INTERNATIONAL STANDARD ISO 2177

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Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution

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FOREWORD

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INTERNATIONAL STANDARD **ISO 2177-1972 (E)**

Metallic coatings – Measurement of coating thickness – Coulometric method by anodic dissolution

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a procedure for measuring the thickness of metallic coatings by the anodic dissolution method. The electrodeposited coatings and substrates to which it applies are given in Table 1. It may also be used to measure the thickness of coatings applied by other means, if due account is taken of special features such as the presence of an alloy layer.

2 PRINCIPLE

Anodic dissolution of the coating from a well-defined area of the coated surface using a suitable electrolyte.

Indication of the end point of the test by the change in electrode potential (and hence cell voltage) occurring when the coating is virtually completely removed.

Calculation of the coating thickness either

– from the time interval between the start and end of the test if the test is conducted at constant current density; or

– from the quantity of electricity, recorded directly by an integrating meter, consumed in dissolving the coating.

3 TEST APPARATUS

Various types of apparatus may be constructed from readily available components (see Annex A). Suitable complete instruments may also be purchased.

4 ELECTROLYTE

The electrolyte to be used shall be such that :

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

– the coating dissolves anodically at 100 % efficiency (or at a nearly constant value close to this);

– a marked change in electrode potential occurs as the coating is penetrated and an increasing area of substrate is exposed.

The electrolyte must be chosen in relation to the coating and substrate metals, the current density and electrolyte circulation within the test cell. Typical electrolytes, which have been found satisfactory for use with two types of test apparatus for testing various electrodeposited coatings on specific substrates, are given in Table 2, their applications being listed in Table 1. The electrolytes satisfactory with one type of apparatus are indicated by symbols A1, A2, etc., and those satisfactory with a second type of apparatus by symbols B1, B2, etc.

TABLE 1 – **Application of typical electrolytes (see Table 2) for electrodeposited coatings**

Coating	**Substrate (Basis metal)**				
	Steel	Copper and copper alloys such as brass	Nickel	Aluminium	Zinc
Cadmium	A1 B1	A1 B1	A1 –	A1 –	– –
Chromium	A2 B2	A3 B3	A2 B4	A2 B4	– –
Copper	A4 B5	– –	A4 B6	A4 –	– B7
Lead	– B8	– B8	– B8	– –	– –
Nickel	A5 B9	A5 B10	– –	A5 B9	– –
Silver	A6 –	A7 B11	A6 –	– –	– –
Tin	A3 B12	A3 B12	A3 –	A2 B13	– –
Zinc	A8 B14	A8 B14	A8 –	A8 –	– –

NOTE – These electrolytes cannot be adapted to all apparatus and when certain commercial instruments are used, it is therefore desirable to follow the supplier's recommendations.

TABLE 2 – **Typical electrolytes**

No.	Electrolyte		Notes
A1	**Potassium iodide** (KI) : 100 g/l, with traces of iodine		
A2	**Sodium sulphate** (Na_2SO_4) : 100 g/l		
A3	**Hydrochloric acid** (HCl) : 73 g/l or **Sodium hydroxide** (NaOH) : 150 g/l		Dilute 175 ml of hydrochloric acid ($d = 1.18$) to 1 litre
A4	**Sodium potassium tartrate** ($NaKC_4H_4O_6$) : 80 g **Ammonium nitrate** (NH_4NO_3) : 100 g	in 1 litre	
A5	**Ammonium nitrate** (NH_4NO_3) : 30 g **Sodium thiocyanate** (NaCNS) : 30 g	in 1 litre	
A6	**Sodium nitrate** ($NaNO_3$) : 100 g **Nitric acid** (HNO_3) : 5 g	in 1 litre	
A7	**Potassium thiocyanate** (KCNS) : 180 g/l		
A8	**Sodium chloride** (NaCl) : 100 g/l		
B1	**Potassium chloride** (KCl) : 30 g **Ammonium chloride** (NH_4Cl) : 30 g	in 1 litre	
B2	**Orthophosphoric acid** (H_3PO_4) : 186 g **Chromic oxide** (CrO_3) : 10 g	in 1 litre	Dilute 118 ml of orthophosphoric acid ($d = 1.75$) and 10 g of chromic oxide to 1 litre
B3	**Sodium carbonate** (Na_2CO_3) : 100 g/l		For coatings up to 5 μm
B4	**Orthophosphoric acid** (H_3PO_4) : 98 g/l		Dilute 64 ml of orthophosphoric acid ($d = 1.75$) to 1 litre
B5	**Ammonium nitrate** (NH_4NO_3) : 800 g **Ammonium hydroxide** (NH_4OH) : ($d = 0.88$) 10 ml	in 1 litre	
B6	**Potassium sulphate** (K_2SO_4) : 100 g **Orthophosphoric acid** (H_3PO_4) ($d = 1.75$) : 20 ml	in 1 litre	
B7	**Fluosilicic acid** (H_2SiF_6) : Pure solution containing not less than 30 % fluosilicic acid		Slightly weaker acid may be used if some magnesium fluosilicate ($MgSiF_6$) is added to the solution
B8	**Sodium acetate** (CH_3COONa) : 200 g **Ammonium acetate** (CH_3COONH_4) : 200 g	in 1 litre	
B9	**Ammonium nitrate** (NH_4NO_3) : 800 g **Thiourea** [$(NH_2)_2CS$] : 3.8 g	in 1 litre	Make up a fresh solution every 5 days
B10	**Hydrochloric acid** (HCl) : 42 g/l		Dilute 100 ml of hydrochloric acid ($d = 1.18$) to 1 litre
B11	**Potassium fluoride** (KF) : 100 g/l		
B12	**Potassium nitrate** (KNO_3) : 100 g **Potassium chloride** (KCl) : 100 g	in 1 litre	
B13	**Sulphuric acid** (H_2SO_4) : 90 g **Potassium fluoride** (KF) : 5 g	in 1 litre	Dilute 50 ml of sulphuric acid ($d = 1.84$) and 5 g of potassium fluoride to 1 litre
B14	**Potassium chloride** (KCl) : 100 g/l or **Sodium chloride** (NaCl) : 100 g/l		

5 PROCEDURE

Clean the area to be tested with a cloth, wetted with an organic solvent for grease removal if necessary. For certain coatings it may be necessary to employ an additional treatment to ensure that the surface is free from passive films or any conversion coating. Press an electrolytic cell, fitted with a flexible sealing ring and incorporating an annular cathode, on the coating so that a circle of known area is exposed to the test solution. Introduce the test solution into the cell and insert and operate the stirrer if appropriate to the instrument used and the thickness of the deposit.

Make the electrical connections, with the specimen anodic.

Continue electrolysis until dissolution of the coating is complete, as indicated by a sudden change in anode potential or cell voltage, and record the quantity of electricity consumed.

Examine the specimen and ensure that removal of the coating is virtually complete over the area enclosed by the seal.

6 CALCULATION OF COATING THICKNESS

Coating thickness is given by the following formula :

$$s = 100k \frac{Q\,E}{A\,\rho}$$

where

s is the coating thickness, in micrometres;

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

Q is the quantity of electricity, in coulombs, passed in dissolving the coating and is given by the formula : $Q = it$

i being the current in amperes;

t being the test duration in seconds.

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

A is the area over which the coating is dissolved, in square centimetres;

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

The formula may also be expressed as :

$$s = C.Q$$

where C is a constant for a given metal coating, electrolyte and cell. The value of C may be determined experimentally by measuring a coating of known thickness and it is recommended that in practice this should normally be done.

7 ACCURACY OF METHOD

The method is normally accurate within ± 10 %. Higher accuracy may be consistently achieved in particular circumstances. The accuracy may be lower when the coating thickness exceeds 50 μm or is less than 0.2 μm.

The presence of an alloy layer at the interface may affect the accuracy of the results obtained.

ANNEX A

DESCRIPTION OF TYPES OF APPARATUS

A.1 GENERAL

With one type of circuit the current through the test cell has to be controlled to a constant value, a clock then being used to measure the time interval between the start and end point of the test. Alternatively an integrating meter may be used to measure the quantity of electricity passed, in which case accurate control of the current and separate measurement of the time interval are unnecessary.

The end point can be determined by observing the sudden change in cell voltage on a suitable voltmeter, or a relay switch can be used to automatically terminate the test. In the latter case, the relay is set to operate at a pre-determined value of either the cell voltage or the rate of increase of cell voltage.

Suitable currents for testing the coating thickness on a defined area of approximately 0.2 cm^2 within a reasonable time are approximately 20 mA for relatively thin coatings, like chromium, and 80 mA for relatively thick coatings, like nickel – i.e. the current densities employed for dissolution should conveniently be approximately 1 to 4 kA/m^2.

A.2 ELECTROLYTIC CELL

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

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

The area of surface enclosed by the seal shall be well defined and small enough (approximately 0.2 cm^2) to permit its application to a surface which is curved. Smaller cells may be required for measuring the thickness of coatings on intricate shapes 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 on which the test is applied.

Normally it is necessary to provide means of agitating the electrolyte in the cell to minimize changes in cell voltage occurring before the end point of the test has been reached.