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ELECTROLIFE

Enhance knowledge on comprehensive electrolysers technologies degradation through modeling, testing and lifetime prevision, toward industrial implementation

Enhance durability of electrolyser technologies
ELECTROLIFE

- Deliverable report

D4.1 – Specification, terminology and Harmonised protocols for LTEL

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Abbreviations & Definitions

Abbreviation	Explanation
AEM	Anion Exchange Membrane
ASR	Area Specific Resistance
AST	Accelerated Stress Test
BET	Specific Surface Area measured by the Brauner–Emmett–Teller method
BoT	Begin-of-Test
DoA	Description of Action
ECSA	ElectroChemical Surface Area
EDX	Energy Dispersive X-ray
EIS	Electrochemical Impedance Spectroscopy
EoT	End-of-Test
GC	Gas Chromatography
GDL	Gas Diffusion Layer
HHV	Higher Heating Value
ICP	Inductively Coupled Plasma
IEC	Ion Exchange Capacity
LTEL	Low Temperature Electrolyser
MEA	Membrane Electrode Assembly
OCV	Open Cell Voltage
PEM	Proton Exchange Membrane
PTL	Porous Transport Layer
RHE	Reversible Hydrogen Electrode
RT	Room Temperature
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
TIP	Test input parameter
TOC	Test operating conditions
TOP	Test output parameter

RW	Real Word
WE	Water Electrolysis
R_Ω	Series Resistance
R_p	Polarization Resistance

Item	Definition
Dwell time	duration for which a specific condition is maintained
V_{th}	Thermoneutral potential
V_{cell}	Cell potential
F	Faradaic constant
n	Number of electrons exchanged during the reaction
area specific resistance (ASR)	Internal resistance of any component of a cell or a stack normalized by the area. The unit is of Ω cm2.
bipolar plate	A bipolar plate is a component in stack, designed to conduct electricity, distribute gases, and manage thermal and fluid flows. It can be equipped with flow fields to facilitate the distribution of reactants and the removal of byproducts, while also serving as a barrier to prevent the mixing of different gases in the stack.
conditioning	Preliminary protocol required to properly operate a stack/component. It is usually provided by the manufacturer.
crossover	Leakage between the positive and negative electrode sides of a single cell or stack, occurring either through the electrolyte or the sealant.
current density	Current per unit active electrode area (expressed in A/ m^2 or A/ cm^2)
degradation rate	Rate at which a cell/stack's performance declines over time. (usually expressed in μV/h). The period over which the degradation rate is assessed should always be specified.
test input parameter (TIP)	Parameters that can be adjusted to establish the operating conditions of the test object. TIPs must be both controllable and measurable, with their values determined prior to the start of the test.
test output parameter (TOP)	Parameters that indicate the response of the test as a result of variation of TIPs. TOPs are measured during the test.
Calculated Values	Quantities derived from TIPs and TOPs
open circuit voltage (OCV)	The measured voltage of an cell/stack without applying external load.
$\dot{n}_{H2\ measured}$	Hydrogen flow rate measured, expressed in moles per second

1   Introduction

This deliverable aims to implement the protocols and procedures for AEMEL, PEMEL, and AEL technologies, through internally defined protocols and based on those developed in other European projects, such as ANIONE, HPEM2GAS, and the protocols developed by the JRC for these technologies [1], [2], [3], [4].

In detail protocols set the overall strategy and it is a high-level guideline outlining what to do and why it should be done during an experiment, focusing on objectives and standardisation. A procedure is a detailed, step-by-step set of instructions describing how to perform a specific task. It is more precise and operational, ensuring consistency and reproducibility in execution.

The main objective of the Electrolife project is to understand the fundamental cause-and-effect chains of aging mechanisms through the study of aging processes occurring in components and stacks.

In this context, a key objective is the selection of reference protocols to assess degradation in terms of performance, reliability, and durability. Operating conditions, such as temperature, pressure, and supply flow, can significantly influence the device behaviour.

The partners involved in Electrolife come from both the industrial and academic sectors and currently use different protocols for the assessment of their devices. The protocols developed in each laboratory are the result of years of experience and research. The main challenge, therefore, is to establish common protocols that can be applied across different laboratories.

To achieve this goal, the strategy was to create two levels of protocols. This document represents the first level, aiming to propose an initial set of protocols to the Electrolife partners. These protocols are based primarily on literature and the experience of the partners. They will be applied in various laboratories using the components and stacks developed within the project.

The results obtained with these proposed protocols will be compared with the existing protocols used by the partners to identify weaknesses and areas for improvement. Feedback from the Joint Research Centre (JRC) will be critical in aligning the developed protocols with the state of the art and ensuring their relevance and accuracy.

Therefore, particular attention has been given to these operating parameters in the definition of the procedures.

Specifically, the protocols and procedures refer to the characterisation of the following components/devices:

- Durability testing of single cells and stacks under both stationary and dynamic conditions;
- Identification of reference components to evaluate performance degradation phenomena;
- Characterisation of the main components both in situ and ex situ (e.g., MEA, catalysts, plates).

The protocols aim to harmonise the tests conducted across the various laboratories of the project partners and will support the testing activities in WP2, WP5, and WP6.

In this project, degradation phenomena will be studied for components and single cells in WP2, aiming to understand the causes of degradation. Short stack prototypes will be developed in WP6 to assess degradation phenomena and compare scientific results among the partners' laboratories. That said, the developed protocols will be used to evaluate the degradation processes of the studied and developed components. Specific dynamic profiles and steady state conditions will be identified, and their effects on degradation phenomena will be investigated. These procedures will thus allow the

project objectives to be validated under different operating conditions and will be the result of an iterative process of comparison among the project partners.

Developing effective test protocols is crucial for several reasons:

- Identifying Degradation Mechanisms: Measuring the operating voltage of a low-temperature electrolysis system provides a general indication of degradation but does not reveal the specific processes behind it, such as membrane thinning, catalyst dissolution, or contamination. Applying advanced diagnostic techniques, such as electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV), is essential for distinguishing the various contributions to overall degradation.

- Consistency in Results: The lack of standardized protocols leads to variable results across different studies, making it difficult to compare data and draw reliable conclusions.

- Supporting Research and Development: Well-defined protocols enable researchers to systematically test new materials and cell configurations, assessing their effectiveness and durability under real operating conditions.

2 Methods and core part of the report

2.1 Background

2.1.1 Studying Degradation in PEMEL, AEMEL, and AEL Electrolysers Under Steady State Conditions

In the field of low-temperature electrolysis, studying the degradation of key components under constant operating conditions is crucial for understanding the long-term durability and performance of electrolyser technologies, including Proton Exchange Membrane (PEMEL), Anion Exchange Membrane (AEMEL), and Alkaline Electrolysers (AEL). These tests allow to isolate fundamental degradation mechanisms without the additional complexity of dynamic load variations.

For all three technologies—PEMEL, AEMEL, and AEL—constant load tests are essential for evaluating the degradation of the membrane-electrode assembly (MEA) or Electrode Assembly Components (AEL). In PEMEL electrolysers, these tests focus on the stability and thinning of the proton-exchange membrane and the loss of active catalytic surfaces. Similarly, in AEMEL electrolysers, the durability of the anion-exchange membrane is assessed, given its critical role in ion transport. In AEL systems, constant operating conditions help to examine the separator or diaphragm, which is key to maintaining the electrolytic process's integrity over time.

The catalyst layer is another critical component across all three technologies. Constant load tests are used to investigate how the catalyst materials degrade, whether due to dissolution, corrosion, or sintering, over prolonged periods. These tests are particularly important for non-precious metal catalysts often used in AEMEL and AEL systems, as they help determine the long-term viability of these cost-effective alternatives.

In PEMEL and AEMEL electrolysers, the PTL supports efficient gas and ion transport, and constant operating condition tests help evaluate its stability over time. For AEMEL electrolysers, these tests reveal how the PTL withstands the chemical environment and maintains its structural and functional properties. In AEL systems, similar tests are conducted on the electrodes and current collectors to assess their durability under continuous exposure to alkaline conditions.

While bipolar plates are most commonly associated with PEMEL electrolysers, they are also present in some AEMEL systems. Constant load testing of these components is crucial for assessing their electrical conductivity, resistance to corrosion, and mechanical stability over time. These tests help ensure that the plates maintain their performance in harsh operating environments.

2.1.2 Studying Degradation in PEMEL, AEMEL, and AEL Electrolysers Under Dynamic Operating Conditions

The concept of Real-World Degradation (RWD) and Laboratory-World Degradation (LWD) profiles, as presented in the document [5], provides key guidance for dynamic load testing. The RWD profiles represent the actual conditions electrolysis systems experience in operation. These profiles are recommended for both system-level and lab-scale testing to avoid the conservatism inherent in LWD profiles, which simplify the external dynamics. LWD profiles, while useful for some laboratory tests, may not fully account for the complex dynamic conditions that electrolysis systems face during actual operation, particularly in renewable energy environments where power supply is highly variable.

Accelerated Stress Testing (AST) and Accelerated Life Testing (ALT) frequently use dynamic profiles as a basis for their design.

Accelerated stress test (AST) protocols are designed to rapidly induce degradation in electrolysers or specific components while still aiming to simulate realistic stress conditions. These protocols are particularly useful for evaluating the stability and durability of electrolysers under conditions that mimic real operational stresses such as fluctuating loads from renewable energy sources (e.g. start up, shut down). An AST is a test that should correlate to long-term degradation, induced over a shorter test period.

ALT protocols apply destructive conditions that exceed normal operating ranges. The target is testing the system's limits to predict its lifespan or identify weak points.

These dynamic tests aim to trigger degradation mechanisms within the shortest possible time, allowing to quickly gain insights into the stability and durability of the system or its components. By simulating varying operational conditions, these protocols help identify which components are most vulnerable to rapid changes in conditions and how these components respond to stress. This information is crucial for improving the design and operational strategies of electrolysers to enhance their longevity and efficiency.

2.2 Protocols

Developing effective protocols requires a systematic approach that integrates best practices from scientific literature and operational experience. Here are the key steps in developing these protocols:

a) Defining Test Objectives

Literature Review: Examine existing studies to identify the main degradation mechanisms, such as membrane thinning, loss of active catalytic surface, and passivation of bipolar plates. The literature provides a reference framework for defining specific test objectives (WP2).

Operational Goals: Establish key parameters to be monitored, such as operating voltage, internal resistance, and faradaic efficiency. These objectives will guide test design and operational condition selection.

b) Designing the Apparatus and Selecting Test Methods

Instrumentation Selection: equipment should be selected based on the accuracy required for the experiments.

Electrochemical Diagnostic Techniques: Implement techniques such as polarisation, EIS, and CV to monitor degradation processes in situ. Additionally, employing ex-situ methods like scanning electron microscopy (SEM), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectroscopy mapping (EDX -mapping), transmission electron microscopy (TEM), and X-ray diffraction (XRD) is essential for capturing topological and structural changes in materials, providing valuable local information for result interpretation.

c) Conducting Tests and Ongoing Monitoring

Accelerated Tests: Use accelerated tests to simulate years of operation in shorter periods, identifying the primary degradation factors such as high current density and rapid load cycling.

Constant load tests: Constant load testing involves operating the electrolyser at a fixed current density and temperature to evaluate the long-term stability and degradation patterns of the devices.

Degradation Parameter Monitoring: The measurement of parameters such as internal resistance, catalyst activity loss, cross-over, and other related factors can provide valuable insights into degradation phenomena.

Comparison with Literature: Compare results with published data to identify common trends or discover new degradation phenomena. Result interpretation must consider operational variables and differences in the protocols used (WP2).

The dual approach of using both constant and dynamic testing protocols allows for a comprehensive evaluation of the electrolyser performance under a range of conditions. Constant load tests are generally simpler and provide baseline data on component stability and degradation rates, which are crucial for long-term operational planning. On the other hand, dynamic tests are more complex and aimed at ensuring the electrolyser's compatibility with the variable outputs typical of renewable energy systems, thereby addressing both technical and practical aspects of electrolyser deployment.

2.3 Data Analysis

2.3.1 Main electrochemical parameters

In this section, the key parameters that can provide fundamental insights into degradation phenomena during testing are presented. These include the Tafel slope, efficiency, Electrochemical Impedance Spectroscopy (EIS), and the calculation of degradation rates. While this overview is not exhaustive, it aims to provide a foundational understanding of the most relevant electrochemical parameters for analysing degradation. For a more comprehensive and detailed explanation, readers are encouraged to refer to existing literature and JRC documents, which are cited in the following sections. These sources offer in-depth discussions on methodology, interpretation, and application, ensuring a more thorough understanding of the subject.

2.3.1.1 Tafel equation

The **Tafel slope** is a fundamental parameter for characterising electrochemical reactions in electrolysers, as it is closely linked to the kinetics of charge transfer. It represents the slope of the Tafel curve, which describes the relationship between the overpotential (η) and the current density (j) in an electrochemical reaction.

The Tafel curve follows the logarithmic relationship:

$$\eta = a + b \cdot \log(j)$$

Where:

η: Overpotential, the difference between the applied potential and the theoretical potential of the reaction.

a: Intercept, associated with the exchange kinetics of the reaction.

b: Tafel slope (expressed in mV/decade), representing the increase in overpotential required to increase the current density by one order of magnitude.

j: Current density (A/cm^2).

The Tafel slope (b) is determined by the slope of the Tafel curve, obtained by plotting ΔV against $\log(j)$. To obtain the Tafel slope, it is necessary to focus on the kinetic region of polarization curve, where the overpotential is primarily determined by the charge transfer kinetics (and not by mass transport phenomena or ohmic resistances).

The polarisation curve of an electrolyser can be divided into three main regions:

1. **Kinetic region (low current density):** In this region, the behaviour is dominated by the kinetics of the electrochemical reactions. This is where the Tafel slope is typically calculated, as the overpotential is primarily influenced by the charge transfer processes.
2. **Ohmic region (intermediate current density):** Here, the system's internal resistance, including the membrane and electrical resistances, has a significant influence on the overpotential.
3. **Mass transport region (high current density):** At higher current densities, the overpotential is dominated by mass transport limitations, where the supply of reactants and removal of products becomes the controlling factor.

The Tafel slope is determined in the kinetic region, generally observed at low to moderate current densities, where the relationship between the overpotential and the current density is dictated by the reaction kinetics.

If voltage-current relationship is plotted in a logarithmic scale, the main parameters a, b, and j_0 are easly detectable. The relationship between a and j_0 (exchange current density at equilibrium) is:

$$a = -b \cdot \log(j_0)$$

The parameter a is the intercept of the Tafel equation with y-axis, representing the overpotential (η) when the current density (j) is unity ($\log(j)=0$). In the Tafel equation, the intercept with the x-axis (logarithmic current axis) represents the exchange current density (i_0). The exchange current density is the current flowing in the both (anodic or cathodic) direction at equilibrium potential. In figure 1 is shown an example of Tafel's plot provided by CNR.
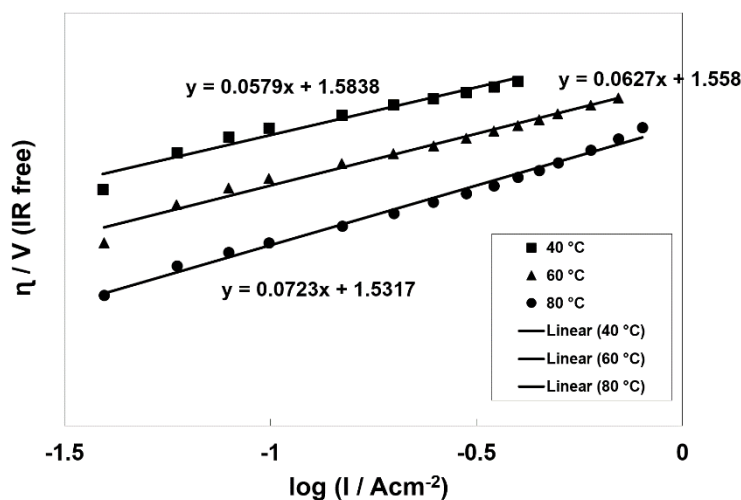
Figure 1. Tafel Plot: graphical determination of the parameters in the Tafel equation (supplied by CNR).

2.3.1.2   Electrochemical Impedance Spectroscopy

Electrochemical Impedance Spectroscopy (EIS) is a sophisticated diagnostic technique used extensively in the characterisation of water electrolysis cells. It is especially valuable for analysing polymer electrolyte membrane water electrolysis (PEMEL), anion-exchange membrane water electrolysis (AEMEL), and alkaline water electrolysis (AEL). Its primary advantage lies in its **non-destructive nature**, allowing detailed insights into cell performance under operating conditions without causing damage. By applying a small sinusoidal perturbation, either as a current or a voltage, across a wide frequency range, EIS measures the cell's impedance response.

EIS permits to separate and quantify different contributions to the cell's total impedance. For instance, it identifies ohmic resistance (R_Ω), which arises from electron and proton conduction in the cell's components, such as the membrane and electrodes. Additionally, EIS can measure polarisation resistance (R_p), which reflects losses due to electrode kinetics and mass transport limitations. Beyond these, other parameters like charge transfer resistance, double-layer capacitance, and adsorption processes can also be analysed.

The frequency range covered by EIS captures phenomena occurring on very different time scales—from microseconds to seconds. This wide range allows to study processes like charge transfer, interfacial capacitance, and even gas diffusion, all of which play a role in the overall performance of the electrolysis cell.

The data collected through EIS is typically represented in two key forms. A **Nyquist plot** visualises the relationship between the real and imaginary components of impedance. A **Bode plot** displays impedance magnitude and phase angle across the frequency spectrum, revealing complementary details.

A Nyquist plot often shows one or more semicircles. Each semicircle represents a specific electrochemical process, such as charge transfer at the electrode/electrolyte interface coupled with the double-layer capacitance.

The **high-frequency intercept** of the plot with the x-axis represents the **ohmic resistance** (R_Ω), which includes the resistance of the electrolyte, components, and electrical connections. In low-impedance systems, such as WE, the parasitic inductance from the wires and connectors often becomes more

visible at higher frequencies. The inductive response can distort the EIS spectrum, leading to incorrect conclusions about the system's behaviour.

The **low-frequency intercept** represents the sum of R_Ω and the polarisation resistance (Rp).

Although frequency is not explicitly displayed on a Nyquist plot, the progression from high to low frequencies can be determined. High-frequency processes (e.g., electron conduction) appear closer to the origin, while low-frequency processes (e.g., mass transport or gas diffusion) appear further away.

The total **polarisation resistance** is the sum of all resistive contributions, excluding the ohmic resistance:

$$R_p = R_{ct} + R_{mt}\_$$

Charge Transfer Resistance (R_{ct}) represents the resistance to the electrochemical reactions occurring at the electrode/electrolyte interface, specifically the transfer of electrons or ions during redox reactions. **Mass transport resistance (R_{mt})** is linked to diffusion of species to the electrode surface. The Nyquist plot provides a visual representation of the total impedance of the system, but it does not directly distinguish the contributions of each process. While R_p can be identified as the difference between the high-frequency and low-frequency intercepts on the x-axis, isolating R_{ct} requires a more detailed approach. An **equivalent circuit model** is necessary to decompose the total impedance into individual contributions.

To account for capacitive effects, the equivalent circuit must explicitly include d**ouble-layer capacitance** (C_{dl}). This is typically represented as a **parallel element** with the charge transfer resistance. The **C_{dl}** significantly influences of the semicircle in the Nyquist plot. This capacitance arises from the accumulation of charges at the electrode/electrolyte interface, and it interacts with the **R_{ct}** to define the characteristic semicircular arc. While the **diameter** of the semicircle is determined by the **charge transfer resistance (Rct)**, C_{dl} affects **frequency range**, and **position** of the semicircle along the frequency spectrum.

The **frequency at which the semicircle peaks** (where the imaginary part −Z'' is maximum) is inversely proportional to C_{dl}; a larger C_{dl} shifts the peak to lower frequencies, a smaller C_{dl} shifts the peak to higher frequencies, compressing the semicircle.

The **Bode plot** is complementary to the **Nyquist plot** in electrochemical impedance spectroscopy (EIS) because it provides a clearer view of how the system's impedance varies across different frequencies. While the Nyquist plot focuses on the relationship between the real and imaginary components of impedance, the Bode plot presents two distinct perspectives:

1. **Magnitude vs Frequency**:

- Shows the absolute value (or modulus) of the impedance (|Z|) as a function of frequency (f).

2. **Phase Angle vs Frequency**:

- Displays the phase angle (φ) between the applied perturbation (voltage or current) and the system's response, also as a function of frequency.

The **Bode plot** shows the behaviour of impedance across the entire frequency spectrum, helping to identify which processes dominate at specific frequencies. It provides a clearer separation of these processes because each phenomenon typically appears as a distinct feature in the modulus and phase angle plots. For instance, at very high frequencies, the phase angle approaches 0°, corresponding to purely resistive behaviour dominated by the ohmic resistance (R_Ω). At intermediate frequencies capacitive behaviour begins to dominate due to C_{dl} and the phase angle moves toward -90° if the capacitance is significant. At low frequencies, the phase angle may deviate as mass transport or diffusion effects begin to influence the system, causing ϕ to rise again. This corresponds to the onset of Warburg impedance or similar phenomena. The Warburg impedance plays a crucial role in describing the impact of diffusion-controlled processes in electrochemical systems, particularly at low frequencies. It represents the resistance to ionic or molecular diffusion within the electrolyte or electrode.

In the magnitude /frequency plot at high frequencies**,** the impedance magnitude typically approaches the value of the ohmic resistance (R_Ω). This reflects the combined electronic and ionic conduction in the cell's components, such as the electrolyte, membrane, and electrode materials.

At intermediate frequencies**,** the impedance magnitude starts to show the influence of the charge transfer resistance (R_{ct}) and the double-layer capacitance (C_{dl}). These contributions are associated with the electrochemical reactions at the electrode/electrolyte interface and the ability of the double layer to store charge dynamically.

At low frequencies**,** the impedance magnitude increases. This is due to limitations in mass transport, such as the diffusion of reactants and products, which become significant at these slower timescales. This behaviour highlights processes related to mass transport resistance (R_{mt}) and other diffusion-driven phenomena.

The use of **Electrochemical Impedance Spectroscopy (EIS)** requires thorough study of the relevant literature to ensure proper understanding and application of the technique. The **JRC protocols** provide specific guidelines and detailed instructions on how to perform EIS effectively, serving as a valuable reference for researchers and practitioners in the field [2].

2.3.1.3   Determination of the degradation rate

During long-term experiments, unplanned interruptions are inevitable, making it difficult to establish a fixed number of operational cycles.

During the shutdown an optimisation of cell/stack components, such as diffusion layers and other structural elements can help to minimise recoverable losses (e.g. reduction of the accumulation of bubbles in the catalyst layer or membrane interface.). Anyway, intentional start-up and shutdown cycles can also be necessary for collecting intermediate diagnostic data, using techniques such as polarisation and impedance analyses. This method ensures that partial results are available, even if unforeseen interruptions occur.

The current JRC methodology described in the harmonisation document for evaluating voltage degradation over time focuses on separating the changes in voltage into reversible and irreversible components [5]. Reversible voltage increases are those that can be recovered after a rest or recovery

period, whereas irreversible voltage increases are permanent changes caused by material degradation or other irreversible processes.

Each test is structured into operational blocks, and voltage measurements are recorded at two critical moments: at the end of each block, just before the system is shut down (referred to as the endpoint voltage), and at the start of the subsequent block, immediately after the cell/stack start up again (the initial voltage). Comparing these two sets of measurements allows to identify and quantify both reversible and irreversible contributions to voltage changes. Figure 2.
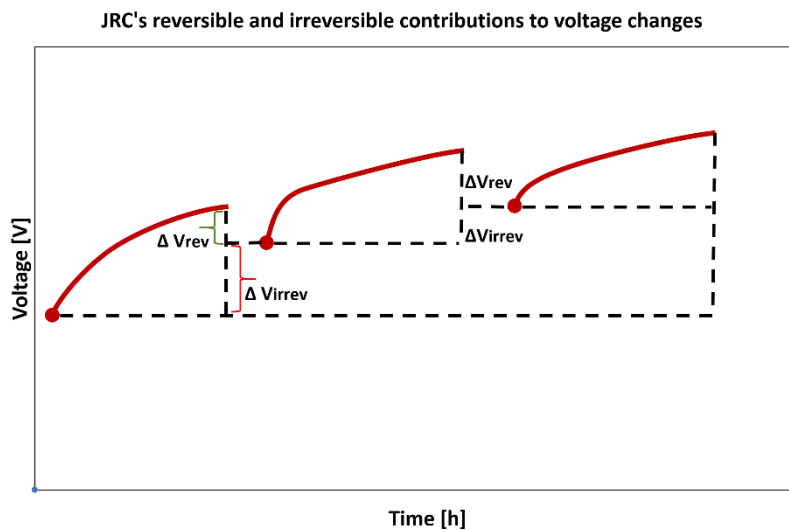

Figure 2. JRC's reversible and irreversible contributions to voltage changes.[5]

The voltage degradation rate is expressed as the total voltage increase (sum of reversible and irreversible contributions) divided by the total operational time (Figure 3):

$$\Delta V_{tot}/\Delta t_{tot} = (\Delta V_{rev} + \Delta V_{irrev})/\Delta t_{tot} \quad \text{(Total voltage degradation rate)}$$
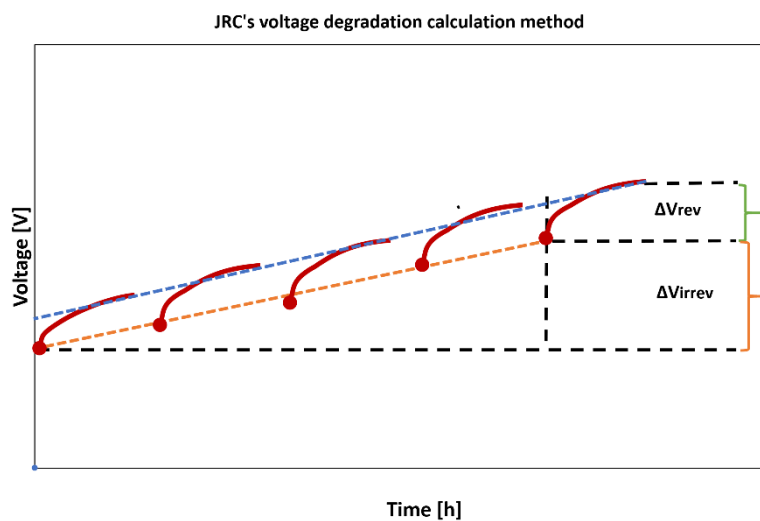

Figure 3. JRC's voltage degradation calculation method.[5]

Here, an alternative method is proposed to assess the voltage degradation rate over a long-term test. In detail, when analysing reversible and irreversible losses, the endpoints of each operational period, recorded immediately before shutdown is considered rather than the initial points taken after start-up. This preference derives from the fact that rest periods at open-circuit voltage, which can differ in length, may influence the initial values.

Endpoints can be employed for linear trend analysis of voltage degradation, provided that the operational periods are sufficiently long to minimise the impact of recoverable losses associated with start-up events (e.g. 500 hrs) Figure 4. The degradation rate is the slope of the linear trend analysis.
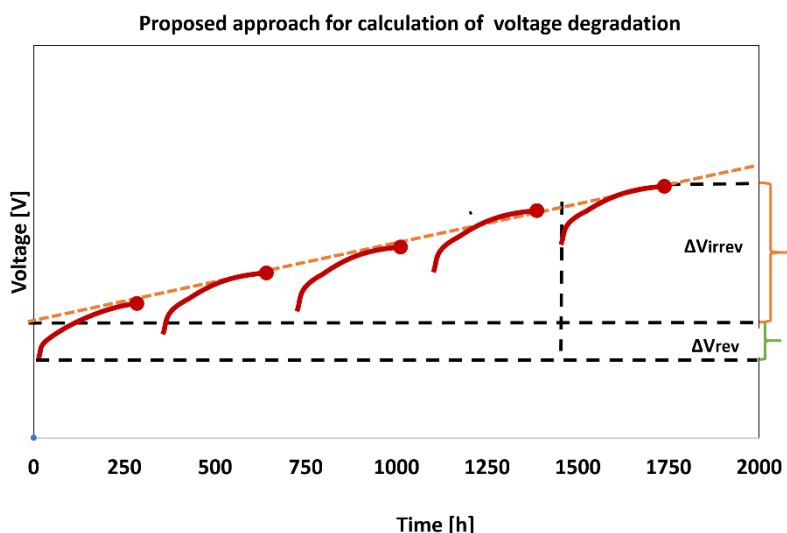

Figure 4. Proposed approach for calculation of voltage degradation.

Both approaches can be applied to ensure comparability among the partners' laboratories, facilitating the assessment of the methods during real durability tests and identifying potential issues in their application. Indeed, real data obtained during stability tests are often difficult to interpret due to problems such as equipment failures or unexpected behaviour of the cell/stack.

2.3.1.4   Calculation of efficiency

Efficiency in low-temperature water electrolysis is a critical parameter that determines the performance of electrolyser cells, stacks, and systems. It quantifies the relationship between the energy input (electricity and heat) and the chemical energy output (hydrogen) during the electrolysis process. Efficiency calculations can be performed at different levels—cell, stack, and system—and are influenced by thermodynamic principles and operational parameters.

The calculation methodology is detailed in the **Joint Research Centre (JRC)** report, which provides a harmonized framework for defining and calculating efficiency across different electrolysis technologies.

Below, the main formulas for determining the efficiency of single cell and stack, focusing on the exothermic operating condition, where the cell voltage exceeds the thermoneutral potential ($V_{cell} > V_{tn}$).

$$\eta_\omega^{cell} = \varepsilon_{cell} \cdot \eta_I^{cell} \qquad\qquad\qquad \textit{(cell overall efficiency)}$$

$$\eta_I^{cell} = 2 \cdot F \cdot \dot{\eta}_{H2measured} \cdot /I_{DC} \qquad\qquad \textit{(cell Faradaic efficiency)}$$

$$\varepsilon_{cell} = (n \cdot F \cdot V_{tn})/(n \cdot F \cdot V_{cell}) = V_{tn}/V_{cell} < 1 \qquad \textit{(cell efficiency in exothermic case)}$$

$$\eta_{I\,stack}^{H2} = 2 \cdot F \cdot \dot{n}_{H2\,measured}/(N \cdot I_{DC}) \qquad\qquad \textit{(stack Faradaic efficiency)}$$

$$\eta^{HHV} = (HHV \cdot \dot{n}_{H2\,measured})/(P_{thermal} + P_{electrical}) \qquad \textit{(overall stack efficiency)}$$

Where:

$\dot{n}_{H2\,measured}$ = flow rate of the produced hydrogen expressed in moles per second;

HHV = Higher Heating Value of hydrogen expressed in joules per mole of hydrogen;

F = Faraday constant;

N = number of cells;

I_{DC} = direct current provided expressed in amperes

V_{tn} = thermoneutral voltage

3 Results & Discussion

3.1 Single-cell and stack protocols

The protocols for evaluating the MEA (Membrane Electrode Assembly) in single cells or in a stack primarily focus on three critical aspects:

- **Efficiency:** Ensuring optimal energy conversion rates.
- **Performance:** Assessing the hydrogen production under various operational conditions.
- **Durability:** Conducting both stationary and accelerated ageing tests to evaluate the long-term stability and reliability of the materials.

The polarization curve, which illustrates the relationship between voltage and current density, will be correlated with key operating parameters, such as pressure, temperature, and current density. This will help in understanding the impact of these factors on the MEA's overall performance.

The Consortium will identify the most suitable single-cell housing for material assessment, considering the available cell sizes and configurations within the partners' laboratories. This selection is crucial for standardising test conditions across different research facilities.

The MEA assessment is not only a method for studying catalysts and membranes but also for understanding the intricate interactions between these components and their assembly in situ. This comprehensive approach allows for the real-time evaluation of material behaviour under practical operating conditions.

Characterising the membrane in MEAs involves conducting in situ conductivity measurements under a variety of operating conditions, including varying pressures, temperatures, and current densities. This step is essential for determining the membrane's ionic conductivity, which directly influences the cell's efficiency.

Tests on catalysts within MEAs will focus on evaluating electrochemical performance across a range of operating conditions, including start-up and shutdown cycles, which are critical for real-world applications. AC impedance spectroscopy will be employed to investigate the internal resistances of the cell, with the series resistance extracted from Nyquist plots. This value will be corrected by subtracting the pure resistances attributed to the cell components. The resulting conductivity could be then be normalised relative to the membrane thickness and electrode area, providing a standardised metric for comparison.

The procedures for in situ gas crossover measurements of the membrane during practical operation will include both sensor-based and electrochemical methods (specifically for H_2). These measurements are vital for assessing the membrane's integrity and preventing unwanted gas permeation, which can lead to performance losses and safety risks.

An incomplete or inadequate activation (or conditioning) procedure for the membrane prior to degradation testing can significantly affect the results and the conclusions drawn from such tests. So, it is necessary defining and carrying out a suitable conditioning procedure before any relevant degradation test. Proper membrane activation is essential to ensure that the results of different degradation tests are comparable and that accurate conclusions can be reached [6].

3.1.1 MEA performance evaluation

The performance evaluation of MEAs (Membrane Electrode Assemblies) begins with initial testing in a small single-cell setup. Before conducting electrochemical tests, it is essential to perform cell compression and leak tests to ensure the structural integrity and reliability of the setup.

MEA assessment is typically conducted at a reference temperature table 1. However, in certain scenarios, the temperature may be adjusted, considering any limitations posed by the membrane or the test setup. This flexibility is important to avoid compromising the membrane's integrity or the accuracy of the evaluation.

In the Electrolife project, the focus is on investigating degradation processes to enhance the efficiency and longevity of low-temperature water electrolysers. Understanding these processes is critical for improving the overall performance and durability of the electrolysers in real-world applications.

One key aspect of this evaluation is managing the temperature gradient within the electrolyser. The temperature difference between the inlet and outlet of the electrolyser should not exceed 5°C. If this limit is approached or exceeded, it is advisable to increase the recirculation flow rate of the water or solution to maintain a stable temperature gradient, which is essential for consistent operation.

The temperature of the cell should be measured at the cathode or anode outlet, as this provides a representative measure of the cell's working conditions. Additionally, temperature measurements at both the inlet and outlet of the cell are recommended to monitor and control the effects of temperature variation on cell performance.

The operating voltage for all three technologies (PEMEL, AEMEL and AEL) typically ranges between 1.5 V and 2.2 V. This range is common for water electrolysis processes, as it represents the voltage required to overcome the electrochemical barriers and initiate the water-splitting reaction. Thus, while the operating voltage is fairly consistent across the technologies, the current density differs significantly, with PEM operating at much higher densities than AEM and AEL (table 1). This variation impacts the efficiency, size, and operational costs of each technology.

The recommended reference operating conditions for a low-temperature water electrolyser stack, as outlined by the JRC (Joint Research Centre), are provided in Table 1[3], [5].

Table 1. Reference operating conditions for typical WE stacks and 2030 SRIA targets (AEL, AEMEL, PEMEL) [3], [5], [7]

Parameter	Unit	AEL	AEMEL	PEMEL
Current density	A/cm2	0.4 /1*	1/1.5*	2/3*
Temperature	°C	80 (±2°C)	60 (±2°C)	80 (±2°C)
Hydrogen pressure	kPa(g)	100 (±2%)	100 (±2%)	100 (±2%)
Water quality used for sol. preparation /Electrolyte concentration	$\mu S.cm^{-1}$/KOH%	30	≤1.0 ISO 3696 Grade 2 @ 25 °C; (target values for KOH based electrolyte < 1.0 % mol)*	≤1.0 ISO 3696 Grade 2 @ 25 °C
Minimum water inlet flow rate (anode/cathode)	$mL/min/cm^2$	1	2	2

D<4.1> – < Specification, terminology and Harmonised protocols for LTEL > (PU)

Degradation*	%/1,000h	0.1	0.5	0.12

*SRIA target 2030 for Renewable Hydrogen production.

In standard conditions, water or solution feeding is provided in both compartments. However, if only one electrode is fed, this must be specified.

Additionally, it must be specified whether the cell operates under differential pressure or balanced pressure.

Before conducting tests, it is necessary to condition the MEA according to the specifications provided by the manufacturer. The operating temperature, duration, and conditioning current must be specified and documented.

After conditioning it is recommended that the cell/stack has reached a steady state condition. The cell should be maintained at low current density and the variation of voltage should not be larger than 1% during the first hour as reported in the document "EU harmonised polarisation curve test method for low-temperature water electrolysis" released by JRC [3].

3.1.2 Polarization curve test procedure

The polarization curve can be obtained either by a linear current sweep or by a stepwise steady-state current sweep. For the linear current sweep, the current is applied first from the lowest to the highest current density (forward scanning), followed by the highest to the lowest current density (backward scanning); the current is linearly varied and the potential recorded.

For the stepwise steady-state current sweep the dwell time should be 60 s. The potential is averaged during the period of acquisition. Alternatively, the last recorded potential point can be considered representative of the stable condition at a specific current density. These two approaches can be compared to highlight their differences. The hydrogen concentration at the anode must be maintained below the flammability limit (e.g. 4% H_2 in O_2 @ 25 °C; 1 bar) during the tests, and a warning signal is suggested around 1% Vol- H_2 in O_2. The flammability limit depends on the operating pressure and temperature. Other pressure explosion limits of H_2 in O_2 are reported in the JRC document [5].

The value of the current density applied for each step is inspirited to the indications from the JRC document "EU Harmonised Polarisation Curve Test Method for Low Temperature Water Electrolysis"[1] and from experience from EU Projects and partners experience.

In Table 2 below, the method used to assess MEA performance in a single cell/stack through the polarisation curve is detailed. If the current density to be achieved exceeds 1 A/cm², the increment in current density should be maintained at 0.1 A/cm².

Table 2. Steps for current density to create a polarization curve.

|	Current Density[1] [A·cm^{-2}]			Cell Voltage	Dwell Time[2]
	PEMEL	AEMEL	AEL	[V]	[s]
1	0.0005	0.0002	0.0002		60
2	0.001	0.0005	0.0005		60
3	0.002	0.001	0.001		60
4	0.005	0.002	0.002		60
5	0.01	0.005	0.005		60
6	0.02	0.01	0.01		60
7	0.03	0.02	0.02		60
8	0.04	0.03	0.03		60
9	0.06	0.04	0.04		60
10	0.08	0.05	0.05		60
11	0.1	0.06	0.06		60
12	0.15	0.08	0.08		60
13	0.2	0.1	0.1		60
14	0.25	0.12	0.12		60
15	0.35	0.15	0.15		60
16	0.4	0.2	0.2		60
17	0.45	0.25	0.25		60
18	0.5	0.3	0.3		60
19	0.6	0.35	0.35		60
20	0.7	0.4	0.4		60
21	0.8	0.45			60
22	0.9	0.5			60
23	1	0.6			60
24	1.1	0.7			60
25	1.2	0.8			60
26	1.3	0.9			60
27	1.4	1			60
28	1.5				60
29	1.6				60
30	1.7				60
31	1.8				60
32	1.9				60
33	2				60

(1) The set points at low current densities may be omitted at the beginning, depending on the specific equipment used for the test.

(2) The dwell time should be chosen so that the cell voltage does not deviate by more than ± 5 mV as reported in JRC document [1]. Anyway, a minimum dwell time of 60 seconds is suggested.

Detailed descriptions of the techniques and steps to follow during the test, including indications on flow rate, pressure, temperature, and how to conduct impedance spectroscopy, are provided. The protocol reported below can be used to test the single cell/stack at different temperature and pressure according to the project targets.

Figure 5. Example of a polarization curve with test conditions included in ascending mode. The polarisation curve should be performed in both ascending and descending modes.
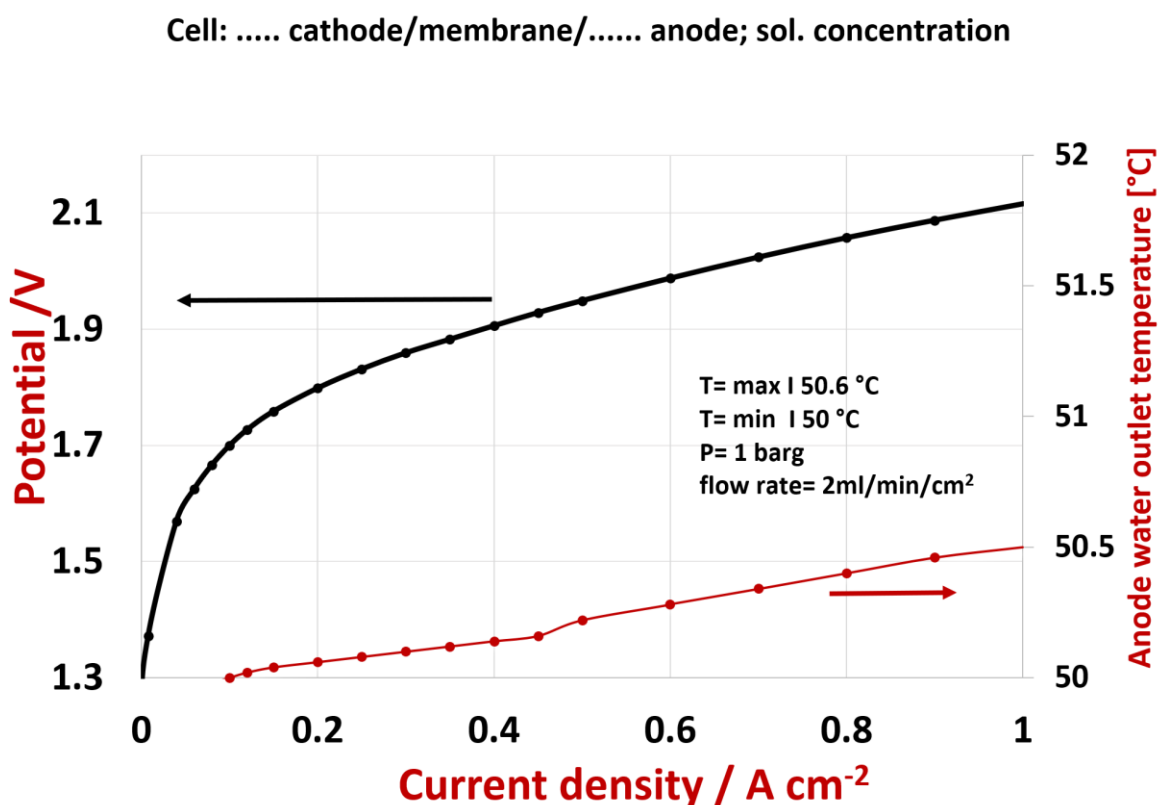
It is suggested to display measurement uncertainty error bars for each point on the curve of the plot. In Table 3, the main Test Input Parameters (TIPs), Test Output Parameters (TOPs), and values derived from the experiment are presented. The list of this parameters is specific for this test; Calculated Values are derived from TIPs and TOPs. With this test, the Area Specific resistance (ASR) is calculated as the slope of the curve at a specific current density. This method is valid in the j-V curve region with almost linearity behaviour.

Table 3. Main Test Input Parameters (TIPs), Test Output Parameters (TOPs) and calculated values for the polarization curve test.

TIP	TOP	Calculated Values
Inlet cell/stack Temperature	Voltage of cell/stack	Current density
Rate of change of current (dwell time)	Outlet cell/stack temperature	Area Specific Resistance (ASR)
Solution/water flow rate	Average temperature of cell/stack	Power density

Current		efficiency
Pressure		

Table 4 provides a detailed description of the steps to be followed during a single cell/stack performance test. The polarization curve and EIS are included in the procedure as methods to assess the device's performance.

Table 4. Single Cell/stack performance evaluation test procedure [1]

MEA conditioning	1. A**s suggested by manufacturer**
Parameters Setting	2. Set the flow rate at the nominal/reference value 3. Check the water quality according to the reference conditions reported in table 1. 4. Establish the potential cut-off, according to the maximum current density or other test constraints. 5. Set pressure and temperature to the reference values;
Cell Performance Evaluation	6. Carry ac-impedance analysis Galvanostatic mode: 0.2 A cm^{-2}, 100 kHz to 100 mHz, amplitude of 5% pk-pk of the applied current Nominal current A cm^{-2}, 100 kHz to 100 mHz, amplitude of 5% pk-pk of the applied current. Potentiostatic mode: 1.5 V and 1.8 V. from 100 kHz to 100 mHz 10 mV rms oscillation. 7. Carry out the polarization curve as previously defined, in both descending and ascending modes. 8. Monitor H_2 concentration in oxygen in the anode compartment throughout the entire duration of the test, and reduce the pressure if the concentration exceeds 3%.
Data Presentation	9. Represent the data in a plot as Potential (V) vs. Current density (A cm^{-2}) 10. Include in the data the temperature at the cell outlet and inlet (if possible), pressure, catalyst loadings, and feed mode.

(I) If the tests are conducted under conditions different from the reference ones, it should be specified. EIS range frequency depends on equipment used in the different labs.
(2) The values reported in the table for EIS represent the minimum requirements to ensure a comparison among the laboratories.

3.1.3 Durability test under steady state condition

Tests conducted under constant operating conditions in low-temperature electrolysis stack or single cell are mainly used to examine how the device degrades over time when external stressors, such as variable loads, are not introduced. These tests help researchers understand the electrolyser's performance and stability under steady-state conditions, allowing them to identify the impact of specific factors like operating current, temperature, pressure and duration on the device's overall durability.

Constant load tests are straightforward to implement and offer a clear picture of the device's inherent degradation patterns. The results from these tests could be used as a baseline, providing essential data that can be compared with findings from more complex tests, such as those involving dynamic loads or stress conditions. This baseline is also useful for validating new materials or design changes in the electrolyser components, ensuring they meet expected performance over time. Below is the procedure for conducting a durability test.

By conducting degradation tests under constant operating conditions across PEMEL, AEMEL, and AEL technologies, researchers can gain valuable insights into the fundamental degradation mechanisms affecting each component.

In table 5, the main TIPs, TOPs, and Calculated Values derived from the experiment are presented. The degradation rate of device tested is calculated as reported in section 2.3.1.4 of this document.

Table 5. Main Test Input Parameters (TIPs), Test Output Parameters (TOPs) and calculated values for steady state test.

TIPs	TOPs	Calculated Values
Inlet cell/stack Temperature	Voltage of cell/stack	Current density
Solution/water flow rate	Outlet cell/stack temperature	Power density
Current		efficiency
Pressure		Degradation rate of cell/stack voltage ($\Delta V/\Delta t$) (see section 2.3.1.4)
		Average temperature of cell/stack

The procedure for performing this type of test is outlined in Table 6. The TIPs should be defined and remain constant throughout the test. Both TIPs and TOPs should be recorded during the testing process. The test duration should be determined based on the test objectives. The JRC recommends 3000 hours for long-term durability tests; however, the test may be terminated earlier if cut-off criteria are met or if issues arise with the device or equipment.

The initial 100 hours are typically excluded from analysis, as the single cell or stack undergoes a transient phase of operation during which the degradation rate may be significantly high.

Table 6. Single Cell/stack durability test procedure steady state condition [1]

MEA Conditioning	1. A**s suggested by manufacturer**
Parameters Setting	2. Set the flow rate at the nominal/reference value 3. Check the water/Electrolyte quality according to the reference conditions reported in table 1 [2]. 4. Establish the potential cut-off, according to Maximum achievable voltage by the cell. 5. Set thermostat to reference/nominal temperature 6. Stabilize the selected pressure (cathode differential pressure or balanced pressure) and temperature at the selected nominal/refence current density.
Initial Assessment	7. Carry ac-impedance analysis _Galvanostatic mode:_ 0.2 A cm^{-2}, 100 kHz to 100 mHz, amplitude of 5% pk-pk of the applied current Nominal current density A cm^{-2}, 100 kHz to 100 mHz, amplitude of 5% pk-pk of the applied current. _Potentiostatic mode:_ 1.5 V and 1.8 V. from 100 kHz to 100 mHz. 10 mV rms oscillation. 8. Carry out the polarization curve at BoT as previously defined, in both descending and ascending modes. 9. Monitor H_2 concentration in oxygen in the anode compartment during the test, and reduce the pressure if the concentration exceeds 3%. 10. measure the O_2 concentration in the hydrogen (if possible).
Durability Test	11. Perform the durability of test (e.g. EoT = 2000 hr or when a test criterion is reached) at constant TIPs. 12. Perform electrochemical diagnostics (impedance and polarization in both descending and ascending modes). 13. Perform electrochemical diagnostics (impedance and polarization) every 500 hours. If necessary, the shutdown and restart procedure should follow the MEA manufacturer's recommendations. Record the cell temperature at least at the anode outlet. 14. Disconnect the current supply and leave the testing set-up under Open Circuit Potential (OCP) conditions for 60 minutes maintaining the water/electrolyte recirculation flowrate and test temperature. 15. Re-apply the TIPs and let cell/stack voltage stabilise for 120 min. If ΔU/Δt calculated over this period is equal or greater than zero, restart with the durability test, otherwise extend the stabilization period for another 60 minutes until reaching a positive ΔU/Δt over the preceding 120 min.
Final Assessment	16. Conduct End-of-Test (EoT) polarization and any additional diagnostics as necessary. 17. Present data as Potential (V) vs. Time (h), including current density, temperature, pressure, catalyst loadings, and feed mode 18. The procedure for calculating the voltage degradation is described

earlier. It is advisable to disregard the first 100 hours of the test and consider the final data point of the analyzed time span.

19. Determine performance and overall efficiency decrease at the nominal current density.

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(1) If the tests are conducted under conditions different from the reference ones, it should be specified.
(2) For AEMEL: If a KOH solution is used instead of water, measure the pH of the KOH solution at the BoT and at least once a week (it is recommended to measure the pH every three days initially to ensure more accurate monitoring over time). Replace the solution every 14 days (unless earlier replacement is required for specific reasons). Any deviation from the protocol should be noted and reported.

The efficiency of device is determined according to G. Tsotridis, A. Pilenga in "EU harmonised terminology for low temperature water electrolysis for energy storage applications", Publications Office of the European Union Publisher, ISBN: 978-92-79-90387-8 (online), 978-92-79-90388-5 [4]. The main equations are reported in the previous section.

3.1.4 Durability test under dynamic profile

The type of current profile applied during electrolysis can significantly influence the overall performance of an electrolyser, affecting its efficiency, behaviour, and degradation over time.
A study on Alkaline electrolyser reports that a Pulsed current profile, which alternate between periods of no current and higher current pulses, can reduce polarisation losses and improve gas bubble release from the electrode surfaces, enhancing charge transfer efficiency [8].
The choice of current profile also affects the degradation rate of the electrolyser. Unoptimized profiles, such as continuous currents with high fluctuations, can lead to increased thermal stress, higher overpotentials, and more rapid wear on electrodes and membranes.
A durability test under dynamic load involves testing the electrolyser's performance and degradation under real-world conditions that simulate how the electrolyser would behave when exposed to variable power inputs, such as from renewable energy sources like wind or solar. The goal is to understand how the system performs over time in these dynamic environments without artificially accelerating degradation [5]. A study on PEM water electrolysers and focused on their performance under voltage fluctuations derived from real wind turbine data, showed that the system spent significantly more time at lower voltages (1.5 V), approximately 13 times longer compared to higher voltage (1.8 V) [9].
With a consistent testing framework, it is possible to directly compare key performance indicators such as:

- Degradation rates.
- Efficiency drops over time.
- Response to variable power input.
- Durability under fluctuating loads.

In this case the procedures proposed by JRC establishes a specific protocol how to perform this kind of test. In the following figure an example of test is shown [5].

Figure 6. Example of a dynamic load profile procedure

Structure of Dynamic Load Degration Protocol
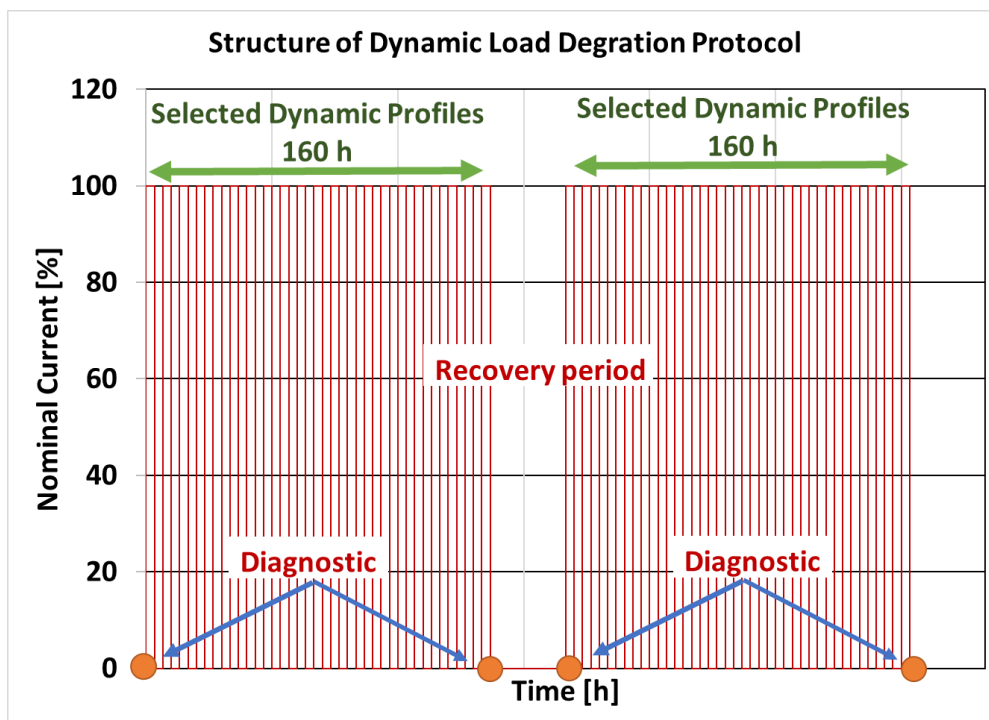
Each red line represents a dynamic cycle derived from a real-world application. The procedure is the same as the durability test in steady state condition from step 1 to step 10 and from step 18 to step 20.

In table 7, the main TIPs, TOPs, and values derived for a test under variable profile are presented. In this case, the degradation rate of the cell/stack is either related to the operating time or the number of cycles. Therefore, it is important to specify the number of cycles completed in the degradation data.

Table 7. Main Test Input Parameters (TIPs), Test Output Parameters (TOPs) and calculated values for dynamic profile tests.

TIPs	TOPs	Calculated Values
Inlet cell/stack Temperature	Voltage of cell/stack	Current density
Solution/water flow rate	Outlet cell/stack temperature	Power density
Current profile		efficiency
Pressure		Degradation rate of cell/stack voltage ($\Delta V/\Delta t$) (see section 2.3.1.4)
Number of cycles		Average temperature of cell/stack

The procedure for performing this type of test is outlined in Table 8. Both TIPs and TOPs should be recorded during the testing process. The test duration should be determined based on the test objectives. The JRC suggests an interruption every 160 hrs to perform diagnostic test.

Table 8. Dynamic Load Degradation Protocol

MEA Conditioning	1. As **suggested by manufacturer**
Parameters Setting	2. Set the flow rate at the nominal/reference value 3. Check the water/Electrolyte quality according to the reference conditions reported in table 1 . 4. Establish the potential cut-off, according to Maximum achievable voltage by the cell. 5. Set thermostat to reference/nominal temperature 6. Stabilize the selected pressure (cathode differential pressure or balanced pressure) and temperature at the selected nominal/refence current density.
Initial Assessment	7. Carry ac-impedance analysis <u>Galvanostatic mode:</u> 0.2 A cm^{-2}, 100 kHz to 100 mHz, amplitude of 5% pk-pk of the applied current Nominal current density A cm^{-2} (AEMWE), 100 kHz to 100 mHz, amplitude of 5% pk-pk of the applied current. <u>Potentiostatic mode:</u> 1.5 V and 1.8 V. from 100 kHz to 100 mHz. 10 mV rms oscillation. 8. Carry out the polarization curve at BoT as previously defined, in both descending and ascending modes. 9. Monitor H_2 concentration in oxygen in the anode compartment during the test, and reduce the pressure if the concentration exceeds 3%. 10. measure the O_2 concentration in the hydrogen (if possible).
Dynamic load test protocol	11. Operate the cell/short stack at the selected RWD load versus time profile for N cycles equivalent to 160 hours (with N rounded to the closest integer). 12. Perform a polarization curve. 13. Disconnect current supply and leave under OCP for 60 minutes maintaining the water recirculation flowrate and test temperature. 14. Re-apply the TIPs of step 1 and let cell/stack voltage stabilize for 120 min. 15. If ΔU/Δt calculated over this period is equal or greater than zero, go to next step, otherwise extend the stabilization period for another 60 minutes until reaching a positive ΔU/Δt over the preceding 120 min. 16. Perform a polarization curve. 17. Repeat steps 11 to 17. The test ends at step 16 after 10 loops for a total of 1,600 hours, or earlier upon reaching one of the EoT criteria.
Final Assessment	18. Conduct End-of-Test (EoT) polarization and any additional diagnostics as necessary. 19. Present data as Potential (V) vs. Time (h), including current density, temperature, pressure, catalyst loadings, and feed mode 20. The procedure for calculating the voltage degradation is described earlier. Determine performance and overall efficiency decrease at the nominal current density.

(1) For AEMEL: If a KOH solution is used instead of water, measure the pH of the KOH solution at the BoT and at least once a week (it is recommended to measure the pH every three days initially to ensure more accurate monitoring over time). Replace the solution every 14 days (unless earlier replacement is required for specific reasons). Any deviation from the protocol should be noted and reported.

(2) AEL: A minimum of approximately 20% of nominal current will reached during the test as suggested by manufacture.

3.1.4.1 *RW-derived profile for Power to Gas Application and Grid balance application*

In this context, an RW-derived profile was selected to assess the behavior of an electrolyser when coupled with a renewable energy source. Specifically, the profile was derived from an 18-month observation period of energy production by a wind farm and demand data from a site in northern Germany, with the final application intended for gas production and grid balancing. Because the energy produced by the wind farm in this location exceeds the maximum power capacity of the electrolyser more than 90% of the time, we assume that the electrolyser operates at full power whenever it is turned on. Figure 7 illustrates the simulated behavior of an electrolyser for this type of application.

Three types of start-up conditions are identified:

- **Warm start:** electrolyser off for less than 15 minutes, pump remains on.
- **Pump-off start:** electrolyser off between 15 minutes and 1 hour, pump is off.
- **Cold start:** electrolyser off for longer than 1 hour, pump is off and water is cold.

The profile (fig. 7) can be used in the procedure outlined in Table 5 to evaluate the response of an electrolyser under these specific operating conditions.

Figure 7. Real-profile derived scenario for to Gas Application and Grid Application and Grid Balance
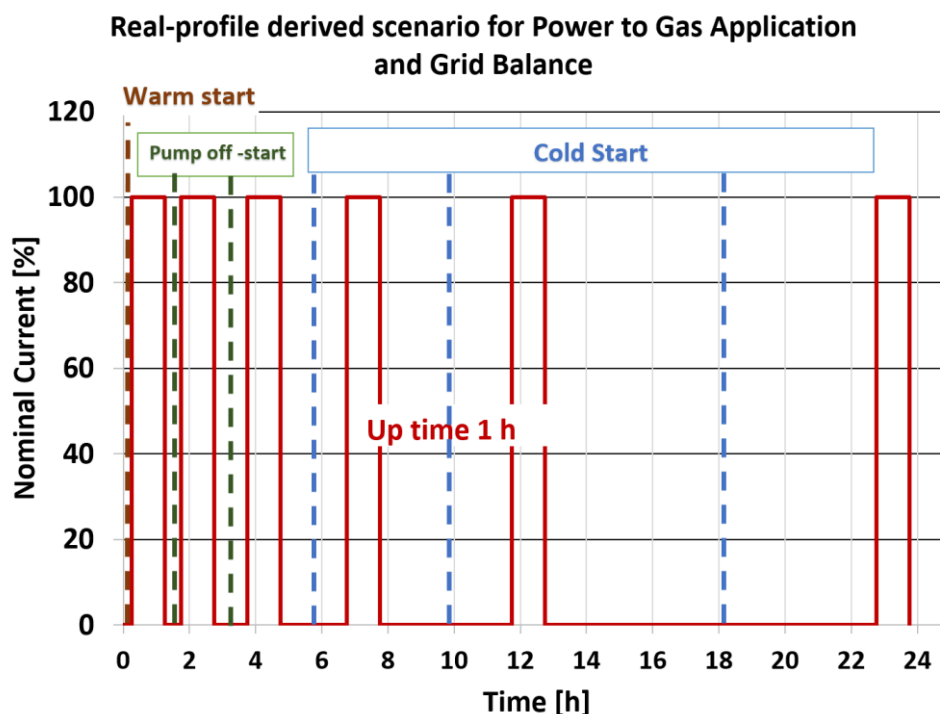
3.1.4.2 Wind and PV derived profiles for the Assessment of Degradation Phenomena under Dynamic Conditions

To evaluate the behavior of electrolysers under varying renewable energy conditions, six representative profiles of standard operating conditions for photovoltaic panels and wind turbine sources were selected. This approach aims to simulate real-world scenarios and provide insights into the operational stability and degradation patterns of electrolysers. The profiles, supplied by the partner EGP and selected from data of a significant set of renewable plants belonging to EGP's fleet, have a sampling time of 10 seconds. They were normalized to the maximum power of the analyzed renewable sources and can be used to simulate a direct connection to an electrolyser with the same power capacity.

In detail, for wind turbines, three distinct profiles were identified: a typical windy day with sustained high wind speeds, a day characterized by minimal wind variations, and one with high overall wind fluctuations. These profiles offer a range of operational scenarios to assess how rapid changes or relative stability in wind energy supply can influence the efficiency and degradation rates of electrolysers.

In the following figures, the selected wind profiles are shown.

Figure 8. Typical representative normalised Wind plant daily production, including examples of high wind speeds, minimal wind variations, and high wind fluctuations.

WINDY DAY - WTG Power % [ ]

DAY WITH LOW VARIATION - WTG Power % [ ]

DAY WITH HIGH VARIATION - WTG Power % [ ]
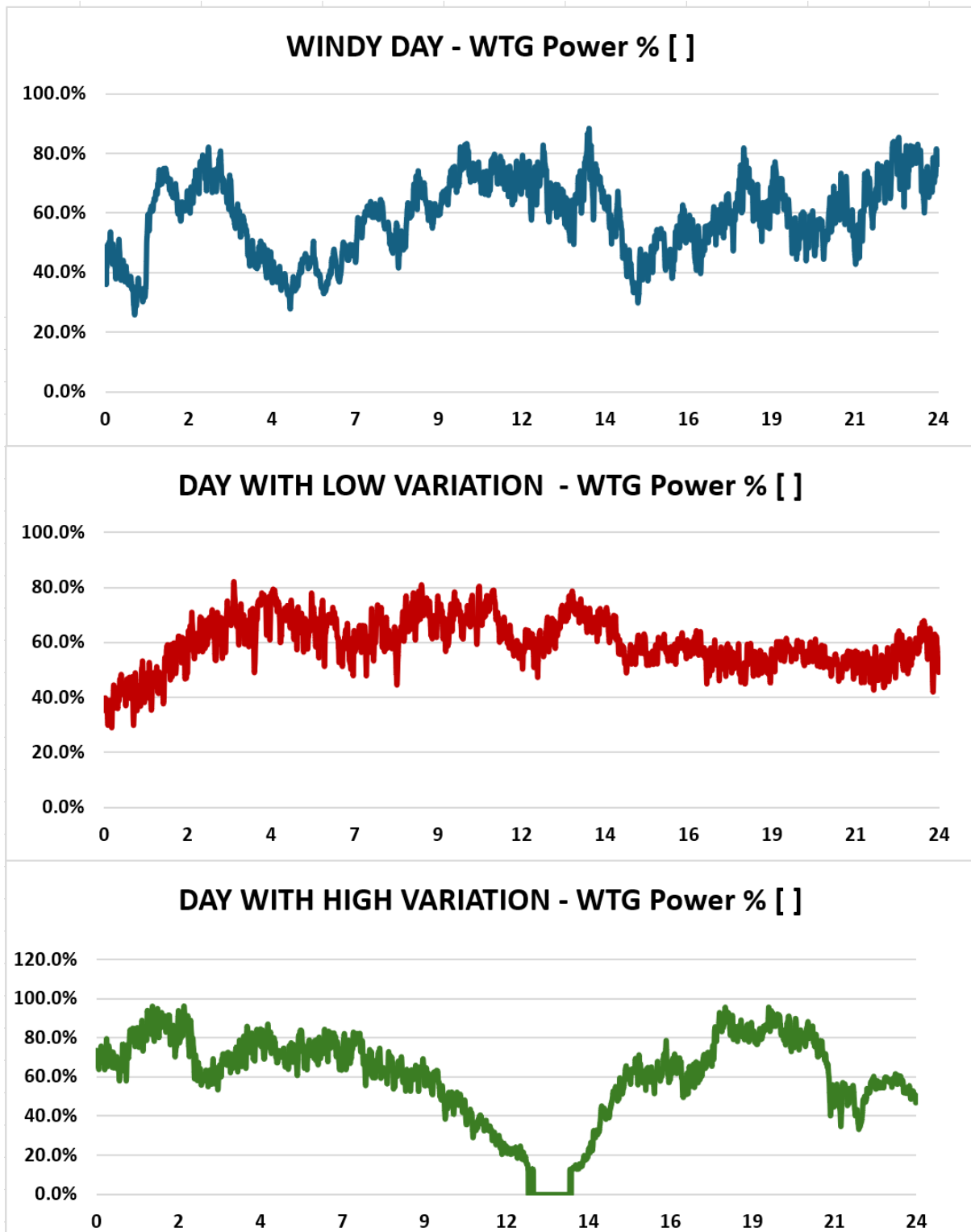
Similarly, three profiles were chosen to represent the output of photovoltaic panels: a sunny day with consistent high solar radiation, a day with low radiation, and a cloudy day marked by significant variability in light intensity. These profiles are important for examining the impact of solar energy fluctuations on the electrochemical performance of electrolysers, particularly regarding cyclic loading, transient power supply, and potential degradation due to repeated shifts in operational conditions.

The application of these renewable energy profiles enables detailed studies into the stress factors affecting electrolysers, such as power intermittency, thermal cycling, and fluctuating operational

states. These insights are crucial for understanding the degradation mechanisms involved and for developing more resilient electrolyser designs, capable of maintaining performance and durability when integrated with renewable energy sources.

Figure 5. Typical representative normalised Photovoltaic plant daily production, including examples of high wind speeds, minimal wind variations, and high wind fluctuations.
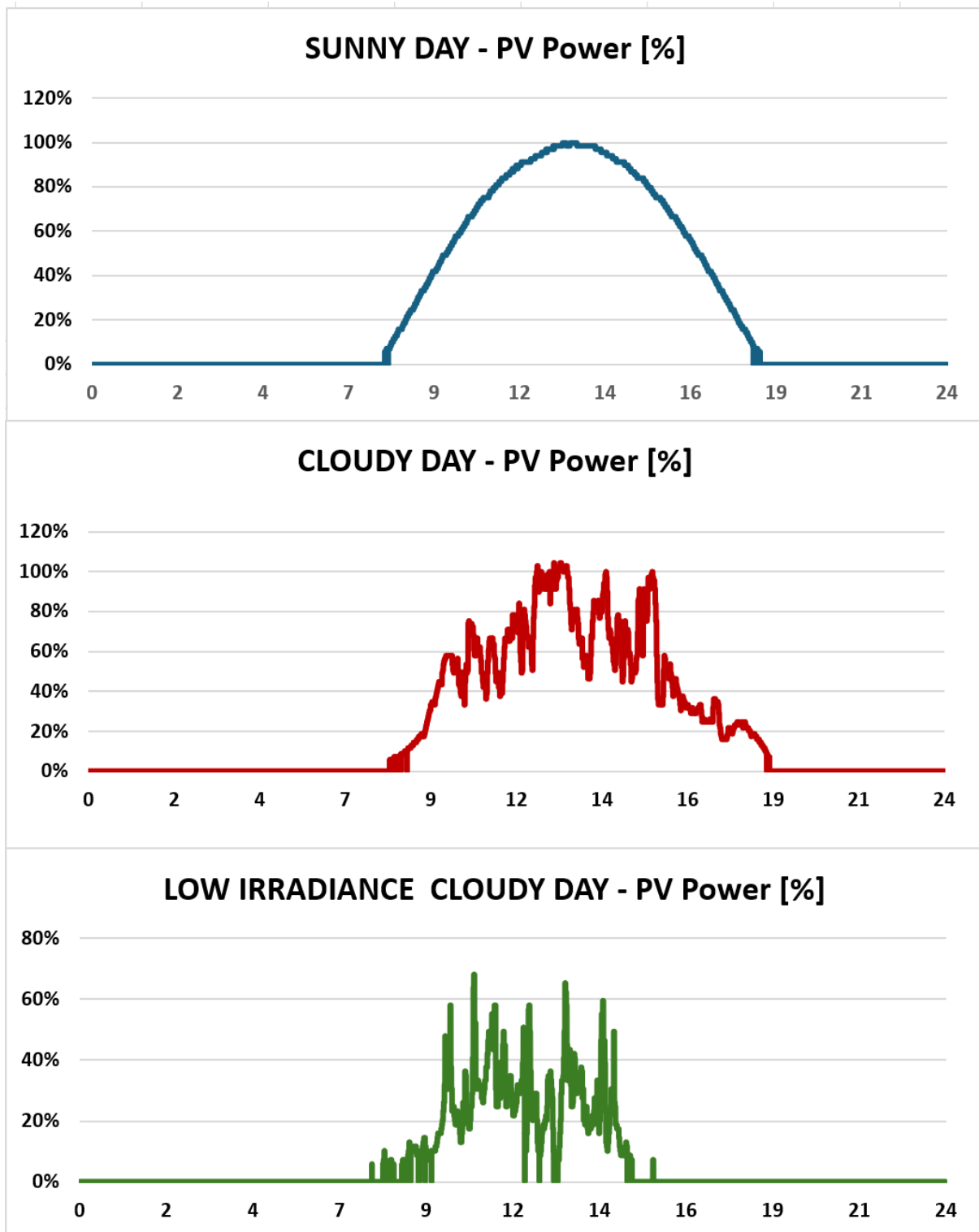
3.1.5 Accelerated Testing protocols and procedures

In accordance with the project objectives, accelerated test protocols will be developed based on the main stressors identified in the protocols by the JRC and recent literature. Dynamic profiles will be defined to study degradation phenomena related to certain stressors such as cycling, start and stop, and variations in internal pressure or temperature.

The test stations will be appropriately equipped to study these phenomena and simulate stress conditions according to the Test Output Parameters (TIP) and Test Input Parameters (TOP) established during the definition phase of the tests to be conducted.

According to the laboratories involved in the project and its testing capability, the main stressors that will be investigates are listened below:

- Dynamic load cycling;
- on/off cycling;
- pressure cycling;
- temperature cycling.

In this context is very important to evaluate behaviour of electrolyser under low current density load cycling and high current density load cycling.

As reported in JRC harmonised testing protocols, accelerated testing can be applied using either Accelerated Life Testing (ALT) or Accelerated Stress Testing (AST) protocols, depending on the specific objectives of the testing as previous mentioned.

Here the protocols proposed for the Electrolife project are focused on AST because the target is to identify and to characterise degradation phenomena and their mechanisms occurring in the test item [3].

A study on AST for PEMEL, found that degradation rate for low load cycling (0-0.5 A/cm^2) and high load cycling (1.2-2 A/cm^2) was respectively 51.4 µV/h and 55.8 µV/h. In contrast, the constant load test (1 A/cm^2) showed lower degradation rate (29.8 µV/h). The high load cycling also led to significant decreases in ohmic resistance, indicating a potential membrane thinning, which could eventually cause membrane failure [10].

These dynamic tests, which are shorter and more cost-effective than traditional long-term degradation tests, provide insights into how different current density intervals affect cell performance. The results could be useful for predicting the lifetime of electrolyser systems and for load balancing applications in systems powered by renewable energy sources like solar and wind.

To develop the procedures, it is necessary to define the value and range of these stressors based on specifications provided by the MEA manufacturer and capabilities of testing station used.

Procedure to carry out for the accelerated stress tests follow the same structure of the "Durability test under dynamic load" (table 5).

- Parameters setting
- Initial assessment
- Stressor application
- Final assessment

Stressor application procedure will be described in deep in dedicated paragraph.

In general, choosing the appropriate dwell time in testing protocols involves balancing several factors to ensure that the test both accelerates degradation and provides meaningful insights.

When determining the appropriate dwell time for testing, the first consideration is the objective of the test. If the goal is to replicate real-world operating conditions, the dwell time should mimic the typical duration the system spends at each power level, which can vary based on daily or seasonal changes in renewable energy inputs. However, in accelerated stress testing (AST), shorter dwell times might be preferred to increase the number of on/off cycles within a shorter period, intensifying stress and speeding up the degradation process.

The frequency of fluctuations is another key factor. A longer dwell time may be necessary to allow certain degradation mechanisms to unfold without interference from frequent switching. This approach helps isolate specific factors, such as the effects of on/off cycling. On the other hand, shorter dwell times are useful when simulating rapid power fluctuations, like those encountered with renewable energy sources such as wind or solar power, to evaluate the single cell's/stack's resilience under dynamic conditions.

Component sensitivity also plays a critical role in selecting the dwell time. Components such as membranes, catalysts, or electrodes may respond differently to thermal, mechanical, or chemical stress during switching events. For instance, if thermal cycling is a concern, a longer dwell time at each power level might be necessary to allow temperature stabilization before switching. However, shorter dwell times could expose sensitive components to frequent transitions, accelerating failure modes. While this helps to identify weaknesses, it may not accurately represent the stresses the system would encounter under normal operation.

In essence, it is necessary to balance dwell times depending on the objectives of the test—whether to study specific degradation mechanisms, assess dynamic resilience, or identify component vulnerabilities.

Finally, operational stability must be considered. If the test involves significant ramping between power levels, a minimum dwell time should be established to ensure that the system reaches stable operation before switching. Too short a dwell time could prevent the electrolyser from stabilizing, resulting in misleading data about its performance.

In the following table 9, the stressors proposed for the AST protocol are listed. The stressor profile is incorporated as an RWD profile in the dynamic load degradation test outlined in Table 5 (steps 11-17). Before to start with the stressor application is recommended to reach a stable condition of cell/stack.

Table 9. Stressors proposed for Accelerated Stress Test protocols [1]

Stressor application	1. **dynamic load cycling**
	2. **shutdown/startup**
	3. **high current operation**
	4. **high voltage operation**
	5. **pressure cycling**
	6. **Temperature cycling**

3.1.5.1 Dynamic Load profiles

The dynamic profile for conducting an accelerated test can be based on a real application profile or simulated stress conditions.
As defined in the AST protocols of the JRC [3], it will be necessary to define compression factors that will be selected based on the equipment that will apply them.
The compression factor is defined as follows:

$$f_{comp} = \frac{t_{compr}(h)}{t_{origin}(h)}, 0 < f_{compr} < 1 \; [3]$$

It is the ratio between the duration of the compressed operation profile (t_{compr}) and the duration of the original profile (t_{origin}).
Before starting the test, it is necessary to activate and condition the MEA as recommended by the manufacturer.
Additionally, it is necessary to set the maximum current and voltage values for safety, along with the acceptable maximum concentrations of H_2 in O_2.
Voltage and current fluctuations can have varying impacts on electrolyser degradation, depending on the technology used.

A study on PEM electrolysers shows as voltage fluctuations can reduce efficiency and accelerate the degradation of critical components, such as the IrO_2 catalyst. This issue is particularly pronounced when operating under the variable conditions typical of renewable energy sources, where maintaining voltage stability is more challenging [11].
Conversely, for alkaline electrolysers, current fluctuations appear more problematic. Current fluctuations, especially those with a high ripple factor, can lead to significant efficiency losses due to increased ohmic losses and instability in the electrolysis process. This results in inefficient energy use and reduced gas production [12], [13].
A review of these scientific papers suggests that the impact of fluctuations on electrolyser performance and degradation depends on the type of technology used, with PEM electrolysers being more sensitive to voltage fluctuations and alkaline electrolysers being more affected by current fluctuations.
A similar approach was followed by Honsho Y and colleagues [11], who developed an accelerated potential fluctuation test protocol to evaluate the durability of a PEM water electrolyser against voltage fluctuations caused by wind energy. This protocol was specifically designed based on the voltage fluctuations observed during a 24-hour period of actual wind turbine operation.
The protocol simulates potential fluctuations equivalent to 160 days of real-world operation. To achieve this, 24-hour voltage fluctuation data from an actual wind turbine were scaled of their original values and applied directly to a single PEM electrolyser cell.
Based on a similar approach and guidance provided by the JRC, a compressed profile can be developed starting from the dynamic profile proposed in the previous paragraphs.
The idea is to appropriately compress the reference profiles in accordance with the technical limitations of the equipment and devices under test.
An example of compressed profile for an accelerated testing protocol is shown in the following figure; a compression factor of ¼ was applied. The aim of this profile is to study the capacity of the electrolyser to withstand this specific application.

The complete AST protocol incorporates this profile as dynamic load degradation test outlined in Table 5.

Figure 9. Compressed profile derived from original RW profile for accelerated testing protocol.
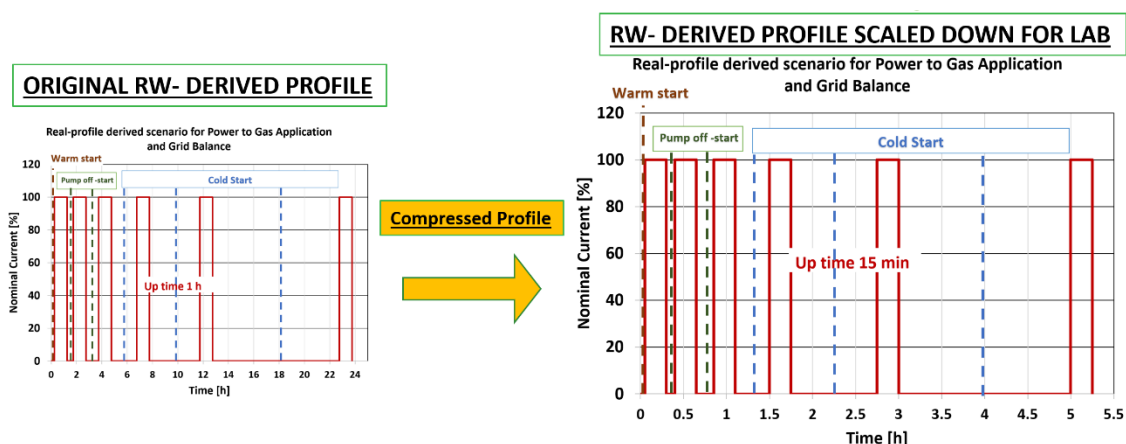
The same approach could be applied to the wind and PV profiles discussed in the previous section. In this case as well, the compression factor should be selected in alignment with the stacks or components manufacturers.

The only difference compared to the TIPs and TOPs reported in Table 7 for the study under dynamic load is the inclusion of the compressor factor specification in the TIPs.

3.1.5.2 *High current and High voltage fluctuating profiles*

Influence of voltage and current on electrolyser performance can be also investigated applying a dynamic load.

High current and high voltage cycling tests are crucial for evaluating the performance, durability, and safety of electrolysers. These tests help in understanding how the electrolyser operates under extreme conditions, where the system is subject to fluctuating or high-demand operations.

The membrane in an electrolyser is particularly sensitive to stress from high currents. Cycling tests help assess the integrity of the membrane under repeated high current loads, which is crucial for preventing failures like membrane tearing or thinning.

Repeated high current cycling can accelerate the wear of electrodes, leading to loss of catalytic activity. These tests provide insights into the rate and nature of electrode degradation, which can inform improvements in electrode design or material selection.

High current cycling tests simulate the conditions that an electrolyser will experience over its operational life. By accelerating the wear and stress on the system, these tests help predict the long-term reliability and lifespan of the electrolyser, allowing manufacturers and users to better plan maintenance and replacement schedules.

The procedure for studying the impact of high current in an AST follows the same structure as that used for the previous dynamic current cycling protocol, but with a current density applied at up to 200% of the MEA's nominal current.

High voltage cycling subjects the materials within the electrolyser, such as the membrane, electrodes, and catalyst layers, to significant stress. Repeated exposure to high voltages can accelerate the degradation of these materials.

The value of high voltage applied is function of electrolysis technology and MEA performance stated by manufacture.

For Alkaline Water Electrolysers (AELs), the nominal cell voltage typically ranges from 1.8 to 2.2 volts per cell during standard operation. Any voltage significantly exceeding this range—often considered to be above 2.2 to 2.5 volts per cell—could be classified as "high voltage."

Anion Exchange Membrane (AEM) electrolysers generally operate at similar voltages to Proton Exchange Membrane (PEM) electrolysers, with nominal values around 1.8 to 2.2 volts per cell. For AEM electrolysers, voltages above 2.3 volts per cell are often considered high, while for PEM electrolysers, voltages exceeding 2.2 volts per cell are typically regarded as high.

The same testing procedure can be applied to high voltage cycling.

A possible profile to study the behaviour of an electrolyser under fluctuating current and voltage conditions for accelerated testing is proposed in Figure 10. The stressor applied are high current, high voltage and frequency (tables 10 and 11). For alkaline electrolysers, current fluctuations appear to be more problematic according to some literature data.

The dwell time of 30 seconds can be reduced to 15 seconds for a more aggressive profile. The maximum current can be adjusted according to the stack/cell manufacturer's recommendations. Additionally, this value should be ≥ 150%. These profiles should be applied like the "Dynamic Load Degradation Protocol", table 5. The dwell time at the plateau for the current or voltage and the number of cycles are, in this case, TIPs to be added to those reported in Table 7. The test duration should be determined based on the test objectives.

Figure 10. Voltage and Current fluctuating profiles for accelerated test protocol.
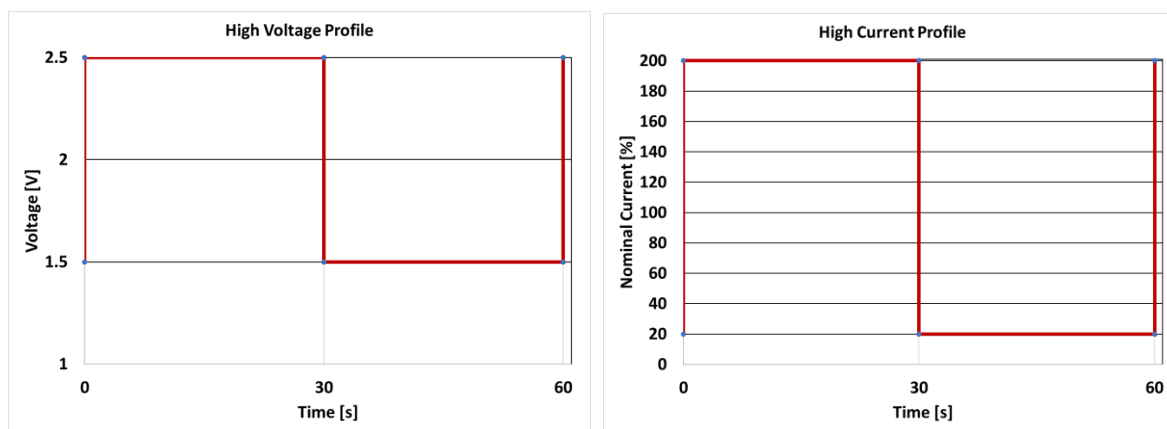
Table 10. Dynamic voltage cycling test procedure

Stress Applied
 	
• High frequency
• High voltage
	1. Apply 1 A cm² (or the selected nominal current) until a stable voltage is reached.
	2. Followed by two-step cycles:
	   a) Step 1: voltage is maintained at 2.0 V (2.5 V severe) for a time interval of 30 sec
	   b) Step 2: Voltage drops to 1.5 V for 30 sec

Table 11. Single cell/stack Dynamic current cycling test procedure

Stress Applied
 	
● High frequency
● High current
	1. Apply 1 A cm² (or the selected nominal current) until a stable voltage is reached.
	2. Followed by two-step cycles:
	   c) Step 1: current nominal current is maintained at 200% of nominal current for a time interval of 30 seconds
	   d) Step 2: current drops to 20% of nominal current for 30 seconds

3.1.5.3 Start-up / shut down profiles

This type of simulation is useful for testing the durability of the electrolyser components, such as membranes and electrodes, and for developing startup/shutdown protocols that minimise degradation and improve device safety.

The simulated on/off cycles for electrolysers are procedures that replicate the startup and shutdown conditions of the device. These simulated cycles are crucial for testing and understanding the behaviour and durability of the electrolyser under realistic operating conditions.

A possible procedure outlining such a cycle for this type of testing is detailed below. For an electrolyser connected to a solar energy source, one start-up and shut down cycle per day can be expected, corresponding to the day/night cycle. However, variable weather conditions could increase this number. As a result, an electrolyser might experience between 365 and 500 start/stop cycles per year, or even more under particularly fluctuating conditions [14]. Considering the maximum number of cycles per year, 2,500 cycles could represent approximately 10 years of operation.

The AST involves current cycling, ranging from OCP to 15% and 120% of nominal current as proposed. A similar approach with two current levels was used to study the stability of an alkaline electrolyser under on/off shutdown cycles [15]. In this case, the dwell time is reduced compared to the literature to create a more severe protocol.

The stressor profile is incorporated as a RWD profile in the dynamic load degradation test outlined in Table 5 (steps 11-17). The table 12 shows the steps for the shut down/start up procedure.
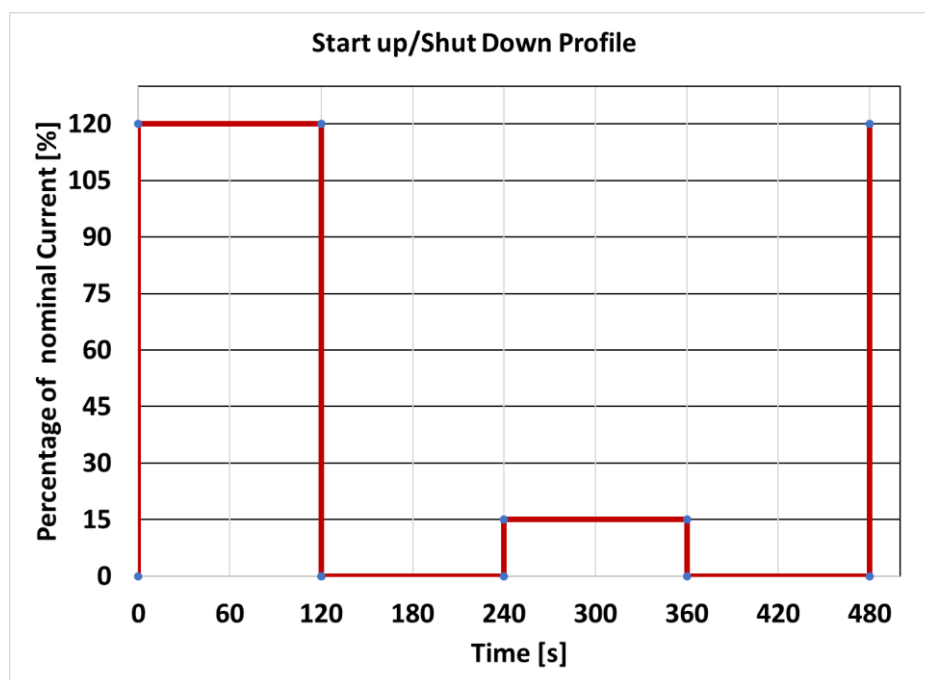
The dwell time at the current plateau and the number of cycles are TIPs that should be added to those listed in Table 7. The test duration should be defined according to the test objectives.

Table 12. Single Cell/stack start-up / shut down procedure

Stress Applied
 	
• High frequency
• Start up/shut down
	• Apply a current density of 1 A cm² (or the selected nominal current) until a stable voltage is reached at ambient pressure.
	• Step 1: high current
	   120% nominal current: dwell time 120 sec
	• Step 2: off phase
	   OCP: Dwell time 120 sec
	• Step 3: low current
	   15% nominal current. Dwell time 120 sec
	• Step 4: off phase
	   OCP: Dwell time 120 min
	• Step 5: Assessment
	   Carry out control polarization and eventually other diagnostics every 160 hrs as suggested in the JRC protocols (table 5, steps 11-17).

Figure 11. Single cell/stack Start-up/Shut down fluctuation profile

3.1.5.4   Pressure Cycling test profiles

Pressure Cycling Accelerated Testing plays a crucial role in assessing how well electrolysers can endure and perform under fluctuating pressure conditions. This method replicates the types of stress that electrolysers typically encounter in real-world situations, especially when they are exposed to frequent pressure variations, as seen in systems powered by renewable energy sources like wind or solar.
The main phenomena that can be observed during such stress testing are:

• Membrane Thinning

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- Fatigue Cracking
- Delamination

Pressure cycling can be performed within different ranges and depending on the testing objective. For example, a test can be conducted from 60% to 100% or from 0% to 100% of nominal pressure (figure 12, table 13). The time required to reach the nominal pressure should be chosen in accordance with the stack/MEA manufacturer's guidelines and the test bench specifications.
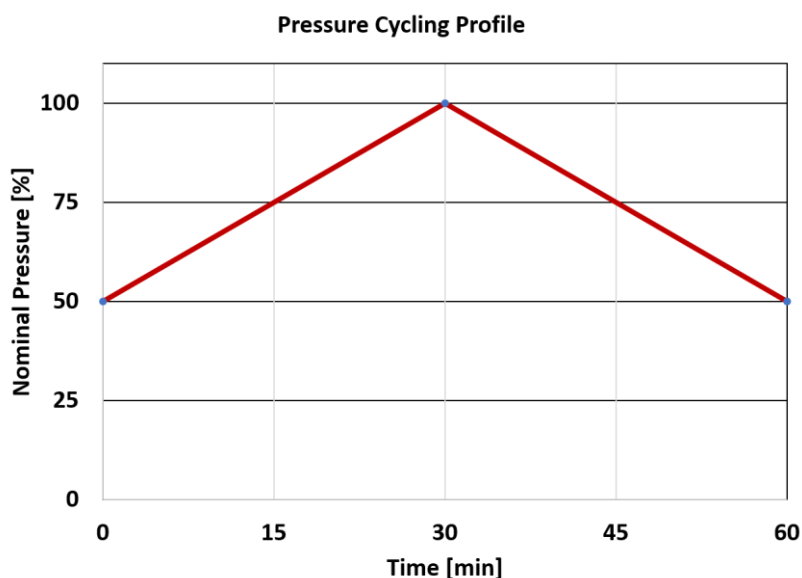
Pressure Cycling Profile

Figure 12. Pressure fluctuation profile.

The rate of pressure change (ΔP/Δt) and the number of cycles are TIPs that should be added to those listed in Table 7. The test duration should be defined according to the test objectives.

Table 13. Single Cell/stack pressure cycling procedure

Stress Applied • High frequency • pressure	• Apply nominal current until a stable value is reached at ambient pressure. • Followed by a 2-step cycles: • Step 1: increase pressure in 30 min to the nominal pressure • Step 2: decrease pressure to 50% of nominal pressure in 30 min (1 bar for a more severe test) • Carry out control polarization and eventually other diagnostics every 160 hrs as suggested in the JRC protocols (table 5, steps 11-17).

* The time required to reach the nominal pressure should be minimised as much as possible, in accordance with the stack/MEA manufacturer's guidelines and the test bench specifications.

3.1.5.5   temperature Cycling test profiles

Temperature cycling is a critical accelerated testing method used to evaluate the performance, durability, and resilience of electrolysers, including Proton Exchange Membrane (PEMEL), Anion Exchange Membrane (AEMEL), and Alkaline Electrolysers (AEL). This type of test involves subjecting the electrolyser components to repeated cycles of heating and cooling, simulating the thermal stresses they may encounter during real-world operation or when integrated with fluctuating renewable energy sources.

Temperature cycling tests can induce several key degradation phenomena in electrolysers, The main degradation phenomena typically observed during these tests include:

- Thermal Fatigue and Mechanical Stress;
- Membrane Thinning and Cracking;
- Catalyst Layer Degradation;
- Seal and Gasket Failure;
- Loss of Electrical Contact and Increased Resistance;
- Delamination of Layers;
- Loss of Mechanical Integrity.

The maximum and minimum temperatures of the cycles should be selected based on the specific electrolysis technology. Table 14 presents the reference conditions applicable to each electrolysis technology [5].

Table 14. Agreed reference and stressor conditions for temperature testing and the electrolysis technologies. [5]

	Reference T [°C]	Stressor Min T	Stressor Max T
PEMEL	80*/60	40	90
AEMEL	50	30	65
AEL	80	50	100

*Alternative value

A proposed temperature cycling profile is shown in Figure 13 for PEM technology, where the minimum and maximum values are 40°C and 90°C, respectively. Table 15 presents the procedure proposed for temperature stress testing. The time required to reach the maximum temperature should be minimised in accordance with the stack/MEA manufacturer's guidelines and the test bench specifications.

The rate of temperature change (ΔT/Δt) and the number of cycles are TIPs that should be added to those listed in Table 7. The test duration should be defined according to the test objectives.

Figure 13. Temperature fluctuation profile.

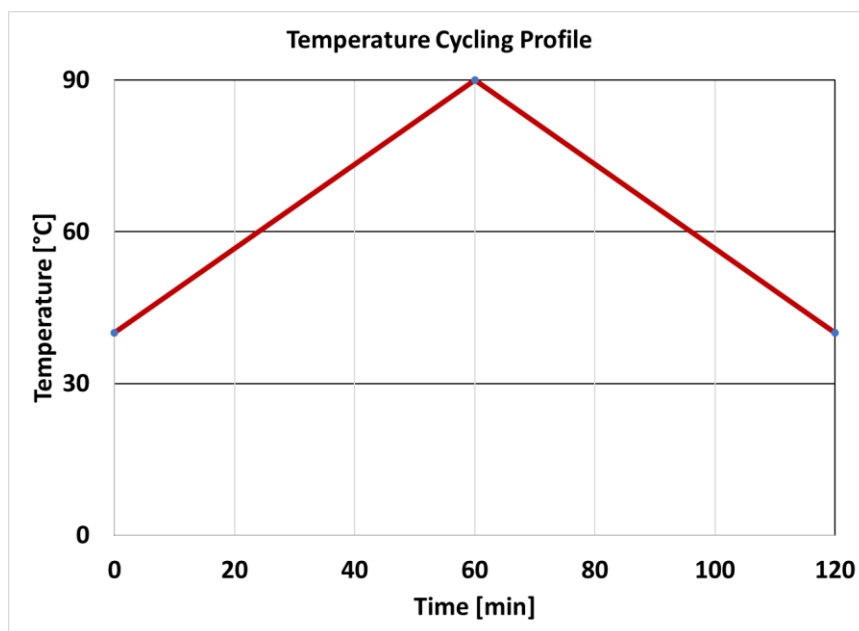*Table 15. Single Cell/stack temperature cycling procedure*

Stress Applied ● Temperature cycling	• Apply nominal current until a stable value is reached at ambient pressure. • Followed by a 2-step cycles: • Step 1: increase temperature to the maximum value in 1 hours • Step 2: decrease temperature to the minimum value in 1 hours. • Carry out control polarization and eventually other diagnostics every 160 hrs as suggested in the JRC protocols [5]. (table 5, steps 11-17).

* The time required to reach the maximum temperature should be minimised as much as possible, in accordance with the stack/MEA manufacturer's guidelines and the test bench specifications.

3.2 Contribution to project (linked) Objectives

Deliverable D4.1 contributes to study the impact of renewable energy source (RES) profiles on electrolyser durability under dynamic conditions; SO3 of DoA "Evaluate the impact of RES electrical profile on electrolysers durability in terms of the dynamic operating conditions". By analysing real RES profiles provided by renewable energy operators, the deliverable has ensured that testing protocols reflect actual operational scenarios. These protocols have been specifically developed to evaluate how dynamic operating conditions influence degradation.

The deliverable also includes detailed guidelines for measuring key parameters, enabling reproducible results. Through these efforts, it will help to identify specific degradation mechanisms, such as

increased cell voltage and reduced efficiency, associated with dynamic operation. This work also helps to develop strategies to enhance the durability and performance of electrolysers across different technologies.

Deliverable D4.1 also contributes to SO8 because represents the first version for developing more robust protocols at the end of project.

3.3 Contribution to major project exploitable result

The deliverable develops harmonised in-situ testing protocols with the aim to standardise the assessment of degradation mechanisms in electrolysis technologies. These protocols enhance the reproducibility of experiments and the evaluation of phenomena, which is essential for improving electrolyser performance and durability.

Through alignment with Joint Research Centre (JRC) protocols, the deliverable supports the development of standardised practices for the hydrogen economy development. This can help develop policy recommendations for adopting testing standards and operational guidelines throughout the industry.

Besides, the protocols outlined in D4.1 help to identify specific degradation issues, allowing for targeted improvements in electrolyser components. This supports the development of more robust and efficient electrolyser technologies, aligning with the project's goals.

4 Conclusion and Recommendation

The deliverable D4.1, titled "Specification, Terminology, and Harmonised Protocols for LTEL," represents a first step forward in establishing a unified framework for testing low-temperature electrolyser (LTEL) technologies, including PEMEL, AEMEL, and AEL. Its main objective is to ensure reproducibility and harmonise research methodologies across laboratories within the Electrolife project.

One of the key challenges addressed by this deliverable is the diversity of testing protocols used by partners from both the industrial and academic sectors. To overcome this, D4.1 proposes an initial set of harmonised protocols based on literature and experience of the project partners. These protocols are designed to be applied in various laboratories using the components and stacks developed within the project. By comparing the results obtained with these protocols to those from partner-specific procedures, it will be possible to identify areas for improvement and refine the methodologies.

The protocols outlined in D4.1 provide a robust framework to isolate critical degradation factors, such as catalyst dissolution, membrane thinning, and mechanical instability. Advanced diagnostic techniques, including electrochemical impedance spectroscopy (EIS), have been incorporated to enhance the accuracy and depth of analysis. Furthermore, the inclusion of accelerated stress testing (AST) procedures allows for the evaluation of long-term durability within shorter timeframes, offering valuable insights into degradation mechanisms.

To address the variability inherent in real-world operations, the protocols are designed for both steady-state and dynamic conditions. Simulated real-world profiles, such as fluctuating power inputs from renewable energy sources, have been integrated into the testing methodologies to evaluate how electrolysers perform and degrade under such conditions. This real-world relevance is critical for assessing the resilience of LTEL technologies.

A strong emphasis is placed on standardising operating conditions, including temperature, pressure, and current density. This ensures that results are consistent, reliable, and aligned with benchmarks provided by the Joint Research Centre (JRC). Feedback from the JRC and experimental data from project partners will play a crucial role in refining the protocols, ensuring they remain adaptable and aligned with the latest advancements in the field. For all aspects not specified in this document, or for further details, please refer to the JRC protocols for LTEL.

This deliverable supports multiple Work Packages within the project, including WP2, which focuses on studying degradation phenomena, and WP6, dedicated to the development of short-stack prototypes. The collaborative and iterative approach used in D4.1 allows for the connection of laboratory research with practical applications, enhancing LTEL technology testing and development.

5   Risks and interconnections

Normal text comes here

5.1   Risks/problems encountered

If applicable (consider using table below to report risks – and solutions ! – encountered for the activities/tasks related to this deliverable)

Risk No.	What is the risk	Probability of risk occurrence[1]	Effect of risk[1]	Solutions to overcome the risk
WP 4. Some protocols may require an updated after the first period of experimental activities	medium	1	2	Update specific protocols according to the experimental evidence and JRC feed back
WP4	The potential for gas crossovers during different stressor operation can lead to safety hazards and system instability, requiring shutdowns.	3	1	Implementing a mixing mode operation can help mitigate this risk by ensuring a more controlled environment, reducing the likelihood of gas crossovers
WP4	The complexity of managing both mixed and separate modes of operation.	1	2	To overcome these challenges, prioritize tests with the stack producer.

[1] Probability risk will occur: 1 = high, 2 = medium, 3 = Low

5.2   Interconnections with other deliverables

This deliverable is closely linked to the activities outlined in Deliverable 2.1, "Degradation Phenomena Compendium," which includes studies and analyses of degradation phenomena. Additionally, this deliverable serves as the first draft of the protocols developed for the Electrolife project. A revised version will be prepared following the validation of these protocols through experimental data and feedback from the Joint Research Centre (JRC).

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Project partners:

#	Partner short name	Partner Full Name
1	POLITO	Politecnico di Torino
2	UNR	Uniresearch B.V.
3	EGP	Enel Green Power SpA
4	FAU	Friedrich-Alexander-Universitaet Erlangen-Nuernberg
5	TUG	Graz University of Technology
6	KER	Kerionics s.l.
7	AAU	Aalborg University
8	FZJ	Forschungszentrum Jülich gmbh
9	ULille	University of Lille
10	STARGATE	Stargate Hydrogen Solutions OU
11	PF	Pietro Fiorentini s.p.a.
11.1	HYT	Hyter s.r.l. (Affiliated)
12	CNR	Consiglio Nazionale delle Ricerche
13	1s1	1s1 Energy Portugal Unipessoal Lda
14	AEA	AEA s.r.l.
15	VDX	Volytica diagnostics GmbH
16	SE	SolydEra SpA

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