

Application Note AN-COR-012

ASTM G5: Potentiodynamic anodic polarization measurements

ASTM-compliant methods from Metrohm Autolab

The ASTM G5 is a standard method to test the corrosion of Type 430 stainless steel with a potentiodynamic anodic polarization measurement. The primary use of this test method is actually not in materials testing – it offers a simple way to confirm the accuracy of the test equipment (i.e., PGSTAT and corrosion cell). With a Metrohm Autolab instrument and our ASTM-compliant corrosion cells, it is possible to meet the requirements of this ASTM standard.

This Application Note describes an example measurement that was made using VIONIC powered by INTELLO according to the ASTM guidelines.

SAMPLE PREPARATION

It is critical that the sample surface is free from contamination. Directly before 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. It is important that this is done just before immersing the sample in order to prevent the surface from recontamination.

EXPERIMENTAL

The sample was immersed in 1 N (0.5 mol/L) aqueous sulfuric acid and two Metrohm platinum sheet electrodes were used as the counter electrode. For the reference electrode, an Ag/AgCl 3 mol/L KCl Metrohm reference electrode was chosen. The cell used in this study was the ASTM-compliant 1 L Metrohm Autolab corrosion cell.

To minimize dissolved oxygen, the sulfuric acid solution was deaerated by bubbling nitrogen gas through it for one hour. The Type 430 stainless steel disk was immersed in the solution 30 minutes before the experiment during the N_2 bubbling step. A nitrogen blanket above the solution was maintained throughout the experiment in order to obstruct any atmospheric oxygen diffusion into the solution.

A VIONIC potentiostat/galvanostat was used for the measurement. The procedure and data treatment were performed with the INTELLO software.

The ASTM G5 standard method requires that a voltage scan is initiated from the corrosion potential (E_{corr}, also known as the open-circuit potential OCP) to 1.60 V vs saturated calomel electrode (SCE) [**1**]. In this case, the reference electrode system employed was Ag/AgCl 3 mol/L KCl, so the end potential was adjusted to 1.630 V.

The data was plotted in accordance with the guidelines laid out with ASTM Standard Practice G3 [**2**].

RESULTS

The plot of current (I) vs potential (E) is shown in Figure 1.

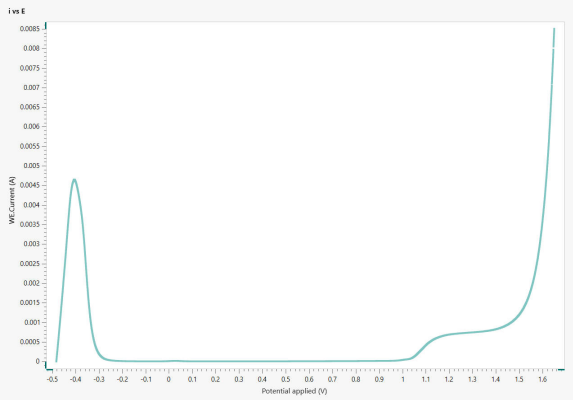

Figure 1. A plot of I vs E for the corroding sample of stainless steel in 0.5 mol/L sulfuric acid solution.

The data is transformed according to ASTM standard G3 in Figure 2, where a plot of the potential (E) vs the log of the current density (j) is shown.

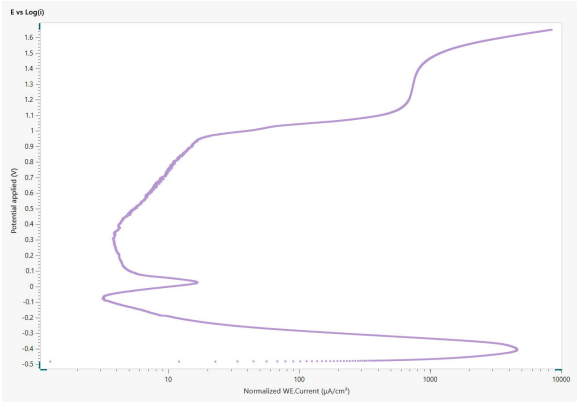

Figure 2. The corresponding plot of log(j) vs E for the corroding sample of stainless steel in 0.5 mol/L sulfuric acid solution.

The common features relating to the oxidation process of stainless steel in acidic solutions are more easily discernible in this plot. These features include the:

1. Active Region
2. Passive Region
3. Transpassive Region
4. Secondary Passivity Region

These four regions are highlighted in the plot In Figure 3.

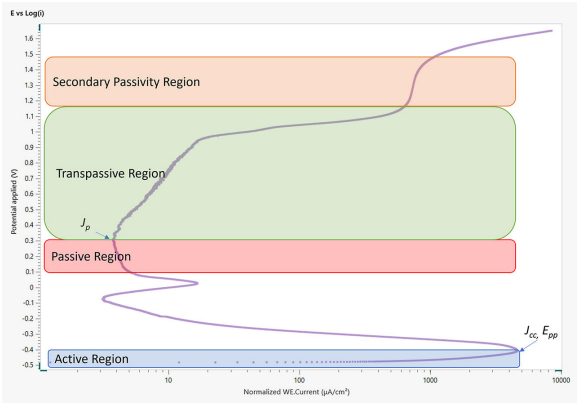

Figure 3. This plot is identical to Figure 2, but the regions in the plot are now highlighted. The Active Region is shown in blue, the Passive Region in red, the Transpassive Region in green, and the Secondary Passivity Region in orange.

The Active Region (Figure 3, in blue) is characterized by a large increase in the current density, corresponding to oxidation (corrosion) of the stainless-steel sample, until the primary passivation potential (E_{pp}) is reached. The E_{pp} also corresponds to a critical current density (j_{cc}). For potentials higher than the E_{pp}, the current density drops off, which is usually associated with the formation of a protective (passivating) layer on the surface of the electrode.

After a small peak, the origin of which is likely related to active dissolution of stainless steel from a Cu-enriched layer [3], the current density does not change substantially from its passive current density value (j_p) even as the potential is increased. This is called the Passive Region (Figure 3, in red).

At potentials higher than where j_p occurs, the current

density increases again due to breaking the passive layer. This is termed the Transpassive Region (Figure 3, in green).
Beyond the Transpassive Region, the curve enters another section where the current density does not increase much with the applied potential. This is termed the Secondary Passivity Region (Figure 3, in orange). Further from this, oxygen evolution can occur, which is not shown in Figure 2.

REFERENCES

1. *Standard Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements*. https://www.astm.org/g0005-14r21.html (accessed 2024-03-08).
2. *Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing*. https://www.astm.org/g0003-14r19.html (accessed 2024-03-08).
3. Ruel, F.; Volovitch, P.; Peguet, L.; et al. On the Origin of the Second Anodic Peak During the Polarization of Stainless Steel in Sulfuric Acid. *Corrosion* **2013**, *69* (6), 536–542. DOI:10.5006/0820

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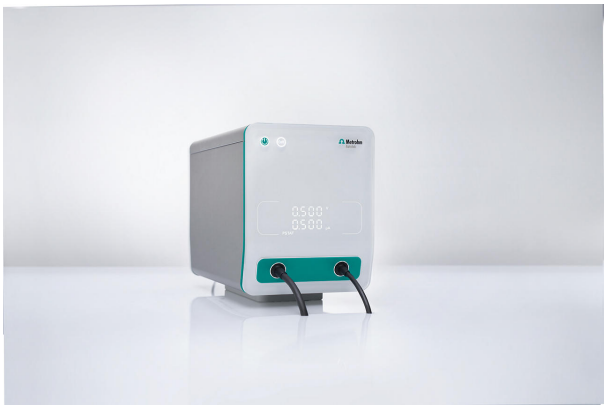
CONFIGURATION



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