre-set to allow a known length of specimen to be immersed. For direct reading instruments, especially those with alternative cell sizes, calculate the length of specimen to be immersed so that the same known surface area is presented to the cathode as for a test cell.

In the majority of applications, the same electrolyte can be used but, in order to obtain the optimum instrument sensitivity and accuracy, the operating conditions may have to be modified, for example the cut-off voltage and deplating current.

NOTE — An exact deplating area is necessary for accuracy and the main source of error is due to the meniscus and current field at the electrolyte surface.

9 Expression of results

The coating thickness, d, in micrometres, is given by the equation

$$d = 100k \frac{QE}{A\varrho}$$

where

k is the current efficiency of the dissolution process (equal to 100 in the case of 100 % efficiency);

E is the electrochemical equivalent, in grams per coulomb, of the coating metal for the conditions of the test;

A is the area, in square centimetres, from which the coating is dissolved, i.e. the measuring area;

ϱ is the density, in grams per cubic centimetre, of the coating;

Q is the quantity of electricity, in coulombs, passed in dissolving the coating; if an integrating meter is not used, calculate Q from the equation

$$Q = It$$

where

I is the current, in amperes,

t is the test duration, in seconds.

The thickness may also be expressed as:

$$XQ$$

where X is a constant for a given metal coating, electrolyte and cell. The value of X can either be calculated theoretically from the area of the test specimen exposed by the sealing ring, the efficiency of anodic dissolution (usually 100 %) and the electrochemical equivalent and density of the coating metal, or can be determined experimentally by measuring a coating of known thickness.

With most commercial instruments, the thickness is either read directly from the instrument or the instrument reading is converted to thickness by applying a factor appropriate to the measuring area exposed by the cell and the coating metal.

10 Measurement uncertainty

The test equipment and the procedure shall be such that the coating thickness can be measured to within 10 % of its true thickness.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) an identification of the test specimen;

c) the area, in square centimetres, over which the measurements were made;

d) the location of the reference area;

e) the location(s) on the coated item at which the tests were carried out;

f) the identity of the electrolyte used;

g) the measured thickness in micrometres at each area at which the tests were carried out; the number of measurements averaged for each reported measurement;

h) the name of the operator and testing laboratory;

j) the date;

k) any deviation from the method specified;

m) any factor that may have influenced the result;

n) an identification of the instrument used.

Annex A

Types of instrument

(This annex is for information only and does not form part of this International Standard.)

A.1 General

The instrument may operate on either of two principles:

a) measurement of the time of anodic dissolution at constant cell current;

b) measurement of the quantity of electricity consumed during the test by current·time integration.

With the former, the current passed through the test cell has to be controlled at a constant value, a timer then being used to measure the time interval between the start and the end-point of the test. With the latter, an integrating meter is used to measure the quantity of electricity passed, in which case accurate knowledge of the magnitude of the current and separate measurement of the time interval are unnecessary.

The results may be displayed as single time units, as the product of current and time (quantity of electricity) units or, more frequently with recent designs of apparatus, directly as thicknesses.

The end-point can be determined by observing the marked change in cell voltage on a suitable voltmeter, or a cut-out can be used to terminate the test automatically. In the latter case, the cut-out is set to operate at a predetermined value[1] of either the cell voltage or the rate of increase of cell voltage.

Other non-mandatory, but advantageous, features of coulometric instruments may include digital results display, electronic timing and switching for greater accuracy of end-point determination, and the capability to accept different test cell sizes or use different current densities. The test cell sealing ring should be capable of being interchanged rapidly.

Many modern instruments display results as direct thickness measurements and may incorporate initial automatic setting of the cut-out at the start of each test to terminate the test at the correct end-point.

A.2 Electrolytic cell

The electrolytic cell consists of a container, usually cylindrical, applied to the article under test through an intermediate non-conductive and elastic seal made, for example, of rubber or plastics material. The cell itself, if it is made of metal, for example stainless steel, can serve as the cathode, in which case the seal will also serve as insulation between the cathode and anode.

If the cell is of insulating material, a separate cathode is used and immersed in the electrolyte before starting the test.

The area of surface enclosed by the seal has to be well defined and small enough to permit its application to a curved surface. Smaller cells may be required for measuring the thickness of coatings on intricately shaped substrates, but these pose problems connected with the definition and measurement of the area of the seal. With any cell, the accuracy of the method is largely controlled by the accuracy of measurement of the area over which the thickness measurement is made. Inaccuracies can occur if the seal defining the test area is applied on a curved surface. A further inaccuracy can also arise from the wear or distortion of the end of seal. This can be assessed visually by examination of the periphery of the circle of stripped coating.

For substantially flat surfaces, a typical deplated area is 0,2 cm^2, but curved surfaces can be measured with cells of the sizes indicated in table 2, depending on the diameters of the deplated areas.

Table 2 — Cell sizes for measurements on curved surfaces

Diameter of deplated area	Deplated area	Minimum diameter of curved surfaces
cm	cm^2	cm
0,32	0,080	3,0
0,22	0,038	1,0
0,15	0,018	0,4

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[1] In the case of hot-dipped coatings, or other coatings where a diffusion layer may have developed between the coatings and the basis metal, this predetermined value may be either that at which the main coating has been removed to expose the diffusion layer or that at which the diffusion layer has been removed to expose completely the basis metal.

Annex B

Typical electrolytes

(This annex is for information only and does not form part of this International Standard.)

B.0 Introduction

Although it is possible to use higher current densities with some proprietary electrolytes (and possibly with some of the electrolytes listed in this annex), the following electrolytes (B.1 to B.16) were formulated to give substantially 100 % anodic efficiency with current densities ranging from 100 to 400 mA/cm^2. However, a few electrolytes were found only to be suitable when used towards the lower or upper ends of this current density range and these electrolytes are marked with an asterisk.

These electrolytes dissolve the metallic coatings at substantially 100 % anodic efficiency. Therefore, the thickness, in micrometres, can be calculated with satisfactory accuracy from the expression

$$10\,000 \frac{QE}{A\rho}$$

or from instrument factors calculated from this expression. (See clause 9 for definitions of the symbols.) There is no need to use coating standards when using these electrolytes. Direct calculation from the above expression will normally give more accurate results than those based on calibration of coulometric instruments with coating standards.[1]

The solutions given in B.1 to B.4 should preferably be made up from reagents of recognized analytical grade and using distilled or demineralized water. Small variations in concentration of the solutions will not affect the accuracy of the results, but may affect the voltage settings in an automatic cut-out mechanism, based on a pre-set voltage, is employed. All the electrolytes, except B.9, have shelf-lives in excess of 6 months.

The instrument manufacturer's advice or instructions shall be followed regarding whether any of these electrolytes may be used with their instruments or whether it is necessary to use proprietary electrolytes for any particular coating/substrate combination. A summary of the applicability of the electrolytes described in clauses B.1 to B.16 is given in table 3.

B.1 Electrolyte for cadmium coatings on steel, copper or brass

Prepare a solution containing 30 g of potassium chloride (KCl) and 30 g of ammonium chloride (NH_4Cl) per litre.

This electrolyte requires a critical pre-set voltage.

Table 3 — Applicability of electrolytes

Coating	Substrate (basis material)					
	Aluminium	Copper and copper alloys (such as brass)	Nickel	Steel	Zinc	Non-metals
Cadmium	—	B.1	—	B.1	—	B.1
Chromium	B.2 and B.4	B.3	B.2 and B.4	B.2	—	B.2 and B.4
Copper	B.5 and B.6	—	B.6	B.5	B.7	B.5, B.6 and B.7
Lead	—	B.8	B.8	B.8	—	B.8
Nickel	B.9	B.10	—	B.9	—	B.9 and B.10
Silver	—	B.11	B.11	—	—	B.11
Tin	B.13	B.12	B.12	B.12	—	B.12 and B.13
Tin-nickel alloys	—	B.16	—	B.15	—	B.15 and B.16
Zinc	—	B.14	—	B.14	—	B.14

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[1] Both direct calculation and use of calibration standards have inherent errors which have to be considered, for example an error in the diameter of the test cell of 3 % will give a 9 % error in measurement.

B.2 *Electrolyte for chromium coatings on steel, nickel or aluminium

Dilute 95 ml of orthophosphoric acid (H_3PO_4) solution, ϱ = 1,75 g/ml, to 1 000 ml with water and add 25 g of chromium(VI) oxide (CrO_3).

WARNING — Orthophosphoric acid causes burns: avoid contact with eyes and skin.

Chromium(VI) oxide may cause fire on contact with combustible material and causes severe burns: avoid inhaling dust and prevent contact with eyes and skin.

This solution is only suitable for use at a current density of about 100 mA/cm^2 and for coatings up to 5 µm thick, when the accuracy of measurement is probably ± 10 %.

NOTE — Anodic dissolution of chromium in this electrolyte — and in electrolytes B.3 and B.4 — produces hexavalent chromium ions Cr(VI) and the electrochemical equivalent for Cr(VI) shall be used when calculating the thickness.

B.3 *Electrolyte for chromium coatings on copper or brass

Prepare a solution containing 100 g of sodium carbonate (Na_2CO_3) per litre.

This electrolyte is only suitable for use at a current density of about 100 mA/cm^2, and for coatings up to 5 µm thick.

B.4 *Electrolyte for chromium coatings on nickel or aluminium

Dilute 64 ml of orthophosphoric acid (H_3PO_4) solution, ϱ = 1,75 g/ml, to 1 000 ml with water.

WARNING — See clause B.2.

This electrolyte is best used at a current density at about 100 mA/cm^2 and is, therefore, particularly useful on thin or decorative chromium coatings. (See also the note to clause B.2.)

B.5 Electrolyte for copper coatings on steel or aluminium

Dissolve 800 g of ammonium nitrate (NH_4NO_3) in water, dilute to 1 000 ml and add 10 ml of ammonia solution (NH_3), ϱ = 0,88 g/ml.

WARNING — Ammonium nitrate may cause fire on contact with combustible material. Keep out of contact with all combustible material.

Ammonia causes burns and is irritating to eyes, respiratory system and skin: avoid breathing vapour and prevent contact with eyes and skin.

This electrolyte gives thickness results that are about 1 to 2 % lower than the correct value.

B.6 Electrolyte for copper coatings on nickel or aluminium

Dissolve 100 g of potassium sulfate (K_2SO_4) in water, dilute to 1 000 ml and then add 20 ml of orthophosphoric acid (H_3PO_4) solution, ϱ = 1,75 g/ml.

WARNING — See clause B.2.

B.7 Electrolyte for copper coatings on zinc or zinc die-cast alloys

Use pure hexafluorosilicic acid (H_2SiF_6), of concentration not lower than approximately 30 % (*m*/*m*).

WARNING — Hexafluorosilicic acid causes burns and is toxic by inhalation, in contact with skin and if swallowed: avoid inhaling vapour and prevent contact with eyes and skin.

This solution dissolves copper coatings with 100 % efficiency at quite low voltages, with practically no anodic attack on the zinc substrate exposed at the end of the test. There is, however, a tendency to leave slight traces of copper, as spots, on the zinc, in the test area but, although noticeable, they are normally too small to affect significantly the accuracy of the results.

It is important to note that:

a) a pure grade of hexafluorosilicic acid is required, substantially free from impurities such as chlorides and sulfates which would allow anodic attack on the zinc substrate when it is exposed at the end of the test;

b) the acid should not have too high a water content, which would lead to an effect similar to that described in a). If the water content of the available acid is too great, its deleterious effect can sometimes be overcome by dissolving a little magnesium hexafluorosilicate in the acid.

B.8 Electrolyte for lead coatings on steel, copper or nickel (with or without tin undercoats)

Prepare a solution containing 200 g of sodium acetate (CH_3COONa) and 200 g of ammonium acetate (CH_3COONH_4) per litre.

This electrolyte tends to give slightly lower than 100 % efficiency, but will probably give results that are not more than about 5 % higher than the correct value.

B.9 Electrolyte for nickel coatings on steel or aluminium

Dissolve 800 g of ammonium nitrate (NH_4NO_3) (see the warning to clause B.5) in water, dilute to 1 000 ml and add 50 ml of a 76 g/l thiourea [$CS(NH_2)_2$] solution.

This mixed solution has rather a short shelf-life and should thus be made up not more than 5 days before use, preferably from stock solutions of 800 g/l ammonium nitrate solution and 76 g/l thiourea solution, which have shelf-lives of at least 6 months.

NOTE — The efficiency of this electrolyte is reduced by passivity of nickel coatings: it will not dissolve nickel with 100 % efficiency if its surface is passive. Nickel surfaces can also become passive by the anodic action of phosphoric acid electrolyte used to remove chromium top coats before measuring the thickness of the nickel coating.

The cell voltage during the test can serve as a guide to this behaviour. If testing at about 400 mA/cm^2, the cell voltage for dissolving nickel with 100 % efficiency is normally less than 2,4 V, while a cell voltage of 2,5 V or more usually indicates either that the nickel is dissolving at much less than 100 % efficiency, usually accompanied by electropolishing of the nickel, or that oxygen is being evolved without any dissolution of the nickel.

If necessary, the nickel can be activated before the test by introducing a little dilute hydrochloric acid [$1\ \text{mol/l} < c(\text{HCl}) < 2\ \text{mol/l}$][1] solution into the cell, removing it after about 0,5 to 1 min and rinsing well before introducing the ammonium nitrate/thiourea electrolyte to test for nickel thickness.

B.10 *Electrolyte for nickel coatings on copper, brass or other copper alloys or on stainless steel

Dilute 100 ml of hydrochloric acid (HCl), ϱ = 1,18 g/ml, to 1 000 ml with water.

WARNING — Hydrochloric acid causes burns and is irritating to the respiratory system: avoid breathing the vapour and prevent contact with eyes and skin.

This electrolyte is only satisfactory for nickel coatings on copper or copper alloys when used at current densities of about 400 mA/cm^2, but not when used at about 100 mA/cm^2.

NOTE — Electrolytes B.9 and B.10 are also suitable for testing the thickness of cobalt, cobalt-nickel or nickel-iron alloy coatings. The electrochemical equivalents of cobalt and iron are very close to that of nickel, thus no significant error occurs when the thicknesses of alloys of these metals are calculated as though they were nickel alone.

B.11 Electrolyte for silver coatings on copper, copper alloys or nickel

Prepare a 100 g/l potassium fluoride (KF) solution.

WARNING — Potassium fluoride is toxic by inhalation, in contact with skin and if swallowed: avoid inhalation of dust and prevent contact with eyes and skin.

This electrolyte is satisfactory for dull silver coatings or bright silver coatings incorporating sulfur-containing brighteners, but is not as satisfactory for bright silver alloy coatings containing small percentages of antimony or bismuth.

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[1] Hitherto expressed as "1 N to 2 N".

NOTE — When testing silver coatings with electrolyte B.11, there is a tendency for the silver to be deposited as metal on the inside of stainless steel cells. The deposit is, however, in the form of a uniform coating, that does not block the aperture, but it can lower the cell voltage needed for dissolution of the silver coatings. It is, therefore, desirable to dissolve away the silver deposit from stainless steel cell bodies with nitric acid after each determination.

B.12 Electrolyte for tin coatings on steel, copper alloys or nickel

Dilute 170 ml of hydrochloric acid (HCl), ϱ = 1,18 g/ml, to 1 000 ml with water.

WARNING — See clause B.10.

This solution dissolves tin coatings at very low cell voltages at which there is no anodic attack on the substrates when they are exposed at the end of the test. There is, however, a tendency for the tin to be deposited out of this solution during the test as a spongy deposit on the cathode, for example on a stainless steel cell body, and this deposit can block the cell aperture after a while and thus prematurely stop the test when testing very thick tin coatings or a number of thinner ones. The aperture of the cell should therefore be cleaned of these growths at fairly frequent intervals.

NOTE — The electrolyte is believed to have 100 % efficiency.

B.13 *Electrolyte for tin coatings on aluminium

Dilute 50 ml of sulfuric acid (H_2SO_4), ϱ = 1,84 g/ml, with water to 1 000 ml by carefully adding the acid, in small portions, to the water, and dissolve 5 g of potassium fluoride (KF) in this solution.

WARNING — See clause B.11. (Sulfuric acid causes severe burns: prevent contact with skin and eyes and do not add water to sulfuric acid.)

B.14 Electrolyte for zinc coatings on steel, copper or brass

Prepare a 100 g/l potassium chloride (KCl) solution.

This electrolyte requires a fairly critical pre-set voltage, but not quite as critical as that for testing cadmium coatings (clause B.1).

B.15 *Electrolyte for tin-nickel alloys on steel

Mix 100 ml of orthophosphoric acid (H_3PO_4) solution, ϱ = 1,75 g/ml, with 50 ml of hydrochloric acid (HCl), ϱ = 1,18 g/ml, and 50 ml of a solution of oxalic acid dihydrate ($C_2H_2O_4 \cdot 2H_2O$), saturated at room temperature.

WARNING — See clauses B.2 and B.10. (Oxalic acid is harmful in contact with skin and if swallowed: avoid contact with eyes and skin.)

This electrolyte was formulated for use at a current density of about 100 mA/cm^2 and at this current density the tin in the alloy was found to dissolve as tin(II) ions. The correct electrochemical equivalent for a 65/35 tin-nickel alloy, with tin in the divalent form, i.e. 0,505 mg/C, should be used for calculating the thickness. For greater accuracy the factor should be adjusted for the actual composition of the alloy (see 7.8).

B.16 *Electrolyte for tin-nickel alloys on copper or brass

Prepare a solution containing 12 g of nickel chloride hexahydrate ($NiCl_2 \cdot 6H_2O$), 13 g of anhydrous tin(IV) chloride ($SnCl_4$), 200 ml of water, 40 ml of hydrochloric acid (HCl), ϱ = 1,18 g/ml, and 50 ml of orthophosphoric acid (H_3PO_4) solution, ϱ = 1,75 g/ml.

WARNING — See clauses B.2 and B.10. (Nickel chloride has a harmful dust and is irritating to skin and eyes: avoid breathing dust and avoid contact with eyes and skin.)

Tin(IV) chloride causes burns and is irritating to the respiratory system: avoid contact with eyes and skin and do not allow to come into contact with water inadvertently.

This electrolyte was formulated for use at a current density of about 400 mA/cm^2 and at this current density the tin in the alloy was found to dissolve as tin(IV) ions. The correct electrochemical equivalent for a 65/35 tin-nickel alloy, with tin in the quadrivalent form, i.e. 0,305 mg/C, should be used for calculating the thickness. For greater accuracy the factor should be adjusted for the actual composition of the alloy (see 7.8).

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