

LOW TEMPERATURE WATER Electrolysers

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1 INTRODUCTION

1.1 Background and purpose

There is an acknowledged need for objective comparative assessment of the behaviour of electrolyser components and devices under conditions foreseen in future applications. For such assessment to be reliable and trustworthy, a number of requirements has to be met.

First, the assessment should be based on tests according to specifications agreed by a broad range of stakeholders, covering both performance and durability aspects. Testing according to these agreed specifications will contribute to improving the repeatability and reproducibility of the generated test results, thereby enhancing their comparability.

Second, agreement on the operating conditions imposed during testing for assessing performance and durability under representative application conditions is needed. This applies in particular for simulation of the dynamic operating conditions for electrolysers connected to fluctuating power sources such as wind or solar for off-grid applications, or subject to partial load operation for electricity grid balancing, or for supplying hydrogen to the gas grid or directly for power-to-gas applications, see Figure 1. This also requires agreement on the definition of appropriate electrolyser system boundaries.

Meeting the above set of requirements will improve the consistency between test results originating from different sources and enhance the representativeness of laboratory test results in simulating real-world applications.

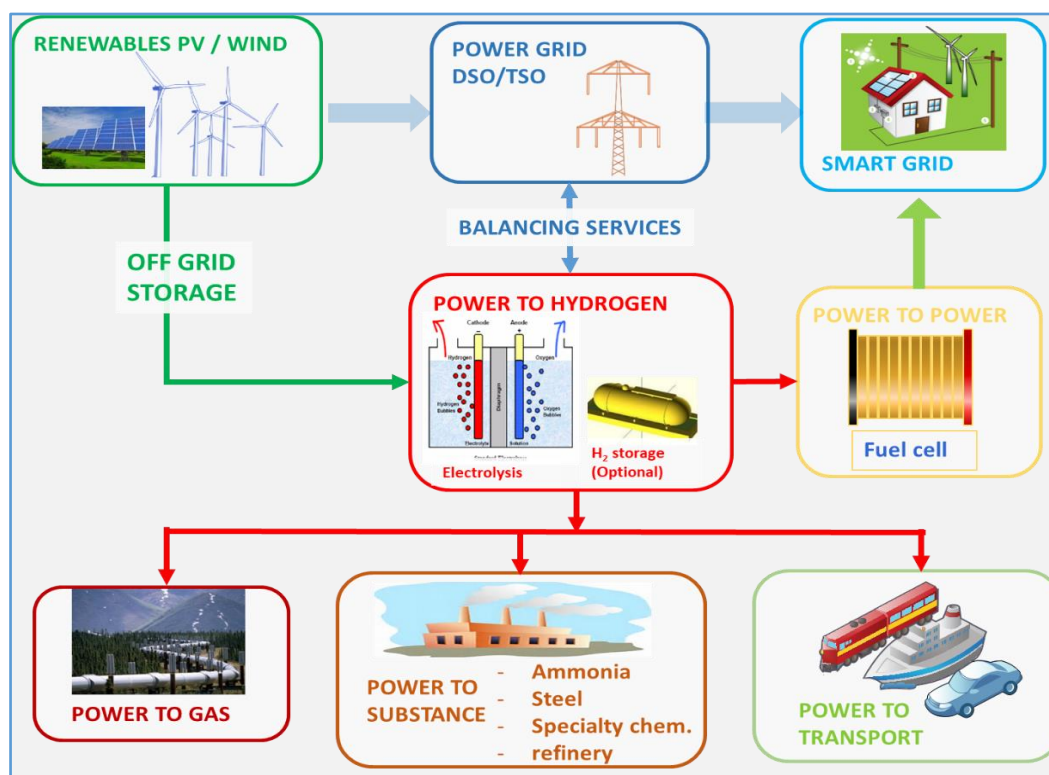


Figure 1. Electrolyser system grid integration

This report addresses the above needs by presenting an agreed set of operating conditions and of testing protocols for assessing both performance and durability of low

temperature water electrolyzers. It also suggests ways of graphically presenting the test results from performance and durability testing, comparing them to the results obtained under the reference operating conditions¹. It does not intend to replace testing practices currently used in various industries and research establishments.

In addition to their primary use for enabling comparison, the results of the "harmonised" performance and durability tests presented in this report are expected to assist FCH2JU through enhanced coherency of data originating from various FCH2JU funded projects. The results obtained from harmonised tests can serve in assessing Key Performance Indicators (KPIs) for low temperature electrolyzers and for proposing improved and/or new indicators for their performance and durability.

¹ The graphical representation of test results is complementary to the mandatory reporting in TRUST

2 OVERVIEW OF LOW-TEMPERATURE WATER ELECTROLYSIS TECHNOLOGIES

2.1 Underlying electrochemistry

In water electrolysis, two main parameters dictate technology differences namely:

- (i) the operating conditions: temperature and pressure (Figure 3a)
- (ii) the pH of the electrolyte (Figure 3b).

2.1.1 operating temperature

As shown in Figure 3a, electrolytic water dissociation is endothermic, i.e. it requires heat input in addition to electricity over the zero to 1,000 °C temperature range. The step change in the required amount of heat and hence in the total energy need (electricity + heat) at 100 °C is due to the water phase transition from liquid to gas. The heat required ($T \cdot \Delta S$) linearly increases with temperature T because the entropy change ΔS is assumed constant. Consequently, the Gibbs free energy change (ΔG) or electricity input required decreases with temperature, whereas the total energy need corresponding to the enthalpy change $\Delta H = \Delta G + T \cdot \Delta S$ only weakly depends on temperature both below and above 100 °C.

According to Faraday's law, the change in Gibbs free energy for an electrochemical system in equilibrium is expressed as

$$\Delta G = n F U_{\text{rev}}$$

with Faraday constant $F = 96,485 \text{ coulombs mole}^{-1}$ and n the number of electrons involved in the electrochemical reaction. U_{rev} represents the reversible cell voltage, which is the minimum voltage needed to drive the reaction. For water electrolysis, U_{rev} is the minimum voltage needed for water splitting. At lower cell voltage water electrolysis is not possible, whereas at higher cell voltage electrolysis is possible and heat is consumed in the reaction. Isothermal cell operation (i.e. reactant and reaction products at the same temperature) hence requires additional heat input from the environment.

However, cell operation generates heat by internal resistance as electric and ionic currents flow through the cell (Joule heating). This internally generated heat reduces the amount of heat to be supplied from the environment to the cell for maintaining thermal equilibrium. With increasing cell voltage, the internal heat generation by the Joule effect increases and at the thermoneutral voltage the internally generated heat equals the amount of heat $T \cdot \Delta S$ required for maintaining the reaction in thermal equilibrium. According to the above, the thermoneutral cell voltage U_{tn} given by

$$\Delta H = n F U_{\text{tn}}$$

is the voltage required for electrolysis without withdrawing heat from the surroundings. In this case, ΔH represents the amount of *electric* energy required for electrolysis in the absence of external heat supply.

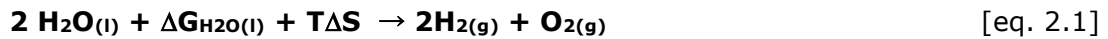
When the cell is operated at higher voltage than U_{tn} , the reaction becomes exothermic and heat needs to be removed for maintaining thermal equilibrium. In practice excess

heat is generated because electrolyzers are operated above the thermoneutral voltage to overcome losses incurred by inefficiencies of the electrochemical reactions and by electrical and ionic resistance as the current flows through the cell.

Because of the phase transition of water upon heating, two different regimes need to be considered for electrolysis of liquid water and for electrolysis of water in the gas phase (water vapour), respectively.

1) liquid water electrolysis

When water is supplied to the electrolyser in the liquid phase, as applies for low-temperature electrolysis, the hydrogen production reaction reads:



The reaction enthalpy $\Delta H_{\text{H}_2\text{O}(\text{l})} = \Delta G_{\text{H}_2\text{O}(\text{l})} + T\Delta S = 237.16 + 48.68 = 285.84 \text{ kJ.mol}^{-1}$. The energy content of the produced hydrogen corresponds to the higher heating value of hydrogen (HHV).

The indicated values for ΔG , $T\Delta S$ and ΔH apply for a perfect cell operating in a thermodynamically reversible manner at standard conditions of temperature (25°C) and pressure (1 bar). Under these conditions ($n=2$ for hydrogen)

$$U_{\text{rev, HHV}} = \Delta G_{\text{H}_2\text{O}(\text{l})} / (n \cdot F) = \mathbf{1.229 \text{ V}} \quad [\text{eq. 2.2}]$$

$$U_{\text{tn, HHV}} = \Delta H_{\text{H}_2\text{O}(\text{l})} / (n \cdot F) = \mathbf{1.481 \text{ V}} \quad [\text{eq. 2.3}]$$

2) water vapour electrolysis

When water to the electrolyser is supplied in the gas phase, the heat energy needed for water vaporization does not need to be provided and the reaction reads:



In this case, the reaction enthalpy $\Delta H_{\text{H}_2\text{O}(\text{g})} = \Delta G_{\text{H}_2\text{O}(\text{g})} + T\Delta S = 228.60 + 13.23 = 241.83 \text{ kJ.mol}^{-1}$ and the energy content of the produced hydrogen corresponds to its lower heating value (LHV). The difference between HHV and LHV originates from the latent heat of water evaporation. The thermoneutral and reversible cell voltages for water vapour electrolysis are:

$$U_{\text{rev, LHV}} = \Delta G_{\text{H}_2\text{O}(\text{g})} / (n \cdot F) = \mathbf{1.185 \text{ V}} \quad [\text{eq. 2.5}]$$

$$U_{\text{tn, LHV}} = \Delta H_{\text{H}_2\text{O}(\text{g})} / (n \cdot F) = \mathbf{1.253 \text{ V}} \quad [\text{eq. 2.6}]$$

For electrolyzers operating at temperatures above the 100°C boiling point of water, the use of LHV rather than HHV is relevant for the produced hydrogen and temperature dependences of ΔH , ΔG and $T\Delta S$ shown in Figure 2a need to be considered.

3) pressure and temperature effects on cell voltage

The effect of the pressure arises from the change in Gibbs free energy. For ideal gases the variation of the cell voltage as a function of pressure is expressed by the equation:

$$\Delta U_{cell} = U_{rev}(T, p) - U_{rev}(T, p^\theta) = \frac{RT}{2F} \ln \left[\left(\frac{p_{O_2}}{p^\theta} \right)^{\frac{1}{2}} \left(\frac{p_{H_2}}{p^\theta} \right) / \left(\frac{p_{H_2O}}{p^\theta} \right) \right] \quad [\text{eq. 2.7}]$$

The overall expression for the cell voltage, U_{cell} , as a function of temperature and pressure is:

$$U_{cell} = U_{rev}^0 + \frac{RT}{2F} \ln \left[(p_{O_2})^{\frac{1}{2}} (p_{H_2}) / (p_{H_2O}) \right] \quad [\text{eq. 2.8}]$$

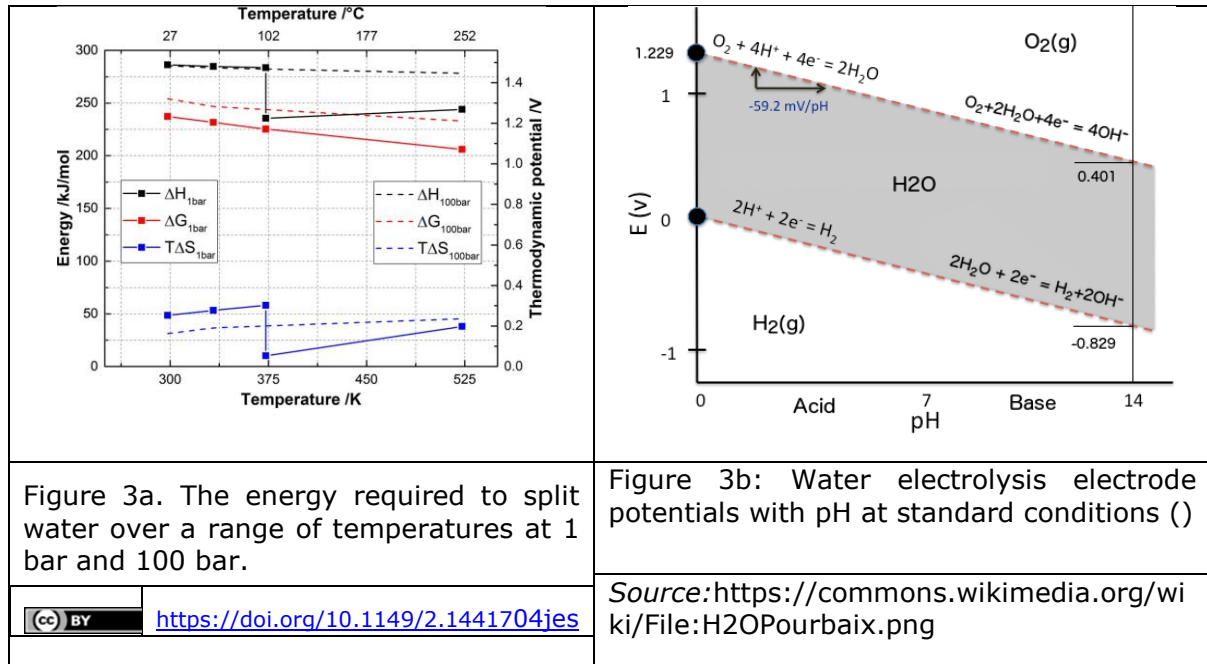


Figure 2. Water splitting characteristics

2.1.2 electrolyte pH

The values for U_{rev} and U_{tn} quoted in the previous section apply for electrolysis of pure water. Because of its low electric conductivity, electrolysis of pure water proceeds very slowly. By adding a water-soluble electrolyte, the conductivity of water rises considerably. The Pourbaix-diagram in Figure 3b shows that by increasing pH of the electrolyte, the half cell (water/hydrogen and water/oxygen) redox potentials shift downwards to a voltage range where conventional metals are usually passivated. This explains why water electrolyzers have traditionally been using an alkaline aqueous solution as electrolyte. This has changed recently with acidic ionomer membranes becoming commercially available, while research on alkaline membranes has started. These three types of electrolyte lie at the basis of the three low-temperature electrolysis technologies included in this report, namely AWE, PEMWE and AEMWE respectively.

2.2 Low-temperature electrolysis technologies

Three different low temperature electrolysis technologies are currently available as commercial products or under development, namely Proton Exchange Membrane (PEM) that uses an acidic polymer membrane sheet as solid electrolyte, Alkaline Water Electrolysis (AWE) that uses a liquid electrolyte (usually an aqueous solution of an

alkaline product, e.g. potassium hydroxide) and a diaphragm and more recently Anion Exchange Membrane (AEM) that uses a hydroxyl-ion conducting polymer membrane sheet as solid electrolyte. Relevant state-of-the-art data for these three technologies are summarized in **Errore. L'origine riferimento non è stata trovata..** A succinct description of these low temperature liquid water electrolysis technologies is given in the sections below. Note that high temperature electrolysis (700–1000 °C), such as Solid Oxide Water Electrolysis is not considered in this report.

Table 1. State of the art low temperature water electrolysis technologies

ELECTROLYSIS TYPE	PEMWE Proton Exchange Membrane	AWE Alkaline	AEMWE Anion Exchange Membrane
Charge carrier	H ⁺	OH ⁻	OH ⁻
Reactant	Liquid Water	Liquid Water	Liquid Water
Electrolyte	Proton exchange membrane	NaOH or KOH 20-40 wt.% /water	Anion exchange membrane
Anode Electrode	IrO ₂ IrO ₂ /Ti ₄ O ₇ Ir _x Ru _y Ta _z O ₂ , Ir black	Co ₃ O ₄ , Fe, Co, Mn Mo, P, S, NiFe(OH) ₂ , Fe(Ni)OOH, oxides, hydroxides, borides, nitrides, carbides based catalyst	IrOx Pb ₂ Ru ₂ O _{6.5} , Bi _{2.4} Ru _{1.6} O ₇ NiOx, Li _x Co _{3-x} O ₄ , Cu _{0.6} Mn _{0.3} Co _{0.21} O ₄ , CuCoOx
Cathode electrode	Pt/C	Raney Ni, Co, Cu, NiCu, NiCuCo, Ni-Co-W, Ni-Cu-Zn-B, Ni-Co, Ni-Fe, Ni-Co-Mo, NiCoZn, Raney Co, Ni-Mo, Ni-S, Ni-rare earth alloys	Raney Ni, NiO, Co based catalyst Ni/(CeO ₂ -La ₂ O ₃)/C
Current density	0.2-8.0 A/cm ²	0.2-2.5 A/cm ²	0.2-0.8 A/cm ²
Temperature	20-80 °C ²	40-90 °C	40-60 °C
Pressure H ₂ out ³	10 ⁵ – 30·10 ⁵ Pa	10 ⁵ – 30·10 ⁵ Pa	10 ⁵ – 30·10 ⁵ Pa
Cathode reaction (H ₂ evolution reaction HER) ⁴	2H ⁺ (aq) + 2e ⁻ → H ₂ (g)	2H ₂ O(l) + 2e ⁻ → H ₂ (g) + 2 HO ⁻ (l)	2H ₂ O(l) + 2e ⁻ → H ₂ (g) + 2 HO ⁻ (aq)

² Research efforts are targeting temperatures up to 150°C and 200°C with water vapour

³ Higher hydrogen output pressure reduces the compression needs for storage or transport of hydrogen,

⁴ (aq), (l) & (g) refers to aqueous, liquid and gaseous state

Anode reaction (O ₂ evolution reaction OER)	$\text{H}_2\text{O(l)} \rightarrow \frac{1}{2} \text{O}_2\text{(g)} + 2\text{H}^+\text{(aq)} + 2\text{e}^-$	$2 \text{HO}^-\text{(aq)} \rightarrow \text{H}_2\text{O(l)} + \frac{1}{2} \text{O}_2\text{(g)} + 2\text{e}^-$	$2 \text{HO}^-\text{(aq)} \rightarrow \text{H}_2\text{O(l)} + \frac{1}{2} \text{O}_2\text{(g)} + 2\text{e}^-$
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2.3 PROTON EXCHANGE MEMBRANE WATER ELECTROLYSIS (PEMWE)

A PEM water electrolysis cell is a zero-gap cell, i.e. the electrodes are directly sandwiched or coated onto the membrane. Reaction gases (H₂ and O₂) are evolved at the rear of the catalytic layers, and not in the inter-polar gap. This compact design allows for high (in the several A·cm⁻² range) current density operation. Figure 3 shows the cross-section of a PEM electrolysis cell. The elementary cell is delimited by two end plates usually made of titanium or of coated stainless steel, see Figure 5. The total cell thickness is typically 5-7 mm.

The central cell component is the proton conducting membrane {region 1} made of Perfluorosulfonic Acid (PFSA) or containing other chemical groups with similar behaviour. The membrane needs hydration to maintain conductivity, which limits the operating temperature, and is surface coated by two catalytic layers. Unsupported or carbon supported Pt nanoparticles are usually used at the cathode for the Hydrogen Evolution Reaction (HER) and supported or unsupported iridium dioxide (IrO₂) or alternative catalyst based particles are used mostly at the anode for the Oxygen Evolution Reaction (OER) {regions 2 and 2'}. Both catalytic layers are microscopically porous to allow gas evolution and contain a mixture of catalyst particles, support particles and ionomer which acts as a binder and provides a high ionic contact with the membrane.

The membrane and its two catalytic layers form a so-called Catalyst Coated Membrane or CCM. The CCM is clamped between two Porous Transport Layers (PTL) {regions 4 and 4'} which are used for water distribution as well as for gas collection and removal. In some cases, the catalyst layer is coated directly on the PTLs and not on the membrane. Whereas sintered titanium disks are usually used at the anode {region 3'}, carbonaceous PTLs are at the cathode {region 3}. PTLs can be subject to physicochemical degradation due to temperature gradients and hotspots, the presence of an acid environment [3], as well as to mechanical degradation caused by compression effects.

Cell spacers, meshes or grids can be placed between the end plates and porous transport layers. They offer an open space allowing water flux through the cell and gas removal from the cell. De-ionised liquid water is pumped through the anodic compartment to feed the electrolysis reaction and to remove heat (when the cell operates above the thermoneutral potential). To assist in heat removal and maintaining temperature constant, in some cases water is also provided to the cathode compartment. The gaseous reaction products H₂ and O₂ need to be de-humidified and the captured water is recirculated to the water inlet.

During operation, protons migrate from the anode (where they form) through the membrane to the cathode (where they reduce to hydrogen gas). During their migration, protons transport a number of water molecules from the anode to the cathode, a process known as electro-osmotic drag. The magnitude of this water flux depends on the type of proton conducting polymer, temperature, pressure and electric current density used in the electrolysis process.

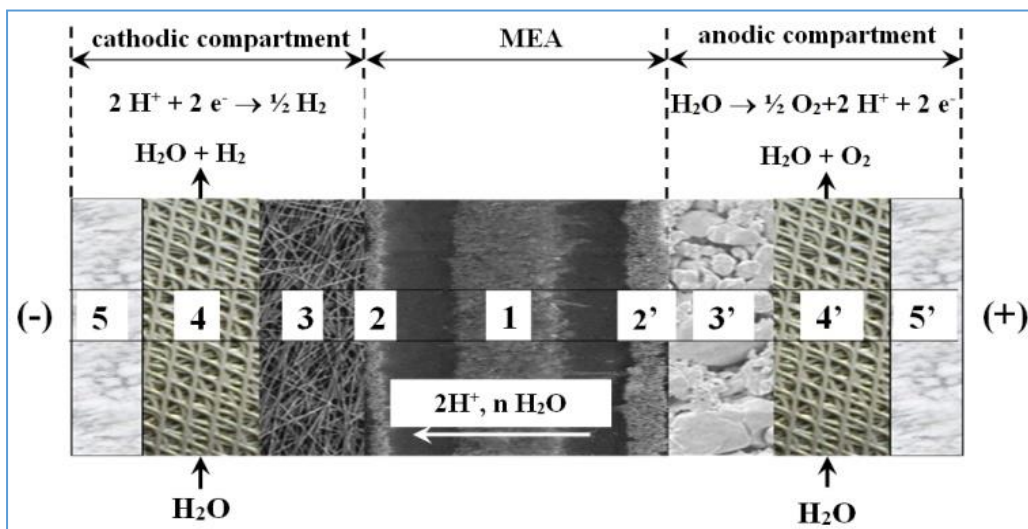


Figure 3. Cross section of a PEMWE cell.

Source: C. Rozain, P. Millet, *Electrochimica Acta* 131 (2014) 160-167

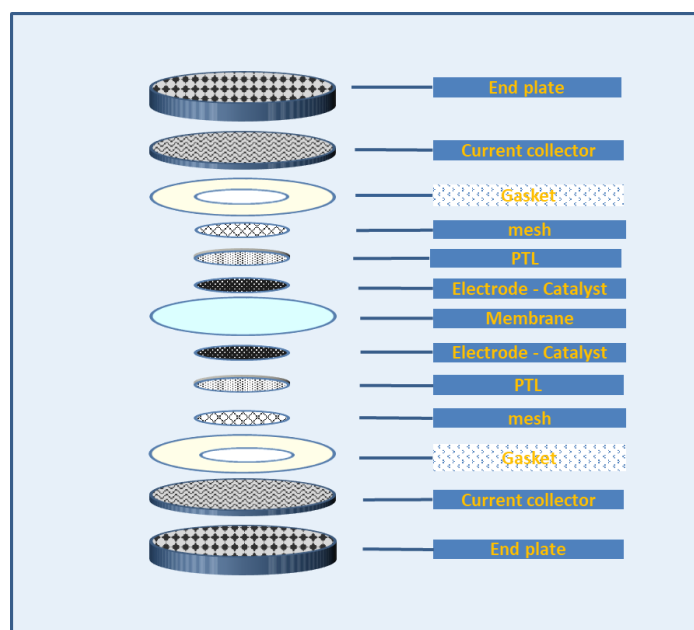


Figure 4. Typical PEM water electrolysis cell components (highlights identify those components for which functional testing is discussed in this report)

To yield higher hydrogen production rates, several single cells, a set of components as shown in Figure 4, are connected electrically in series and hydraulically in parallel as a stack. A metallic plate separates two adjacent cells and simultaneously acts as anode of one cell and cathode of the adjacent cell, hence the term bipolar plate (BPP). Pressure plates fix the components of the cells and provide the clamping force by threaded bolts and nuts.

Industrial PEM electrolyzers have a typical hydrogen discharge pressure of 10-30 bar. High pressure operation is possible in two modes: either with anode and cathode at the same pressure ("equibar"), or in differential mode with the hydrogen compartment at

higher pressure. In the latter case, the BoP of the oxygen compartment is simpler, but with the drawback of the additional stress on the MEA.

Compared to the main other low temperature electrolyser technology, alkaline electrolysis, a concentrated electrolytic solution is not required and the advantages include high current density, high energy efficiencies, and ease of gas separation. These advantages enable high performance and excellent load-following at a low safety risk and make PEM electrolyzers the most suited technology for coupling with RES. However, the acidic environment of PEM limits the materials of bipolar plates and current collectors, and in particular the catalysts to expensive platinum-group metals (PGMs). Also durability related to catalyst loss and membrane lifetime is an issue.

Hydrogen and oxygen produced with this technique have a very low level of contaminants. In the hydrogen gas, the main other compounds are water that can be easily removed and oxygen due to gas crossover that also can easily be removed (e.g. with catalytic conversion). The final hydrogen purity can reach 99.99%.

The power of PEM electrolyzers ranges from a few kilowatts to several megawatts. The system power, for equal cell area and current density depends on the number of stacks contained in the system.

Water purification treatment at 1 MΩ·cm level is recommended to minimize negative impact of impurities on membrane operation.

2.4 ALKALINE WATER ELECTROLYSIS (AWE)

Alkaline water electrolysis is a mature technology industrialised since the nineteenth century. In an alkaline water electrolyser (AWE), water molecules are decomposed electrochemically at the cathode to hydrogen molecules and hydroxide ions; the latter diffuse through the alkaline electrolyte and a diaphragm, and discharge at the anode releasing oxygen molecules.

The major components of an AWE single cell are the diaphragm and the two electrodes. The diaphragm has a microporous structure, allowing the alkaline electrolyte to seep through for sufficient ion-conductance. The electrolyte is an aqueous solution containing either sodium hydroxide (NaOH) or potassium hydroxide (KOH). The latter is usually preferred for its higher OH⁻ conductivity for the same molarities. The typical concentration of 20-40 wt.% corresponds to the highest conductivity; at higher concentrations the conductivity decreases due to Coulombic force interactions.

The KOH concentration can be expressed in % or in molar unit, hereafter a table and formulas for the conversion.

KOH [M]	KOH [wt.%]
1	5,45
2	10,61
3	15,50
4	20,13
5	24,54
6	28,72
7	32,71
8	36,51
9	40,14
10	43,61
11	46,92
12	50,10

M – molar mass of KOH = 56.10564 g·mol⁻¹

ρ - density of dry KOH = 2.044 g·cm⁻³

n – molarity of the KOH solution in water [M] \equiv [mol·l⁻¹]

c – KOH concentration [wt.%]

m – mass of dry KOH for 1l KOH solution in water = $M \cdot n$

$V_{\text{KOH solution}}$ = volume of KOH solution = $M n / \rho$

Formula for conversion from mol·l⁻¹ to wt.%:

$$c [\text{wt.\%}] = (M \cdot n) / (M \cdot n + (1000 - (M \cdot n / \rho))) \quad [\text{eq. 2.9}]$$

Formula for conversion from wt.% to mol·l⁻¹:

$$M [\text{mol} \cdot \text{l}^{-1}] = c (M \cdot n + (1000 - (M \cdot n / \rho))) / n \quad [\text{eq. 2.10}]$$

Anode and cathode are separated into two compartments by the diaphragm. Two configurations exist: Figure 6a depicts the cross-section of a gap-cell, in which a void between electrodes and diaphragm is filled with electrolyte. Gases evolve on both sides of each electrode, especially in the inter-polar domain, which results in ohmic losses. Figure 6b shows the cross-section of a zero-gap cell. Electrodes with latticed or porous structures (grids, meshes, fibre felts, porous sinters or foamed metals) are pressed against the central diaphragm. Gases evolve at the rear of both electrodes; leading to a reduction of ohmic losses caused by the gaps and gaseous films. The zero-gap design is widely used in modern alkaline water electrolyzers. The thickness of commercial diaphragms (e.g. Zirfon®, Supor-200®), which is equal to the distance between two electrodes, is typically 0.5 mm for the former and 0.14 mm for the latter. Some manufacturers integrate the electrodes and the diaphragm into a single component to achieve a true zero gap.

The cathode catalyst typically is a high-area nickel foam or nickel supported on stainless steel. Alternatives are Ni-Mo on a ZrO₂-TiO₂ support. The anode catalyst is usually made of Ni₂CoO₄, La-Sr-CoO₃ or Co₃O₄. Current distributors are typically nickel (the electrodes are directly pressed or welded onto the bipolar plates) with the main containment material being Ni-coated steel.

Alkaline electrolyzers like PEM ones have a very wide range of rated power, from a few kilowatts to several megawatts. The system power is a function on the number of stacks present in the system. Operation temperatures are in the range of 60 - 80 °C to ensure electrolyte conductivity and to promote reaction kinetics. Typical electrolysis pressure is 10-30 bar. The current density is typically 200 to 400 mA·cm⁻². In the latest technology

development, replacement of diaphragms by membranes with lower ohmic resistance has resulted in current density increases up to 1.5 A cm^{-2} and even to 2.5 A cm^{-2} by increasing the surface area of the Ni mesh electrode.

For AWE, water needs to be purified before use and the product gases must be dried before release. The purity level of hydrogen and oxygen can reach 99.9 and 99.7 vol. %, respectively, without auxiliary purification equipment.

Advantages of alkaline electrolysis are that it does not use expensive noble metal catalysts and that it is stable over a long lifetime, often in excess of 100,000 hours. One drawback is that alkaline electrolyzers experience high ohmic losses across the electrolyte and separator and hence operate at relatively low current densities compared to PEM electrolyzers. Historically alkaline electrolysis systems have shown poor dynamic behaviour, with limited load flexibility. This occurs because separator materials are not very effective at preventing cross-diffusion of gases. Specifically, oxygen crossing to the cathode leads to a lowering in efficiency (as it can be converted back to water) and to potential safety issues, particularly in low load scenarios. These challenges need to be overcome when using alkaline electrolyzers with RES such as wind or solar PV.

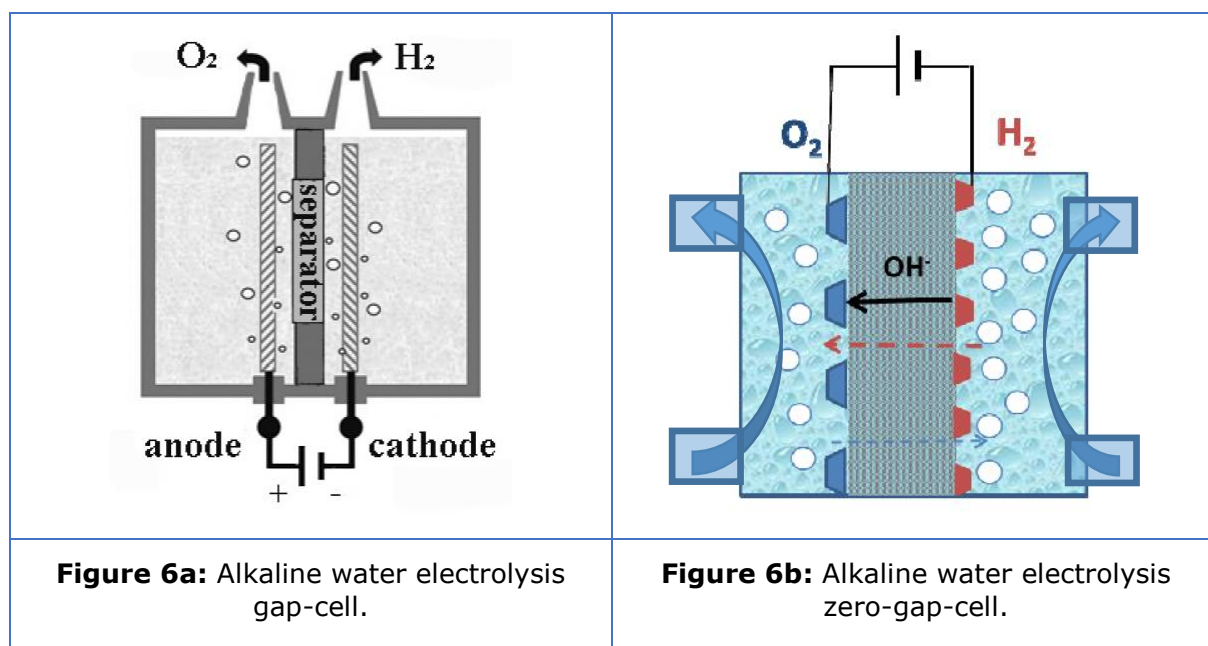


Figure 5. Alkaline electrolysis cell

2.5 ANION EXCHANGE MEMBRANE WATER ELETROLYSIS (AEMWE)

AEM (anion exchange membrane) electrolysis is a technology still under development. The main difference with alkaline electrolysis is the replacement of the diaphragm with a solid AEM, which allows the use of distilled water or of a low concentration alkaline solution as electrolyte instead of concentrated KOH or NaOH. The membrane serves as solid electrolyte for conducting OH^- ions and as separator for hydrogen and oxygen gasses. Compared to PEM electrolysis, switching the working condition from acidic to alkaline, relaxes the material restriction for the cell components. In particular, bipolar plates can be made from a cheaper material such as stainless steel, significantly reducing cost. In addition, alkaline conditions can enable the use of a lower amount of PGM catalyst, or even their substitution with catalysts based on lower cost transition metals, such as Ni, Co, Fe, Cu.

Figure 6 shows the schematic diagram of an anion exchange membrane (AEM) water electrolysis cell.

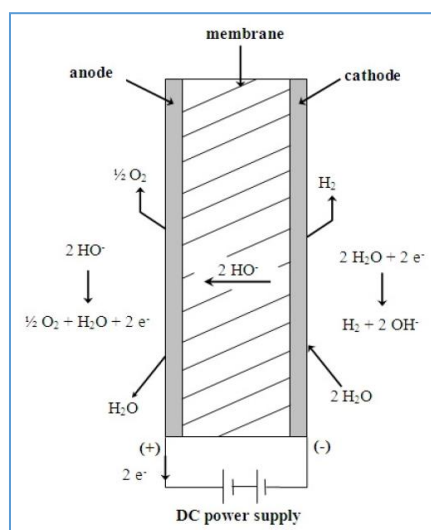


Figure 6. Schematic diagram of an AEM water electrolysis cell

Water quality requirements for AEMWE are similar to those for PEM WE. As for PEM electrolysis, the produced hydrogen can be easily pressurized. Because mechanical properties of the membrane and other components are almost the same for both PEM and AEM electrolyzers, safe pressurized operation of an AEM electrolyser is expected to be possible. However, for pressurized operation, the increase in hydrogen cross-permeation through the membrane needs to be addressed.

With a view to achieving commercially viable hydrogen production, AEM electrolysis requires further improvements, specifically regarding efficiency, membrane stability, robustness, and cost reduction [reference:]

3 MATERIALS TESTING for electrolyser applications

Improvement of properties of materials and of components for electrolyser application is based on a number of innovative methods, processes and manufacturing techniques. In a first step, "*screening*" the functional properties of newly developed or improved materials making up the different cell components is needed. Such "*screening*" is performed by two testing approaches, namely "*ex-situ*" and "*in-situ*", as depicted in Figure 7. When performing screening tests, either *ex-situ* or *in-situ*, care must be taken that the test outcomes are not affected by experimental artefacts or biased measurements resulting from testing in different environments, from damages during preparation (cutting, crushing, etc.), storing and handling, or from unintended damages induced by the testing technique itself ((i.e. rotating disk electrode measurements [4])).

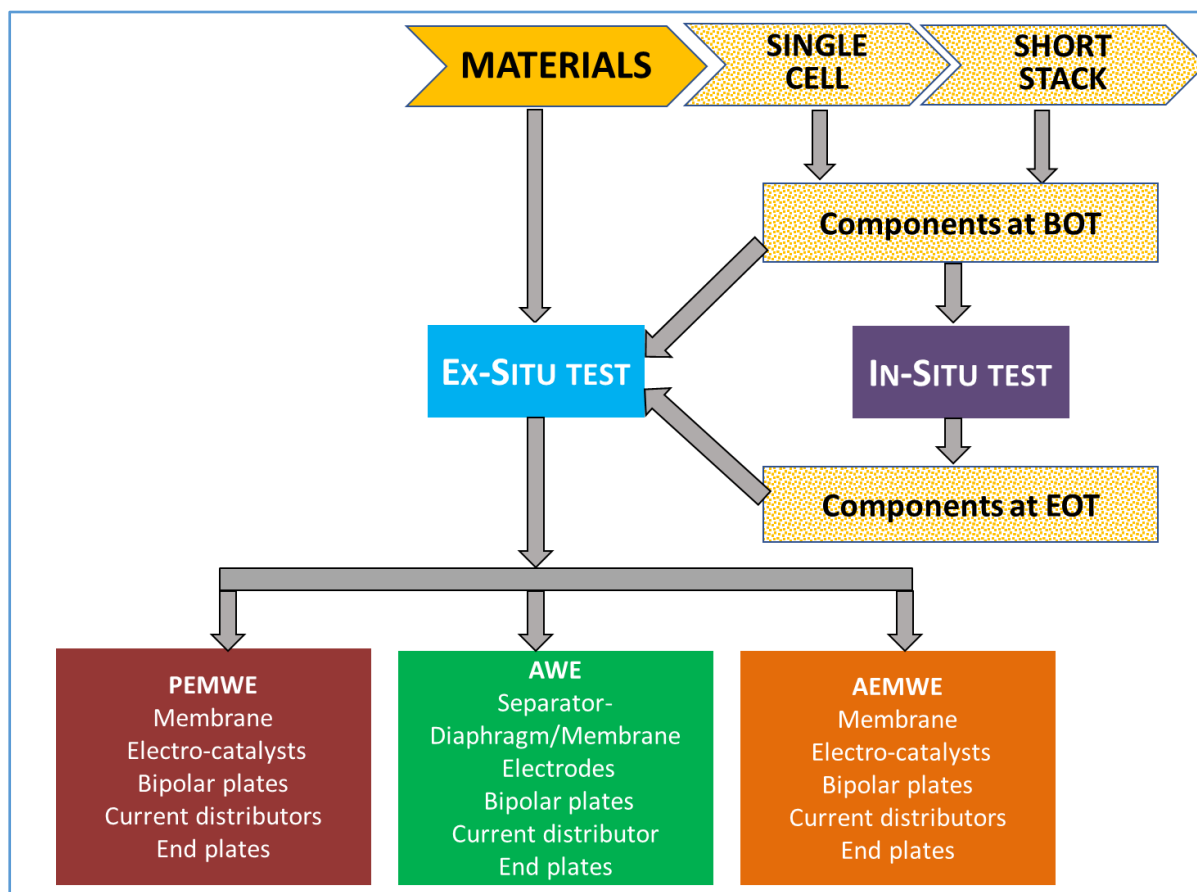


Figure 7. flow chart for functional materials testing according to the two approaches

"*Ex-situ*" tests discussed in this chapter support the development or improvement of materials to better meet the requirements for use in electrolysis cell components such as e.g. Catalyst Coated Membranes, CCMs, or Membrane Electrode Assemblies, MEAs. Such tests in which materials under test are not integrated in an electrochemical cell, may also be performed on materials that have been previously assembled in a cell structure for *in-situ* testing (which will be treated in chapter 4). In this case, *ex-situ* testing aims at evaluating the effect of material modifications that may result from electrolyser operation.

Ex-situ testing covers a wide range of analyses, aimed at assessing materials used in PEMWE, AWE and AEMWE components. The material properties to be characterised are those that affect cell performance. **Errore. L'origine riferimento non è stata trovata.** provides a list of such relevant functional properties and identifies associated test methods.

Table 2. Ex-situ test methods

Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Ultimate tensile strength	Tensile Test
	Elongation at break	Tensile Test
	Young's Modulus	Tensile Test
	Permeability	Mercury intrusion method, Mercury porosimeter
		Potential sweep (H2 crossover test)
	Hydrogen uptake and permeation	Potentiodynamic polarization test - ASTM G148 -97(2018)
	Porosity	Bubble point pressure test cell
		Mercury intrusion method, Mercury porosimeter
	Tortuosity	Mercury intrusion method, Mercury porosimeter
	Pore size distribution	Mercury intrusion method, Mercury porosimeter
	Mass loss in water (dissolution)	Hydrolytic stability test
	Water content	Water absorption test (ASTM D570)
	Water uptake	Water absorption (ASTM D570)
	volumetric expansion (swelling)	Water absorption test
	Hydrophobicity/hydrophilicity	Water contact angle test
	Surface Roughness	Profilometer; interferometer

LOW TEMPERATURE WATER ELECTROLYSERS

PHYSICO-CHEMICAL	Bulk / surface chemical composition Material microstructure <ul style="list-style-type: none"> • Grain size • Crystallographic phases • Crystal Orientation 	Cyclic voltammetry (CV)
		Atomic emission spectroscopy (AES)
		Atomic force microscopy (AFM)
		Brunauer-Emmett-Teller (BET) surface area measurement
		Energy dispersive X-ray spectroscopy (EDX)
		Field emission gun-scanning electron microscopes energy dispersive X-Ray analysis (FEG SEM-EDX)
		Fourier transformed infrared analysis (FTIR)
		Neutron tomography
		Scanning electron microscopy (SEM)
		Secondary ion mass spectroscopy (SIMS)
		Transmission Electron Microscopy (TEM)
		Extended X-ray absorption fine structure (EXAFS)
		X-ray absorption near-edge structure (XANES)
		X-ray diffraction (XRD)
		X-ray fluorescence (XRF)
		X-ray micro computed tomography (μ -CT)
		X-ray photoelectron spectroscopy (XPS)
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test
	Contact Resistance	4-wire Kelvin method
	Ionic Conductivity	In-Plane/through plane conductivity test

CHEMICAL	Reaction Kinetics	Rotating disk electrode (RDE) test
	Oxidative Stability (mass loss)	Fenton's reagent test
	Ion exchange capacity (IEC)	Chemical Titration
	Equivalent weight (EW)	Chemical Titration
	Metal dissolution	Inductively coupled plasma mass spectrometry (ICP-MS)
THERMAL	Thermal conductivity	thermal conductivity measurement
	Glass Transition Temperature	Dynamic mechanical analysis DMA(Tg)
	Thermal decomposition	Thermogravimetric Analyser/Differential Scanning Calorimeter (TGA-DSC)

Additional information on physico-chemical methods are presented in appendix A.

The cell components and functional property tests from the above table relevant for the individual low temperature electrolysis technologies are discussed in the following sections of this chapter.

Inputs to the test methods which are treated successively in the following sections have been gathered from literature or provided by the FCH-JU funded project ELECTROHYPEM, HPEM2GAS, NEPTUNE, NOVEL for PEMWE; ELYGRID, ELYNTEGRATION for AWE, ANIONE, CHANNEL and NEWELY for AEMWE.

3.1 PEMWE functional property testing

For PEMWE components ex-situ testing aims at establishing the functional properties of materials used in

- Membranes
- Electrodes and electrocatalysts layer
- Porous Transport Layers (PTLs)
- Current collectors, meshes, bipolar plates and separator plates
- End plates

3.1.1 PEMWE MEMBRANE MATERIALS

Key requirements for the membrane relate to its barrier effect to transfer electrons and gases between the anode and cathode compartments and its capacity of acting as electrolyte for ionic species. For the barrier effect gas transport properties are the most relevant, whereas the ionic resistance serves as a metric for electrolyte properties. The ionic resistance depends on:

- Ion exchange capacity (IEC, mmol ion·g⁻¹ polymer) or equivalent weight (EW, g polymer per mol, IEC = 1000 / EW).
- amount of water uptake
- cross-permeation of gasses, which is affected by permeability and depends on the applied current density
- Thickness
- Geometrical area

The ionic resistance can be characterised by two parameters: area resistance [$\Omega\cdot\text{cm}^2$], i.e. measured resistance [Ω] multiplied by the geometric area [cm^2], or resistivity [$\Omega\cdot\text{cm}^2/\text{cm}$], i.e. area resistance divided by thickness.

A list of functional properties of membrane materials is presented in **Errore. L'origine riferimento non è stata trovata.**. The protocols agreed for performing the associated ex-situ tests are identified in the table and described in detail in Appendix B. Because functional properties may depend on temperature and on pressure, assessment of PEMWE membrane materials may require performing ex-situ tests over a range of temperatures or pressures.

Table 3. PEMWE membrane material ex-situ tests

MEMBRANE			
Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL	Ref.
PHYSICAL	Thickness	micrometer screw gauge	B5
	Ultimate tensile strength	Tensile Test (ASTM D882-09, ASTM D638)	
	Elongation at break	Tensile Test	B9
	Young's Modulus	Tensile Test (ASTM D638 type V)	
	Permeability	Potential sweep (H2 crossover test)	B7
	Mass loss in water (dissolution)	Hydrolytic stability test	B2
	Water content	Water absorption test (ASTM D570)	
	Water uptake	Water absorption (ASTM D570)	
	volumetric expansion (swelling)	Water absorption test (ASTM D 756) <ul style="list-style-type: none"> ➤ Thickness increase Δz in H2O at specific T ➤ Machine Direction (MD) thickness 	B6

		increase Δx in H ₂ O at specific T ➤ Transverse Direction (TD) thickness increase Δy in H ₂ O at specific T	
	Surface Roughness	Profilometer; interferometer	
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test (Four-electrode chronopotentiometry)	
	Contact Resistance	4-wire Kelvin method	
	Ionic Conductivity	In-Plane/through plane conductivity test	B4
CHEMICAL	Reaction Kinetics	Rotating disk electrode (RDE) test	
	Oxidative Stability (mass loss)	Fenton's reagent test	B3
	Ion exchange capacity (IEC)	Chemical Titration	B1
	Equivalent weight (EW)	Chemical Titration	B1
THERMAL	Thermal conductivity	thermal conductivity measurement	
	Glass Transition Temperature	Dynamic mechanical analysis DMA(T _g)	B8
	Thermal decomposition	Thermogravimetric Analyser/Differential Scanning Calorimeter (TGA-DSC)	B8

3.1.2 PEMWE ELECTRODE AND ELECTROCATALYSTS

The function of an electrocatalyst is to increase the electrochemical reaction rate, in this case of water splitting, by reducing the activation energy. When precious materials are used such as Platinum or Iridium are used as catalysts, it is important to maximise their use by increasing the surface area. For this reason, some porous supporting medium like porous Titanium, Magneli phase is used for the anode electrode. Carbon fibres are suitable for the cathode only, because their corrosion resistance is too low in the anodic environment.

For functional property evaluation of electrocatalyst materials, ex-situ tests include determination of morphology, particle size and dispersion of the catalysts (see [Table 4. PEMWE electrode and electrocatalyst ex-situ tests](#) Table 4). For electrode materials, surface homogeneity, hydrophobicity/hydrophilicity, chemical resistance and electrical properties are the most relevant to be investigated.

Table 4. PEMWE electrode and electrocatalyst ex-situ tests

ELECTRODE AND ELECTROCATALYST		
Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Hydrophobicity/hydrophilicity	Water contact angle test
	Surface Roughness	Profilometer; interferometer
PHYSICO-CHEMICAL	Bulk / surface chemical composition Material microstructure <ul style="list-style-type: none"> • Grain size • Crystallographic phases • Crystal Orientation 	Cyclic voltammetry (CV)
		Atomic emission spectroscopy (AES)
		Atomic force microscopy (AFM)
		Brunauer-Emmett-Teller (BET) surface area measurement
		Energy dispersive X-ray spectroscopy (EDX)
		Field emission gun-scanning electron microscopes energy dispersive X-Ray analysis (FEG SEM-EDX)
		Fourier transformed infrared analysis (FTIR)
		Neutron tomography
		Scanning electron microscopy (SEM)
		Secondary ion mass spectroscopy (SIMS)
		Transmission Electron Microscopy (TEM)
		Extended X-ray absorption fine structure (EXAFS)
		X-ray absorption near-edge structure (XANES)
		X-ray diffraction (XRD)

		X-ray fluorescence (XRF)
		X-ray micro computed tomography (μ -CT)
		X-ray photoelectron spectroscopy (XPS)
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test
	Contact Resistance	4-wire Kelvin method
	Ionic Conductivity	In-Plane/through plane conductivity test
CHEMICAL	Reaction Kinetics	Rotating disk electrode (RDE) test
	Metal dissolution	Inductively coupled plasma mass spectrometry (ICP-MS)
THERMAL	Thermal conductivity	through-plane thermal conductivity measurement

3.1.3 PEMWE POROUS TRANSPORT LAYER MATERIALS

The porous transport layer acts as electroconductive diffusion layer facilitating mass transport of reactants and removal of reaction products between electrode and BPP. It is made of a porous medium or a combination of different porous media forming adjacent layers or a composite layer.

PTLs can be subject to electrochemical degradation due the combination of thermal variation and presence of an acid environment [3], as well as some mechanical degradation due to the compression effects. When differential pressure is applied, the PTL has to provide also mechanical support for the membrane.

The properties of PTLs are measured with tests similar to those used for the bipolar plate materials (see 3.1.4). Additional characterization methods are: gas/water permeation, mechanical tests, X-ray micro computed tomography, and neutron tomography for assessing water/gas presence

A list of ex-situ tests used for PTL materials is provided in Table 5.

Table 5. PEMWE PTL ex-situ tests

POROUS TRANSPORT LAYER		
Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Ultimate tensile strength	Tensile Test
	Elongation at break	Tensile Test
	Young's Modulus	Tensile Test
	Permeability	Mercury intrusion method, Mercury porosimeter
	Hydrogen uptake and permeation	Potentiodynamic polarization test - ASTM G148 -97(2018)
	Porosity	Mercury intrusion method, Mercury porosimeter
	Tortuosity	Mercury intrusion method, Mercury porosimeter
	Pore size distribution	Mercury intrusion method, Mercury porosimeter
	Hydrophobicity/hydrophilicity	Water contact angle test
	Surface Roughness	Profilometer; interferometer
PHYSICO-CHEMICAL	Bulk / surface chemical composition	Neutron tomography
	Material microstructure	Scanning electron microscopy (SEM)
	<ul style="list-style-type: none"> Grain size Crystallographic phases Crystal Orientation	X-ray micro computed tomography (μ -CT)
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test
	Contact Resistance	4-wire Kelvin method
CHEMICAL	Metal dissolution	Inductively coupled plasma mass spectrometry (ICP-MS)
THERMAL	Thermal conductivity	thermal conductivity measurement

3.1.4 PEMWE BIPOLAR PLATES AND CURRENT DISTRIBUTOR materials

A BPP is an electrically conductive gastight plate separating individual cells in a stack. It distributes current and reagents flows and provides mechanical support for the electrodes or membrane electrode assembly (MEA). For BPP materials the relevant functional properties include electrical/thermal conductivity, corrosion resistance and characterization of applied coating materials. A list of ex-situ tests used for bipolar plates and current distributor materials is reported in Table 6.

BPPs should have sufficient electrical and thermal conductivity. Often a flow field based on a channel structure, typically in the mm-range, is used to distribute the reactant water evenly over the active area and remove product gases and waste heat.

Table 6. PEMWE BPPs and current distributor ex-situ tests

Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Ultimate tensile strength	Tensile Test
	Elongation at break	Tensile Test
	Young's Modulus	Tensile Test
	Hydrogen uptake and permeation	Potentiodynamic polarization test - ASTM G148 -97(2018)
	Hydrophobicity/hydrophilicity	Water contact angle test
	Surface Roughness	Profilometer; interferometer
PHYSICO-CHEMICAL	Bulk / surface chemical composition	Atomic force microscopy (AFM)
		Field emission gun-scanning electron microscopes energy dispersive X-Ray analysis (FEG SEM-EDX)
		Neutron tomography
		Scanning electron microscopy (SEM)
		X-ray diffraction (XRD)
		X-ray micro computed tomography (μ -CT)
		X-ray photoelectron spectroscopy

		(XPS)
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test
	Contact Resistance	4-wire Kelvin method
CHEMICAL	Metal dissolution	Inductively coupled plasma mass spectrometry (ICP-MS)
THERMAL	Thermal conductivity	thermal conductivity measurement

3.1.5 PEMWE END PLATE materials

End plates are components located on either end of the electrolyser cell/stack serving to transmit the required compression to the stacked cells. The end plates may comprise ports, ducts and manifolds for transporting fluids (reactants, coolant) to and from the stack.

For end plates components the mechanical and thermal properties are relevant. A list of ex-situ tests used for end plate material assessment is given in Table 7.

Table 7. PEMWE end plate ex-situ tests

Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Ultimate tensile strength	Tensile Test
	Elongation at break	Tensile Test
	Young's Modulus	Tensile Test
	Surface Roughness	Profilometer
THERMAL	Thermal conductivity	thermal conductivity measurement

3.2 AWE FUNCTIONAL PROPERTIES

For AWE systems, materials of the following components require functional testing:

- Diaphragm
- Membranes (for newer AWE technology)
- Electrodes
- Support plates

3.2.1 AWE DIAPHRAGM MATERIALS

The main requirements for the diaphragm are ionic conductivity and gas separation capability. Ionic conductivity should be high to minimise ohmic losses, and it depends on material composition, porosity and wettability, as well as on thickness and assembly. Gas separation capability should also be high to withstand the anode-cathode differential pressure measurable with bubble point pressure (BPP) test cell. Measures to increase gas separation normally negatively affect ionic conductivity.

Other relevant properties are mechanical form stability, flexibility and chemical resistance.

Table 8. AWE Diaphragm ex-situ tests

DIAPHRAGM		
Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Ultimate tensile strength	Tensile Test (ASTM D882-09, ASTM D638)
	Young's Modulus	Tensile Test (ASTM D638 type V)
	Porosity	Bubble point pressure test cell
	Tortuosity	Mercury intrusion method, Mercury porosimeter
	Pore size distribution	Mercury intrusion method, Mercury porosimeter
	Surface Roughness	Profilometer; interferometer
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test (Four-electrode chronopotentiometry)
	Contact Resistance	4-wire Kelvin method
	Ionic Conductivity	In-Plane/through plane conductivity test

3.2.2 AWE MEMBRANE MATERIALS

With the latest technology developments polymeric membranes have been introduced in alkaline electrolyzers to improve their efficiency. The lower thickness of Nafion® or m-PBI (poly(2,2-(m-phenylene)-5,5-bibenzimidazole) membranes (30-180 µm compared to 500 µm or more of diaphragms) provides acceptable mechanical resistance, whilst it increases ionic conductivity.

For the applicable ex-situ tests see table 3.

3.2.3 AWE ELECTRODE MATERIALS

Electrodes are in contact with the electrolyte for the supply of electrical energy for the electrochemical reaction. Electrode materials normally consist of metal porous structures that exhibit high electrical conductivity, high surface area and high corrosion resistance in the electrolyte caustic environment.

Table 9- AWE Electrode ex-situ tests

ELECTRODE		
Property Classification	PROPERTY	EX-SITU TEST METHOD OR MEASUREMENT TOOL
PHYSICAL	Thickness	micrometer screw gauge
	Ultimate tensile strength	Tensile Test (ASTM D882-09, ASTM D638)
	Young's Modulus	Tensile Test (ASTM D638 type V)
	Porosity	Bubble point pressure test cell
	Tortuosity	Mercury intrusion method, Mercury porosimeter
	Hydrophobicity/hydrophilicity	Water contact angle test
	Surface Roughness	Profilometer; interferometer
PHYSICO-CHEMICAL	Bulk / surface chemical composition	Atomic emission spectroscopy (AES)
	Material microstructure	Atomic force microscopy (AFM)
	<ul style="list-style-type: none"> Grain size Crystallographic phases Crystal Orientation 	Energy dispersive X-ray spectroscopy (EDX)
		Neutron tomography
		Scanning electron microscopy (SEM)

		X-ray diffraction (XRD)
		X-ray fluorescence (XRF)
		X-ray micro computed tomography (μ -CT)
		X-ray photoelectron spectroscopy (XPS)
ELECTRICAL	Electrical Conductivity	In-plane/through-plane conductivity test
	Contact Resistance	4-wire Kelvin method
	Ionic Conductivity	In-Plane/through plane conductivity test
CHEMICAL	Metal dissolution	Inductively coupled plasma mass spectrometry (ICP-MS)
THERMAL	Thermal conductivity	thermal conductivity measurement

3.2.4 AEM SUPPORT PLATE MATERIALS

Support plates or end plates have mainly a mechanical support function and are made from metals. For bipolar plates, considering that their electric conductivity is four orders of magnitude, or more, higher than that of the electrolyte, the electrical properties are not critical. Normally nickel is used as it is cheap, stable and has low contact resistance. However use of passivating layers to protect against corrosion can significantly increase contact resistances which may become an important contribution to the total cell resistance. For the applicable ex-situ tests see Table 6.

3.3 AEMWE FUNCTIONAL PROPERTIES

Notwithstanding the difference between PEMWE and AEMWE related to the use of an acidic, resp. alkaline solid electrolyte, the cells and stacks of both technologies contain similar components assembled in a similar way. Hence, the materials of interest as well as the applicable ex-situ tests to establish their functional properties, are the same as for PEMWE (see 3.1).

4 IN-SITU TESTS

In "*in-situ*" tests the performance of materials is assessed by using measurement devices and sample connectors which are compatible with the operational environment that the materials are expected to experience in actual applications. These tests aim at evaluating performance under operating conditions in single cell or short stack arrangement, by measuring the electro-chemical properties in terms of voltage, current and time. Test campaigns should only start after appropriate Cell/Stack Activation and Conditioning according to the manufacturer's instructions has been performed.

In-situ testing can be performed over a wide range of testing conditions and at different moments in time, as indicated schematically in figure 9 below:

Testing conditions include static and dynamic operating conditions (the latter according to a given load-versus-time profile) imposed by the test hardware on the single cell or short stack. To be exhaustive, such conditions should cover normal and out-of-normal or "stressor" operation for the cell or stack components. Hence, four dimensions apply for in-situ test conditions: static/dynamic/normal/stressor.

The time dimension enters because the performance determined from results of in-situ tests constitutes an instantaneous measure that depends on the material properties of the components making up the single cell or short stack at a given time, as affected by their actual exposure history corresponding to a given load-versus-time profile. Interrupting the above-mentioned dynamic loading and subjecting a single cell or short stack to in-situ tests allows assessing the change of performance induced by a change in material properties⁵. Execution of in-situ tests upon repeated interruption thus allows monitoring performance degradation or, in other words, assessing durability.

To reduce testing effort and time required for assessing durability through performance degradation monitoring, the imposed load profiles may require adaptation to accelerate the induced degradation. This approach, known as accelerated in-situ testing, is based on the assumption of an increase in the rate of degradation, with the mechanism(s) causing degradation in the accelerated tests remaining the same as in actual service.

⁵ Performing ex-situ tests (as described in chapter 3) allows measuring the functional material properties at any moment during their exposure history.

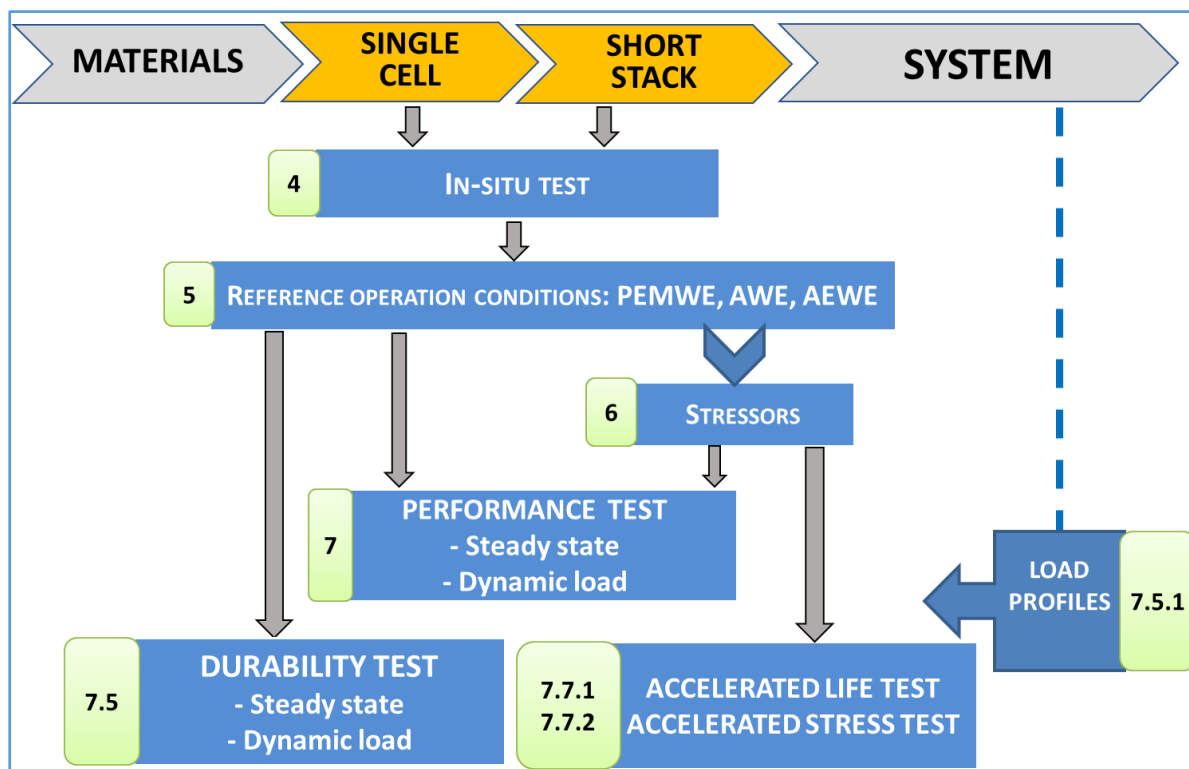


Figure 8. scheme for in-situ single cell and short stack testing. Number labels to boxes refer to chapters in this report

In-situ cell or short stack tests enable evaluating performance in terms of a number of indicators derived from the test outcomes, either as a direct experimental result, or indirectly from an analysis of the test outcomes. The most frequently used in-situ tests are:

- ❖ **Polarization curve (I-V Curve)** for overall electrochemical performance evaluation
- ❖ **Cyclic Voltammetry (CV)** to measure reaction kinetics for determination of electro-chemical active surface area
- ❖ **Electrochemical Impedance spectroscopy (EIS)** for separating ohmic, activation and concentration losses for evaluation of reaction rates, diffusion coefficients, charge transfer resistance and double layer capacity values

A detailed description of the associated test protocols is presented in JRC test reports for Polarisation Curve Measurements [7]; Cyclic Voltammetry [8] and Electrochemical Impedance Spectroscopy [9].

An orientation for the in-situ test approach is presented in the following table, where the last column shows the section of the present report in which this aspect is addressed.

Table 10: in-situ testing orientation table

In-situ Test level	Subject of the assessment	Topic	Section
--------------------	---------------------------	-------	---------

Single cell/ short stack	performance	Set of reference operating conditions	5
		Type and intensity of stressors	6
		Identification of performance indicators	7.1, table 22
		Definitions for efficiency	7.2
		Performance assessment	7.3
	Durability (through monitoring of performance degradation)	Identification of durability indicators	7.4.1-3, table 25
		Formulation of durability test protocol for steady state loading	7.5.1-2
		Formulation of durability test protocol for dynamic loading	7.5.3-4
	Accelerated testing	Formulation of protocol for accelerated life testing	7.7.1
		Formulation of protocol for accelerated stress testing	7.7.2
System	Performance	Identification of performance indicator	8.7, table 48
		Formulation of grid-balancing fit-for-purpose test	8.4
		Formulation of harmonised grid services protocols	8.5
		Definitions for efficiency	8.6

5 REFERENCE OPERATING CONDITIONS FOR TESTING OF SINGLE CELLS AND OF SHORT STACKS

Electrolyser materials are subjected to a variety of operating conditions. The aim of establishing a set of Reference Operating Conditions is to be able to test and evaluate the Reference Performance of different materials in-situ in single cell or short stack configuration and to provide a means for objective comparison of test results. The reference operating conditions should be representative as far as possible of current and future electrolyser applications.

The operating conditions selected as reference should ideally represent the centre of the window of normal operating conditions. However, in some cases electrolyzers are bound to operate under conditions outside of the normal operation window. Some of these conditions could be severe and act as stress factors to cell and short stack materials. These stressor conditions are elaborated upon in chapter 6.

Operating Conditions consist of two sets of parameters:

First, test parameters with a set-point value that can be controlled using a feedback loop within the test hardware are defined as **Test Input Parameters (TIP)**.

Second, non-adjustable parameters whose measured value depends on the values imposed by the TIPs are defined as **Test Output Parameters (TOP)**.

Whereas the instrumentation for experimental measurement of TIPs (measurement and control) and TOPs (measurement only) should in principle be located as close as possible to the relevant position of interest in the cell, this is not always feasible in practice. Therefore, in the following sections TIPs and TOPs are identified for properties that are experimentally accessible at the level of the test bench and of the anode and cathode compartments⁶. For test results to be valid, the values assumed by the TIPs have to fall within specific tolerances, independently or in combination with specific TOPs (see Table 14).

The following sections of this chapter list the agreed reference operating conditions (TIPs and TOPs) for the three considered low temperature electrolysis technologies.

⁶ In addition, electrical parameters current (density) and cell voltage serve as TIP and TOP, respectively.

5.1 REFERENCE OPERATING CONDITIONS for PEMWE CELL/SHORT STACK TESTING

5.1.1 CELL TEMPERATURE (*TIP*)

Because cell temperature is one of the most important parameters affecting performance, it should be controlled as accurately as possible. Hence, to minimize temperature variations from the intended setting, cell temperature is controlled by a temperature control system incorporated in the recirculating water loop which is adequately insulated to minimize thermal losses to the environment.

The TIP cell temperature should be representative of the temperature of the MEA where the water electrolysis reaction occurs. The temperature sensor for monitoring and controlling cell temperature should hence be located as close as possible to the MEA. Depending on test bench configuration, a number of cases can be differentiated for the location of the temperature sensor:

- i. Temperature sensor located inside the anode and cathode bipolar plate close to the MEA
 - The cell temperature is that indicated by the sensor at the anode side

For a temperature sensor placed in the water recirculation loop, two cases should be considered:

- ii. The liquid water is fed to the anode compartment only:
 - The cell temperature is equal to the temperature of the water measured as close as possible to the cell inlet.
- iii. The liquid water is fed to both anode and cathode compartments.
 - The cell temperature is equal to the average water temperature of the water measured at anode and cathode inlets.

For the latter two cases, the uniformity of the water temperature between inlet and outlet of the cell is important and the water temperature difference between outlet and inlet should be minimised.

5.1.2 WATER QUALITY (*TIP*)

The indicator used for the quality of the de-ionised water is its electrical conductivity measured at the cell inlet. Recirculation of the water may deteriorate the water quality because of possible accumulation of ions or impurities such as organic carbonaceous and non-conducting pollutants. Whereas the presence of such impurities may not show up in the results of electric conductivity measurements, cell performance/durability may nevertheless be affected. It is therefore recommended to fit a purification stage in the recirculation loop upstream of the cell inlet, and to measure water conductivity downstream of the purification system.

The water reacted is replenished in the recirculation loop through a deionisation water treatment system ("water make up" unit).

5.1.3 ANODE CONDITIONS

These are split in two classes: TIPs and TOPs which are discussed consecutively.

Anode Test Input Parameters

i. Anode water quality

➤ See 5.1.2

ii. Anode water inlet temperature

The anode water inlet temperature serves as TIP when the cell temperature cannot be measured in the cell bipolar plate close to the MEA. The temperature in this case should be measured as close as possible at the inlet of the cell/stack (case ii, 5.1.1).

iii. Water inlet pressure

Pressure is normally controlled indirectly with a feed-back control loop based on the gas outlet pressure that is adjusted from ambient to the maximum design pressure with a backpressure regulation valve.

iv. Water inlet Flow rate

The water inlet flow rate should in principle be set based on the cell or short stack active area perpendicular to the direction of the current, corresponding to the geometrical electrode area in contact with the membrane.

However, next to serving as feedstock for the electrolysis reaction, water also contributes to heat management of the cell/short stack by removing or minimising heat produced by the electrolysis reaction, thereby maintaining the correct temperature at the reaction site of the MEA. The water flow rate is related to the temperature difference between cell input and output through the dimensionless Lambda factor λ_{H_2O} [5] defined as:

$$\lambda_{H_2O} = \frac{2F}{M_{H_2O} C_{H_2O}^p \Delta T} (U^{cell} - U^{tn}) \quad [\text{eq. 5.1}]$$

Where

- λ_{H_2O} is the dimensionless and time independent ratio of the actual water flow rate to that of the electrolysed water;
- F = Faraday constant;
- M_{H_2O} = molar weight of water;
- $C_{H_2O}^p$ = heat capacity of liquid water at constant pressure;
- ΔT = water temperature difference between cell outlet and inlet;
- U^{cell} = cell voltage at operational T,p;
- U^{tn} = thermoneutral cell voltage at operational T,p.

The unit to be used for each of the parameters is that indicated in **Errore. L'origine riferimento non è stata trovata.**, Symbols.

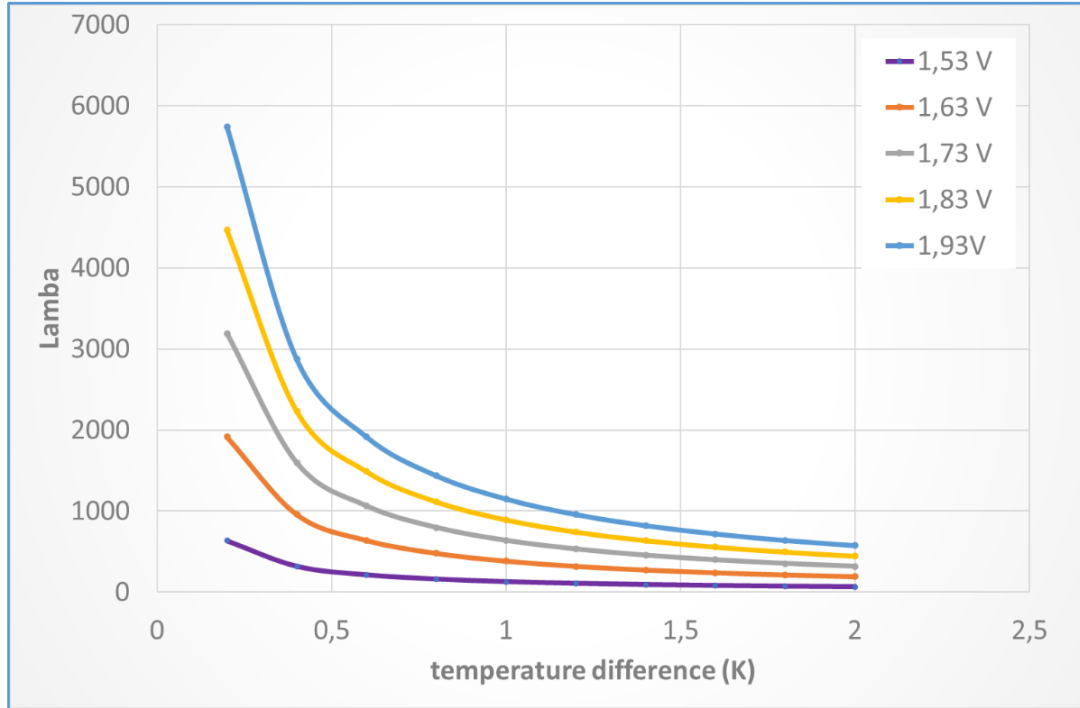


Figure 9. lambda plot for various cell voltages and temperature differences

As shown in Figure 10 lambda increases with decreasing temperature difference and increasing operating voltage, indicating the need for higher water flow rates to maintain thermal equilibrium under these conditions.

Another parameter that can be used for determining an appropriate water inlet flow rate to minimise the temperature difference between inlet and outlet is the water utilization factor (UF_w). It is defined as the ratio of the water reacted at a given current and the corresponding water flow fed to the anode and calculated as follows:

The relationship between water reacted and total current is

$$\dot{n}_{H_2O,reacted} = \frac{i \times A \times N}{2F} \quad [\text{eq. 5.2}]$$

Therefore with the specific feed water flowrate expressed in $\text{ml} \cdot \text{min}^{-1} \cdot \text{cm}^{-2}$ of active area, UF_w can be calculated as

$$UF_w = \left(\frac{i \times A \times N}{2F} \times M_{H_2O} \times 60 \right) / (\text{specific feed water flow rate} \times A \times N) \quad [\text{eq. 5.3}]$$

$$UF_w = \left(\frac{i}{96485,33} \times 18,015 \times 30 \right) / (\text{Feed water flow rate}) \quad [\text{eq. 5.4}]$$

$$UF_w = (i \times 0,0056) / (\text{Feed water flow rate}) \quad [\text{eq. 5.5}]$$

For typical water electrolysis operation, UF_w is commonly set at 0.5%. As shown in Figure 11, this corresponds to flow rates higher than 2 ml/min for a typical current density of 2 A cm^{-2} . For operation at higher current density, a higher water utilization factor is required for minimizing temperature difference while maintaining the reaction rate.

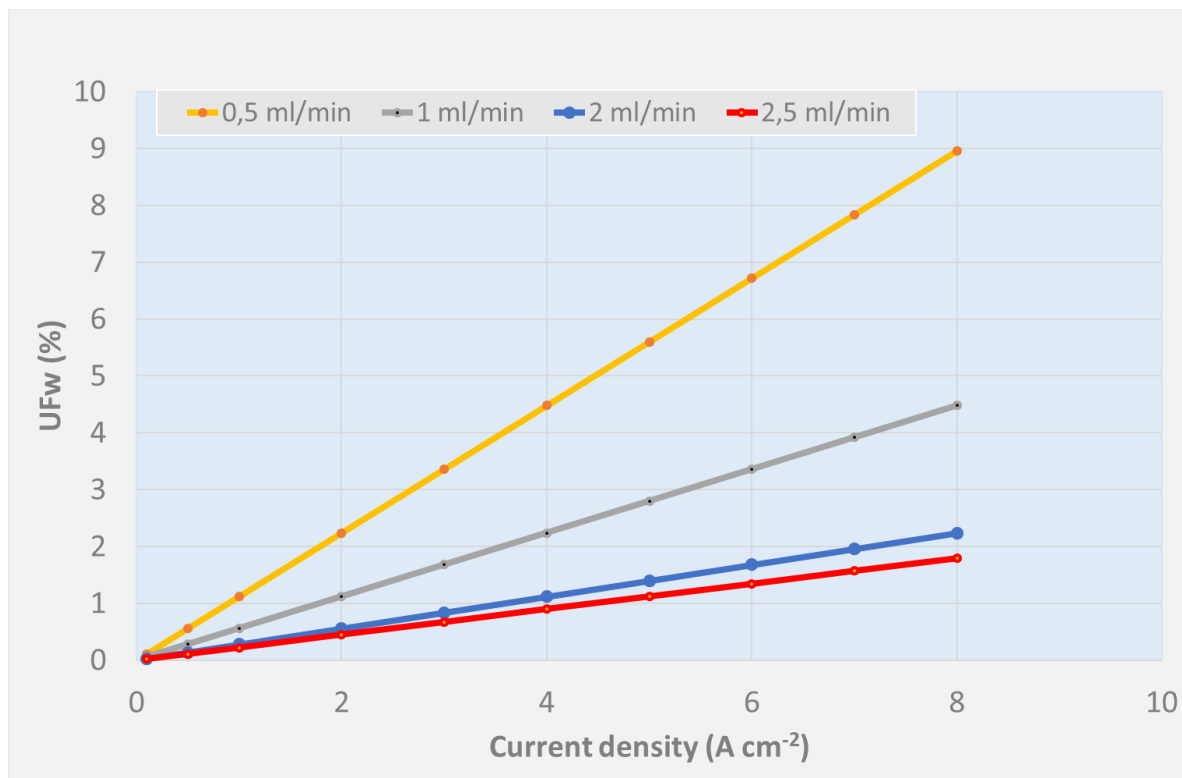


Figure 10. Water utilization factor (UFw) evolution Vs current density and water inlet flowrate

In addition to the water flowrate requirements corresponding to the use of water as reagent and its contribution to heat management, water flow rate also affects gas bubble evolution and removal at the electrodes, with higher flow rates having a beneficial effect on cell performance.

For the two above reasons, the water inlet flowrate is usually set considerably higher than that required for converting it into hydrogen and oxygen for a given active cell area and current density. Accordingly, the minimum specific feed flowrate in the water recirculation loop is set to 2 ml·min⁻¹·cm⁻² of active area.

v. Oxygen outlet pressure

Depending on the anticipated use of the produced oxygen, its pressure may serve as a TIP. In this case, oxygen pressure should be measured at the outlet of the electrolysis cell/short stack, after the water removal components, and before the backpressure regulation valve. The measured oxygen pressure is then fed as a feedback control signal to the anodic water circulation pressure control loop.

In pressurized test hardware, anode and cathode semi-cells can be designed to operate at the same or at different pressure; in the first case differential pressure effects on the MEA are minimized, while in the second case the oxygen line can be kept at lower (ambient) pressure, thereby reducing possible safety issues and simplifying BoP.

Anode Test Output Parameters

i. Water outlet temperature

The water outlet temperature depends on the water inlet temperature, flowrates of water and of oxygen, heat transfer resulting from ohmic losses in the MEA, conductive heat losses from piping and from hardware surfaces.

The temperature difference between water outlet and inlet serves as a test validity criterion and should not exceed +/- 2K (see Table 14).

ii. Oxygen Quality

When the produced oxygen is to be used as feedstock, its quality should be measured in real time at the cell or stack exit after water removal, according to the specifications for its use.

Due to the high diffusion coefficient of hydrogen, a certain amount of it is expected to be present in the oxygen gas stream. Experimental data shows that anodic hydrogen contamination decreases with increasing applied current density because the associated increase of oxygen evolution rate dilutes the permeating hydrogen, the amount of which is considered rather constant. The operating pressure is another important parameter affecting hydrogen concentration in the anodic compartment, and is limited to approximately 30 bar.

In the absence of a real-time quality control system, for safety reason it is recommended to monitor at least the presence of hydrogen in the oxygen outlet stream using a safety sensor to ensure that the hydrogen concentration does not reach the Lower Explosion Limit (LEL) of a hydrogen-oxygen gas mixture. LEL decreases with temperature and increases with pressure as shown in Table 11 and Figure 11, indicating that higher temperatures and lower pressures merit more attention [6]. In practice, when the hydrogen concentration exceeds 50% of LEL, appropriate safety measures should be triggered.

Table 11. Influence of pressure on explosion limits of H₂-O₂ mixtures at room temperature, 25 °C, and 80 °C

[Source: Schroeder, V., Sicherheitstechnische Untersuchungen für die Hochdruck-wasserelektrolyse zur Speicherung regenerativer Energie, Report No. VH2226, BAM Berlin, 2002]

Pressure in bar	LEL in mol% H ₂		UEL in mol% H ₂	
	25°C	80°C	25°C	80°C
1	4.0	3.8	95.2	95.4
5	4.6	4.4	94.6	95.0
10	5.0	4.8	94.2	94.6
20	5.4	5.2	94.2	94.6
50	5.5	5.3	94.6	95.0
100	5.7	5.7	94.9	95.3
150	5.7	5.3	95.1	95.5

200	5.9	5.7	95.1	95.5
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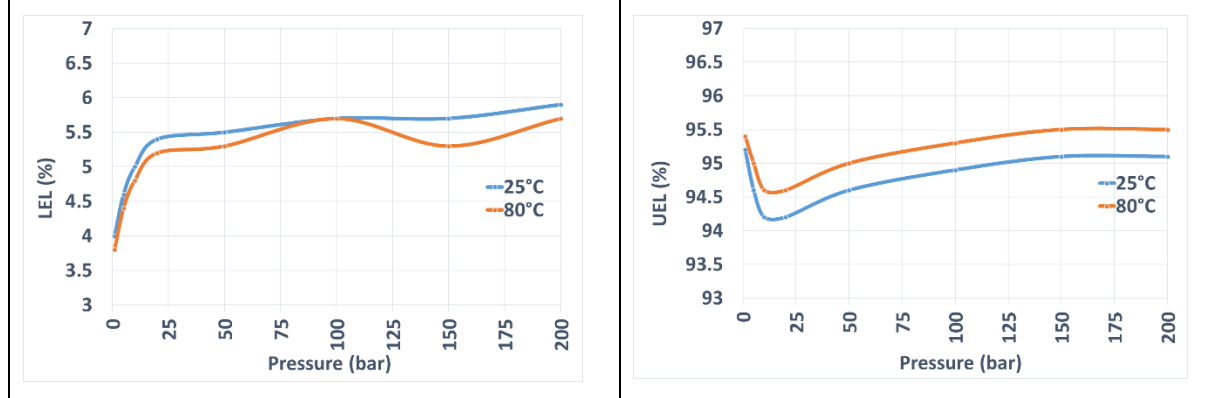


Figure 11. Pressure and temperature effect on LEL and UEL mixture H₂-O₂

iii. Oxygen production rate

The oxygen production rate is a direct consequence of the current applied with some loss depending by the overall system efficiency. Considering 100% faradaic efficiency the oxygen production rate is:

$$\dot{n}_{O_2,out} = \frac{I}{4F} \quad [\text{eq. 5.6}]$$

5.1.4. CATHODE CONDITIONS

These are split in two classes: TIPs and TOPs which are discussed consecutively.

Cathode Test Input Parameters

For heat management purposes, liquid de-ionised water can also be fed to the cathode compartment, in parallel to the anode water feed. The related TIPs are identical to those for the anode water inlet (temperature, quality, pressure) discussed before. An additional TIP at cathode side is

i. Hydrogen outlet pressure

The hydrogen pressure is measured at the outlet of the electrolysis cell/stack after the water mist and vapour removal components and before the backpressure regulation valve. The measured hydrogen pressure is fed as a feedback control signal to the water circulation pressure control loops for tests with the same pressure at anode and cathode side. For differential operation mode, the hydrogen outlet pressure is the relevant TIP, because for safety reason oxygen outlet pressure is set at ambient or lower pressure.

Cathode Test Output Parameters

When optional cathode water feed is used for heat management purposes, the same TOPs apply as for the anode water feed. Additional TOPs at cathode side are:

i. Hydrogen purity

The hydrogen purity should be measured in real time at the exit of the cell or stack, after water removal (when the cathode operates under water flow) by techniques such as Gas Chromatography (GC), Mass Spectrometry (MS), Thermal Conductivity (TC) detector, or Galvanic Electrochemical Cell. The time interval between measurements is determined by the selected technique and the response characteristics of the method used. For research and characterization purposes, the composition of the hydrogen stream should be checked at least every 30 minutes.

Hydrogen purity levels not achievable directly by electrolysis can be increased by additional purification step(s). The main purification technologies available are Pressure Swing Absorption (PSA), cryogenic condensation, getter/palladium membrane adsorption.

The required hydrogen purity for commercial electrolyser systems depends on its subsequent use. For example, for use in fuel cells, hydrogen purity should comply with the Fuel Quality Standards ISO 14687-2:2012 for fuel cell automotive applications and ISO 14687-3:2014 for fuel cell stationary applications. For all other industrial applications, the hydrogen purity level achieved by the electrolysis cell / stack should be mentioned.

In the absence of a real-time quality control system, for safety reason it is recommended to monitor the presence of oxygen in the hydrogen outlet stream using a safety sensor to ensure that the oxygen concentration does not drop below the Upper Explosion Limit (UEL) of hydrogen in a hydrogen-oxygen gas mixture. UEL increases with temperature and changes non-linearly with pressure as shown in Table 11. In practice, when the detected oxygen concentration exceeds 50% of the (100-UEL)% difference appropriate safety measures should be triggered.

ii. Hydrogen production rate

The hydrogen production rate is a direct consequence of the current applied with some loss depending by the overall system efficiency. With 100% faradaic efficiency the hydrogen production rate is:

$$\dot{n}_{H_2,out} = \frac{I}{2F} = \dot{n}_{H_2O,reacted} \quad [eq. 5.7]$$

5.1.5 SETTINGS of TIPS for PEMWE REFERENCE OPERATING CONDITIONS

The settings of the TIPS for the Reference Operating Conditions ("reference settings") for PEM Water Electrolysis for single cell and short stack are agreed as:

Table 12. Agreed reference settings for TIPS for PEMWE single cell and short stack testing

	Test Input Parameters	Unit	Reference Settings
Cell/short stack	Cell / Stack temperature	°C	60
	Water quality (conductivity) at recirculation loop <i>inlet</i>	$\mu\text{S}\cdot\text{cm}^{-1}$	≤ 1.0 ISO 3696 Grade 2 @ 25 °C
ANODE	Water inlet temperature	°C	60
	Water inlet pressure (absolute)	kPa	100
	Water quality (conductivity) <i>within recirculation loop</i>	$\mu\text{S}\cdot\text{cm}^{-1}$	≤ 1.0 ISO 3696 Grade 2 @ 25 °C
	Minimum Water inlet flowrate	$\text{ml}\cdot\text{min}^{-1}\cdot\text{cm}^{-2}$	2.0
	Oxygen outlet pressure (absolute)	kPa	100
CATHODE	Water inlet temperature	°C	60
	Minimum Water inlet flowrate (if applied)	$\text{ml}\cdot\text{min}^{-1}\cdot\text{cm}^{-2}$	2.0
	Hydrogen outlet pressure (abs)	kPa	100

5.1.6. TEST HARDWARE CONFIGURATION AND REQUIREMENTS FOR MEASUREMENT DEVICES OF TIPS AND TOPS for PEMWE CELL/STACK TESTING

To enable accurate control of the experimental conditions during cell and stack testing, the characteristics and location of the required instrumentation should be carefully considered. A general overall hardware configuration with location of TIPS and TOPs to be measured is schematically shown in

Figure 12.

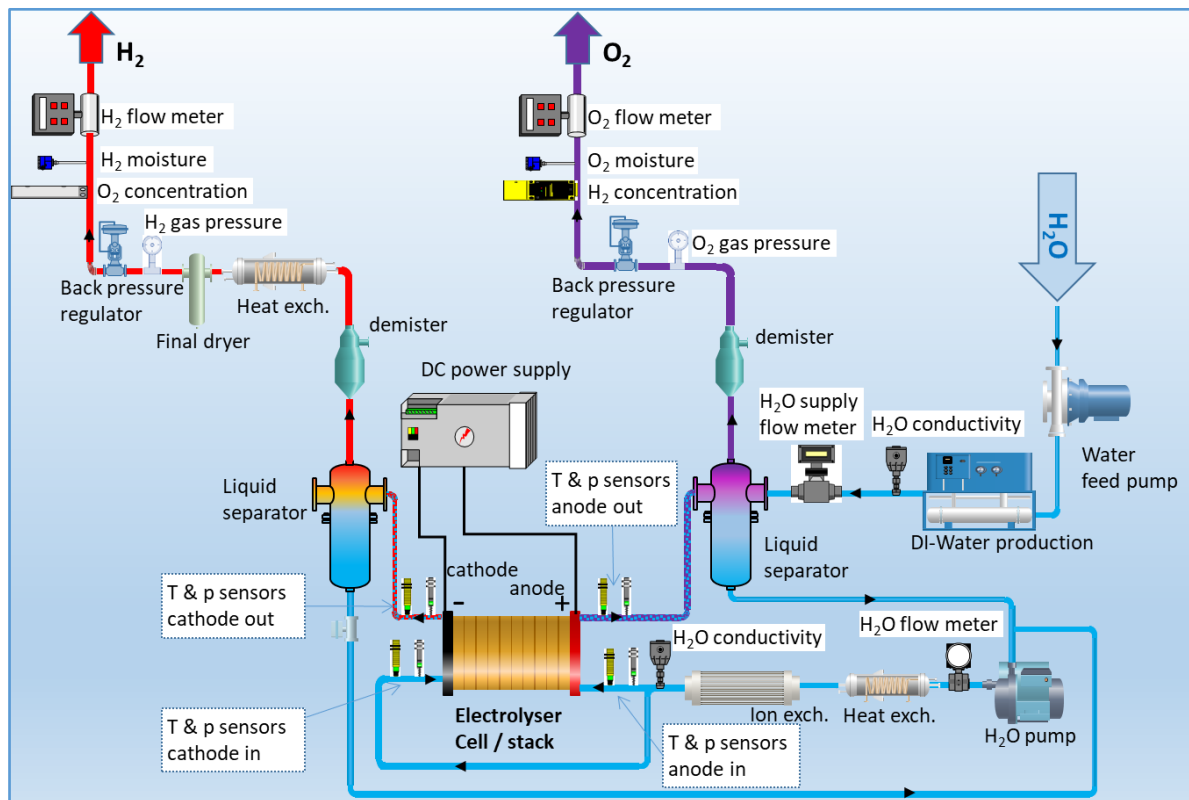


Figure 12: Scheme of PEM single cell/stack testing apparatus including position of the monitoring devices

Table 13 summarises the location of sensors or measuring devices (as in the figure above).

Table 13. Sensor type/location for PEMWE cell/stack testing

INPUT / OUTPUT PARAMETER	TIP/TOP	POSITION OF SENSORS
Current (or current density)	TIP	Power supply module
Cell/Stack voltage	TOP	Cell Hardware, Current collectors, Voltage terminals
Temperatures		
Cell	TIP	anode (TIP), and cathode BPPs, as close as possible to MEA
Water, anode inlet	TIP	as close as possible to cell/stack hardware inlet
Water, anode outlet	TOP	as close as possible to cell/stack hardware outlet
Water, cathode inlet (optional)	TIP	as close as possible to cell/stack hardware inlet
Water, cathode outlet (optional)	TOP	as close as possible to cell/stack hardware outlet
Pressures		
Water, anode inlet	TIP	as close as possible to cell/stack hardware inlet
Water, anode outlet	TOP	as close as possible to cell/stack hardware outlet
Water, cathode inlet (optional)	TIP	as close as possible to cell/stack hardware inlet, if in use
Water, cathode outlet (optional)	TOP	as close as possible to cell/stack hardware outlet
H ₂ , outlet	TIP	after liquid and vapour separation
O ₂ , outlet	TIP	after liquid and vapour separation
Flow rates		
Water feed to cell/stack	TIP	deionised water (DIW) cell inlet
Water make-up	TOP	outlet deionised water (DIW) production
Hydrogen	TOP	after water knockout
Oxygen	TOP	after water knockout
Water quality		
Water conductivity	TIP	outlet DIW production & recirculation loop
Gas safety sensors		
Hydrogen concentration	TOP	H ₂ gas sensor in O ₂ outlet
Oxygen concentration	TOP	O ₂ gas sensor in H ₂ outlet

The type of measurement devices for the TIPs and TOPs should be selected based on the required range, accuracy, and sampling rate as indicated in Table 14. Measuring equipment should be regularly calibrated.

Table 14. Required measurement accuracy and sampling rate

Parameter to be measured	Unit	Required Measurement accuracy	Sampling rate Performance test	Sampling rate Durability test
Current	A	0.001A	≥1 Hz	≥0,0166 Hz
Temperature	°C	± 2 K	≥1 Hz	≥0,0166 Hz
voltage	V	± 0.5% ⁵	≥1 Hz	≥0,0166 Hz
Pressure	kPa	± 2%	≥1 Hz	≥0,0166 Hz
Water Flow rate	l·min ⁻¹	± 2%	≥1 Hz	≥0,0166 Hz
Gas Flow rate	l·min ⁻¹	± 2%	≥1 Hz	≥0,0166 Hz
Gas concentration	%	± 1%	≥1 Hz	≥0,0166 Hz

For the generation of valid results from in-situ tests the following conditions have to be met during the full test duration:

- As a minimum all the TIPs and TOPs listed in Table 13 shall be measured
- The measurement accuracy and sampling rate shall meet the specifications listed in Table 14
- The temperature difference between water outlet and water inlet shall not exceed +/- 2K.
- Any deviation from the suggested hardware configuration and/or from the location of measuring devices shall be reported

The incorporation of additional temperature sensors at appropriate locations in the test rig layout of Figure 12 can provide supplementary information on performance in terms of efficiency of the cell/short stack (see chapter 7). Next to depending on the functional performance of its components and of the cell/short stack itself, efficiency may be affected e.g. by recuperating heat by the introduction of exchangers at different locations, as shown in Figure 12).

5.2 REFERENCE OPERATING CONDITIONS for AWE CELL/SHORT STACK TESTING

This subsection discusses TIPs and TOPs for in-situ testing of alkaline water electrolysis single cells and short stacks. Where possible and relevant, it refers back to those for PEMWE, while differences with PEMWE are elaborated specifically.

5.2.1 CELL TEMPERATURE (TIP)

Depending on test bench configuration, a number of cases can be differentiated for the location of the temperature sensor:

- i. Temperature sensor located close to the electrodes:
 - The cell temperature is that indicated by the sensor in the anode side.
- ii. Temperature sensor located in the electrolyte circuit:
 - The cell temperature is equal to the average temperature of the electrolyte measured at anode and cathode inlets.

For the second case, the uniformity of the electrolyte temperature between inlet and outlet of the cell is important and the temperature difference between outlet and inlet should be minimised.

5.2.2 Water Quality (TIP)

Considering the high alkalinity of the electrolyte it is important to reduce impurities in the replenishing water by a demineralisation treatment (or possibly de-ionization for higher purity levels) to minimize the concentration of magnesium and calcium in the feed water to avoid precipitation of their hydroxides, which may cause a performance reduction over time. The solvation of CO₂ (from the ambient air) should also be minimized. Requirements at system level are given by the manufacturer (e.g. concentration for Ca/Mg/Fe, Cl⁻, CO₃²⁻, SO₄²⁻, Si, etc.); for laboratory testing de-ionised water with 1μS·cm⁻¹ conductivity has been agreed as the reference to be used.

5.2.3 Electrolyte TIPs

The circulation of the electrolyte can be implemented in two different ways, (i) mixed circuit and (ii) separated circuit:

- i- mixed circuit: after separation of product gas from the electrolyte exiting each semi-cell, both electrolyte streams are mixed and the concentration is adjusted by water replenishment. As both electrolyte streams still contain remaining product gases soluble in the electrolyte solution, their mixing causes losses and higher gas impurities.
- ii- separated circuit: the electrolyte streams are kept separated. Water formation on the anode side results in a decreased concentration, while water consumption on the cathode side increases the concentration. Occasional mixing of the two electrolyte streams is needed to re-establish the optimal concentration.

The TIPs below relate to the presence of an aqueous alkaline solution acting as liquid electrolyte and contributing to heat management.

i. Inlet temperature

The electrolyte temperature at anode inlet serves as TIP when the cell temperature cannot be measured in the bipolar plate. The temperature in this case should be measured as close as possible at the inlet of the cell/stack.

ii. inlet pressure

Typically test benches have controlled back pressure valves positioned in the exhaust gas stream close to the cell outlet. Therefore, the pressure regulation is normally performed at cell outlet. Alkaline electrolyzers are operated with the same pressure (balanced pressure) on both compartments, or with a small differential pressure to limit cross-permeation of the electrolyte with its dissolved gases through the porous separator.

iii. inlet concentration

The concentration of the aqueous solution electrolyte is measured at the inlet of the cell and adjusted to the set value with fresh demineralised water replenishment to maintain the correct specific conductivity of the electrolyte. Examples of the specific conductivity change with electrolyte concentration for NaOH and KOH are given in **Errore. L'origine riferimento non è stata trovata..** The ionic conductivity increases with electrolyte concentration up to a value beyond which interactions by Coulombic force interactions between ions result in a reduction of the conductivity.

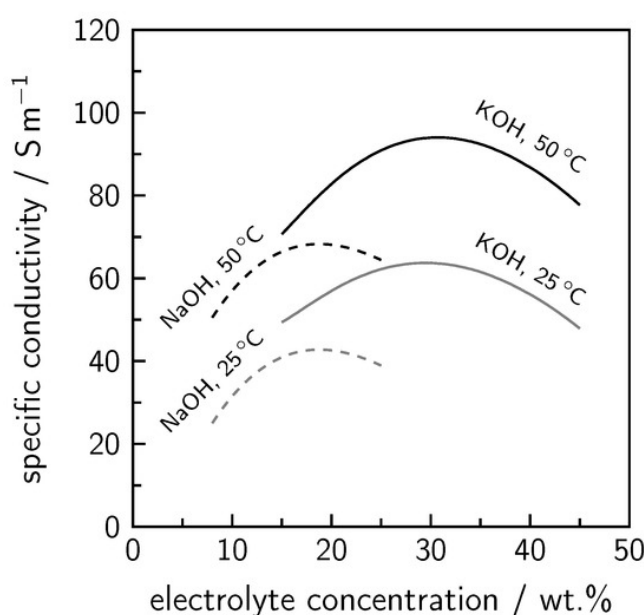


Figure 13. calculated specific electrolyte conductivity as a function of the electrolyte concentrations and temperature



Processes 2020, 8(2), 248; <https://doi.org/10.3390/pr8020248>

iv. inlet Flow rate

Electrolyte flow operation can be done without pump aid through natural convection. The associated savings of the reduced BOPs are counterbalanced by the risk of increasing the gas coverage of electrodes, hence requiring a higher operation cell voltage.

Nevertheless, for optimal process control the electrolyte is constantly pumped through the cell to maintain the electrolysis reaction and contribute to heat management. The circulation flowrate is hence set to minimise both the concentration difference and the temperature difference between inlet and outlet.

5.2.4 ANODE CONDITIONS

i. Oxygen outlet pressure (*TIP*)

In pressure balanced operation mode, the oxygen outlet pressure equals the hydrogen outlet pressure *TIP*. High outlet pressures increase the concentration of H₂ in O₂ at anode outlet and O₂ in H₂ at cathode outlet and thereby the risk of explosion, as shown in Figure 14 for the anode side.

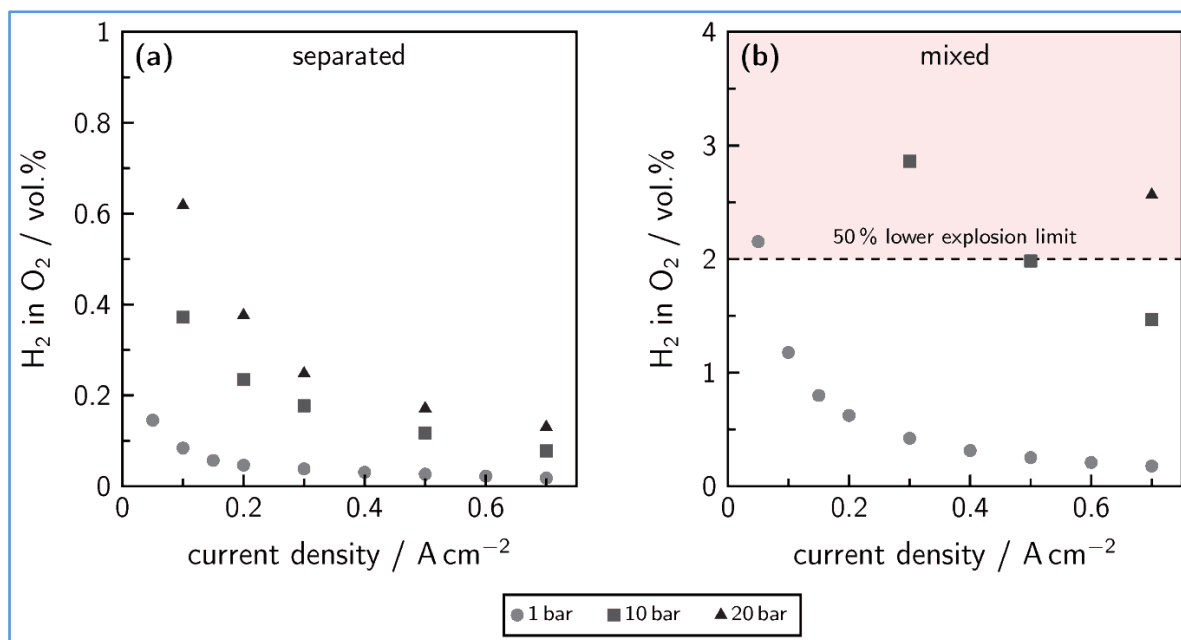


Figure 14 Anodic gas impurity (H₂ in O₂) in relation to current density at different pressure levels for (a) separated and (b) mixed electrolyte cycles

i. Oxygen Quality (*TOP*)

The typical oxygen purity is in the range 99.0 to 99.5% Vol. The most relevant impurity is hydrogen crossing the separator. The gas crossover is mainly due to gas dissolution in the electrolyte and diffusion through the separator, and hence depends on pressure, current density and electrolyte circulation configuration, either with separate or mixed cycle. With increasing pressure more hydrogen is dissolved in the electrolyte and therefore more hydrogen can reach the other semi-cell. Increasing current density results in higher hydrogen generation, and considering that current does not affect the amount of gas crossing the separator, higher dilution of the crossing hydrogen. Operation with mixed electrolyte circulation increases the level of contamination.

An example of hydrogen concentration in the oxygen outlet stream by changing pressure, current and electrolyte flow segregation is given in Figure 15 for a 32% concentrated electrolyte at 60°C and 0.35 L·min⁻¹ flow rate.

ii. oxygen production rate (*TOP*)

See 5.1.3

iii. electrolyte concentration at anode outlet (*TOP*)

The concentration difference between electrolyte outlet and inlet serves as a test validity criterion and should not exceed +/- 5wt% (see table 15).

iv. electrolyte temperature at anode outlet (*TOP*)

The temperature difference between electrolyte outlet and inlet serves as a test validity criterion and should not exceed +/- 2K (see table 15).

5.2.5 CATHODE CONDITIONS

The related TIPS are identical to those for the anode electrolyte inlet (temperature, quality, pressure) discussed before. An additional TIP at cathode side is

i. Hydrogen outlet pressure (*TIP*)

see 5.2.4 i

TOPs at cathode side are:

ii. Hydrogen quality (*TOP*)

See 5.1.4

iii. hydrogen production rate (*TOP*)

see 5.1.4

5.2.6. SETTINGS of TIPS for AWE REFERENCE OPERATING CONDITIONS

The settings of the TIPS for the Reference Operating Conditions ("reference settings") for alkaline water electrolysis for single cell and short stack are agreed as:

Table 15. Agreed reference settings for TIPS for AWE single cell and short stack testing

	Test input parameters	Unit	Reference Settings
cell / short stack	Cell / stack temperature	°C	65
	conductivity of water used for electrolyte preparation and supply to electrolyser	$\mu\text{S}\cdot\text{cm}^{-1}$	≤ 1 , ISO 3696 Grade 2 @ 25 °C
ANODE	electrolyte inlet temperature	°C	65
	electrolyte inlet pressure	kPa	100
	electrolyte inlet concentration	wt. %	30
	Min. electrolyte inlet flow rate	$\text{ml}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$	1
	Oxygen outlet pressure (absolute)	kPa	100
CATHODE	electrolyte inlet temperature	°C	65
	electrolyte inlet pressure	kPa	100
	electrolyte inlet concentration	wt. %	30
	Min. electrolyte inlet flow rate	$\text{ml}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$	1
	Hydrogen outlet pressure (absolute)	kPa	100

5.2.7 TEST HARDWARE CONFIGURATION AND REQUIREMENTS FOR MEASUREMENT DEVICES OF TIPS AND TOPS

Figure 15 shows a scheme with the location of the instrument measuring points for an alkaline water electrolysis experimental set-up.

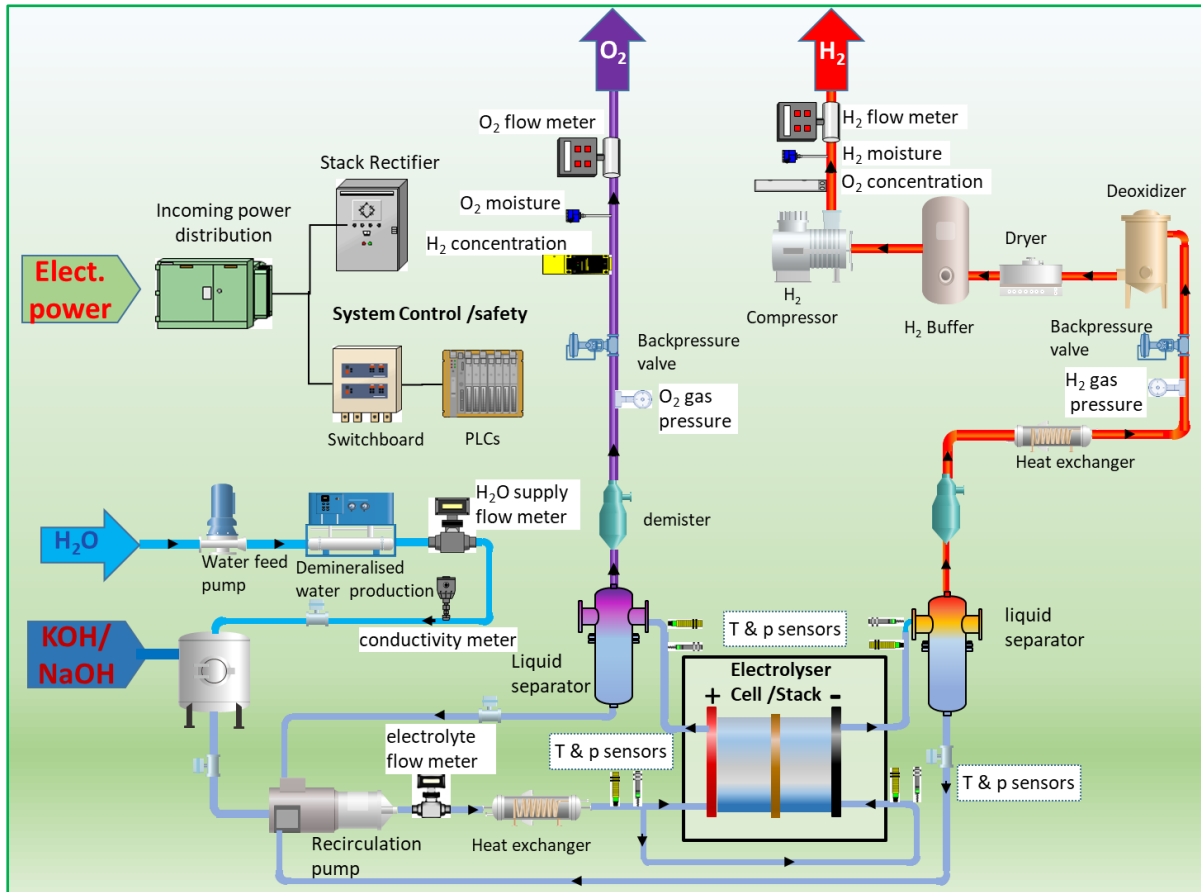


Figure 15. Scheme of AW Electrolyser with the position of monitoring devices

Table 16 summarises the location of sensors or measuring devices (as in the figure above).

The incorporation of additional temperature sensors at appropriate locations in the test rig layout of Figure 15 can provide supplementary information on performance in terms of efficiency of the cell/short stack (see chapter 7). Next to depending on the functional performance of its components and of the cell/short stack itself, efficiency may be affected e.g. by recuperating heat by the introduction of exchangers at different locations, as shown in Figure 15).

Table 16. Sensor type/location for AWE cell/stack testing

INPUT / OUTPUT PARAMETER	TIP/TOP	LOCATION OF SENSOR
-----------------------------	---------	--------------------

LOW TEMPERATURE WATER ELECTROLYSERS

Current (or current density)	TIP	Power supply module
Cell/Stack voltage	TOP	Cell Hardware, Current collectors, Voltage terminals
Temperatures		
Cell Temperature	TIP	Close to electrodes, TIP uses anode as reference
Anode inlet	TIP	as close as possible to cell/stack hardware inlet
Anode outlet	TOP	as close as possible to cell/stack hardware outlet
Cathode inlet	TIP	as close as possible to cell/stack hardware inlet
Cathode outlet	TOP	as close as possible to cell/stack hardware outlet
Pressures		
Electrolyte Anode inlet	TIP	as close as possible to cell/stack hardware inlet
Electrolyte Anode outlet	TOP	as close as possible to cell/stack hardware outlet
Electrolyte Cathode inlet	TIP	as close as possible to cell/stack hardware inlet
Electrolyte Cathode outlet	TOP	as close as possible to cell/stack hardware outlet
H ₂ outlet	TIP	after liquid and vapour separation
O ₂ outlet	TIP	after liquid and vapour separation
Flow rates		
electrolyte recirculation	TIP	recirculation loop
Water make-up	TOP	outlet demineralised/DI water production
Hydrogen	TOP	Mass Flow Meter after water knockout
Oxygen	TOP	Mass Flow Meter after water knockout
Water quality		
Water conductivity	TIP	outlet demineralised/DI production & recirculation loop
Gas safety sensor		
Hydrogen concentration	TOP	H ₂ gas sensor in O ₂ outlet
Oxygen concentration	TOP	O ₂ gas sensor in H ₂ outlet

For the generation of valid results from in-situ tests the following conditions have to be met during the full test duration:

- As a minimum all the TIPs and TOPs listed in Table 15 shall be measured
- The measurement accuracy and sampling rate shall meet the specifications listed in Table 14
- The temperature difference between electrolyte outlet and electrolyte inlet shall not exceed +/- 2K.
- The concentration difference between electrolyte outlet and electrolyte inlet shall not exceed +/- 5 wt%.
- Any deviation from the suggested hardware configuration and/or from the location of measuring devices shall be reported

5.3 AEMWE REFERENCE OPERATING CONDITIONS

Because of the similarity between PEMWE and AEMWE, the same set of Reference Operating Conditions as those for PEMWE can be used for AEMWE for both single cells and short stacks.

5.3.1 CELL TEMPERATURE

See 5.1.1

5.3.2 WATER QUALITY

See 5.1.2

5.3.3 ANODE OPERATING CONDITIONS

See 5.1.3

5.3.4 CATHODE OPERATING CONDITIONS

See 5.1.4

5.3.5 SETTINGS of TIPS for AEMWE REFERENCE OPERATING CONDITIONS

The settings of the TIPS for the Reference Operating Conditions ("reference settings") for Anion Exchange Membrane Water Electrolysis for single cell and short stack are agreed as shown in Table 17.

For further testing in alkaline environment it is possible to replace water as electrolyte using a KOH solution with molarity equal to 0.2M or 1 M.

Table 17: agreed reference settings for TIPs for AEMWE single cell and short stack testing

	Test Input Parameters	Unit	Reference Settings
Cell/short stack	cell / stack temperature	°C	50
	Water quality (conductivity) at recirculation loop <u>inlet</u>	$\mu\text{S}\cdot\text{cm}^{-1}$	≤ 1.0 ISO 3696 Grade 2 @ 25 °C
ANODE	water inlet temperature	°C	50
	water inlet pressure (absolute)	kPa	100
	Water quality (conductivity) <u>within recirculation loop</u>	$\mu\text{S}\cdot\text{cm}^{-1}$	< 1.0 ISO 3696 Grade 2 @ 25 °C
	Minimum Water inlet flowrate	$\text{ml}\cdot\text{min}^{-1}\cdot\text{cm}^{-2}$	1
	Oxygen outlet pressure	kPa	100
CATHODE	water inlet temperature	°C	50
	water inlet pressure (absolute)	kPa	100
	Water quality	$\mu\text{S}\cdot\text{cm}^{-1}$	< 1.0 ISO 3696 Grade 2 @ 25 °C
	Minimum Water inlet flowrate	$\text{ml}\cdot\text{min}^{-1}\cdot\text{cm}^{-2}$	1.0
	Hydrogen outlet pressure (abs)	kPa	100

5.3.6 TEST HARDWARE CONFIGURATION AND REQUIREMENTS FOR MEASUREMENT DEVICES OF TIPS AND TOPS

Figure 16 shows a scheme with the location of the instrument measuring points for an AEM water electrolysis experimental set-up.

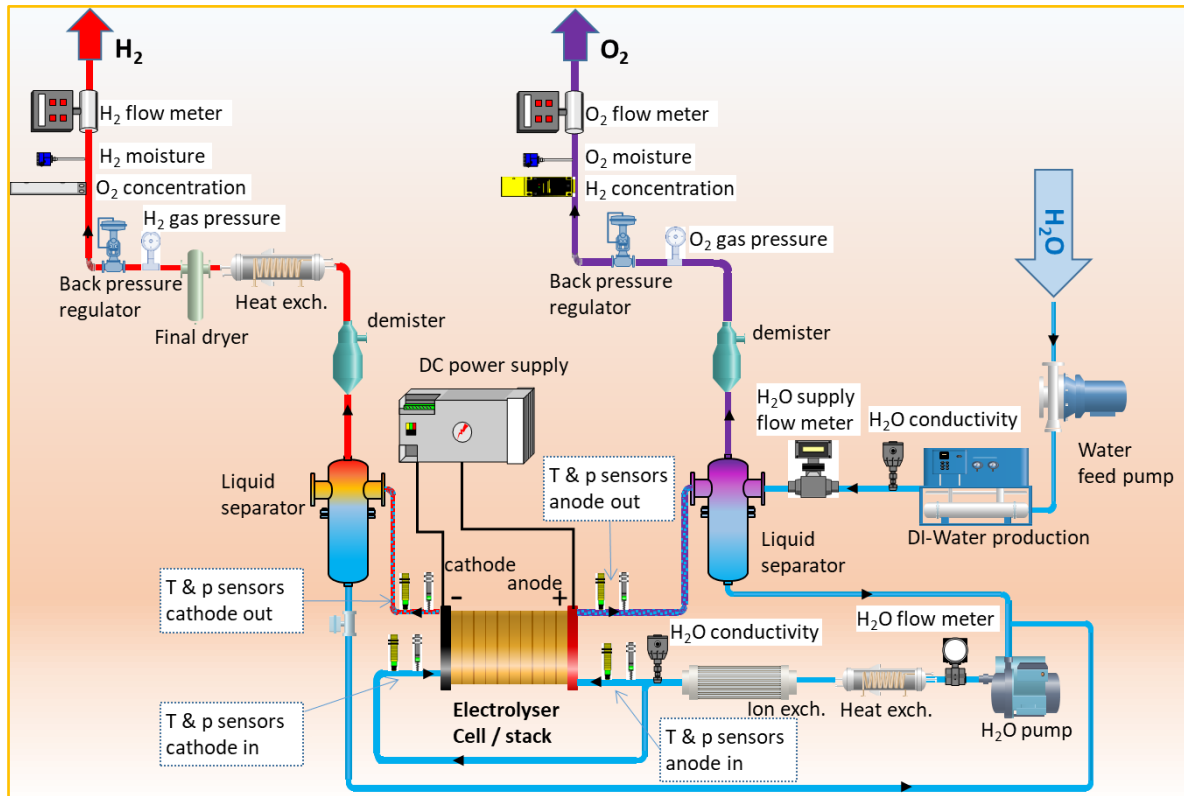


Figure 16. Scheme of AEMW electrolyser with the position of monitoring devices

For the generation of valid results from in-situ tests the following conditions have to be met during the full test duration:

- Considering the similarity with PEMWE the minimum set of TIPs and TOPs listed in Table 13 shall be measured
- The measurement accuracy and sampling rate shall meet the specifications listed in Table 14
- The temperature difference between water outlet and water inlet shall not exceed +/- 2K.
- Any deviation from the suggested hardware configuration and/or from the location of measuring devices shall be reported

The incorporation of additional temperature sensors at appropriate locations in the test rig layout of Figure 16 can provide supplementary information on performance in terms of efficiency of the cell/short stack (see chapter 7). Next to depending on the functional performance of its components and of the cell/short stack itself, efficiency may be affected e.g. by recuperating heat by the introduction of exchangers at different locations, as shown in Figure 16).

