

# Electrochemical systems for corrosion measurements



# Corrosion impact

Corrosion is the term generally used to describe oxidative processes that result from the interaction of a metal with environmental elements like oxygen or water. The World Corrosion Organization estimates that corrosion has a yearly cost of more than the 3% of GDP world-wide. In the United States, e.g., this translates into 2.2 trillion dollars<sup>1)</sup>.

For this reason, clear financial and technological benefits result from monitoring these processes and developing effective strategies against them.

<sup>1)</sup> [http://corrosion.org/wco\\_media/nowisthetime.pdf](http://corrosion.org/wco_media/nowisthetime.pdf)

## Reliable corrosion measurements via electrochemistry

### Electrochemical measurements of corrosion processes

Over the past three decades, several methods have been introduced to measure corrosion. Many of these traditional methods, such as weight loss and spray test analysis, can be quick and cost-effective. Unfortunately, the results that they yield are only qualitative. By contrast, electrochemical analysis techniques yield accurate and reproducible quantitative data.

Depending on the nature of the application, different electrochemical techniques can be applied in order to determine the parameters of interest. Metrohm offers fully customizable hardware and software solutions to perform electrochemical experiments and calculate corrosion parameters. The software package offers dedicated methods for corrosion analyses that are in compliance with ASTM standards and industry best practices.

Electrochemical techniques	Parameters of interest	ASTM standards
<b>DC techniques</b> <ul style="list-style-type: none"><li>• Linear sweep voltammetry</li><li>• Tafel slope analysis</li><li>• Potentiodynamic polarization</li></ul>	<ul style="list-style-type: none"><li>• Polarization resistance (Rp)</li><li>• Corrosion rate (mm/year)</li><li>• Corrosion current</li><li>• Corrosion potential</li></ul>	<ul style="list-style-type: none"><li>• ASTM G102</li><li>• ASTM G59</li></ul>
<b>AC techniques</b> <ul style="list-style-type: none"><li>• Electrochemical Impedance Spectroscopy (EIS)</li><li>• Electrochemical Frequency Modulation (EFM)</li></ul>	<ul style="list-style-type: none"><li>• Film resistance and conductivity</li><li>• Charge-transfer resistance</li><li>• Solution resistance</li><li>• Polarization resistance</li></ul>	<ul style="list-style-type: none"><li>• ASTM G59</li><li>• ASTM G106</li></ul>
<b>Chrono and Other Techniques</b> <ul style="list-style-type: none"><li>• Electrochemical Noise (ECN)</li><li>• Critical Pitting Temperature (CPT)</li><li>• Hydrogen permeation</li><li>• Cyclic polarization</li><li>• Forced convection measurements</li></ul>	<ul style="list-style-type: none"><li>• Redox kinetics</li><li>• Pit initiation</li><li>• Crevice progression</li><li>• Hydrogen resistance</li><li>• Surface morphology</li></ul>	<ul style="list-style-type: none"><li>• ASTM G150</li><li>• ASTM G148</li><li>• ASTM G100</li></ul>

# Corrosion parameters by Linear Polarization Resistance (LPR) measurements

The use of a potentiodynamic linear sweep measurement to quantify the corrosion processes is described in the ASTM G59 standard. Typically, this technique is used to measure and monitor corrosion on a day-to-day basis. In this method, a three-electrode setup is employed with the sample of interest acting as the working electrode. A potential sweep is applied and the resulting current is measured. The polarization resistance can be extracted from the resulting data and the Tafel approximation can be applied to calculate the corrosion

rate. Every instrument offered by Metrohm Autolab is equipped with a protocol for conducting a typical LPR measurement consistent with the ASTM G59 standard.

## Application

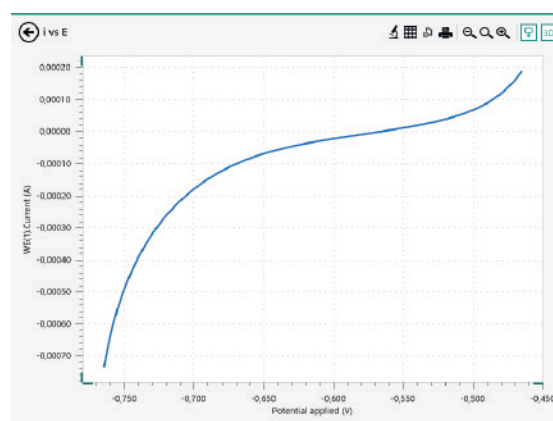
Corrosion monitoring and measurements in industries and corrosion labs.

## Recommended configuration

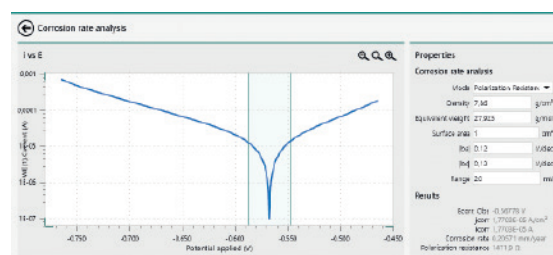
PGSTAT204 + 1L ASTM corrosion cell

### Unique software features for LPR measurements

- Linear polarization protocol with built-in OCP evaluation
- Change voltage or scan rate during a measurement
- Procedure scheduler: For long measurements or multiple runs, experiments can be scheduled with added wait times
- High-resolution vectorial graphics with export and printing options
- Automatic calculation of kinetic rate constants
- Direct determination of polarization resistance and corrosion rates



Polarization resistance



Linear polarization analysis

## Corrosion parameters by Electrochemical Impedance Spectroscopy (EIS)

The use of electrochemical impedance spectroscopy (EIS) is illustrated in the ASTM G106 standard. Rather than a typical DC-based current-vs-voltage study, an AC signal is applied to the sample, the response is measured, and the impedance is then calculated. The data are then analyzed via fit and simulation, to yield film resistance, reaction kinetics, polarization resistance, and other parameters which govern the corrosion processes and reaction mechanisms. The EIS technique is advantageous for the development of corrosion inhibitors and anti-corrosive films.

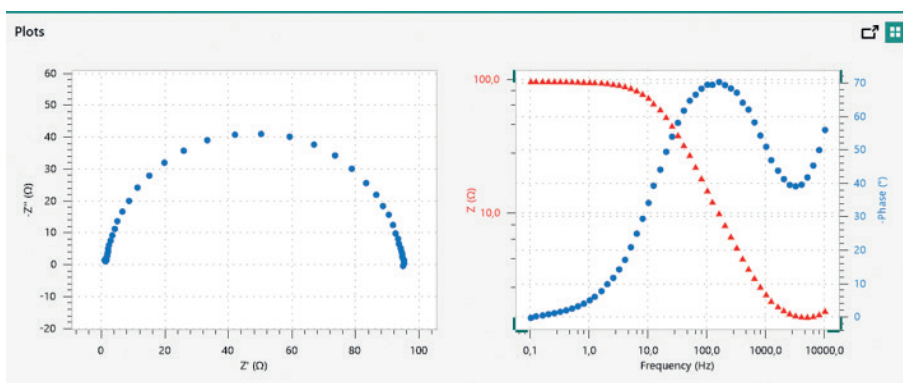
With the FRA32M module, Metrohm Autolab offers the possibility of performing EIS measurements with potentiostatic or galvanostatic control, over a frequency range of 10  $\mu$ Hz to 1 MHz, extendable to 10 MHz with the ECI10M module. We provide dedicated setups for EIS measurements according to the ASTM G106 standard, on single and multiple cells.

### Application

Coatings, corrosion inhibitor testing, and redox kinetics analysis at corrosion research and testing labs.

### Unique software features for EIS analysis

- Real-time EIS analysis and plot generation for:
  - Equivalent circuit fitting and simulation
  - Real-time AC signals, in time domain and frequency domain
  - Lissajous plots
- Six predefined EIS protocols
- Multiple EIS measurements with a single click
- Multi-sine technique for quick low-frequency measurements
- Possibility to sample the potential, current, time, and frequency domains
- Electrochemical frequency modulation (EFM) measurements



Impedance Nyquist and Bode plots





# Corrosion parameters by Electrochemical Noise (ECN) analysis

ECN measures the fluctuations of the raw current and potential, coming from the system, using a low-noise reference electrode or a zero-resistance ammeter (ZRA). The data is examined visually for spikes, or a Fourier transform is applied to obtain a power density spectrum. This yields important parameters for multiple applications in corrosion research.

Metrohm Autolab instruments can be equipped with the dedicated ECN module which increases the potential resolution to 760 nV.

## Application

Corrosion in  $\text{Cl}^-$ -containing electrolytes, pit initiation studies, and crevice propagation.

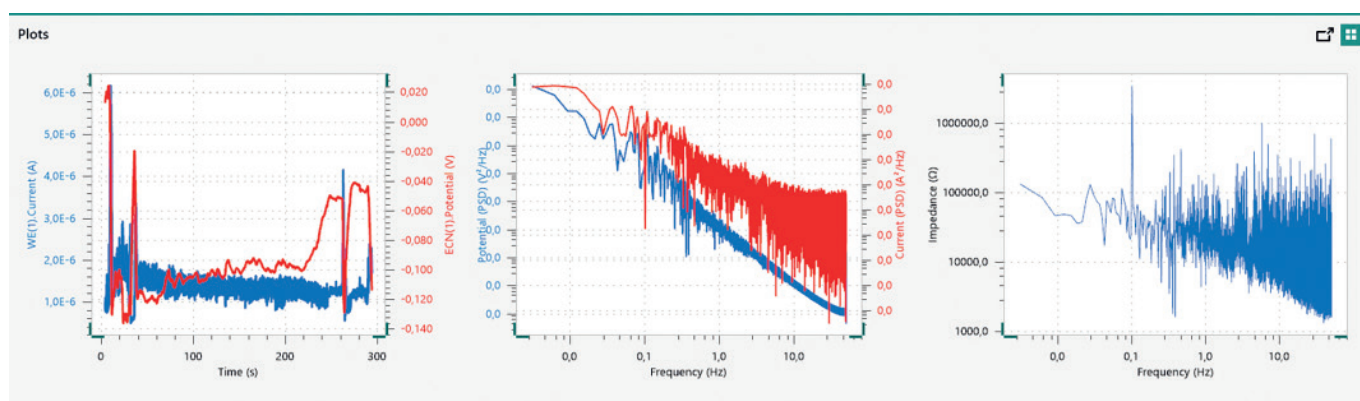
## Recommended configuration

PGSTAT302N + ECN module

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### Unique software features for ECN analysis

- Protocol for power density plots
- Automated ECN spectrum analysis
- Built-in calculation of statistical indicators:
  - Skewness and kurtosis for current
  - Noise resistance
  - Pitting index



ECN spectrum

## Critical Pitting Temperature (CPT) analysis

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The ASTM standard G150 describes the use of CPT for stainless steel samples. This technique is mainly used to obtain relevant information on pit propagation and critical pitting temperature of the samples. At a constant anodic potential, a simultaneous temperature increase is applied at a rate of 1 °C/min and the resulting current is measured. The temperature at which a rapid increase in current is observed is the so-called critical pitting temperature of the sample.

### Application

Pit propagation, pitting resistance, and critical pitting temperature for lab testing of stainless steel samples.

### Recommended configuration

PGSTAT302N + pX1000 module

### Unique software features for CPT analysis

- Temperature control in NOVA through RS232 control
- Auto calibration routine for pX1000 temperature sensor
- Multiple signal sampling for pH, voltage, and temperature
- Procedure sequence editor

*Metrohm Autolab's customized CPT analysis setup provides fully automated temperature control and real-time plotting for current and temperature signals.*



## Hydrogen permeation test

Hydrogen permeation is the process in which hydrogen penetrates and accumulates into the bulk of high-tensile-stress stainless steel alloys; this introduces a cracking hazard. The use of the hydrogen permeation test is described in the ASTM G148 standard using a Devanathan-Stachurski cell.

Electrochemically controlled hydrogen permeation is measured using two electrolytic cells that are separated by the sample under study. Hydrogen is generated electrochemically at the cathode, whereas the hydrogen that has diffused through the specimen is oxidized at the anode. The oxidation current is directly proportional to the amount of hydrogen diffusing through the metal membrane.

### Application




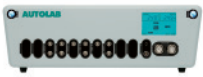

Hydrogen permeation study on metal sheets.

### Recommended configuration

Two floating instruments PGSTAT302F

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## Potentiostats that meet your application needs

					
Parameter	Compact Series	N Series	Multichannel	Special – High Volt	Floating Ground
Compliance volts (max)	10 V, 20 V	12 V, 30 V	10 V, 20 V	100 V	30 V
System current (max)	100 mA, 400 mA, 10 mA	1 A, 2 A, 10 A, 20 A	100 mA, 400 mA, 10 mA	250 mA, 10 A	2 A
Applications	LPR, EIS, CPT, Multiplexing	LPR, EIS, CPT, ECN, Multiplexing	LPR, EIS, CPT, Multiplexing	Concrete corrosion testing, EIS, LPR	Hydrogen permeation, LPR, EIS

## Corrosion cells that fulfill industrial guidelines



### ASTM Grade 1 L Corrosion Cell

- Exposed surface area: 1 cm<sup>2</sup>
- Sample diameter: 14–16 mm
- Sample holder: PP
- Seal: Rubber



### Complete 0.4 L Corrosion Cell

- Exposed surface area: 0.785 cm<sup>2</sup>
- Sample diameter: 14 mm
- Sample holder: POM
- Seal: Viton



### Flat Corrosion Cell

- Exposed surface area: 16.9 cm<sup>2</sup>
- Sample holder: PVC
- Seal: Viton O-ring

### Expand your applications with additional modules

- **FRA32M** – Corrosion inhibitors study, research, and development
- **ECN** – Characterization of coating failures
- **pX1000** – Critical pitting temperature measurements
- **MUX.MULTI4** – Sequential measurements on up to 64 cells



### Measurement and results

Integrating over three decades of user experience, the NOVA software is designed to meet the requirements of industrial and research users by providing the necessary functionality along with user-friendly flexibility. NOVA is equipped to insert fully automated post-data analysis commands in every predefined procedure setup. The drag-and-drop functionality allows easy operation for both basic and advanced users.

### Product warranty

All Metrohm Autolab instruments are backed by our industry-leading 3-year warranty.



[www.metrohm.com](http://www.metrohm.com)

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