Alkaline electrolysers with enhanced durability

ENDURE

Report on harmonised test protocols adopted for ENDURE

(Deliverable D1.2)

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NOTICES

For information, please contact the project coordinator, Mari Šavel, e-mail: mari.savel@stargatehydrogen.com. This document is intended to fulfil the contractual obligations of the ENDURE project, which has received funding from the Clean Hydrogen Partnership and its members, concerning deliverable D1.2 described in contract 101137925.

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Table of revisions

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List of Partners

Stargate Hydrogen Solutions OÜ (Stargate)
Fraunhofer-Institut für Fertigungstechnik und Angewandte Materialforschung (IFAM)
Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (ZSW)
Fundación para el Desarrollo de las Nuevas Tecnologías del Hidrógeno en Aragón (FHa)
Permascand AB (Permascand)
Université catholique de Louvain (UCLouvain)

List of Abbreviations

AEL: alkaline electrolysis

Ao: after operation

Bo: before operation

EIS: electrochemical impedance spectroscopy

HER: hydrogen evolution reaction

HTO: hydrogen-to-oxygen ratio

OTH: oxygen-to-hydrogen ratio

JRC: European Commission's Joint Research Centre

KOH: potassium hydroxide

KPI: key performance indicator

OCP: open circuit potential

OER: oxygen evolution reaction

RHE: reversible hydrogen electrode

TIPs: test input parameters

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1 Executive Summary

The project "alkaline electrolysers with enhanced durability" (ENDURE) is focused on bringing the performance and durability of alkaline electrolysers to a new level. The main objective is to drastically reduce the degradation rate and increase the efficiency of alkaline cells and stacks via the development of hierarchically structures flow-engineered monolithic porous transport electrodes, via design/material improvements on stack level, and via accelerated testing procedures.

To validate stack level improvements and performance, ENDURE partners have agreed on common harmonised test protocols for carrying out cell- and stack- level testing in the project. These protocols are presented in this document and rely heavily on the harmonised test protocols developed by the European Commission's Joint Research Centre (JRC). Furthermore, they expand the scope of the existing protocols to include a complete procedure for stack and single cell level testing.

The definition of test protocols within this project has been carried out in task 1.4 and have started with a review of the most recent JRC's documents about testing procedures and other available public reports produced by previous projects. Furthermore, existing experience in cell and stack testing from partners have also been considered in protocols definition.

It must be noted that the test protocols detailed in this deliverable have been agreed with all partners before the beginning of tests as they will be used by all partners. In particular, we have agreed on specifications for the electrochemical analysis for oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) which can be harmonised by all partners and generally speaking by other research institutes even though the characteristics of the test setup differ in some aspects. This covers aspects like cell temperature, electrolyte concentration, electrolyte flow rate, cell operating pressure and nominal current density.

2 Objectives

Currently, the comparison of different alkaline electrolyser (AEL) components from literature sources becomes difficult due to the differences in the followed testing procedures. With the definition of testing protocols within ENDURE, we aim at enabling a higher comparability of different AEL components.

3 Introduction

As previously stated, the goal of this document is to provide a testing protocol for alkaline technology cells and stacks based on currently standardised protocols to be followed in ENDURE project. It proposes tests that allow validating the correct operation of the materials developed within the project, as well as evaluating the improvement of the performance of components.

The definition of a test plan based on harmonised protocols aims to ensure reliable, optimised and cost-competitive production, as well as the validation of key performance indicators (KPIs) as a basis for operational decisions. These protocols will be followed by all partners and in particular by ZSW, Fraunhofer-IFAM and FHA in the test benches they have available at their facilities, which allow working with cells and stacks with a power up to 10 kW.

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The main reference that has been considered for the definition of these protocols is the technical document published in 2024 by the Joint Research Center (JRC) "**EU harmonised testing procedure: determination of water electrolyser energy performance**", whose main objective is to establish a procedure to calculate the energy efficiency of an AEL system to produce hydrogen. Moreover, previous reports by the JRC have also been considered, such as the "**EU harmonized protocols for testing of low temperature water electrolysis**" published in 2021, and the "**EU harmonised polarisation curve test method for low-temperature water electrolysis**" published in 2018.

4 Cell level test protocols

Within the ENDURE project the project partners agree on a harmonised protocol for single cell testing including half-cell arrangements and full cell arrangements, for the evaluation of electrode materials. The protocols are based on the agreements of the JRC on alkaline electrolysis, namely

JRC 122565 (Tsotridis and Pilenga 2021)

JRC 111151 (Malkow et al. 2018a)

JRC 120120 (Malkow et al. 2021)

JRC 104045 (Malkow et al. 2018b)

JRC 107053 (Malkow et al. 2018c)

For the test input parameters (TIPs) the test protocol follows the agreed settings of the JRC 122565 (Table 1), where applicable:

Table 1. Agreed reference settings for TIPs for AEL single cell and short stack testing by JRC 122565 (Tsotridis and Pilenga 2021)

	Test input parameters	Unit	Reference Settings
cell / short stack	Cell / stack temperature	°C	80
	Conductivity of water used for electrolyte preparation and supply to electrolyser	µS.cm^{-1}	≤1, ISO 3696 Grade 2 @ 25 °C
ANODE	Electrolyte inlet temperature	°C	80
	Electrolyte inlet pressure	kPa; (bar)	100; (1)
	Electrolyte inlet concentration	KOH wt.%	30
	Minimum electrolyte inlet flow rate	mL.cm^{-2}.min^{-1}	1
	Oxygen outlet pressure (absolute)	kPa; (bar)	500, (5)
CATHODE	Electrolyte inlet temperature	°C	80
	Electrolyte inlet pressure	kPa, (bar)	100, (1)
	Electrolyte inlet concentration	KOH wt.%	30
	Minimum electrolyte inlet flow rate	mL.cm^{-2}.min^{-1}	1
	Hydrogen outlet pressure (absolute)	kPa; (bar)	500; (5)

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Since there is no precise protocol for single cell test of alkaline electrolysis, the consortium partners of this project agreed on most of the testing procedure based on their scientific experience. The aim of this protocol is furthermore to create a uniform test protocol which is applicable for different test setups, while generating results which are comparable to each other.

4.1 Half-cell arrangement

The following table (Table 2) shows the testing specifications for the half-cell arrangement:

Table 2. Agreed reference setting defined for half-cell arrangements.

Test input parameter	Reference setting	Comments
Cell temperature	80 °C	According to JRC 122565 chapter 5.2.6 (Malkow et al. 2021)
Conductivity of water used for electrolyte preparation	$\leq 1\ \mu S/cm$	According to JRC 122565 chapter 5.2.6 (Malkow et al. 2021)
Lye concentration	30 wt. % KOH	According to JRC 122565 chapter 5.2.6 (Malkow et al. 2021)
Impurities of the lye	-	Not specified
Iron content in the lye	-	Not specified, to be measured and stated if > 10 ppm in relation to total weight
Pressure	atmospheric	
Volume flow	-	Not specified
Type of reference electrode	-	Not specified
Potential definition	vs. RHE	Either by direct measurement vs. RHE or recalculation towards RHE potential
Potentiostat	-	Not specified
Active electrode area	-	Not specified
IR drop compensation	Yes	Either by Current Interrupt or by Impedance spectroscopy (EIS) to estimate the cell resistance
Cell design	-	Not specified

The required measurement accuracy and sampling rate shall comply with the agreed values by the JRC → compare JRC 122565 table 14 (Malkow et al. 2021)

The test protocol for the half-cell arrangement consists out of the following steps:

- **Step 1:** Conditioning η_{500} (5 h) - overpotential towards HER/OER for 5 h at 0.5 A cm^{-2}
    - Overpotential IR compensated

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- **Step 1.2:** Relaxation for 5 minutes at open circuit potential (OCP)
  - ➔ Comment: The conditioning might change depending on the electrode

- **Step 2:** Tafel plot / polarization curve (ascending) up to 0.5 A cm^{-2} with stepwise galvanostatic measurements between 0.1 to 500 mA cm^{-2}. (See table 3)
  - Overpotential IR compensated

- **Step 3:** Impedance spectroscopy at various overpotentials between η=300 - 0 mV vs HER/OER (potentiostatic) or between j=0 - 100 mA cm^{-2} (galvanostatic) in a frequency range of 300 kHz – 0.1 Hz
  - **Specific suggestion:**
    Galvanostatic EIS
    DC current: 40 mA cm^{-2}
    AC current: 6 mA cm^{-2} (15 % of DC)
    Initial frequency: 300 kHz
    Final frequency: 0.1 Hz
    Points/decade: 10

- **Step 4:** η_{1000}(5 h): overpotential towards HER/OER for 5 h at 1.0 Acm^{-2}
    - Overpotential IR compensated
  - **Step 4.1:** Relaxation for 5 minutes at OCP

- **Step 5:** Tafel plot / polarization curve (ascending) up to 1 Acm^{-2} with stepwise galvanostatic measurements between 0.1 to 1000 mAcm^{-2}. (See table 3)
  - Overpotential IR compensated

- **Step 6:** Impedance spectroscopy at various overpotentials between η=300 - 0 mV vs HER/OER (potentiostatic) or between j=0 - 100 mA cm^{-2} (galvanostatic) in a frequency range of 300 kHz – 0.1 Hz
  - **Specific suggestion:**
    Galvanostatic EIS
    DC current: 40 mA cm^{-2}
    AC current: 6 mA cm^{-2} (15 % of DC)
    Initial frequency: 300 kHz
    Final frequency: 0.1 Hz
    Points/decade: 10

- **Step 7:** Cyclic voltammetry +50 mV to -50 mV vs. OCP or a specific potential (+0.4 V vs RHE) with varying sweep rate (10 – 1000 mV s^{-1}) (a pretreatment step is sometimes used before step 7).

The polarisation curves will be performed with stepwise galvanostatic measurements. Each step will be held for a minimum of 30 seconds (dwell time) with a sampling rate of 1 point/second. The following steps will be performed (Table 3):

Table 3. Specification of the steps for the polarization curve.

Step	1	2	3	4	5	6	7	8	9	10

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mA/cm^2	0.1	0.16	0.25	0.4	0.63	1	1.26	1.58	2	2.51
Step	11	12	13	14	15	16	17	18	19	20
mA/cm^2	3.16	3.98	5	6.31	7.94	10	12.59	15.85	19.95	25.12
Step	21	22	23	24	25	26	27	28	29	30
mA/cm^2	31.62	39.81	50.12	63.1	79.43	100	112.2	125.9	141.25	158.5
Step	31	32	33	34	35	36	37	38	39	40
mA/cm^2	177.8	200	223.9	251.2	281.8	316.23	354.81	398.81	446.68	500
Step	41	42	43	44	45					
mA/cm^2	600	700	800	900	1000					

4.2 Full-cell arrangement

The following table (Table 4) shows the testing specifications for the full-cell arrangement:

Table 4. Agreed reference setting defined for full-cell arrangements.

Test input parameter	Reference setting	commentary
Cell temperature	80 °C	According to JRC 122565 chapter 5.2.6 (Malkow et al. 2021)
Conductivity of water used for electrolyte preparation	$\leq 1\ \mu S/cm$	According to JRC 122565 chapter 5.2.6 (Malkow et al. 2021)
Lye concentration	30 wt. % KOH	According to JRC 122565 chapter 5.2.6 (Malkow et al. 2021)
Impurities of the lye	-	Not specified
Iron content in the lye	-	Not specified, to be measured and stated if > 10 ppm in relation to total weight
Pressure	atmospheric	
Volume flow	-	Not specified
Potentiostat	-	Not specified
Active electrode area	-	Not specified
Cell design	-	Not specified

The test protocol for the full-cell arrangement consists out of the following steps:

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- **Step 1:** Determining single cell resistance via EIS (e.g. membrane- / contact resistance) (Bo)
- **Step 2:** Conditioning - electrolysis at a specific current of 600 mA/cm^2 for at least 30 minutes and then until the deviation of the potential is < 0.5 mV/minute, 5 mV/10 minutes

  Comment: The conditioning might change depending on the electrode material
- **Step 3:** Current-voltage curves / polarization curves up to 1.0 A/cm^2 with a sweep rate of 0.08 mA/cm^2 per minute → single cell performance (Bo) [1]
- **Step 4:** Impedance spectroscopy (EIS) at various overpotentials between $U_{cell}(DC)$ = 1.2-1.6 V (potentiostatic) or between j= 0 – 100 mA cm^{-2} (galvanostatic) in a frequency range of 100 kHz – 0.1 Hz
  - **Specific suggestion:**
    Galvanostatic EIS
    DC current: 40 mA cm^{-2}
    AC current: 6 mA cm^{-2} (15 % of DC)
    Initial frequency: 100 kHz
    Final frequency: 0.1 Hz
    Points/decade: 10

- **Step 5:** electrolysis at 1.0 A/cm^2 for 100 h
- **Step 6:** current-voltage curves / polarization curves up to 1.0 A/cm^2 with a sweep rate of 0.08 mA/cm^2 per minute → single cell performance (Ao) [2]
- **Step 7:** Impedance spectroscopy at various overpotentials between $U_{cell}(DC)$ = 1.2-1.6 V (potentiostatic) or between j= 0 – 100 mA/cm^2 (galvanostatic) in a frequency range of 100 kHz – 0.1 Hz
  - **Specific suggestion:**
    Galvanostatic EIS
    DC current: 40 mA cm^{-2}
    AC current: 6 mA cm^{-2} (15 % of DC)
    Initial frequency: 100 kHz
    Final frequency: 0.1 Hz
    Points/decade: 10

5 Stack level test protocols

In this section, the test protocols for short stack testing are detailed. The test will consist of 3 stages: a first fingerprint performance test to identify the operation window of the stack. A long-term galvanostatic steady state test, which will be carried out for 400 h, and a second

[1] According to JRC104045 testing method A

[2] According to JRC104045 testing method A

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fingerprint performance test where the most relevant conditions of the first fingerprint test will be replicated.

For the three phases of the test, the following specifications will be considered:

- Stack temperature: 80 ^oC
- Lye concentration: 30 wt% KOH
- Impurities of the lye: not specified
- Iron content in the lye: not specified. To be measured and stated if > 10 ppm in relation to total weight

Before the beginning of the tests, a leak test will be performed on the stack and the balance of plant (BoP). The stack pressure test will be carried out according to the manufacturer specifications (Stargate in ENDURE project). The BoP pressure test will be performed pressurising with N_2 to 1.1 times the maximum pressure expected during the tests (30 barg) at ambient temperature. This final pressure will be maintained for 30 minutes to observe any external leaks using soapy water. If the leak test is successful (no leaks identified), testing will commence.

5.1 First fingerprint performance test

This test will be carried out in the first place to identify the stack **operation window**. The test will last approximately 50 h and will consist of changes from low to high pressure, current density and KOH flow while monitoring cell voltage and gas purities to determine the limits for safe and correct operation.

To be more specific, this first fingerprint test will consist of the following. From 5 up to 30 barg (steps of 5 barg) an extensive characterization is carried out to determine the operation range of the main parameters. Different values of KOH flow and current density will be tested at each pressure (see ranges in Table 5). For every permutation, cell voltages and gas purity (HTO and OTH) will be obtained. HTO will be continuously monitored for safety reasons. Each permutation will be maintained until the output variables are stable.

If possible, a more extensive investigation will be conducted at 15 barg in order to compare the results obtained at FHA and ZSW test rigs, that pressure being the highest that can be reached at ZSW.

EIS measurements will be conducted under operation on the stack during this first tests, between j= jmin* - 1 A cm^{-2} (galvanostatic) in a frequency range of 13 kHz and 100 Hz.

- **Specific suggestion:**
  Galvanostatic EIS
  DC current: 0.5 A cm^{-2}
  AC current: 6.25 mA cm^{-2} (1.25 % of DC)
  Points/decade: 10 (approx.)

Table 5. Conditions to be tested at FHa's test-bench.

Current density (A/cm^2)	Pressure (barg)	KOH flow (mL/min.cm^2)**
J_{min}* – 1	5 – 30	0.2 - 0.75

*j_{min} will be defined based on the results of purity levels obtained in each permutation.

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**Full-cell flow rate.

5.2 Long-term galvanostatic steady-state test

After the first fingerprint test, a long-term galvanostatic steady state test will be conducted with the following specifications:

- Stack temperature: 80 ºC
- Lye concentration: 30 wt% KOH
- Impurities of the lye: not specified
- Iron content in the lye: not specified. To be measured and stated if > 10 ppm in relation to total weight
- Pressure (P_{test})*: 30 barg
- Volume flow (VF_{test}): defined in the first fingerprint test
- Current density (j_{test})*: 1 A/cm^2

*Target values, which may be modified if the performance of the stack under these conditions does not guarantee safe operation.

Prior starting this test, j_{test}, P_{test}, VF_{test} and j_{min} are defined considering the results of the first fingerprint tests.

Four steps will be followed during the test:

- Step 1: Conditioning at j_{test} and P_{test} until a stable voltage and pressure is reached.

- Step 2: At P_{test}, current-voltage curves / polarization curves from j_{test} to j_{min} (descending part of the polarization curve), followed by a measurement in reverse order (ascending part of the polarization curve). A step rate of 0.016 mA/cm^2 per minute will be applied, with a dwell time of 30 seconds and data acquisition time of 30 seconds.

- Step 3: Steady state at j_{test}, 80 ºC, P_{test}, for 400 h. KOH sample collection each 100 h to verify lye concentration. EIS measurement during the whole test at j_{test} (galvanostatic) in a frequency range of 13 kHz and 100 Hz.
  - Specific suggestion:
    Galvanostatic EIS
    DC current: 1 A cm^{-2}
    AC current: 6.25 mA cm^{-2} (0.625 % of DC)
    Points/decade: 10 (aprox)

- Step 4: Polarization curve as in step 2.

5.3 Second fingerprint performance test (approx. 50h)

Following the long-term galvanostatic steady state test, a second fingerprint performance test will be conducted, where the most critical conditions from the first fingerprint performance test will be replicated with the aim of detecting any possible deviation due to stack degradation.

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6 Deviations

Deviations may arise if 1 A/cm^2 cannot be achieved due to cell voltage restrictions and/or HTO values exceeding the safety limit (2 vol%). If this occurs, the target current density should be reduced to 0.7 A/cm^2 or a current density resulting in a cell voltage below 2.2 V/cell and HTO below 2 vol%.

Deviations due to high HTO/OTH may also arise in conditions where high KOH flows and pressure are used. If this happens, pressure and flows should be limited to lower values so that the safety limit threshold is not reached (2 vol%)

7 Conclusions

In this document the test protocols for carrying out cell- and stack- level testing in the ENDURE project are detailed. These protocols rely heavily on the harmonised test protocols developed by the JRC and expand the scope of the existing protocols to include a procedure for stack level testing.

Despite certain differences in the parameters of the testing setups, they consider the requirements for the electrochemical analysis for OER and HER, which can be standardized by all partners and, in general, by other research institutes. This includes variables like nominal current density, cell operating pressure, electrolyte concentration, flow rate, and temperature of the cell.

It must be noted that these protocols have been agreed by all partners and will be followed in all the tests performed in the frame of ENDURE.

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