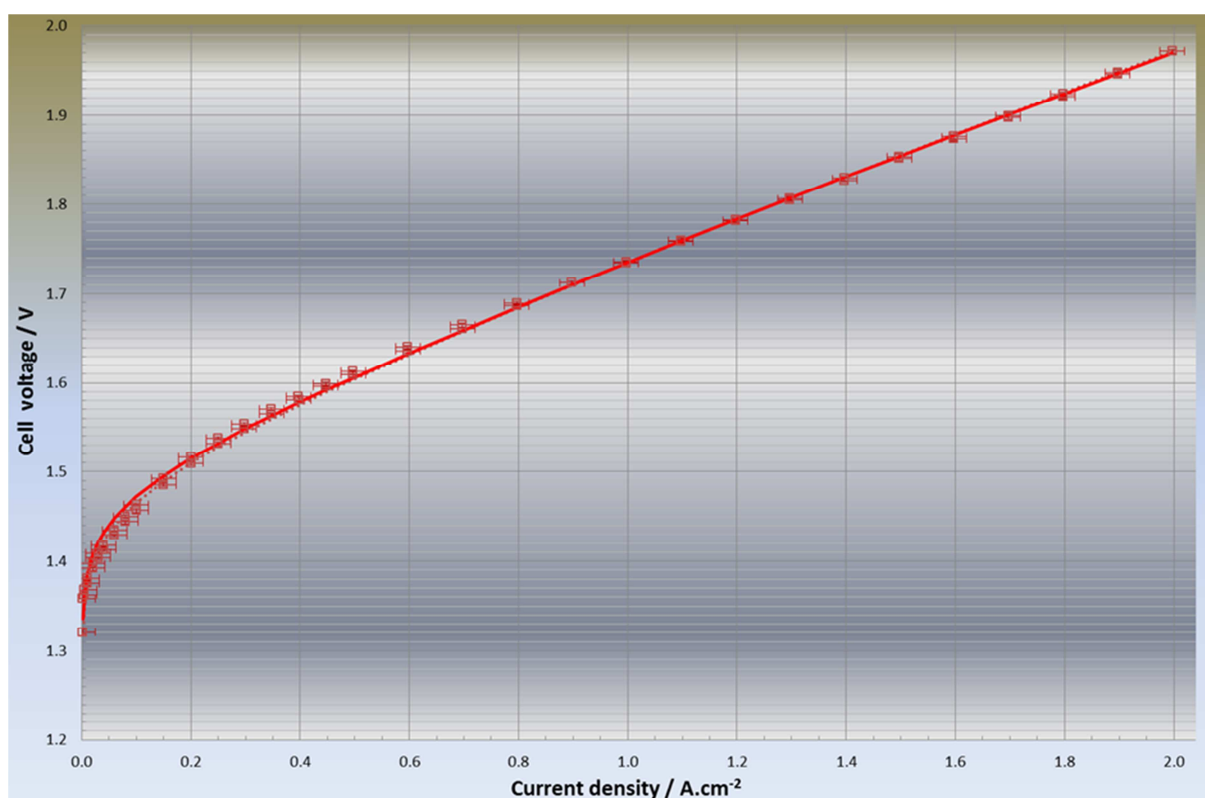


JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

EU harmonised polarisation curve test method for low-temperature water electrolysis

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De Marco G.

2018



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Contents

Contents	i
Foreword.....	ii
List of contributors	iii
Acknowledgments	v
1. Introduction.....	1
2. Objective and scope.....	2
3. Terminology, definitions and symbols.....	3
3.1. Terminology and definitions	3
3.2. Abbreviations	3
3.3. Symbols	5
4. Test equipment and set-up	8
5. Test input parameter and test output parameter	11
5.1. Test input parameter.....	11
5.2. Test output parameter.....	13
6. Test procedure	15
7. Data processing and representation	19
Bibliography	23
Appendix A Statistical analysis of test results	24
Appendix B Measurement statistics of TIPs and TOPs	27
Appendix C Measurement uncertainties of TIPs and TOPs	33
Appendix D Example of measurement data of Figure 1	36



Foreword

This report was carried out under the Framework Contract between the Joint Research Centre (JRC) and the Fuel Cells and Hydrogen second Joint Undertaking (FCH2JU) implemented by annual Rolling Plans.

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1. Introduction

This report on *EU harmonised polarisation curve test method for low-temperature water electrolysis* was carried out under the framework contract between the Joint Research Centre and the Fuel Cells and Hydrogen 2 Joint Undertaking, rolling plan 2017.

The polarisation curve measurement is a basic test method used to characterise low-temperature water electrolysis (WE) single cells and stacks at specified operating conditions (temperature, T and pressure, p). The procedure is applicable to polymer electrolyte membrane water electrolyser (PEMWE), alkaline water electrolyser (AWE) and anion exchange membrane water electrolyser (AEMWE) single cells and stacks.

A polarisation curve is the plot of the voltage, U expressed in volts (V) or the power density, P_d expressed in watts per square centimetre (W/cm^2) vs the current, I or the current density, j expressed in amperes per square centimetre (A/cm^2) of the cell/stack.

It provides information on the **reaction kinetics**, **ohmic resistance** and **mass transport resistance** of the cell/stack.

In almost every test campaign the testing protocol starts with a polarisation curve test and ends with another polarisation curve test. The comparison between the curves performed at beginning of life (BoL) and at end of life (EoL) provides information on performance and on degradation.

The current procedure is a general characterisation method and should be used in both research and development of low-temperature WE single cells and stacks.

2. Objective and scope

The purpose of this generic testing procedure is to characterise the performance of a low-temperature WE cell or stack in terms of polarisation curves at specified operating conditions.

The present procedure can be considered generic and useful for testing low-temperature WE for various applications, whether single cells or stacks.

At ambient pressure, the performance is measured from the lowest to the highest current density (**ascending part** of the polarisation curve), followed by a measurement in reverse order from the highest to the lowest current density (**descending part** of the polarisation curve), considering the specifications of the manufacturer and the objective of the test.

For operation under pressure, the performance is alternatively measured from the highest to the lowest current density (**descending part** of the polarisation curve), followed by a measurement in reverse order (**ascending part** of the polarisation curve).

3. Terminology, definitions and symbols

3.1. Terminology and definitions

All the terminology definitions used in this document are defined in [1].

3.2. Abbreviations

The abbreviations used in this document are given in Table 1.

Table 1. Description of abbreviations

Abbreviation	Description
AEMWE	Anion exchange membrane water electrolyser
AWE	Alkaline water electrolyser
Avg	Average
BoL	Beginning of life
Cov	Covariance
CVM	Cell voltage monitoring (and recording)
DAQ	Data acquisition
DC	Direct current
EoL	End of life
ERR	Measurement error
FM	Flow meter
FS	Full scale
GUM	Guide to the Expression of Uncertainty in Measurement
MEA	Membrane electrode assembly
MFC	Mass flow controller
NLLS	Non-linear least square

Abbreviation	Description
PEMWE	Polymer electrolyte membrane water electrolyser
QTY	Quantity
RMU	Relative measurement uncertainty
RSD	Relative standard deviation
STD	Standard deviation
STP	Standard temperature and pressure (273.15 K, 101.325 kPa)
TIP	Test input parameter
TOP	Test output parameter
Var	Variance
WE	Water electrolysis

3.3. Symbols

The symbols used in this document are given in Table 2.

Table 2. Definition of symbols

Symbol	Description
A	Active area of the cell (geometric)
F	Faraday's constant ($F = 96\,485.3\text{ C/mol}$)
j	Current density ($j = I/A$) of the cell/stack
j_0	Exchange current density (rate of the electro-catalytic reaction at equilibrium, zero net current) for the for the anode, a and cathode, c of the WE cell
I	Electrical current of the cell/stack
k	Step (set point) of the polarisation curve measurement
M	Number of values of a quantity measured
N	Total number of test conditions
p_A	Ambient pressure (absolute)
p_{H_2}	Pressure (gauge) of hydrogen after the gas liquid separator
p_{O_2}	Pressure (gauge) of oxygen after the gas liquid separator
$p_{x, y}$	Pressure (gauge) of cell/stack fluid x at cell/stack location y
$P_{d, \text{cell/stack}}$	Electric power density of the cell/stack
Q_{in}	Heat supplied by an external source
Q_{rev}	Reversible heat in the overall liquid WE reaction
$Q_{v, x, y}$	Volumetric flow rate of cell/stack fluid x at cell/stack location y under STP conditions
R	Universal gas constant ($R = 8.31446\text{ J.mol}^{-1}\text{.K}^{-1}$)
R_Ω	Total series resistance of components and their interface of the WE cell

Symbol	Description
T	Temperature
T _{x, y}	Temperature of cell/stack fluid x at cell location y (either inlet = in or outlet = out)
T _a	Ambient temperature
T _c	Cell/stack temperature
u	Measurement uncertainty
U _{cell}	Cell voltage
U _{on}	Onset voltage
U _{rev}	Reversible cell voltage
U _{stack}	Stack terminal voltage
U _{tn}	Thermal neutral voltage
X	Placeholder for a quantity (test variable)
Y	Placeholder for a function of test variable(s) X's (TIP or TOP)
z	Number of electrons exchanged in the cell reaction for one mole of gas
Subscripts	
avg	Average of a quantity (test variable)
err	Measurement error
H ₂	Reference to hydrogen or the hydrogen evolution electrode
k	Quantity at step (set point) k of the polarisation curve measurement
max	Maximum of a quantity
O ₂	Reference to oxygen or the oxygen evolution electrode
rmu	Relative measurement error

Symbol	Description
rsd	Relative standard deviation
std	Standard deviation
sterr	Standard error
X	Reference to quantity X
Superscripts	
a	Reference to the oxygen evolution electrode (anode) of the WE cell
c	Reference to the hydrogen evolution electrode (cathode) of the WE cell
Greek symbols	
α	Charge transfer coefficient
η	Overtoltage of the electrode of the WE cell
ΔG	Gibbs free energy in the overall liquid water electrolysis reaction
ΔG^0	Gibbs free energy in the overall liquid water electrolysis reaction at 101.325 kPa absolute pressure and 25 °C (298.15 K) temperature
ΔH	Enthalpy change in the overall liquid WE reaction
ΔS	Entropy change in the overall liquid WE reaction
ρ_x	Density of cell/stack fluid x under STP conditions

4. Test equipment and set-up

This test procedure does not prescribe the type, geometry and size of the single cell or stack unless proprietary information, materials, designs, geometry and sizes of the MEA, mono-polar plates and cell have to be described in the test report to meet the test objective.

The test bench comprises subsystems to provide the cell with fluids in a defined manner (flow rate, pressure and temperature), a DC power supply and a heating/cooling subsystem for controlling the cell/stack temperature.

The test bench is controlled by a computer, which also acts as a data-acquisition unit. Table 3 lists the recommended test bench and sensor requirements with their main specification.

Table 3. Recommended test bench requirements and main specifications

Description	Specification	QTY
Hydrogen evolution electrode pressure sensor*	Range dependent on applied pressure	1 (minimum)
Oxygen evolution electrode pressure sensor*	Range dependent on applied pressure	1 (minimum)
Hydrogen evolution electrode temperature sensor**	Minimum range: 15-100 °C	2
Oxygen evolution electrode temperature sensor**	Minimum range: 15-100 °C	2
Hydrogen evolution electrode flow meter	Range dependent on anticipated flow	1 (optional)
Oxygen evolution electrode flow meter	Range dependent on anticipated flow	1 (optional)
Hydrogen safety sensor for the oxygen evolution electrode outlet	Range suitable to detect hydrogen in wet oxygen-rich gas to prevent occurrence of an explosive atmosphere	1
Oxygen safety sensor for the hydrogen evolution electrode outlet	Range suitable to detect hydrogen in (wet) hydrogen-rich gas to prevent occurrence of an explosive atmosphere	1
Cell/stack temperature sensor***	Minimum range: 15-100 °C	1 (optional)

Description	Specification	QTY
Adjustable flow meter for inert gas (i.e. nitrogen)	Range of flow rates suitable to dilute the evolved hydrogen to prevent occurrence of an explosive atmosphere	1 (optional)
Gas liquid separator		2
Cell/stack heating (cooling) devices	Minimum temperature range: ambient to 100 °C	Minimum 1
Water/liquid circulation pump	Range dependent on applied current density (galvanostatic control) or cut-off voltage (potentiostatic control)	Minimum 1
Water treatment device	-	1
Water conductivity meter	Minimum range: 0.1-10 ⁻⁶ S.cm	1
DC power supply	Dependent on maximum reachable current density $\geq 2 \text{ A/cm}^2$ (galvanostatic control) Dependent on maximum cut-off voltage $\geq 2 \text{ V/cell}$ (potentiostatic control) (optionally)	1
Control and data-acquisition hardware including software (e.g. computer)	The capacity of the data-acquisition system has to be sufficient to record all test variables with the sample rates defined	1

* The location of the sensor, usually directly after the gas liquid separator, should be mentioned in the test report. An additional sensor may be placed directly at the cell/stack inlet.

** The location of the sensor, usually directly at the cell/stack (water/liquid) outlet, should be mentioned in the test report. An additional sensor should be placed directly at the cell/stack (water/liquid) inlet.

*** The location of this sensor should be mentioned in the test report. It should be chosen so as to best represent the temperature of the cell/stack, taking into account the control equipment used in the test and the internal configuration and dimension of the cell/stack; see the recommendation of T_c in Section 5.

Important

During the test the hydrogen concentration in the gas stream from the oxygen evolution electrode should be monitored by a hydrogen gas safety sensor to trigger appropriate safety measures in case a specified threshold is approached. A threshold value of 10 % of the lower explosive limit (LEL) of hydrogen in oxygen (about 0.8 Vol-% H₂ in O₂) is advised.

Likewise, the oxygen concentration in the gas stream from the hydrogen evolution electrode should be monitored by an oxygen gas safety sensor to



EU harmonised polarisation curve test method for low-temperature water electrolysis



trigger appropriate safety measures in case a specified threshold is approached. A threshold value of 10 % of the upper explosive limit (UEL) of oxygen in hydrogen (about 5 Vol-% O₂ in H₂) is advised.

5. Test input parameter and test output parameter

The **test input parameter** (TIP) is a physical quantity which sets the testing conditions. There are variable TIPs which are changed during testing and static TIPs which are kept constant during testing.

The **test output parameter** (TOP) is a physical quantity resulting from carrying out the tests.

Note that the control accuracy of the TIPs (see Table 4 & Table 5 below) and the measurement uncertainty of the TOPs (see Table 6 below) for stacks (with a large cell area) will depend respectively on the control equipment and the measurement equipment used in the test.

It is recommended that the cell/stack temperature, T_c (**set point**) be measured directly at the cell/stack water outlet, $T_{\text{water, out}}$ of the oxygen evolution electrode. For a cell/stack with liquid on both the oxygen evolution electrode and the hydrogen evolution electrode, it is recommended that the cell/stack temperature be measured directly at the cell/stack water/liquid outlet, $T_{\text{water/liquid, out}}$ of the hydrogen evolution electrode.

This implies that the regulation of the cell/stack temperature, which is monitored at the cell/stack outlet, is at the cell/stack inlet of the oxygen evolution electrode and/or hydrogen evolution electrode where the fluid flows and/or fluid temperatures are controlled.

Where the monitoring and regulation of the cell/stack temperature is different from this recommendation, this should be mentioned in the test report.

5.1. Test input parameter

The static TIPs applied during the test are given in Table 4.

Table 4. Static TIPs

TIP	Control accuracy	Sampling rate
p_{H_2} (gauge) (set point)	$\pm 2 \%$	≥ 1 Hz
p_{O_2} (gauge) (set point)	$\pm 2 \%$	≥ 1 Hz
$p_{water/liquid, in}^{**}$ (gauge) (hydrogen evolution electrode)	$\pm 2 \%$	≥ 1 Hz
$p_{water/liquid, in}$ (gauge) (oxygen evolution electrode)	$\pm 2 \%$	≥ 1 Hz
$p_{N_2, in}^{***}$ (gauge)	$\pm 2 \%$	≥ 1 Hz
$Q_{v, N_2, in}^{**}$ (hydrogen evolution electrode)**	$\pm 1 \%$ FS	≥ 1 Hz
$Q_{v, water/liquid, in}^{**}$ (hydrogen evolution electrode)****	$\pm 1 \%$ FS	≥ 1 Hz
$Q_{v, water/liquid, in}$ (oxygen evolution electrode)****	$\pm 1 \%$ FS	≥ 1 Hz
T_c^{*****} (set point)	± 2 K	≥ 1 Hz
$T_{water/liquid, out}^{**}$ (hydrogen evolution electrode)*****	± 2 K	≥ 1 Hz
$T_{water/liquid, out}$ (oxygen evolution electrode)*****	± 2 K	≥ 1 Hz

* Commonly, these inlet flows are kept constant during the entire test. Usually, the digital flow controllers are provided with an accuracy level of 1 % of full scale (FS) or maximum flow and, in general, a minimum measurable flow of 10 % of FS. This means that the measurement uncertainty decreases with the flow rate and so decreases while increasing the current density.

** Only relevant for PEMWE with intended presence of liquid water.

*** Only relevant when used for PEMWE with no intended presence of liquid water.

**** Usually, the water inlet flow is kept constant during the entire test. The flow rate should be selected to be high enough to replenish the water. Also, the flow rate can greatly influence the water temperature. When the flow rate is variable as a result of the regulation of the cell/stack temperature, this should be mentioned in the test report.

***** Only relevant for AEMWE and for PEMWE with intended presence of liquid water.

***** The sensor location and method of regulation of the cell/stack water/liquid temperature should be mentioned in the test report.

***** The regulation of the cell/stack (water/liquid) outlet temperature **set point** is assumed to take place by the variation of the cell/stack (water/liquid) inlet temperature. If the cell/stack (water/liquid) outlet temperature **set point** is regulated by the variation of the cell/stack

(water/liquid) outlet temperature, $T_{water/liquid, in}$ are static TIPS (**set points**) and $T_{water/liquid, out}$ are variable TIPS.

The variable TIPS applied during the test are given in Table 5.

Table 5. Variable TIPS

TIP	Control accuracy	Sampling rate
j (galvanostatic control)	$\pm 2 \%$ for $j < 0.1 \text{ A/cm}^2$ $\pm 1 \%$ for $j \geq 0.1 \text{ A/cm}^2$	$\geq 1 \text{ Hz}$
$P_{N2, in}^*$ (gauge)	$\pm 2 \%$	$\geq 1 \text{ Hz}$
$T_{N2, in}^*$ (hydrogen evolution electrode) ^{***}	$\pm 2 \text{ K}$	$\geq 1 \text{ Hz}$
$T_{water/liquid, in}^{**}$ (hydrogen evolution electrode) ^{***}	$\pm 2 \text{ K}$	$\geq 1 \text{ Hz}$
$T_{water/liquid, in}$ (oxygen evolution electrode) ^{***}	$\pm 2 \text{ K}$	$\geq 1 \text{ Hz}$
U (potentiostatic control)	$\pm 1 \text{ mV/cell}$	$\geq 1 \text{ Hz}$

* Only relevant when used for PEMWE with no intended presence of liquid water.

** Only relevant for PEMWE with intended presence of liquid water.

*** The temperature may vary during the test depending on the regulation of the cell/stack temperature. If the cell/stack (water/liquid) outlet temperature **set point** is regulated by the variation of the cell/stack (water/liquid) outlet temperature, $T_{water/liquid, in}$ are static TIPS (**set points**) and $T_{water/liquid, out}$ are variable TIPS.

5.2. Test output parameter

Table 6 below lists the TOPs.

Table 6. TOPs

TOP	Measurement uncertainty	Sampling rate
j (potentiostatic control)	Calculated*	$\geq 1 \text{ Hz}$
P_d^{**}	Calculated*	-
$p_{H2 \text{ or } H2/N2, out}$ (gauge)	Calculated*	$\geq 1 \text{ Hz}$
$p_{O2, out}$ (gauge)	Calculated*	$\geq 1 \text{ Hz}$

TOP	Measurement uncertainty	Sampling rate
$T_{H_2 \text{ or } H_2/N_2, \text{ out}}$ ***	Calculated*	$\geq 1 \text{ Hz}$
U (galvanostatic control)	Calculated*	$\geq 1 \text{ Hz}$

* see Appendices B and C for the calculations of the statics of the test results and the measurement errors of the quantity concerned.

** Calculated according to equation (5) when applying method B. For method A, the measurement uncertainty results from the uncertainty of the measurement equipment used in the test.

*** Only relevant for PEMWE with no intended presence of liquid water.

6. Test procedure

The main objective of the polarisation curve measurement is to determine the change in the cell/stack voltage (and consequently in supplied power or power density) generated by the variation in the supplied current under steady-state conditions, and in particular at a constant cell/stack temperature and a constant hydrogen (and/or oxygen) pressure.

The complete testing procedure is composed of the following steps:

- leak test (not part of this procedure);
- break-in (not part of this procedure) and cell/stack conditioning;
- polarisation curve test;
- break-out (not part of this procedure).

Upon break-in of the MEA in accordance with the manufacturer's instructions, it is recommended that the cell/stack be operated for a specified duration to ensure the MEA has/all MEA have reached a steady state.

It is recommended that the cell/stack current density be kept at 0.1 A/cm^2 for 5 minutes to ensure the catalysts are in the proper oxidation state. This is followed by setting the lowest current value (Table 7) in steps of 0.025 A/cm^2 per minute prior to the actual polarisation curve measurement. The variation in cell voltage at the lowest current should not be more than 1 % during 1 hour of hold in this condition.

The polarisation curve measurement is performed under galvanostatic control. The measurement should be aborted when the cell voltage is 2 V or above (cut-off voltage). Note that a higher cut-off voltage, for example 2.4 V, may be applied where EoL testing is aimed at.

At ambient pressure, it is recommended that a polarisation curve measurement from lowest to highest current density (ascending polarisation curve) be performed, followed by a polarisation curve measurement in reverse order (descending polarisation curve).

For operation of the cell/stack at above-ambient pressure, the cell/stack should be operated at the highest current density for the specified pressure until a stable voltage and pressure is established for a specified duration prior to measuring the performance from the highest to the lowest current density (descending part of the polarisation curve), followed by a measurement in reverse order (ascending part of the polarisation curve).

As the hydrogen concentration in the gas stream from the oxygen evolution electrode could increase significantly, especially under high hydrogen-to-oxygen differential pressure operation for low current densities, e.g. $0.3\text{-}0.5 \text{ A/cm}^2$, the hydrogen pressure as a precautionary measure may be reduced where the electrical load is lower than 20 % of the nominal load or where the current density is lower than 0.5 A/cm^2 .

The difference between the ascending polarisation curve and the descending polarisation curve (hysteresis) provides information on whether or not the cell/stack was in (thermal) equilibrium during the measurement. A small hysteresis implies that this was the case.

The static TIPs are to be maintained at their values/ranges during the measurement.

All TIPs and TOPs should be recorded versus the entire test duration with the specified sampling rate (see Table 4, Table 5 and Table 6).

The polarisation curve measurement is performed up to a maximum/minimum value (Table 7) either by:

- **method A** (linear current sweep): continuous increase/decrease of current density at a specified rate; or
- **method B** (stepwise steady-state current sweep): applying consecutive current density steps (Table 7).

For method A, the current is linearly varied, e.g. 0.080 A/cm² (PEMWE) and 0.016 A/cm² (AWE and AEMWE) per minute.

Note that the actual rate of the current density to be used in the test will depend on the equipment used.

Table 7. Polarisation curve set points for method B (galvanostatic control)

Set point k^*	Current density (A/cm ²)			Step duration (s)	
	PEMWE	Alkaline WE	AEMWE	Dwell time ^{**}	Data acquisition ^{***}
1	0.001	0.0002	0.0002	30	30
2	0.005	0.0010	0.0010	30	30
3	0.010	0.0020	0.0020	30	30
4	0.025	0.0050	0.0050	30	30
5	0.050	0.0100	0.0100	30	30
6	0.075	0.0150	0.0150	30	30
7	0.100	0.0200	0.0200	30	30
8	0.150	0.0300	0.0300	30	30
9	0.200	0.0400	0.0400	30	30
10	0.250	0.0500	0.0500	30	30

Set point k^*	Current density (A/cm ²)			Step duration (s)	
	PEMWE	Alkaline WE	AEMWE	Dwell time ^{**}	Data acquisition ^{***}
11	0.300	0.0600	0.0600	30	30
12	0.350	0.0700	0.0700	30	30
13	0.400	0.0800	0.0800	30	30
14	0.450	0.0900	0.0900	30	30
15	0.500	0.1000	0.1000	30	30
16	0.600	0.1200	0.1200	30	30
17	0.700	0.1400	0.1400	30	30
18	0.800	0.1600	0.1600	30	30
19	0.900	0.1800	0.1800	30	30
20	1.000	0.2000	0.2000	30	30
21	1.100	0.2200	0.2200	30	30
22	1.200	0.2400	0.2400	30	30
23	1.300	0.2600	0.2600	30	30
24	1.400	0.2800	0.2800	30	30
25	1.500	0.3000	0.3000	30	30
26	1.600	0.3200	0.3200	30	30
27	1.700	0.3400	0.3400	30	30
28	1.800	0.3600	0.3600	30	30
29	1.900	0.3800	0.3800	30	30
30****	2.000	0.4000	0.4000	30	30

* Depending on the actual equipment used in the test, the set points at low current densities may be skipped to start, for example at or above 0.1 A/cm².

** Generally, the dwell time should be chosen so that the cell voltage does not deviate by more than ± 5 mV over a duration of 30 s. For stacks this criterion may also be applied by dividing the stack voltage by the number of cells in the stack to check on the deviation of the resulting mean cell voltage. Particularly for stacks (with a large cell area), the dwell time should be chosen so as to allow thermal equilibrium to be obtained, for example by checking whether the cell/stack temperature and/or its gradient is within a specified range (depending on the measurement equipment used in the test) over a specified duration (depending on the control equipment used in

the test) following the application of a set point. Once thermal equilibrium is obtained in this way, the deviation in the cell voltage should be checked. The temperature range and duration of check on thermal equilibrium should be mentioned in the test report, along with any greater-than-specified deviation.

**** The period of data acquisition is preceded by the dwell time.*

***** Any possible increase beyond the given maximum current density should be made in steps consisting of the difference between this maximum value and its preceding value.*

When the polarisation curve measurement cannot be performed under galvanostatic control or when the cell/stack needs to be protected from high voltages, the measurement should be performed under potentiostatic control by varying the voltage of the cell/stack between two specified voltages (lowest cell voltage and highest cell voltage) either at a specified rate (**method A**) or by specified voltage steps (**method B**).

Under potentiostatic control, it is recommended that a cell voltage above the thermal neutral voltage be applied as the lowest cell voltage and 2 V or more as the highest cell voltage.

Upon performing the polarisation curve measurement, and depending on the objective of the test, a break-out procedure may be applied in accordance with the manufacturer's instructions. This procedure purges the hydrogen evolution electrode compartment (cathode) of the cell/stack with an inert gas to prevent cathode ageing and mixing of hydrogen and air during long exposures, and also to inhibit possible reactions in the presence of electrode catalyst.

7. Data processing and representation

For method A the TIPs (pressure, temperature and flow) are averaged.

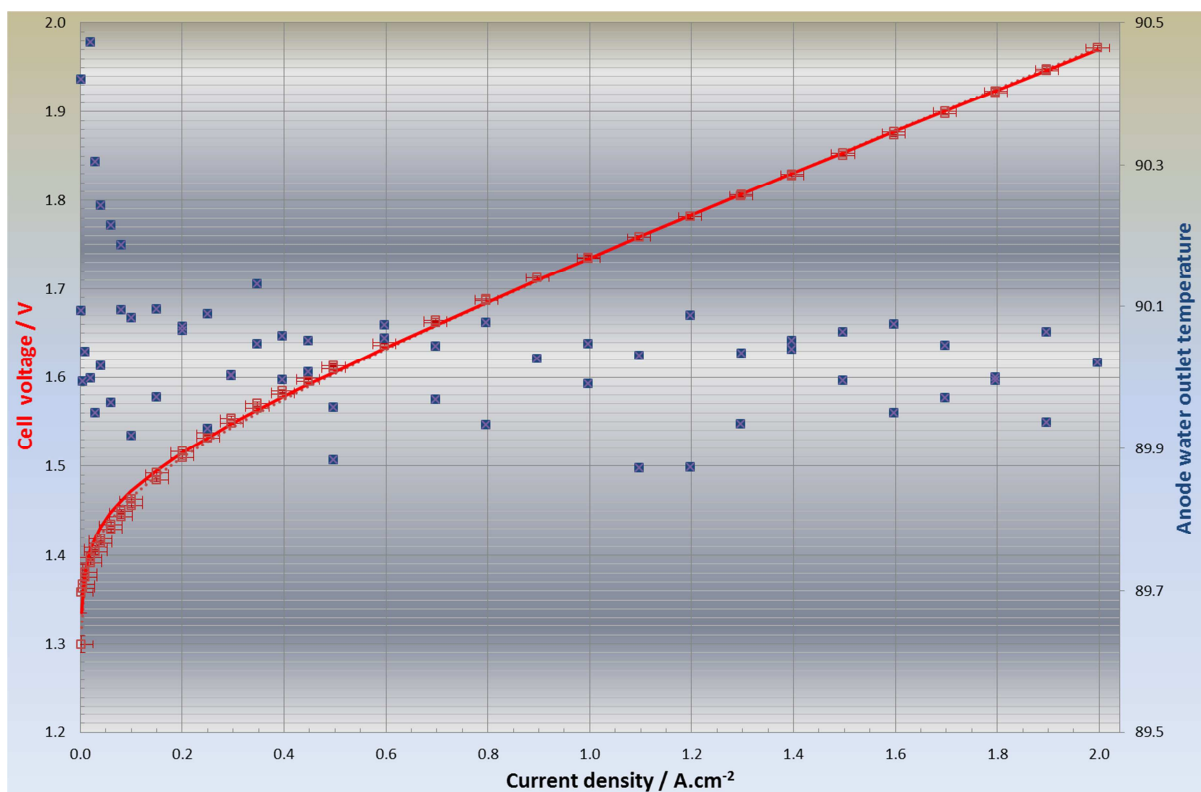
For method B the TIPs and TOPs are averaged for a sufficient number of data points, for example a minimum of 10 data points recorded during the period of data acquisition at each current density step.

Along with the average the standard deviation (STD) should be calculated according to GUM Guide [2] (see Appendix A).

For method A the instantaneous voltage of the cell/stack, U versus that of the current density, j is the polarisation curve in the ascending and descending parts for a given cell/stack temperature, T_c and pressure, p .

For method B the average of U versus j (see Appendix B for their calculation) is the polarisation in either part (Figure 1).

Figure 1. Example of the polarisation curve (ascending and descending) measurement of a WE single cell at 90 °C cell temperature



The displayed error bars in the graphs in Figure 1 constitute the measurement uncertainty (see Appendix C) of the cell voltage and current density expanded twofold (expansion factor $k = 2$ according to GUM [2]). The dotted line and the solid line are the non-linear least square (NLLS) fits with positivity constrains (for R_Ω , α^a , α^c , j_0^a & j_0^c) of equation (1) to the average of the measured values of respectively the ascending part ($R_\Omega = 26.8 \text{ m}\Omega$, $\alpha^a = 0.29$, $\alpha^c = 0.71$, $j_0^a =$

24.1 mA/cm² and $j_0^c = 24.6$ mA/cm²) and descending part of the polarisation curve ($R_\Omega = 26.8$ m Ω , $\alpha^a = 0.34$, $j_0^a = 15.6$ mA/cm² & $j_0^c = 19.7$ mA/cm²); see Appendix D for the tabulation of the averaged measurement data. In this example, the anode water inlet temperature constituting the cell temperature is kept constant in the test while the anode water outlet temperature is monitored (see Figure 1).

Further analysis of the polarisation curve for given operating conditions (i.e. temperature, pressure) may provide additional insight into MEA performance. Assuming the following:

- i. no mass transport limitations;
- ii. homogeneous current distribution over the active area (1D model);
- iii. neglecting the reverse term in the Butler-Volmer reaction corresponding to assuming a very low reaction rate for oxygen reduction;
- iv. provided that all the reversible heat, $Q_{rev}(T_c, p) = T_c \Delta S$ in the endothermic WE operation is supplied by an external source,

the cell/stack voltage, $U(j, T, p)$ at a given cell/stack temperature, T expressed in Kelvin (K) and pressure, p expressed in kilopascals (kPa) as a function of current density, j can be fit by the NLLS method [4,5] with positivity (non-negativity) constrain for the model parameter [3].

$$U(j, T_c, p) = U_{rev}(T_c, p) + IR_\Omega + \eta_{H_2}(j, T_c) + \eta_{O_2}(j, T_c) \quad (1)$$

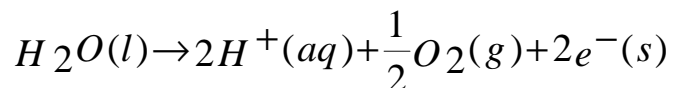
with the cell/stack temperature expressed in degrees Celsius (°C), the cell/stack current, $I = A \cdot j$ expressed in amperes (A), where A is the active area (geometric) of the cell/stack expressed in square centimetres (cm²) and the reversible cell voltage, $U_{rev}(T_c, p) = \frac{\Delta G(T_c, p)}{zF}$, e.g. $U_{rev}(25^\circ\text{C}, 1 \text{ atm}) \approx 1.23 \text{ V}$ [1,6] ⁽¹⁾,

$$U_{rev}(T_c, 1 \text{ atm}) = 1.5184 - 1.5421 \cdot 10^{-3} \cdot T_c + 9.523 \cdot 10^{-5} \cdot T_c \log_e T_c + 9.84 \cdot 10^{-8} \cdot T_c^2, \quad (2)$$

ΔS is the change in entropy ($\Delta S(25^\circ\text{C}, 1 \text{ atm}) = \Delta S^0 \approx 0.16$ kJ.mol⁻¹.K⁻¹ at 101.325 kPa absolute pressure), ΔG is the change in Gibbs free energy ($\Delta G(25^\circ\text{C}, 1 \text{ atm}) = \Delta G^0 \approx 237.21$ kJ.mol⁻¹ at 298.15 K absolute temperature and 101.325 kPa absolute pressure) [1,6], $F = 96\,485.3$ C/mol is Faraday's constant and $z = 2$ is the number of exchanged electrons in the liquid WE reaction:

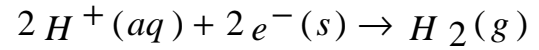
PEMWE cells

anode (oxygen electrode)



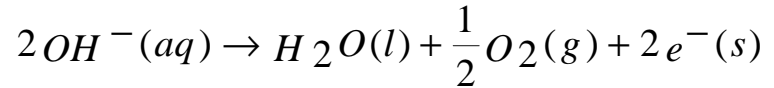
⁽¹⁾ Equation (2) applies up to 18 bar pressure.

cathode (hydrogen electrode)

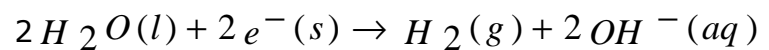


AWE cells & AEMWE cells

anode (oxygen electrode)



cathode (hydrogen electrode)



Note that $U_{rev}(T_c, 1 atm)$ in equation (1) is to be replaced by the thermal neutral voltage,

$$U_{tn}(T_c, 1 atm) = U_{tn}(T_c, 1 atm) + \frac{T_c \Delta S}{z F} \quad (3)$$

if heat is not supplied by an external source, for example $U_{tn}(25^{\circ}C, 1 atm) \approx 1.48 V$. For a partial heat supply, $U_{rev}(T_c, 1 atm)$ in equation (1) is to be replaced by the onset voltage,

$$U_{on}(T_c, 1 atm) = U_{rev}(T_c, 1 atm) + \frac{T_c \Delta S - Q_{in}}{z F} \quad (4)$$

where Q_{in} is the heat supplied by the external source.

The total ionic/electronic cell resistance (bulk and surface/interface), R_{Ω} is expressed in ohms (Ω).

$$\eta_{H_2}(j, T_c) = \frac{RT_c}{\alpha c n^c F} \ln \left(\frac{j}{j_0^c} \right) \quad (5)$$

and

$$\eta_{O_2}(j, T_c) = \frac{RT_c}{\alpha^a n^a F} \ln \left(\frac{j}{j_0^a} \right) \quad (6)$$

are the overvoltage expressed in volts (V) for the hydrogen evolution reaction (HER) at the cathode (c superscript) and the oxygen evolution reaction (OER) at the anode (a superscript), respectively, j_0 , is the exchange current density, $R = 8.31446 J.mol^{-1}.K^{-1}$ is the universal gas constant and n is the number of electrons involved in the rate determining step (rds) of the electrode evolution process; note, the transfer coefficient, $\alpha = -\frac{RT_c}{nF} \frac{d \ln k}{dU}$ is related to the symmetry of the activation (energy) barrier in the evolution process which proceed with reaction rate, k . Usually, α assumes a value of between zero and unity.

An output which can directly be derived from a polarisation curve measurement without any need for additional measurement is the power density,

$$P_d(j, T_c, p) = U(j, T_c, p) \cdot j \quad (7)$$

as the product of the measured cell voltage and the current density.

The calculation of the statistics of the TIPs and TOPs and of their measurement errors is outlined respectively in Appendix B and Appendix C.

Due to the nonlinearities in equation (5) and equation (6), the value and error in the fit parameter R_{Ω} , $\alpha^{a,c}$ and $j_0^{a,c}$ should be determined through the fitting of equation (1), or equation (3) or equation (4) as appropriate using the average and the error of U , j and T_c .

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Appendix A Statistical analysis of test results

From M sampled data under N sets of test conditions, the statistical values such as sample average (avg), sample variance (Var), sample covariance (Cov), sample standard deviation, relative standard deviation (RSD) and sample standard error values for test variable (TIP or TOP), X_k ($0 < k \leq M$) and $Y_k (X^1 \pm X^1_{stdev}, \dots, X^i \pm X^i_{stdev}, \dots, X^N \pm X^N_{stdev})$ ($0 < i \leq N$) are calculated according to GUM [2].

Also, the calculation of the measurement uncertainty, $u(X_k)$ considering the instrument uncertainty, u_x of the measurand X is analogous to GUM [2].

These calculations are commonly performed by a spreadsheet program, but due attention should be paid to the calculations involving combined quantities such as electric power density.

A.1. Statistics on test variables

Sample average or arithmetic mean (measure of central tendency):

$$X_{k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} X_{k,l} \quad (8)$$

Sample variance:

$$Var(X_k) = \frac{1}{M-1} \sum_{l=0}^{M-1} (X_{k,l} - X_{k,avg})^2 \quad (9)$$

Sample standard deviation (measure for absolute variability):

$$X_{k,std} = \sqrt{Var(X_k)} \quad (10)$$

Sample covariance:

$$Cov(X_k^i, X_k^j) = \frac{1}{M(M-1)} \sum_{l=0}^{M-1} (X_{k,l}^i - X_{k,avg}^i) \cdot (X_{k,l}^j - X_{k,avg}^j) \quad (11)$$

Combined standard deviation:

$$Y_{k,std} = \sqrt{\sum_{i=1}^N \left(\frac{\partial Y_k}{\partial X_k^i} \Big|_{X_{k,avg}^i} \right)^2 \cdot Var(X_k^i) + \frac{N-1}{2} \sum_{i=1}^N \sum_{j=i+1}^N \frac{\partial Y_k}{\partial X_k^i} \frac{\partial Y_k}{\partial X_k^j} \Big|_{X_{k,avg}^{i,j}} \cdot Cov(X_k^i, X_k^j)} \quad (12)$$

Sample RSD (measure of relative variability):

$$X_{k,rsd} = \frac{X_{k,std}}{X_{k,avg}} \cdot 100\% \quad (13)$$

Note that the RSD provides the mean to compare the variability of the measurand X at the different steps k of the polarisation curve for the same number of measurements M .

Sample standard error (measure for normalised variability):

$$X_{k,sterr} = \frac{X_{k,std}}{\sqrt{M}} \quad (14)$$

Note that the standard error provides the mean to compare the variability of the measurand X for different numbers of measurements M at step k of the polarisation curve.

The significant figures of a test variable to be reported should be consistent with its measurement uncertainty.

For a calculated test variable, the lesser number of significant figures of all of the involved test variables should determine the significant figure.

The standard deviation and standard error should be expressed with at least one more figure than the value of the test variable and the RSD should be reported with two significant digits.

A.2. Calculation of measurement uncertainties

Measurement uncertainty:

$$u(X_k) = \sqrt{Var(X_k) + u_X^2} \quad (15)$$

Combined measurement uncertainty:

$$u(Y_k) = \sqrt{\sum_{i=1}^N \left(\frac{\partial Y_k}{\partial X_k^i} \Big|_{X_{k,avg}^i} \right)^2 \left[\text{Var}(X_k^i) + u_{X^i}^2 \right] + 2 \sum_{i=1}^{N-1} \sum_{j=i+1}^N \frac{\partial Y_k}{\partial X_k^i} \frac{\partial Y_k}{\partial X_k^j} \Big|_{X_{k,avg}^{i,j}} \left[\text{Cov}(X_k^i, X_k^j) + u_{X^i} u_{X^j} \right]} \quad (16)$$

Relative measurement uncertainty (RMU):

$$u_{rmu}(X_k) = \frac{u(X_k)}{|X_{k,avg}|} \cdot 100\% \quad (17)$$

Note that the RMU provides the mean to compare the variability of the measurand X at the different steps k of the polarisation curve for the same number of measurements M .

Measurement error:

$$u_{err}(X_k) = \frac{X_{k,std}}{\sqrt{M}} \quad (18)$$

Note that the measurement error provides the mean to compare the variability of the measurand X for different numbers of measurements M at step k of the polarisation curve.

The significant figures of a test variable to be reported should be consistent with its measurement uncertainty.

For a calculated test variable, the lesser number of significant figures of all of the involved test variables should determine the significant figure.

The standard deviation and standard error should be expressed with at least one more figure than the value of the test variable and the RSD should be reported with two significant digits.

Appendix B Measurement statistics of TIPs and TOPs

B.1. Current density statistics

The average of the current density of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve (see Table 7) is:

$$j_{k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} j_{k,l} \quad (19)$$

with standard deviation:

$$j_{k,std} = \sqrt{\text{Var}(j_k)} \quad (20)$$

and variance:

$$\text{Var}(j_k) = \frac{1}{M-1} \sum_{l=0}^{M-1} (j_{k,l} - j_{k,avg})^2 \quad (21)$$

The RSD is:

$$j_{k,rstd} = \frac{j_{k,std}}{|j_{k,avg}|} \cdot 100\% \quad (22)$$

and the standard error is:

$$j_{k,sterr} = \frac{j_{k,std}}{\sqrt{M}} \quad (23)$$

B.2. Voltage statistics

The average of the cell/stack voltage from the M measurements during the acquisition period at step k of the polarisation curve is:

$$U_{k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} U_{k,l} \quad (24)$$

with standard deviation:

$$U_{k,std} = \sqrt{\text{Var}(U_k)} \quad (25)$$

and variance:

$$\text{Var}(U_k) = \frac{1}{M-1} \sum_{l=0}^{M-1} (U_{k,l} - U_{k,avg})^2 \quad (26)$$

The RSD is:

$$U_{k,rsd} = \frac{U_{k,std}}{|U_{k,avg}|} \cdot 100\% \quad (27)$$

and the standard error is:

$$U_{k,sterr} = \frac{U_{k,std}}{\sqrt{M}} \quad (28)$$

B.3. Electric power density statistics

The average electric power density of the cell/stack at step k of the polarisation curve is:

$$P_{d,k,avg} = U_{k,avg} \cdot j_{k,avg} \quad (29)$$

with standard deviation:

$$P_{d,k,std} = P_{d,k,avg} \sqrt{\frac{\text{Var}(j_k)}{j_{k,avg}^2} + \frac{\text{Var}(U_k)}{U_{k,avg}^2} + 2 \frac{\text{Cov}(j_k, U_k)}{P_{d,k,avg}}} \quad (30)$$

and covariance:

$$\text{Cov}(j_k, U_k) = \frac{1}{M(M-1)} \sum_{l=0}^{M-1} (j_{k,l} - j_{k,avg}) \cdot (U_{k,l} - U_{k,avg}) \quad (31)$$

Note, power density has a combined standard deviation as voltage and current are correlated to each other in the WE reaction.

The RSD is:

$$P_{d,k,rsd} = \frac{P_{d,k,std}}{|P_{d,k,avg}|} \cdot 100\% \quad (32)$$

and the standard error is:

$$P_{d,k,sterr} = \frac{P_{d,k,std}}{\sqrt{M}} \quad (33)$$

B.4. Temperature statistics

The average water inlet temperature of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$T_{water,in,k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} T_{water,in,k,l} \quad (34)$$

with standard deviation:

$$T_{water,in,k,stdev} = \sqrt{\text{Var}(T_{water,in,k})} \quad (35)$$

and variance:

$$\text{Var}(T_{water,in,k}) = \frac{1}{M-1} \sum_{l=0}^{M-1} (T_{water,in,k,l} - T_{water,in,k,avg})^2 \quad (36)$$

The average water outlet temperature of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$T_{water,out,k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} T_{water,out,k,l} \quad (37)$$

with standard deviation:

$$T_{water,out,k,stdev} = \sqrt{\text{Var}(T_{water,out,k})} \quad (38)$$

and variance:

$$\text{Var}(T_{water,out,k}) = \frac{1}{M-1} \sum_{l=0}^{M-1} (T_{water,out,k,l} - T_{water,out,k,avg})^2 \quad (39)$$

The temperature of the cell/stack at the oxygen evolution electrode is:

$$T_c = T_{water,in} + \frac{T_{water,out} - T_{water}}{2} \quad (40)$$

The average temperature of the cell/stack at step k of the polarisation curve is:

$$T_{c,k,avg} = T_{water,in,k,avg} + \frac{T_{water,out,k,avg} - T_{water,in,k,avg}}{2} \quad (41)$$

with standard deviation:

$$T_{c,k,std} = \frac{\sqrt{(\text{Var}(T_{water,in,k}) + \text{Var}(T_{water,out,k}))}}{2} \quad (42)$$

The RSD is:

$$T_{c,k,rsd} = \frac{T_{c,k,std}}{|T_{c,k,avg}|} \cdot 100\% \quad (43)$$

and the standard error is:

$$T_{c,k,sterr} = \frac{T_{c,k,std}}{\sqrt{M}} \quad (44)$$

The average hydrogen outlet temperature of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$T_{H2,out,k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} T_{H2,out,k,l} \quad (45)$$

with standard deviation:

$$T_{H2,out,k,std} = \sqrt{\text{Var}(T_{H2,out,k})} \quad (46)$$

and variance:

$$\text{Var}(T_{H2,out,k}) = \frac{1}{M-1} \sum_{l=0}^{M-1} (T_{H2,out,k,l} - T_{H2,out,k,avg})^2 \quad (47)$$

The RSD is:

$$T_{H2,out,k,rsd} = \frac{T_{H2,out,k,std}}{|T_{H2,out,k,avg}|} \cdot 100\% \quad (48)$$

and the standard error is:

$$T_{H2,out,k,sterr} = \frac{T_{H2,out,k,std}}{\sqrt{M}} \quad (49)$$

B.5. Pressure statistics

The average water inlet pressure of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$P_{water,in,k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} P_{water,in,k,l} \quad (50)$$

with standard deviation:

$$P_{water,in,k,std} = \sqrt{\text{Var}(P_{water,in,k})} \quad (51)$$

and variance:

$$\text{Var}(P_{water,in,k}) = \frac{1}{M-1} \sum_{l=0}^{M-1} (P_{water,in,k,l} - P_{water,in,k,avg})^2 \quad (52)$$

The RSD is:

$$P_{water,in,k,rsd} = \frac{P_{water,in,k,std}}{P_{water,in,k,avg}} \cdot 100\% \quad (53)$$

and the standard error is:

$$P_{water,in,k,sterr} = \frac{P_{water,in,k,std}}{\sqrt{M}} \quad (54)$$

The average hydrogen outlet pressure of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$P_{H2,out,k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} P_{H2,out,k,l} \quad (55)$$

with standard deviation:

$$P_{H2,out,k,std} = \sqrt{\text{Var}(P_{H2,out,k})} \quad (56)$$

and variance:

$$\text{Var}(P_{H2,out,k}) = \frac{1}{M-1} \sum_{l=0}^{M-1} (P_{H2,out,k,l} - P_{H2,out,k,avg})^2 \quad (57)$$

The RSD is:

$$P_{H2,out,k,rsd} = \frac{P_{H2,out,k,std}}{P_{H2,out,k,avg}} \cdot 100\% \quad (58)$$

and the standard error is:

$$P_{H2,out,k,sterr} = \frac{P_{H2,out,k,std}}{\sqrt{M}} \quad (59)$$

The average oxygen outlet pressure of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$P_{O2,out,k,avg} = \frac{1}{M} \sum_{l=0}^{M-1} P_{O2,out,k,l} \quad (60)$$

with standard deviation:

$$P_{O2,out,k,std} = \sqrt{\text{Var}(P_{O2,out,k})} \quad (61)$$

and variance:

$$\text{Var}(P_{O2,out,k}) = \frac{1}{M-1} \sum_{l=0}^{M-1} (P_{O2,out,k,l} - P_{O2,out,k,avg})^2 \quad (62)$$

The RSD is:

$$P_{O2,out,k,rsd} = \frac{P_{O2,out,k,std}}{P_{O2,out,k,avg}} \cdot 100\% \quad (63)$$

and the standard error is:

$$P_{O2,out,k,sterr} = \frac{P_{O2,out,k,std}}{\sqrt{M}} \quad (64)$$

Appendix C Measurement uncertainties of TIPs and TOPs

C.1. Current density uncertainty

The measurement uncertainty of the current density of the cell/stack from the M measurements during the acquisition period at step k of the polarisation curve is:

$$u(j_k) = \sqrt{\text{Var}(j_k) + u_j^2} \quad (65)$$

The RMU is:

$$u_{rmu}(j_k) = \frac{u(j_k)}{|j_{k,avg}|} \cdot 100\% \quad (66)$$

and the measurement error is:

$$u_{err}(j_k) = \frac{u(j_k)}{\sqrt{M}} \quad (67)$$

C.2. Voltage uncertainty

The measurement uncertainty of the cell/stack voltage from the M measurements during the acquisition period at step k of the polarisation curve is:

$$u(U_k) = \sqrt{\text{Var}(U_k) + u_U^2} \quad (68)$$

The RMU is:

$$u_{rmu}(U_k) = \frac{u(U_k)}{|U_{k,avg}|} \cdot 100\% \quad (69)$$

and the measurement error is:

$$u_{err}(U_k) = \frac{u(U_k)}{\sqrt{M}} \quad (70)$$

C.3. Electric power density uncertainty

The measurement uncertainty of the electric power density of the cell/stack voltage from the M measurements during the acquisition period at step k of the polarisation curve is:

$$u(P_{d,k}) = P_{d,k,avg} \sqrt{\frac{\text{Var}(j_k) + u^2(j_k)}{j_{k,avg}^2} + \frac{\text{Var}(U_k) + u^2(U_k)}{U_{k,avg}^2} + 2 \frac{\text{Cov}(j_k, U_k)}{P_{d,k,avg}}}. \quad (71)$$

The RMU is:

$$u_{rmu}(P_{d,k}) = \frac{u(P_{d,k})}{|P_{d,k,avg}|} \cdot 100\% \quad (72)$$

and the measurement error is:

$$u_{err}(P_{d,k}) = \frac{u(P_{d,k})}{\sqrt{M}}. \quad (73)$$

C.4. Cell/stack temperature uncertainty

The measurement uncertainty of the temperature of the cell/stack voltage from the M measurements during the acquisition period at step k of the polarisation curve is:

$$u(T_{c,k}) = \sqrt{\frac{\text{Var}(T_{water,in,k}) + \text{Var}(T_{water,out,k}) + 2u^2(T)}{2}}. \quad (74)$$

The RMU is:

$$u_{rmu}(T_{c,k}) = \frac{u(T_{c,k})}{|T_{c,k,avg}|} \cdot 100\% \quad (75)$$

and the measurement error is:

$$u_{err}(T_{c,k}) = \frac{u(T_{c,k})}{\sqrt{M}}. \quad (76)$$

C.5. Pressure uncertainty

The measurement uncertainty of the hydrogen pressure of the cell/stack voltage from the M measurements during the acquisition period at step k of the polarisation curve is:

$$u(p_{H2,k}) = \sqrt{\text{Var}(p_{H2,out,k}) + u^2(p)} \quad (77)$$

The RMU is:

$$u_{rmu}(p_{H2,k}) = \frac{u(p_{H2,k})}{|p_{H2,k}|} \cdot 100\% \quad (78)$$

and the measurement error is:

$$u_{err}(p_{H2,k}) = \frac{u(p_{H2,k})}{\sqrt{M}} \quad (79)$$

The measurement uncertainty of the oxygen pressure of the cell/stack voltage from the M measurements during the acquisition period at step k of the polarisation curve is:

$$u(p_{O2,k}) = \sqrt{\text{Var}(p_{O2,out,k}) + u^2(p)} \quad (80)$$

The RMU is:

$$u_{rmu}(p_{O2,k}) = \frac{u(p_{O2,k})}{|p_{O2,k}|} \cdot 100\% \quad (81)$$

and the measurement error is:

$$u_{err}(p_{O2,k}) = \frac{u(p_{O2,k})}{\sqrt{M}} \quad (82)$$

Appendix D Example of measurement data of Figure 1

Table 8 provides the average data of measurement of the ascending part and the descending part of the polarisation curve of the example given in Figure 1.

Table 8. Example of polarisation curve measurement data

Ascending		Descending	
Current density (A/cm ²)	Cell voltage, (V)	Current density (A/cm ²)	Cell voltage, (V)
0.002196	1.320659	1.997028	1.971923
0.005037	1.36234	1.897006	1.945978
0.010062	1.375311	1.797147	1.921179
0.020117	1.392194	1.696957	1.897333
0.030138	1.403544	1.597124	1.873583
0.040142	1.412891	1.497197	1.851072
0.060187	1.429582	1.397212	1.827036
0.080224	1.444366	1.297258	1.804812
0.100238	1.456766	1.197299	1.781062
0.150326	1.485284	1.097249	1.759601
0.200385	1.509416	0.997313	1.735278
0.250495	1.531258	0.897482	1.712101
0.297237	1.548522	0.797049	1.689114
0.347374	1.565118	0.697107	1.664982
0.397259	1.580856	0.597099	1.640373
0.447324	1.596021	0.497116	1.612904
0.497136	1.60947	0.447302	1.599169
0.597415	1.636368	0.397207	1.584576
0.697348	1.660976	0.347321	1.570555
0.79707	1.686156	0.297202	1.553672
0.99733	1.734229	0.250492	1.537553
1.097498	1.758361	0.200378	1.516188

Ascending		Descending	
Current density (A/cm ²)	Cell voltage, (V)	Current density (A/cm ²)	Cell voltage, (V)
1.197311	1.782015	0.150317	1.492438
1.297265	1.806052	0.100234	1.463251
1.397199	1.829134	0.080205	1.449898
1.497192	1.853552	0.060172	1.435019
1.597162	1.875967	0.040124	1.418232
1.696959	1.899622	0.030118	1.40898
1.79709	1.924231	0.020112	1.397249
1.896997	1.948363	0.01006	1.380557
1.997028	1.971923	0.005034	1.368348

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