



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 cm² disk of Type 430 stainless steel) was cleaned by a

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 $\mu\text{V/s}$ 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).

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RESULTS AND DISCUSSION

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

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

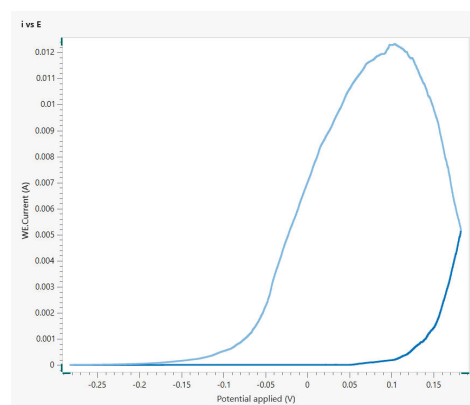


Figure 1. Voltammogram (I vs E) of the Type 430 stainless steel sample under investigation.

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 (E_{corr}), pitting potential (E_{pitt}) 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

CONTACT

Metrohm Suisse SA
Industriestrasse 13
4800 Zofingen

info@metrohm.ch

CONFIGURATION



Électrode à feuille de platine

Cette électrode consiste en une mince feuille de platine insérée dans une tige en verre. Cette électrode s'utilise comme contre-électrode pour la plupart des mesures électrochimiques. La surface active est d'environ 1 cm².



Électrode de référence Ag/AgCl avec KCl (longueur 12,5 cm)

Électrode de référence argent/chlorure d'argent avec système double jonction, longueur d'insertion 10 cm. Le rodage normalisé 14/15 facilite le montage et le diaphragme rodé flexible insensible à la contamination peut être changé à tout moment. L'électrolyte de référence et l'électrolyte intermédiaire peuvent être sélectionnés librement selon l'utilisation et sont faciles à remplacer. Ce capteur est prérempli de $c(\text{KCl}) = 3 \text{ mol/L}$ comme électrolyte de référence et électrolyte intermédiaire.



Cellule de corrosion de 1 L

La cellule de corrosion Autolab 1 L convient aux mesures de la corrosion conformément aux normes ASTM. Cette cellule possède une gaine thermostatique pour réguler la température et toute une série d'orifices pour contre-électrodes, capteur pH, thermomètre, capillaire de Luggin-Haber et purge des gaz.

La cellule de corrosion de 1 L a été conçue pour mesurer les propriétés de corrosion d'échantillons de 14,7 à 16 mm de diamètre et de 0,5 à 4 mm d'épaisseur. La surface exposée est de 1 cm² et les joints sont en caoutchouc naturel.



VIONIC

VIONIC est notre potentiostat/galvanostat de dernière génération piloté par le nouveau logiciel d'Autolab, INTELLO.

VIONIC offre les **spécifications combinées les plus polyvalentes pour un appareil unique** actuellement sur le marché.

- Tension disponible : ± 50 V
- Intensité standard : ± 6 A
- Fréquence de SIE : jusqu'à 10 MHz
- Intervalle d'échantillonnage : jusqu'à 1 μ s

Le prix de VIONIC inclut également des fonctions qui impliquent généralement des coûts supplémentaires avec la plupart des autres appareils, telles que :

- Spectroscopie d'impédance électrochimique (SIE)
- Mode flottant sélectionnable
- Seconde électrode de détection (S2)
- Scan analogique