



Technical Report

ISO/TR 5604

Electrochemical noise measurement for assessing the protection of metal afforded by organic coatings

*Mesurage du bruit électrochimique pour l'évaluation de la
protection du métal induit par des revêtements organiques*

**First edition
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Foreword

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Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

There are several test methods for assessing the protection afforded by organic coatings on metal, particularly on low carbon steel, using the available electrochemical measurements. The most commonly used technique is electrochemical impedance spectroscopy (EIS), which is detailed in the ISO 16773 series, and is well suited for laboratory use. However, there is a strong need for a non-intrusive technique that can be used on site to monitor or for quality control that is quick and is relatively simple to use and interpret.

This document gives the current state of the art for such a technique, which is electrochemical noise measurement (EN). The developments described in this document suggest that electrochemical noise measurement can be used as an alternative to and potentially even as the preferred process for field or on-site use.

For further information, a detailed description of the EN methodology when applied to bare metal can be found in ISO 17093. The differences associated with using the technique for organic coatings are described in this document, which is intended to complement ISO 17093.

The EN approach has been directly compared with methodologies such as EIS and DC resistance, with good agreement.^{[3][4]}

Furthermore, electrochemical noise applied to assess coatings has been reviewed in References [5] and [6], showing the potential for this technique.

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Electrochemical noise measurement for assessing the protection of metal afforded by organic coatings

1 Scope

This document describes the principle of electrochemical noise measurement (EN), specifically focusing on the application of the technique to indicate the level of protection provided by an organic coating to the underlying metal. It discusses the principles behind the measurement method but also the type of electrochemical apparatus, the experimental set-up and electrodes configurations, the presentation of measured data, and analysis of results that have been used in the work done so far and reported in the references. This work has mainly been done in the laboratory, and there are some examples of work conducted in the field.

NOTE At the time of publication of this document, electrochemical noise measurement for coatings is mainly used by a number of academic groups. This document aims to encourage more general application of this method, particularly to field work.

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 16773-1, *Electrochemical impedance spectroscopy (EIS) on coated and uncoated metallic specimens — Part 1: Terms and definitions*

ISO 17093, *Corrosion of metals and alloys — Guidelines for corrosion test by electrochemical noise measurements*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16773-1, ISO 17093 and the following apply.

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/>

3.1 electrochemical noise

EN
fluctuation typically at low frequencies (≤ 1 Hz) and low amplitude in current and potential, generated by electrochemical reactions and other processes on the surface, for example, bubble evolution

3.2 electrochemical potential noise

fluctuation in potential of an electrode relative to a reference electrode or fluctuation in potential between two similar electrodes

Note 1 to entry: The electrochemical potential noise is expressed in microvolts (μV) or millivolts (mV).

3.3

electrochemical current noise

fluctuation in current to one electrode or between two electrodes

Note 1 to entry: The electrochemical current noise is normally expressed in nanoamperes (nA).

3.4

electrochemical noise resistance

R_n
resistance obtained by dividing the standard deviation of potential noise by the standard deviation of current noise from the time record

3.5

working electrode

WE

coated metal in contact with the electrolyte, made out of the investigated material(s)

3.6

pseudo-working electrode

PWE

working electrode (3.5) other than coated metal

Note 1 to entry: A silver/silver chloride electrode (Ag/AgCl) in laboratory work or a noble metal like silver, platinum or gold can be used as part of a sensing probe in field work. PWEs are needed for configurations other than the standard bridge.

3.7

pseudo reference electrode

P_{Ref}

electrode used as a reference electrode in field work

Note 1 to entry: The metal itself (for single substrate configuration), platinum wire or sheet, copper, and silver.

3.8

electrode configuration

arrangement by which the electrodes are connected to the measuring device, i.e. bridge (Bridge), single Substrate (SS), no connection to the substrate (NOCS)

Note 1 to entry: The configuration used depends on the particular circumstances of the measurement.

3.9

zero resistance amperemeter

ZRA

electronic circuit which measures current but has itself no significant impedance

Note 1 to entry: For coatings, a ZRA capable of measuring low levels of current down to picoamperes (pA) is usually needed.

4 Principles

4.1 Organically coated metal exposed to a corrosive environment – how a resistance measurement can indicate protection

Water and oxygen penetrate through an organic coating leading to electrochemical reactions occurring at the interface between the metal and the coating.^[7] In aqueous corrosion, discrete anodic and cathodic areas arise, and these allow corrosion to proceed whenever there is an ionically conductive pathway between the substrate and the surrounding environment. However, if a high ionic resistance is introduced into the circuit between anodes and cathodes, e.g. by the coating, the rate of corrosion will decrease to a low value. This is one accepted mechanism by which the coatings can operate to prevent corrosion. Another mechanism, which can take place concurrently, is interface reactions when aided by an oxidizing agent, e.g. oxygen,

hydrogen peroxide or chemicals within the coating. These can result in the formation of a passive coherent, protective, chemically stable oxide layer which slows the corrosion rate. For this “passivity” to pertain for any length of time, the coating acts to prevent the arrival of incoming aggressive ions. In both mechanism routes, a high resistance (R) between the anodes and cathodes prevent aggressive ions arriving at the interface and the rate of the corrosion process is largely determined by the through film ionic resistivity of the coating system. Overall, the resistance whether measured by a DC technique as R_{dc} , by EIS as $R_{0,1Hz}$, or by electrochemical noise resistance as R_n , is believed to provide a quantitative measure for the extent to which the coating is protecting the underlying metal. However, the influence of, for example, the binder type on coating resistance, is not fully understood. Therefore under certain circumstances, it is possible that the above statement on coating resistance is not fully justified.

4.2 Generation of electrochemical noise from bare and coated metal

A metal in contact with any solution generates both current noise and potential or voltage noise, due to small random fluctuations which arise stochastically during electrochemical processes.^[8] The voltage noise can be easily measured with respect to a stable reference electrode. In the case of current noise, it is important to simulate the situation within the metal by using two identical but separated metal specimens and measuring the current in between using a zero resistance amperemeter (ZRA). The current noise is the level of fluctuation on the current value. It has been theoretically shown^[5] that the standard deviation of the voltage values, i.e. voltage noise, divided by the standard deviation of the current values, i.e. current noise, gives rise to a parameter called electrochemical noise resistance (R_n). In the case of bare metal, R_n has been shown to be equivalent to the resistance obtained using the linear polarization technique.

When a coating is present, R_n can be attenuated in the case of current noise and amplified in the case of potential noise, through the coating. The level of both potential and current noise that is sensed by the instrument can be converted to R_n . The largest value of resistance R_n is very likely the resistance of the coating system to the movement of ions (R_{paint}) and this in turn has a direct bearing on the rate of corrosion, as described in 4.1. An exception would be when the coating has broken down and corrosion is occurring, and in that case a low measured value will be indicative of failure.

4.3 Evaluation criteria

The measurement of the electrochemical noise resistance (R_n) provides a manner of assessing the protection capability of the coating.

When $R_n > 1 \times 10^8 \Omega \cdot \text{cm}^2$ indicates good protection, $1 \times 10^6 \Omega \cdot \text{cm}^2$ to $1 \times 10^8 \Omega \cdot \text{cm}^2$ is the borderline and $R_n < 1 \times 10^6 \Omega \cdot \text{cm}^2$ affords poor protection.^[9] The rate of reduction of R_n with time will indicate the rate at which the coating system is breaking down.

5 Apparatus

5.1 Test cell

The test cell is a leak proof cell constructed of material that will not corrode, deteriorate or contaminate the solution. The test cell was used to contain the electrolyte. The electrode holder and mounting material for the electrodes were mounted so as to have no influence on the measurement.

5.2 Field or site work

Although in laboratory work leak proof cells can be permanently attached, in field work a different approach is used since a dismountable probe is needed. One solution is to hold the electrolyte solution in a filter paper pad cut to the size of the pseudo reference electrode (P_{Ref}) or pseudo working electrodes (PWE), whose area defines the measurement area.

5.3 Reference electrode

In the field a robust electrode, such as a silver/silver chloride (Ag/AgCl) or copper/copper sulfate (Cu/CuSO₄) was used.

5.4 Working electrode

In almost all the work described in the references, the configurations were not just the standard bridge (see 6.2) where two nominally identical and separate coated samples are needed, and the two working electrodes will be the metal under the coating in each sample. The two most common alternatives are single substrate (see 6.3) configuration, where the reference becomes the metal under the coating, two standard reference electrodes (SRE) become the working electrodes, and no connection to the substrate (NOCS) configuration which requires three SREs (see 6.3) or P_{Ref} (field work).

5.5 Electrolyte

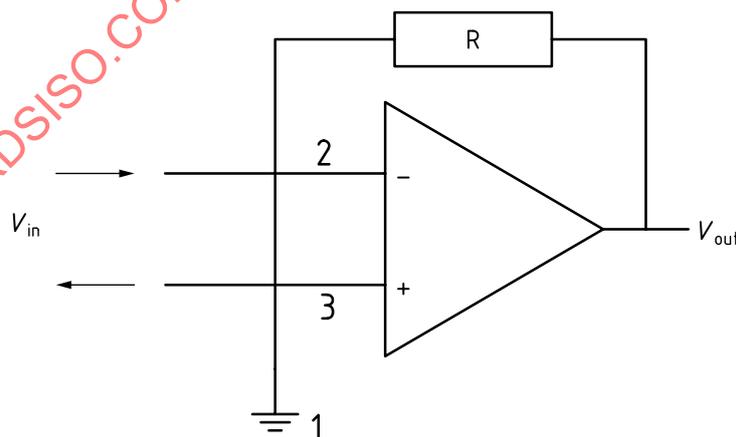
The test solution most commonly used in the references was NaCl solution, $c = 0,6 \text{ mol/l}$, because it typically has a corrosivity similar to sea water. This solution has a low resistance in comparison with the resistance of the investigated system. Sometimes other electrolytes more in keeping with the likely exposure environment were used. In all cases, the electrolyte is normally conductive enough to not become the limiting factor.

6 Description of the measurement system

This clause describes how the experiments were normally done in the work which generated the results in the referenced papers and presented in [Annex A](#), [Annex B](#) and [Annex C](#).

A measurement system consisting of an electrochemical cell connected to instrumentation and software for the characterization of materials using standard precision electrical measurement techniques was employed. Decisions were made as to the length of time that the data are gathered and at what frequency. Typically, data was obtained at 0,5 s (2 Hz), collecting 512 data points over 256 s with the measurement repeated twice. Experiments done many years ago showed this to be optimal for coatings work

A potentiometer with a very high input impedance (i.e. $10^{11} \Omega$ to $10^{14} \Omega$) to minimize the current drawn from the working electrode(s) during measurement was used to measure the potential difference between the reference and working electrodes, along with a zero-resistance amperemeter, ZRA with high sensitivity to measure currents as low as picoampere (see [Figure 1](#)).



Key

- 1 ground
- 2 working electrode 1
- 3 working electrode 2

R resistor
 V_{in} voltage in
 V_{out} voltage out

NOTE This figure shows the operational amplifier which is the basis for the zero-resistance amperemeter (ZRA) with an indication of connection to working electrodes (key references 2 and 3).

Figure 1 — Schematic representation of an operational amplifier

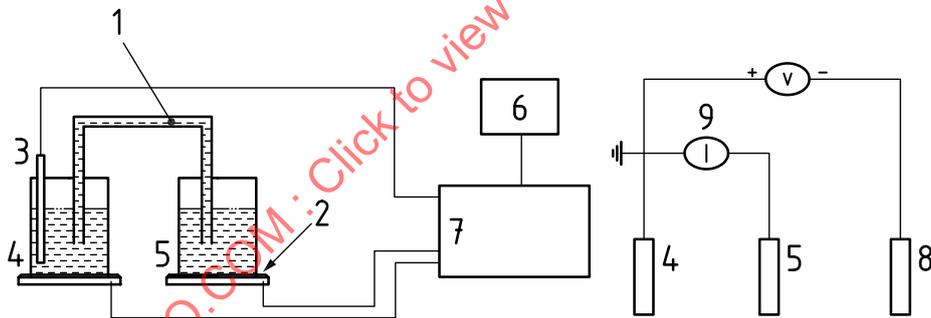
7 Electrode configurations

7.1 General

There are three common electrode configurations which have been used for coated metal and these are described in 7.2 to 7.4. The first one is the standard arrangement (as shown in Figure 2). The other two have been developed specifically for coatings particularly to make application of the method in the field possible (see Figures 3 and 4).

7.2 Bridge (laboratory use)

This arrangement, shown in Figure 2, was one of the three methods used in the references for laboratory-based investigations. It involves a bridge between the two nominally identical and electrically separated working electrodes both covered with the same coating at the same thickness applied at the same time and hence nominally identical. The current between these can be measured via a ZRA and simultaneously the potential of these electrodes, joined together internally in the equipment, can be measured with respect to a low noise standard reference electrode.



Key

1	salt bridge	6	personal computer
2	coating	7	zero resistance amperemeter (data logger)
3	standard reference electrode	8	reference electrode
4	working electrode 1	9	zero resistance amperemeter
5	working electrode 2	V	voltage

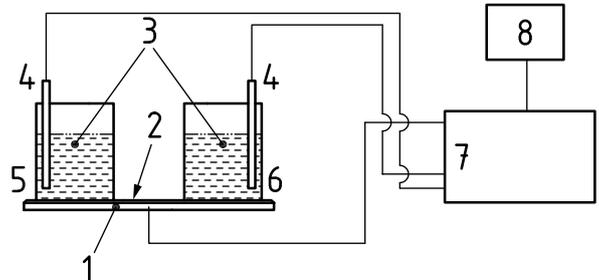
Figure 2 — Standard “salt-bridge” configuration

7.3 Single substrate (laboratory use)

In practical (field/on-site) applications, it is extremely rare to find two nominally identical but separate working electrodes. Therefore, in field work, the single substrate (SS) configuration can be used (see Figure 3). It is a re-arrangement of the original salt bridge configuration which is electrically equivalent. It replaces the working electrodes, (the two coated metal substrates) by very low noise reference electrodes and uses the coated metal substrate as the pseudo-reference. The reference electrodes make electrolytic contact with the corroding surface via solution held in a well-defined area, e.g. a glass cell held in place

by pressure or a plastic pipe adhered on the surface in laboratory use. The current perturbation that is measured by the ZRA originates from the electrochemical activity of the two coupled areas of the specimen.

When EN values from coated substrates have been obtained using the SS arrangement and compared with those obtained from the bridge arrangement, good agreement [10] has been shown. In cases where it is suspected that the two areas have different value of resistance, three pairs can be examined allowing the individual values to be computed. Recent equipment developed for noise measurements on coated metal has this facility available. Since the currents are so small (measured in nA), the reference electrodes are considered to act reversibly.



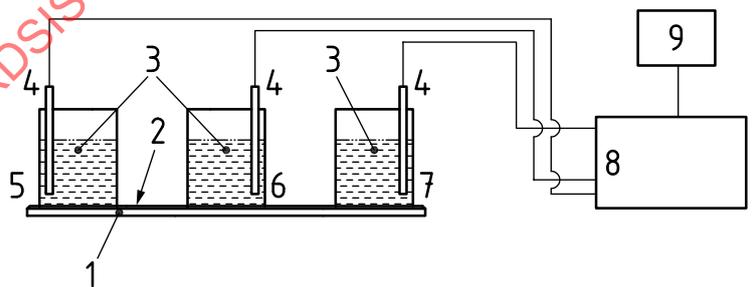
Key

- | | |
|--------------------------------|---|
| 1 substrate | 5 working electrode 1 |
| 2 coating | 6 working electrode 2 |
| 3 solution | 7 zero resistance amperemeter (data logger) |
| 4 standard reference electrode | 8 personal computer |

Figure 3 — Single substrate (SS) configuration as used in a laboratory setting

7.4 No connection to the substrate as used in the laboratory

In the no connection to the substrate (NOCS) arrangement shown in Figure 4, potential noise can be measured against a third stable low noise reference electrode. Like the working electrodes, the NOCS arrangement is in electrolytic contact with the specimen. Hence, any wired electrical contact to the substrate is eliminated. This configuration is preferred where connection to the metal substrate is difficult. It is possible that the three areas which constitute the measurement have different resistances. Obtaining information about individual coated areas is difficult. It has been shown [11] that some idea of the values can be achieved by making three consecutive measurements with the coated areas with the terminals on the measuring box, differently connected.



Key

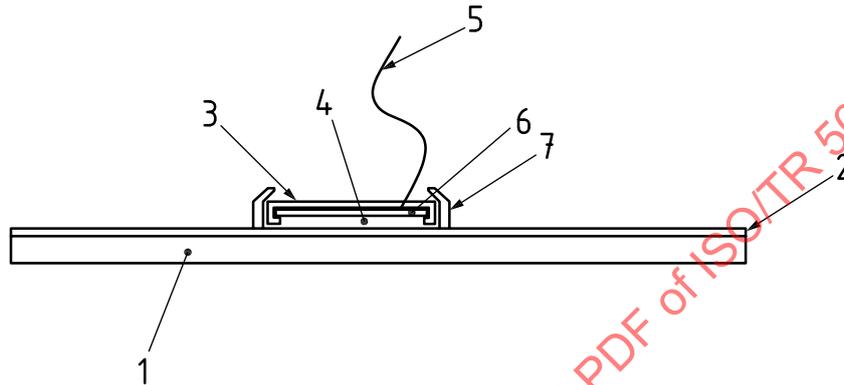
- | | |
|--------------------------------|---|
| 1 substrate | 6 working electrode 2 |
| 2 coating | 7 pseudo reference electrode |
| 3 solution | 8 zero resistance amperemeter (data logger) |
| 4 standard reference electrode | 9 personal computer |
| 5 working electrode 1 | |

Figure 4 — Standard “salt-bridge” configuration

7.5 Field measurement

For field work, the pseudo reference and working electrodes replace the SREs, and they then constitute part of a “probe”. One such design using copper probes is shown in [Figure 5](#). A study from 2021 showed that Ag/AgCl electrodes are preferable to (Cu/CuSO₄).^[6]

In [Figure 5](#), the pseudo reference or pseudo working electrode contacts a filter paper “pad”. The ionically conductive pad can be made by cutting filter paper into 3 cm × 3 cm pieces, soaking in 3 % NaCl solution or another agreed solution and placing the pad between the electrode and the coated surface. This has found to be essential for efficient penetration of the electrolyte into the coating. When in contact with a solution with a fixed chloride concentration the Ag/AgCl electrode has been shown to develop a steady voltage within a short period of time (few minutes) making it possible for it to act as a P_{Ref} or a PWE. The probe can be fixed temporarily by tape or silicone adhesive as shown in [Figure 5](#), to the coated metal.



Key

- | | | | |
|---|--|---|--------------------------------------|
| 1 | substrate | 5 | wire to connect to ZRA |
| 2 | anti-corrosive coating | 6 | silicone to cover pad |
| 3 | electrode (Cu/CuSO ₄ or Ag/AgCl) | 7 | silicone to fix pad to the substrate |
| 4 | filter paper soaked in the appropriate electrolyte | | |

Figure 5 — Pseudo reference/working electrode for on-site measurement

8 Dummy cell and checking/calibration

It is essential to eliminate unwanted environmental and instrumental noise to allow accurate measurement of the electrochemical noise in a laboratory situation. This can be achieved by use of a Faraday cage. In addition, this can be supported by implementation of appropriate filters that are integrated in the noise measurement device. Reference [\[12\]](#) provides guidelines for the calibration of noise measuring device including the use of a dummy cell with known level of noise from a “sine wave” signal generator or measuring the instrument noise level in an open circuit with a hanging leads configuration.

9 Measurement

The values of voltage and current are measured against time. It is the fluctuations in these values that are of interest.

The standard deviation resistance noise, R_n , was calculated in accordance with Ohm’s law, as shown in [Formula \(1\)](#):

$$R_n = \left[\frac{\sigma(V)}{\sigma(I)} \right] \quad (1)$$

where

$\sigma(V)$ is the standard deviation of potential or voltage fluctuation;

$\sigma(I)$ is the standard deviation of current fluctuation.

Coating degradation can be accompanied by a decrease in $\sigma(V)$ or a significant increase in $\sigma(I)$ or changes in $\sigma(V)$ and $\sigma(I)$ so that the $\sigma(V)/\sigma(I)$ ratio decreases.^[8]

The work that has been done indicates that the difference in potential between the reference electrode and the validation electrode is an important variable and a small value of this is useful.

The exposed surface area of the specimen is an important variable.

One of several electrode configurations (shown in [Figures 2, 3, 4](#) and [5](#)), can be chosen for measurement. The configuration depends on circumstances, e.g. if no easy connection can be made to the metal, then the NOCS ([Figure 4](#)) would be the preferred arrangement.

Depending on the type of cell and probe, the sample is mounted before or after the addition of the test solution. The test solution is added to the electrochemical cell and probe.

In both laboratory and field environments, the temperature is an important variable.

Normally noise data are gathered for 256 s at 0,5 Hz a total of 512 data points, in duplicate. This is compromise between keeping the measurement time short and having enough data to give an accurate value of R_n .

10 Data analysis and data treatment

To achieve acceptable accurate values of electrochemical noise resistance, R_n , some data treatment is typically carried out. The need for this will be indicated by inspection of the data sets, voltage against time and current against time. A typical requirement can be "drift" removal although there are other factors (see [Annex B](#)); many software packages also offer the facility to use selected data and discount outlying data points.

11 Summary

The results given in References [\[3\]](#), [\[5\]](#), [\[6\]](#), [\[10\]](#), [\[11\]](#) and [\[13\]](#) have shown that the method can offer considerable promise as a method of assessing coated metal substrates. More experiments are needed. This document is intended to encourage more extensive use of the technique.

[Annexes A, B](#) and [C](#) provide details on some specific experiments on electrochemical noise measurements.

Annex A (informative)

Description of an experiment with resistors simulating a paint system

A.1 General

This annex describes two specific experiments which were performed, which are relevant to the technique and process of making EN measurements for assessing coated substrates.

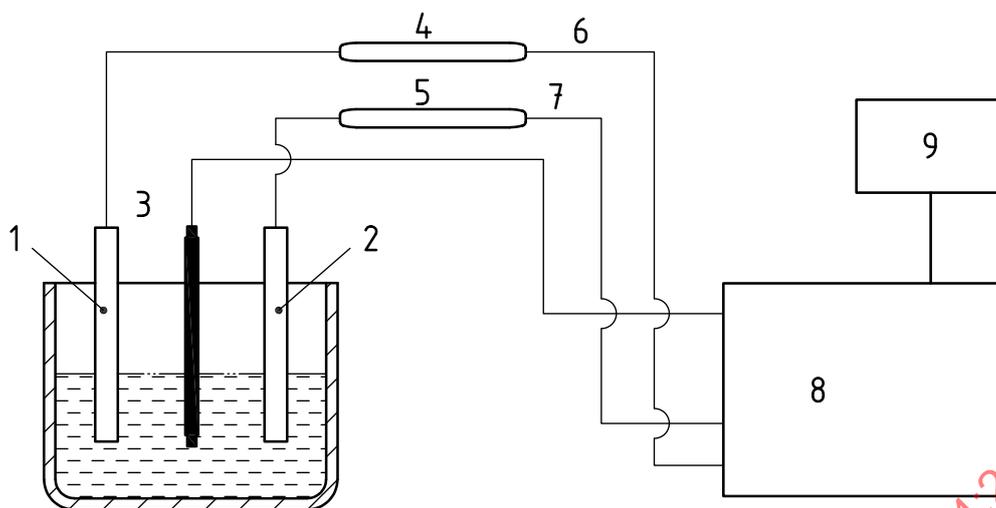
A.2 Experiment 1 — technique

This experiment concerns the technique of making EN measurements for assessing coated substrates. Two 1 cm diameter steel electrodes were abraded to 600 grit. The exposed area was about 10 cm² with the rest of the specimen blanked off, e.g. using beeswax and colophony rosin. These working electrodes were immersed in a solution of citric acid, $c = 0,1$ mol/l, at 25 °C. They corrode, generate noise and provide a data set that is approximately Gaussian. The steel rods were left in the solution for 30 min before the measurements commenced, to allow a steady corrosion rate to be reached. The reference electrode was a standard calomel electrode SCE. A range of resistors from $10^5 \Omega$ to $10^{10} \Omega$ were available. Each working electrode was connected via the same value resistor (e.g. working electrode 1 is connected via resistor 1, working electrode 2 via resistor 2 - see [Figure A.1](#)) to the cell. The values of resistor 1 (R1) and resistor 2 (R2) vary typically from $1 \times 10^6 \Omega$ to $1 \times 10^9 \Omega$ (R1 = R2 in all cases). With each resistor the measurement was repeated five times. The electrochemical noise resistance value, R_n , calculated from these measurements was reproducible and equal to the resistance value of the resistors. A ± 20 % variation of R_n values among the five measurements is acceptable.

NOTE If in practice a data treatment regime is envisaged to be used, then the dummy cell data are put through this regime before calculating the value of R_n .

A.3 Experiment 2 — process

This experiment concerns the process of making EN measurements for assessing coated substrates. The procedure for checking the equipment was as follows: two steel electrodes together with the reference electrode were immersed in the solution and connected to the noise measuring box via R1/C1 or R2/C2 as shown in [Figure A.1](#). The potential and the current noise under standard conditions were recorded. The data was inspected. If necessary, the data was treated for drift removal. The value of R_n was then calculated. The obtained values of R_n were then compared with the nominal values in the dummy circuit as shown in [Figure A.1](#). The difference was within ± 20 % of the nominal value.



Key

- | | | | |
|---|------------------------------|---|---|
| 1 | metal rod 1 | 6 | working electrode 1 |
| 2 | metal rod 2 | 7 | working electrode 2 |
| 3 | standard reference electrode | 8 | zero resistance amperemeter (data logger) |
| 4 | resistor 1 | 9 | personal computer |
| 5 | resistor 2 | | |

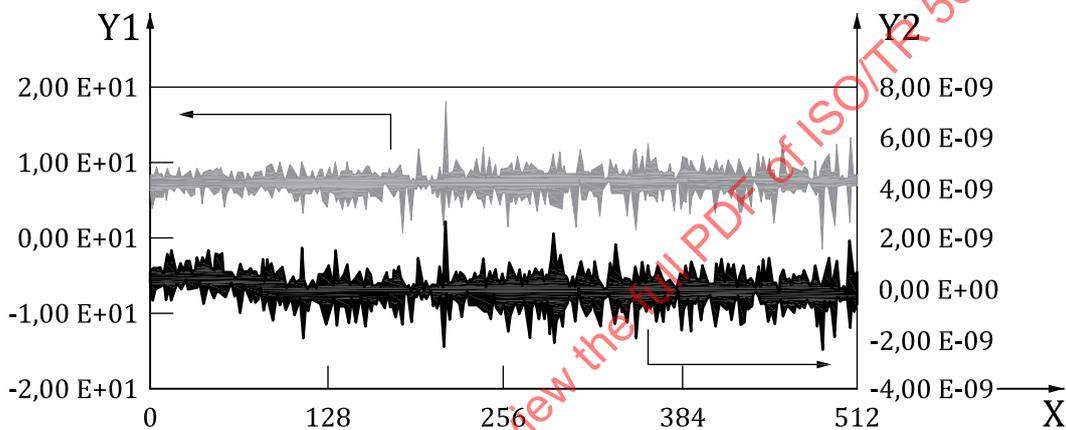
Figure A.1 — Circuit diagram for dummy cell and the connections for checking/calibration of the electronic instrumentation for noise measuring

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Annex B (informative)

Data analysis and data treatment

When it comes to data analysis, the simplest approach is to make a visual assessment of the noise data. To accurately calculate the electrochemical noise resistance, R_n , for coated metal, the data set ideally follows a Gaussian distribution, which would be indicated normally by very few transients. Figure B.1 shows raw noise data collected from a steel surface coated with a soya-based alkyd in NaCl solution, $c(\text{NaCl}) = 0,5 \text{ mol/l}$ with R_n of approximately $2,7 \times 10^9 \Omega \cdot \text{cm}^2$. This data set contains one or two spikes, which is not uncommon. Owing to the very large data set (600 points), these spikes only have a minimal effect on the result. This was shown by taking out the spikes and the value of R_n was virtually the same. Overall, this is an example of useful data.



Key

- X time, in seconds (s)
- Y1 electrochemical potential noise, in millivolts (mV)
- Y2 electrochemical current noise, in nanoamperes (nA)

NOTE The steel was coated with a soya-based alkyd varnish with thickness of $90 \mu\text{m}$ immersed in NaCl solution, $c(\text{NaCl}) = 0,5 \text{ mol/l}$, exhibiting $R_n = 2,7 \times 10^9 \Omega \cdot \text{cm}^2$ (unpublished data)

Figure B.1 – Time records of the potential and current signals from coated

A common problem detected in this data analysis is drift. If significant drift is suspected, the data can be put through a drift removing programme such as the freely available EnAnaliz programme.^[14] These normally operate by subtracting a linear regression line, linear method or a polynomial trend from the raw data resulting in a smaller variation around the mean value zero.

A part of the data set that is giving more Gaussian type of behaviour can be selected using software options.

There is sometimes a case for taking the data into the frequency domain (R_{sn}) for analysis by maximum entropy method (MEM) or fast Fourier transform (FFT) approach. Application of these approaches is outside the scope of this document. Some general information is available in ISO 17093 and specific results obtained in application of these approaches to coated substrates can also be found in Reference [13]. However, for coatings on steel, it has been found that in the majority of cases, there is little advantage in converting the data to obtain R_{sn} rather than use R_n .