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Determination of galvanic corrosion rate for assembled forms of carbon fibre reinforced plastics (CFRPs) and protection-coated metal — Electrochemical tests in neutral sodium chloride solution

Détermination du taux de corrosion galvanique pour les formes assemblées de plastiques renforcés de fibres de carbone (CFRP) et de métal revêtu de protection — Essais électrochimiques en solution neutre de chlorure de sodium

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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

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Determination of galvanic corrosion rate for assembled forms of carbon fibre reinforced plastics (CFRPs) and protection-coated metal — Electrochemical tests in neutral sodium chloride solution

1 Scope

1.1 This document specifies the electrochemical test for determining galvanic corrosion rate of CFRPs and metal assemblies with protection-coating, subjected to the corrosive environment of electrolyte diffusion through the coating. It specifies the apparatus, the test solutions, and the procedure to be used in conducting the electrochemical tests for

a) the assessment of the Fick's diffusion parameter for protective coating on metallic materials, and

b) the estimation of the galvanic corrosion rates with the conversion of ISO 21746 coating-free sample data.

1.2 The following are intended situations of implementing an electrochemical test based on this document:

a) when interested parties estimate the galvanic corrosion rate of bonded joints relating engineering metals with protection-coating and CFRPs of the potential drastically nobler than those of most metals, utilizing the resources of ISO 17475;

b) when expanding CFRP-metal bonded joints applications using coatings to the fields of corrosion-sensitive environments caused by electrolytes.

1.3 It is not the intent of this document to fulfil the need for:

- omitting relevant field tests for the applications in corrosive environment;
- superimposing test data for specific applications for the range of relevant data;
- comparative testing as a means of ranking different protections with respect to corrosion rates;
- ignoring the field hazards such as erosion, abrasion, and ultraviolet irradiation.

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 2808, *Paints and varnishes — Determination of film thickness*

ISO 17475, *Corrosion of metals and alloys — Electrochemical test methods — Guidelines for conducting potentiostatic and potentiodynamic polarization measurements*

ISO 21746, *Composites and metal assemblies — Galvanic corrosion tests of carbon fibre reinforced plastics (CFRPs) related bonded or fastened structures in artificial atmospheres — Salt spray tests*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Symbols

a	slope of Q-t plot
b	error in Q-t plot owing to charge duration and other factors
C	static capacity of capacitor
c_{O}	saturated oxygen density in water
$c(x)$	electrolyte density function
d	coating thickness
D	Fick's diffusion parameter
D_{ox}	oxygen diffusion constant in water
E_{cr}	estimation of galvanic corrosion rate for coated sample
F_{pe}	protection efficiency
J	diffusion flux
J_{ASO}	oxygen flux in air saturated static water
J_{PC}	steady flux through thin protective coating
J_{PCO}	oxygen flux through protection coating
J_{PFO}	oxygen flux of air saturated water for protection free sample
J_{OMax}	the maximum through coating flux of oxygen in the form of water solution
K_{cr}	galvanic corrosion rate of coating-free sample derived with ISO 21746
Q	charge at a time t
Q_0	charge at linearly extrapolated time point t_0
S	grid area of capacitor
t	time
t_0	flux onset time
t_1	upper bound time of linear section in time-charge plot
V	inter-grid potential of capacitor
x_1	inter-grid distance of capacitor

x_2 locational dimension in flux direction

x_{DL} diffusion layer thickness

ε_0 permittivity of vacuum

ε_r relative permittivity

5 Principle

5.1 General

Protection coating behaves as a capacitor in the form between conductive materials and conductive electrolytes. Focusing on the capacitance, Fick's diffusion parameter is monitored by analysing the drift in charge due to the distance shrinkage with electrolyte absorption from the surface of the protective coating in the plate-shaped sample. The effect of the protective coating is evaluated by converting the value into the salt spray flux in the galvanic corrosion test.

5.2 Capacitor model

Static capacity, C, of a capacitor in Figure 1 is expressed as Formula (1):

$$C = \varepsilon_r \varepsilon_0 \frac{S}{x_1} \tag{1}$$

where $\varepsilon_0 = 8{,}854\,187\,62 \times 10^{-12}$ is the permittivity of vacuum.

The charge, Q, is expressed as follows when inter-grid potential, V, is loaded.

$$Q = CV \tag{2}$$

Formulae (1) and (2) lead to the following expression.

$$Q = \varepsilon_r \varepsilon_0 \frac{SV}{x_1} \tag{3}$$

When the inter-grid distance x_1 shrinks to x_1-Δx_1 through diffusion, the charge drift ΔQ is expressed using Formula (3) as follows.

$$x_1 \Delta Q + Q \Delta x_1 = 0 \tag{4}$$

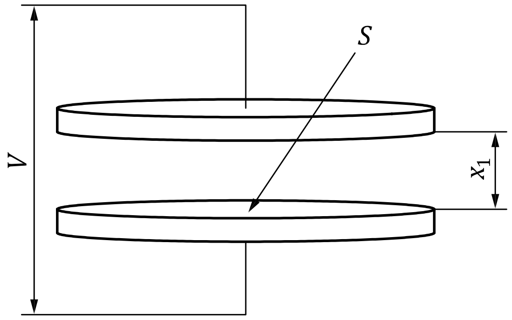
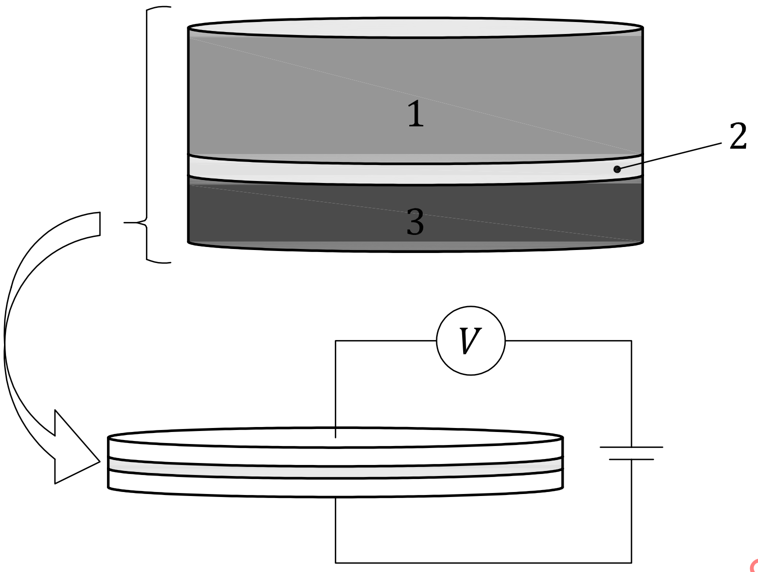


Figure 1 — Schematic diagram of capacitor

A capacitor in Figure 1 is expressed as is shown in Figure 2 for a grid of conductive base material, a grid of electrolyte, and thin coating with the thickness, x_1, to separate the grids.



Key

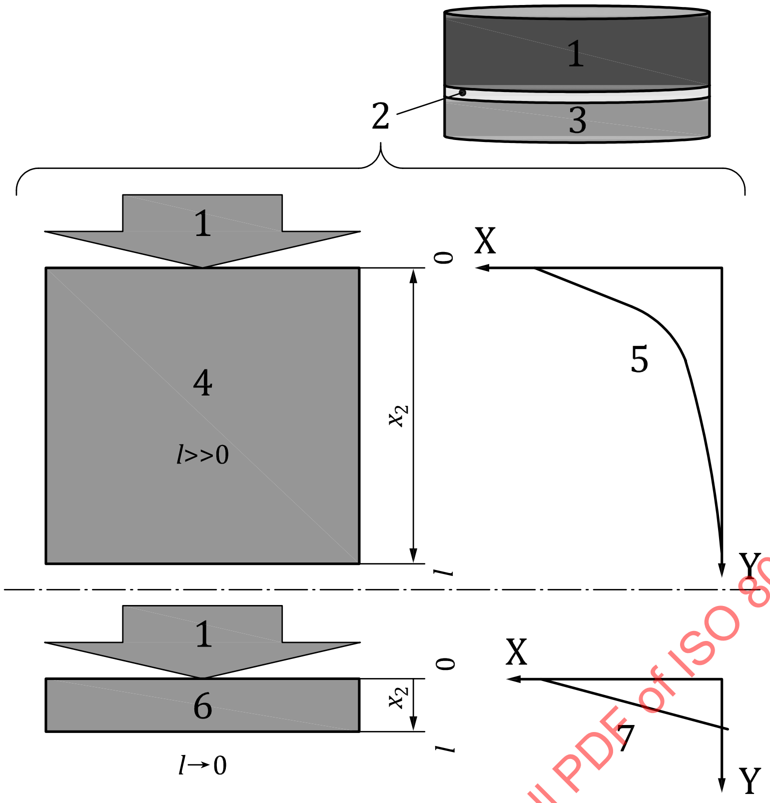
1 water/electrolyte
2 thin coating layer
3 conductive base

Figure 2 — Capacitor formation with thin coating layer

5.3 Fick's diffusion coefficient for thin coating layer

A. Fick expressed the phenomenon of water diffusion in a mathematical form using an analogy with the laws of heat conduction derived by Fourier and the diffusion of electricity in a conductor derived by Ohm. The diffusive flux occurring in a unit of time through a medium of small distance is expressed in Fick's first law proportional to the difference of density and inversely proportional to the distance of the medium. Fick's second law is derived assuming the proportional coefficient constant – Fick's diffusion constant – and the conservation of mass for the unsteady state of diffusion. See Annex A.

For thick media, Fick's second law has been applied assuming the density distribution is regular along the thickness during the diffusion of unsteady flux. However, Fick's first law provides an acceptable approximation for thin region of media in the form of linear density distribution along the thickness at the diffusion of steady flux as illustrated in Figure 3.



Key

X	density
Y	thickness
1	electrolyte
2	coating layer
3	conductive base material
4	thick coating media
5	regular
6	thin coating media
7	linear

Figure 3 — Fick's diffusion model for a thick coating and the thin region

Fick's diffusion coefficient D is defined as follows in Fick's first law.

$$J = -D\frac{dc(x_2)}{dx} \tag{5}$$

where $c(x_2)$ is the electrolyte density function, and x_2 is the locational dimension in the flux direction. Formula (5) is approximated for a thin protective coating of the thickness $d \rightarrow 0$ with steady flux through the thickness J_{PC} as follows.

$$J_{\mathrm{PC}} \approx -D\frac{c(0)-c(d)}{d} \tag{6}$$

The flux J_{PC} is also approximated as follows atop of the coating in a short period of time Δt.

$$J_{\mathrm{PC}} \approx [c(0)-c(d)]\frac{\Delta x_2}{\Delta t} \tag{7}$$

Fick's diffusion coefficient D is therefore approximated with the Formulae (6) and (7) as follows.

$$D = -d \cdot \frac{\Delta x_2}{\Delta t} \tag{8}$$

Formulae (4) and (8) lead to an expression as follows for the case $x_1 = x_2 = d$ in Formulae (4) and(7).

$$D = d^2 \cdot \frac{\left(\frac{\Delta Q}{Q}\right)}{\Delta t} \tag{9}$$

i.e.

$$\frac{\Delta Q}{Q} = \left(D/_{d^2}\right) \cdot \Delta t \tag{10}$$

Thereby Fick's diffusion coefficient D is given with a slope $a = D/_{d^2}$ on a $\Delta Q/Q - t$ plot, as $D = a \cdot d^2$.

5.4 Corrosion rate estimation

The galvanic corrosion rate is diffusion-limited of the oxygen supply for the anodic metal. Therefore, a known corrosion rate of protection free sample K_{cr} derived with ISO 21746 on the oxygen saturated saltwater mist is converted by multiplying a factor protection efficiency F_{pe} into an estimation for the corrosion rate E_{cr} with coating case as follows.

$$F_{\mathrm{pe}} \equiv \frac{J_{\mathrm{PCO}}}{J_{\mathrm{PFO}}} \leq \frac{J_{\mathrm{OMax}}}{J_{\mathrm{ASO}}} \tag{11}$$

The J_{OMax} is approximated for a coating thickness d as follows.

$$J_{\mathrm{OMax}} \approx D\frac{c_{\mathrm{O}}}{d} \tag{12}$$

The oxygen flux in air saturated static water J_{ASO} is approximated for the diffusion layer thickness x_{DL} as follows.

$$J_{\mathrm{ASO}} \approx D_{\mathrm{ox}} \cdot {}^{c_{\mathrm{O}}}\!/\!{}_{x_{\mathrm{DL}}} \tag{13}$$

Therefore, relationships are drawn with the Formulae (11) to (13) as follows:

$$F_{\mathrm{pe}} \leq \frac{D}{d} \times \frac{x_{\mathrm{DL}}}{D_{\mathrm{ox}}} \tag{14}$$

and

$$E_{\mathrm{cr}} \leq F_{\mathrm{pe}} \times K_{\mathrm{cr}} \tag{15}$$

Therefore, an estimation for the corrosion rate E_{cr} with Formula (15) provides accurate or larger value, or safer side value for most of engineering applications.

NOTE The oxygen flux in air saturated static water J_{ASO} at 25 °C is given as follows.

Dissolved oxygen concentration in air saturated fresh water is 8 ppm, or 8/32=1/4 mol/m^3. The diffusion constant of oxygen, D_{ox}, is $2{\times}10^{-9}$ m^2/s. Diffusion layer thickness, x_{DL}, is $5{\times}10^{-4}$ m in static water. Therefore, oxygen flux is given as $2{\times}10^{-9}$ [m^2/s] ×8/32[mol/m^3] $\times(5 \times 10^{-4}$ [m]$)^{-1} = 1 \times 10^{-6}$ [mol·m^{-2}·s^{-1}].

6 Test solutions

The compounds in test solutions, their density, and the temperature, shall be selected in the combination with the applicational environments and the specification for the coatings or product being tested and used. When not specified, the details shall be mutually agreed upon between all interested parties. Unless otherwise specified or agreed, aqueous sodium chloride in ISO 21746 shall be selected as a representative corrosive test solution as follows.

Dissolve a sufficient mass of sodium chloride in distilled or deionized water with a conductivity not higher than 20 µS/cm at 25 °C ± 2 °C to produce a concentration of 50 g/l ± 5 g/l. The sodium chloride concentration of the sprayed solution collected shall be 50 g/l ± 5 g/l. The specific gravity range of 50 g/l ± 5 g/l solution is from 1,029 to 1,036 at 25 °C.

The sodium chloride shall contain less than 0,001 % mass fraction of copper and less than 0,001 % mass fraction of nickel, as determined by atomic absorption spectrophotometry or another analytical method of similar sensitivity. It shall not contain more than 0,1 % of a mass fraction of sodium iodide, or more than 0,5 % of a mass fraction of total impurities calculated for dry salt.

NOTE If the pH of the prepared solution at 25 °C ± 2 °C is outside the range 6,0 to 7,0, investigate the presence of undesirable impurities in the salt and/or the water.

7 Apparatus

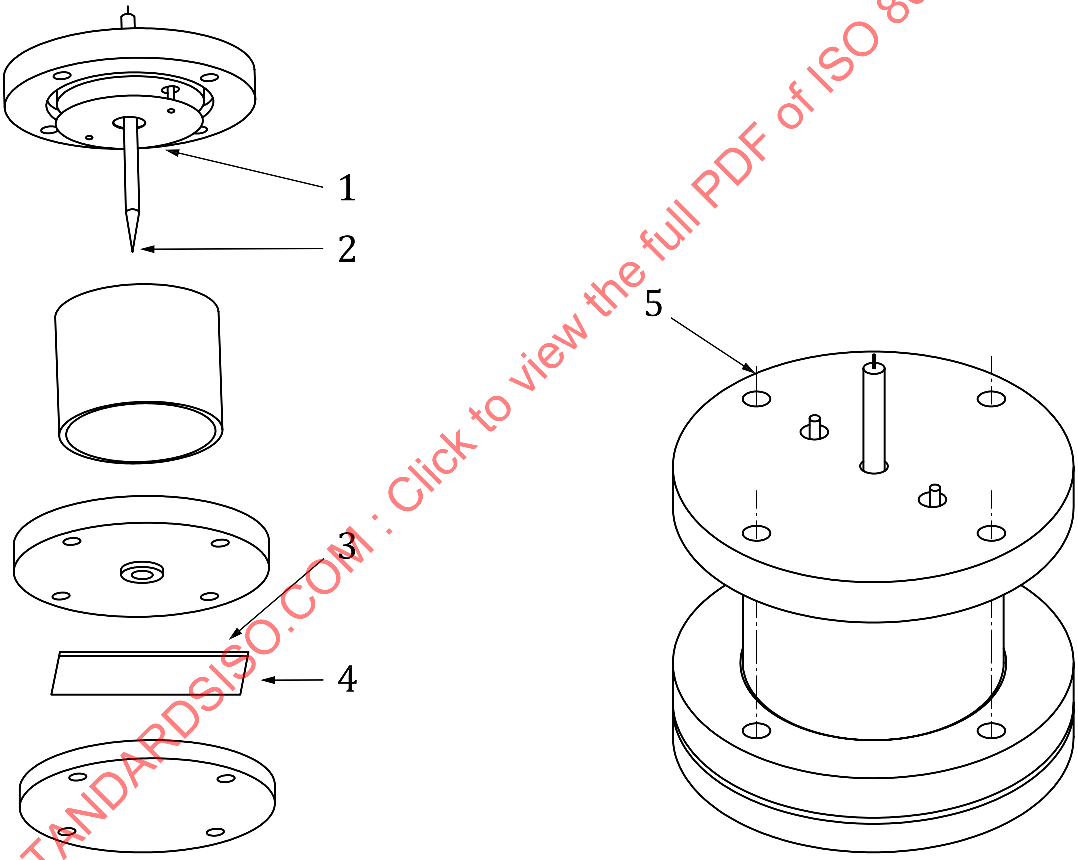
7.1 Potentio/Galvanostat

The potentio/galvanostat should be capable of chronocoulometric measurement with automatically controlling the electrode potential within ±1 mV to a preset value. The sensitivity and accuracy of the charge measurement unit for the chronocoulometric measurement should be sufficient to detect $\pm 1{,}0 \times 10^{-8}$ mAh. Unless otherwise specified, the potentio/galvanostat in ISO 17475 shall be applied.

NOTE $\pm 1{,}0 \times 10^{-8}$ mAh is converted to $\pm 3{,}6 \times 10^{-8}$ C.

7.2 Test cell

The test cell should contain the working electrode, which is the specimen with protection coating, a reference electrode for measuring the electrode potential, and counter electrode, which is typically Pt mesh. The detailed construction of the test cell depends on the specimen specification. Figure 4 shows an example commonly used for plate type specimen.



a) Disassembled form

b) Assembled form

Key

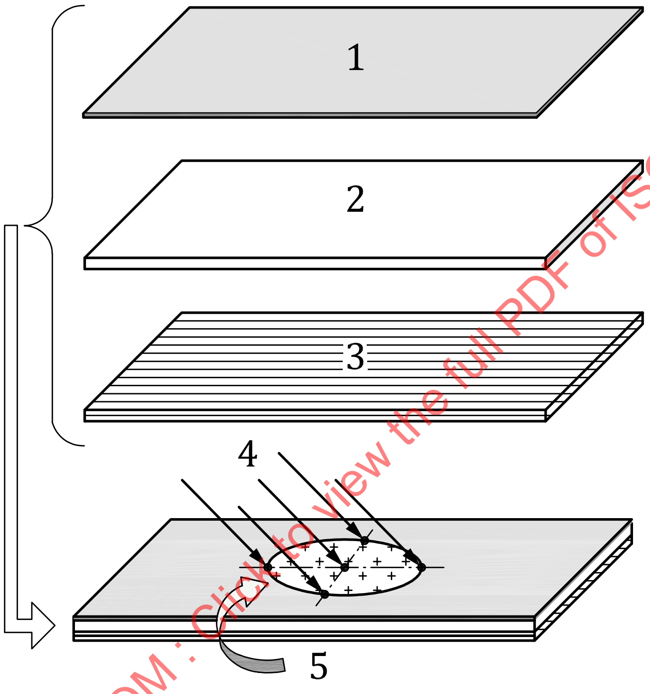
1	counter electrode (Pt mesh)
2	reference electrode
3	protection coating side
4	working electrode (specimen)
5	fastening

Figure 4 — Schematic overview of chronocoulometric measurement cell

In chronocoulometric measurement, the working electrode is kept constant potential for monitoring the relationship between charge and time.

8 Test specimen preparation

The type of test specimens, their number, the shape, and the dimensions, shall be selected in the combination with the cell type and the specification for the material or product being tested. When not specified, the details should be mutually agreed upon between all interested parties. Unless otherwise specified or agreed, plate type specimen as described in ISO 21746 shall be selected to serve as working electrode in combination with flat type cell, with a surface finish according to the applicational requirements. A typical area of the working electrode is 100,0 ± 0,4 mm^2 for a circular form as depicted in Figure 5. The coating thickness, *d*, of the sample shall be measured as described in ISO 2808 for the average value of five points on the circumference per 90 degrees and the centre.



Key

1 coating

2 metal

3 CFRP

4 measurement points for coating thickness

5 contact area to test solution

Figure 5 — Flat type test specimen

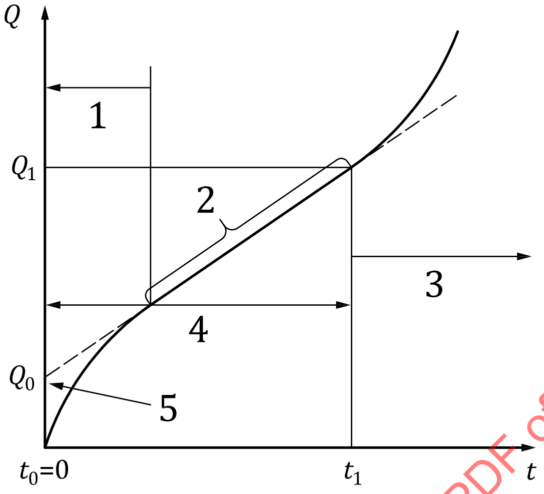
9 Test procedure

9.1 Fick’s diffusion parameter — Setups

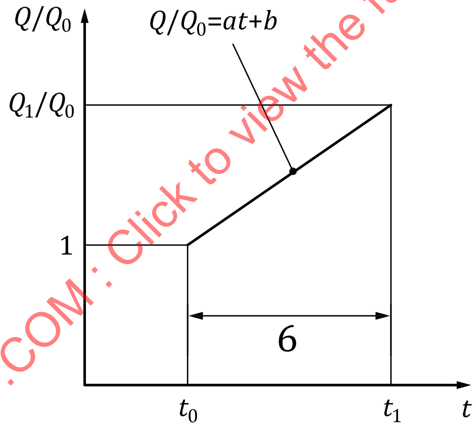
The specimen shall be set to test cell to form a working electrode in the coating side bounding to test solution. Wiring shall be made as specified for the potentio/galvanostat in the chronocoulometric measurement mode to record both current and charge under a constant potential less than the level of electrolysis. Unless specified, the wiring shall be for the three-grid method, the constant potential set is 0,350 ± 0,004 V, and the recording interval of 30 s.

9.2 Fick’s diffusion parameter — Calculation

Time domain is empirically defined between t_0 =0 and t_1 are defined for the current peak and the bottom, and empirical time points t_0 and t_1, where t_0 is the flux onset time and t_1 is the upper bound time of the linear section in time-charge plot as depicted in Figure 6 a). A plot of normalized charge $\Delta Q/Q_0$ from t_0 to t_1, where Q is a charge at a time t and Q_0 is defined at the linearly extrapolated point at t_0 in Figure 6 b), provides a linear relationship $\Delta Q/Q_0 = a{\cdot}t + b$ where b represents an error owing to charge duration and other factors. The Fick’s diffusion parameter D is given using the slope a and the coating thickness d as $D = a{\cdot}d^2$.



a) Schematic time-charge plot



b) Empirical domain of definition

Key

t	time
Q	charge
Q/Q_0	normalized charge
1	charge duration
2	linear
3	in-plane diffusion
4	domain of definition
5	extrapolation
6	domain of definition

Figure 6 — Schematic procedure of chronocoulometry data analysis

9.3 Corrosion rate estimation

The estimation of the galvanic corrosion rates for coated samples, E_{cr}, is given with the conversion of ISO 21746 coating-free sample data K_{cr} as depicted in Formula (16).

$$E_{cr} = F_{pe} \times K_{cr} \tag{16}$$

where, a factor “protection efficiency” F_{pe} is given as follows.

$$F_{pe} = \frac{D}{d} \times \frac{x_{DL}}{D_{ox}} \tag{17}$$

NOTE At 25 °C, the diffusion constant of oxygen, D_{ox}, is 2×10^{-9} m^2/s, and the diffusion layer thickness, x_{DL}, is 5×10^{-4} m, both in static water. Therefore, the F_{pe} is given as follows.

$$\begin{aligned} F_{pe} &= \frac{D\left[m^2/s\right]}{d\left[m\right]} \times \left(\frac{5}{2}\right) \times 10^5 \left[\frac{s}{m}\right] \\ &= 2{,}5\times10^5 \frac{D}{d} \end{aligned} \tag{18}$$

10 Test report

The test report shall contain information about the test procedure. This information may vary according to the purpose of the test and the guidelines prescribed, but a general list of the details likely to be required is as follows:

a) a reference to this document, i.e. ISO 8057:2024;

b) the solution composition, pH, prepared volume, temperature, and any variations during the tests;

c) description of the specimen material or product tested, dimensions, shape, surface treatment, and the area of the surface tested;

d) preparation of the test specimen, including any cleaning treatment applied, any protection given to edges or other special areas, and dry treatment conditions prior to test;

e) number of test specimens subjected to the test representing each material or product;

f) duration of the test and results of any intermediate inspections;

g) current and charge at each measurement time;

h) the potential shall be quoted with respect to the standard hydrogen electrode;

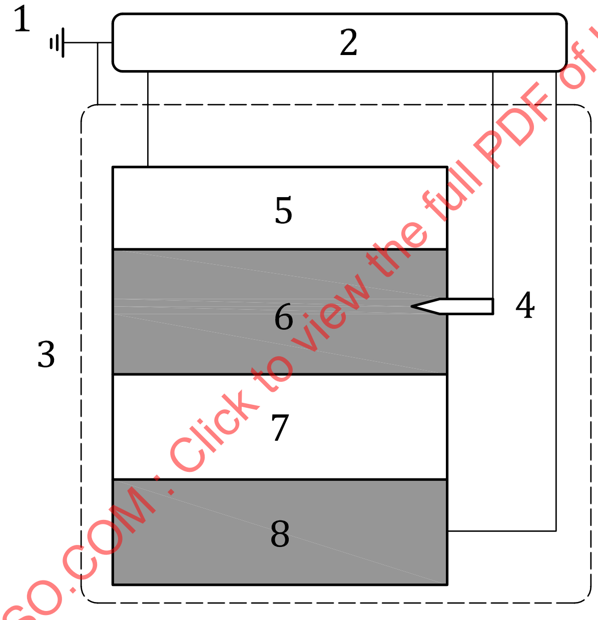
i) any abnormality or incident occurring during the entire test procedure.

Annex A
(informative)

Example of measurement for Fick’s diffusion parameter

A.1 Sample materials and test setup

PA6[1] roll-processed sheets with an area of 150 mm ×70 mm and a nominal thickness of 1,0 mm were selected as a representative material used for the metal coating and show a mild water diffusivity. The sheets were vacuum dried for 2 weeks at less than 10 Pa before the experiments, and the water content was verified using a Karl-Fischer moisture analyser MKH-710M equipped with a moisture vaporizer ADP-611[2]. The chronocoulometry measurements were conducted using a VSP-300[3] at a fixed potential of 0,35 V with Coulomb-gage-contained flat-type cells having a working electrode area of 100 mm^2. The electrolyte was prepared at 5 wt. % using high-purity water and NaCl.



a) Schematic configuration

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1) PA6 is polyamide 6. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) MKH-710M and ADP-611 are the trade names of a product supplied by Kyoto Electronics Co. Ltd., Japan. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3) VSP-300 is the trade name of a product supplied by Bio-Logic Science Instruments, France. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.