

STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 2331.1.2—2001

Methods of test for metallic and related coatings

Method 1.2: Local thickness tests—Coulometric method

RECONFIRMATION NOTICE

Technical Committee MT-009 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

Certain documents referenced in the publication may have been amended since the original date of publication. Users are advised to ensure that they are using the latest versions of such documents as appropriate, unless advised otherwise in this Reconfirmation Notice.

Approved for reconfirmation in accordance with Standards Australia procedures for reconfirmation on 20 March 2017.

The following are represented on Technical Committee MT-009:

Australasian Institute of Surface Finishing
Australian Chamber of Commerce and Industry
Australian Industry Group
Australian Steel Institute
Bureau of Steel Manufacturers of Australia
Galvanizers Association of Australia
Galvanizing Association of New Zealand
New Zealand Metal Roofing Manufacturers

NOTES

Australian Standard™

AS 2331.1.2

Methods of test for metallic and related coatings**Method 1.2: Local thickness tests—Coulometric method**

PREFACE

This Standard has been prepared by the Joint Standards Australia/Standards New Zealand Committee MT-009, Metal Finishing, to supersede AS 2331.1.2—1980. It is the result of a consensus among representatives on the Joint Committee to produce it as an Australian Standard. The objective of this revision is to upgrade the requirements so that they closely align with ISO 2177:1985, *Metallic coatings—Measurement of coating thickness—Coulometric method by anodic dissolution*.

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is only for information and guidance.

METHOD

1 SCOPE

This Standard specifies a coulometric method based on 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, or with new electrolytes developed for them; 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 Clause 7.6).

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 thickness of diffusion layers can also be measured. The method can also be used to measure the thickness of coatings on cylindrical specimens and wires (see Clause 7.8).

NOTES:

- 1 The test procedures described in this Standard do not necessarily include all of the precautions required to satisfy health and safety aspects. Care should be taken to ensure that the procedures are carried out only by people who have received suitable training. Guidance in the handling and use of hazardous chemicals is given in AS/NZS 2243.1 and AS/NZS 2243.2.
- 2 Factors relating to measuring accuracy are given in Appendix A.



2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS/NZS

- 2243 Safety in laboratories
- 2243.1 Part 1: General
- 2243.2 Part 2: Chemical aspects

ISO

- 2064 Metallic and other inorganic coatings—Definitions and conventions concerning the measurement of thickness

3 DEFINITIONS

For the purpose of this Standard, the definitions in ISO 2064, and those below apply.

3.1 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.

3.2 Reference area

The area within which a specific number of single measurements is required to be made.

4 PRINCIPLE

A well-defined area of the coating is dissolved anodically using a suitable electrolyte. The virtually complete dissolution of the coating is indicated by a rapid change in cell voltage. The coating thickness is determined from the quantity of electricity (in coulombs) used, which may be calculated from—

- (a) the time interval between the start and the end of the test, if it is conducted at constant current density; or
- (b) the integrated quantity of electricity used in dissolving the coatings.

5 INSTRUMENTATION

Suitable instruments may be constructed from readily available components; however, proprietary instruments are usually used.

NOTE: The principle of operation of various types of instruments is given in Appendix B.

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 3.1), 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.

The performance of the instrument shall be checked using specimens of known coating thickness. If the instrument readings agree to within $\pm 5\%$ of the known thickness 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 manufacturers' instructions.

Reference specimens of known coating thickness shall consist of the same type of coating and substrate as the specimen to be measured, and the thickness shall be known to an

accuracy of 5% or better. When measuring the thickness of alloy coatings, the use of correct reference specimens is of particular importance.

6 ELECTROLYTE

The electrolyte shall have a known and 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) it causes the coating to dissolve anodically at an efficiency as close to 100% as possible;
- (c) a detectable sharp change in electrode potential occurs as the coating is penetrated and an increasing area of substrate is exposed; and
- (d) the test area exposed in the test cell is completely wetted.

The choice of electrolyte shall take into account the coating and substrate materials, the current density and electrolyte circulation within the test cell.

NOTE: 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 Appendix C.

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

7 PROCEDURE

7.1 General

If commercial equipment is used, the manufacturer's instructions shall be followed with respect to the operating procedure for measurement, the electrolytes and, if necessary, calibration (see Clause 5). Otherwise, the general test procedure set out in Clauses 7.3 to 7.8 shall be followed.

NOTE: Appropriate attention should be given to the factors listed in Appendix A.

7.2 Pre-set voltage

If using instruments that require a pre-set voltage, the actual value of this voltage is dependent on the particular metallic coating, the current density, electrolyte concentration and temperature, and circuit resistance. Because of these factors, it is advisable to first perform an evaluation test.

7.3 Preparation of test surface

Test pieces shall be thoroughly cleaned before testing. The cleaning method employed shall depend on the nature of the surface and the contaminants, and shall not include the use of any abrasives or solvents that may attack the surface of the test piece.

Care shall be taken to ensure that test pieces are not recontaminated by excessive or careless handling after cleaning.

7.4 Cell application

Press the electrolytic cell, fitted with its flexible sealing ring, onto 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; otherwise, insert a suitable cathode (incorporated in some instruments as part of the electrolyte agitation mechanism).

7.5 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.

7.6 Undercoats

When one or more undercoats are to be measured after measuring the top coat, 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 coat, introduce the appropriate electrolyte and continue the test as before.

7.7 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.

NOTE: Information on non-uniform dissolution is given in Appendix A, Paragraph A13.

7.8 Coatings on cylindrical specimens

If the surface area is too small to use the normal cell with a flexible gasket, replace the gasket with an electrolyte container and an adjustable holding device, using an agitator if necessary. Adjustability of the holding device is necessary so that it can 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, for example the cut-off voltage and deplating current, may have to be modified.

NOTE: The exact deplating area needs to be known to obtain an accurate result. The main source of error is due to the meniscus and current field at the electrolyte surface.

8 EXPRESSION OF RESULTS

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

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

where

- k = the current efficiency of the dissolution process (equal to 100 in the case of 100% efficiency)
- E = the electrochemical equivalent of the coating metal for the conditions of the test, in grams per coulomb
- A = the area from which the coating is dissolved, in square centimetres, i.e. the measuring area

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

Q = 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 = the current, in amperes

t = 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.

9 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.

10 TEST REPORT

The test report shall include the following information:

- (a) Reference to this Australian Standard, i.e. AS 2331.1.2.
- (b) Identification of the test specimen.
- (c) The area, in square centimetres, over which the measurements were made.
- (d) The location(s) on the coated item at which the tests were carried out.
- (e) The identity of the electrolyte used.
- (f) The measured thickness, in micrometres, at each area at which the tests were carried out and the number of measurements, averaged for each reported measurement.
- (g) The name of the testing laboratory.
- (h) The report number and date.
- (i) Any deviation from the method specified.
- (j) Any factor that may have influenced the result.
- (k) Identification of the instrument used.