

Application Note AN-COR-016

ASTM G61: Standard test method for conducting cyclic potentiodynamic polarization

ASTM-compliant methods from Metrohm Autolab

ASTM G61 is a standardized method to test susceptibility of various alloys of iron, nickel, and cobalt to localized corrosion within a chloride environment [1]. The potential at which the anodic current increases rapidly is an indication of pitting. The higher the potential, for the same scan rates, indicates better protection against pitting corrosion.

By using a Metrohm Autolab instrument and our ASTM-compliant corrosion cells, it is possible to fully meet the requirements of this ASTM standard. The following Application Note describes an example measurement that was made using VIONIC powered by INTELLO according to the guidelines of ASTM G61.

SAMPLE PREPARATION

It is essential that the surface of the sample is free from contamination. Therefore just prior to immersion in the corrosive medium, the sample (a 1 cm^2 disk of Type 430 stainless steel) was cleaned by a combination of mechanical polishing with sandpaper and alternatively rinsing with ultrapure water and isopropyl alcohol.

EXPERIMENTAL

The test consists of polarizing the system toward potentials that are more positive than the open-circuit potential (OCP) until the current reaches a predetermined value (usually 5 mA). Then the scan is reversed, forming a hysteresis loop on the voltammogram. The higher the potential at which the hysteresis loop is closed, the less prone the alloy is to pitting corrosion.

For this experiment, the sample (430 SS) was used as a working electrode (WE). Two Metrohm platinum sheet electrodes were used as the counter electrode. As a reference electrode, an Ag/AgCl 3 mol/L KCl electrode was chosen. The cell used in this study was the ASTM-compliant Metrohm Autolab 1 L corrosion cell. The electrolyte was a 3.5% NaCl solution (artificial seawater).

Nitrogen gas was bubbled into the solution for one hour during the preparation step to remove any oxygen dissolved in the electrolyte. After one hour, the sample was immersed in the electrolyte, and the degassing continued for another hour.

Then 10 minutes before the start of the polarization (i.e., 50 minutes after insertion), the OCP of the counter electrode was recorded using the S2 connection of VIONIC. The OCP of the sample (WE) was measured, and the scan started from 0 V vs OCP. The scan rate used was 167 µV/s with a step potential of 150 µV.

The anodic scan continues until a current cut-off of 5 mA is reached, at which point the scan direction is reversed. The measurement stops when either the corrosion potential (E_{corr}) is reached, or the hysteresis is closed (noted by manual observation).

RESULTS AND DISCUSSION

The OCP (platinum potential) of the counter electrode was recorded as $E_{C\text{-}OCP}$ = 0.24 V. The corrosion potential of the working electrode was recorded as E_{corr} = -0.28 V vs Ag/AgCl.

In Figure 1, the resulting voltammogram (I vs E) is shown.

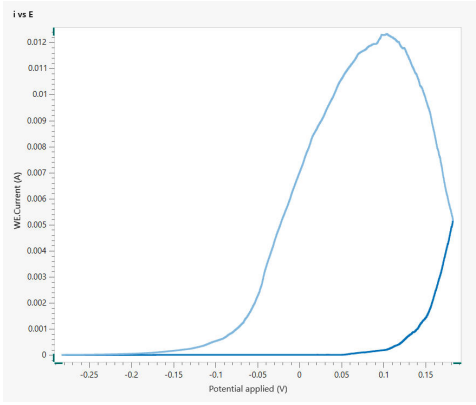

Figure 1. Voltammogram (I vs E) of thev Type 430 stainless steel sample under investigation.

The data is transformed according to ASTM standard G3 [2] in Figure 2, where a plot of the potential (E) vs the log of the current density (j) is shown.
In this case, this sample exhibited an open hysteresis, so the measurement ended once the corrosion potential was reached again. E_{pitt} is the pitting potential and corresponds to the potential at which the pitting (localized) corrosion begins. Between the initial E_{corr} and E_{pitt} is a current density passivity range where new pits are unable to form, but existing ones can propagate [3]. The high hysteresis indicates that the sample has undergone pitting corrosion.

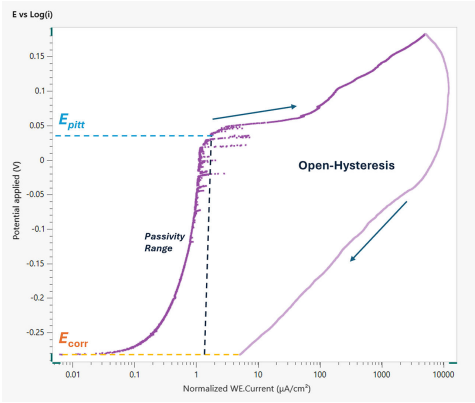

Figure 2. E vs log(j) plot of the sample under investigation. The corrosion potential (Ecorr), pitting potential (Epitt) and the passivity range are all indicated on the plot. The arrows indicate the scan direction.

REFERENCES

1. G61 Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys. https://www.astm.org/standards/g61 (accessed 2024-05-24).
2. Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing. https://www.astm.org/g0003-14r19.html (accessed 2024-03-08).
3. Bellezze, T.; Viceré, A.; Giuliani, G.; et al. Study of Localized Corrosion of AISI 430 and AISI 304 Batches Having Different Roughness. Metals **2018**, 8 (4), 244. DOI:10.3390/met8040244

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CONFIGURATION



Platinum sheet electrode

This electrode consists of a thin sheet of platinum embedded into a glass shaft. This electrode can be used as a counter electrode for most electrochemical measurements. The surface area is approximatively 1 cm2.



Ag/AgCl reference electrode with KCl (length 12.5 cm)

Silver / silver chloride reference electrode with double junction system, installation length 10 cm.

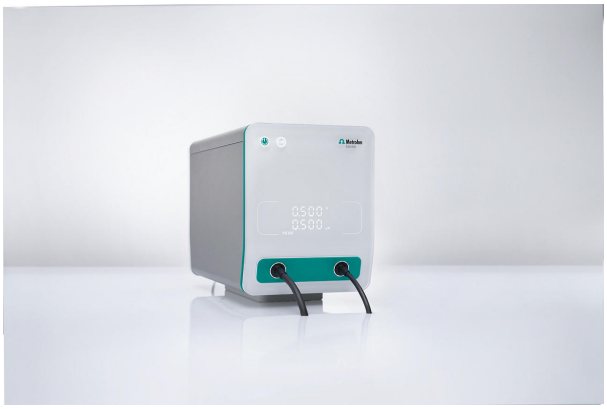
The SGJ 14/15 enables easy assembly and the flexible ground-joint diaphragm, which is insensitive to contamination, can be easily replaced. The reference and bridge electrolyte can be freely selected according to the application and are easy to exchange. This sensor is pre-filled with c(KCl) = 3 mol/L as reference and bridge electrolyte.



1 L corrosion cell

The Autolab 1 L corrosion cell is suitable for corrosion measurements according to ASTM standards. The cell has a thermostatic jacket for temperature control and a series of openings for counter electrodes, pH sensor, thermometer, Luggin-Haber capillary and gas purging.

The 1 ll corrosion cell has been designed to measure the corrosion properties samples with a diameter ranging from 14.7 mm to 16 mm and a thickness from 0.5 mm up to 4 mm. The exposed surface is 1 cm2 and the seals are made out of natural rubber.



VIONIC

VIONIC is our new-generation potentiostat/galvanostat that is powered by Autolab's new INTELLO software.

VIONIC offers the **most versatile combined specifications of any single instrument** currently on the market.

- Compliance voltage: ± 50 V
- Standard current ± 6 A
- EIS frequency: up to 10 MHz
- Sampling interval: down to 1 µs

Also included in VIONIC's price are features that would usually carry an additional cost with most other instruments such as:

- Electrochemical Impedance Spectroscopy (EIS)
- Selectable Floating
- Second Sense (S2)
- Analog Scan