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Fuel cell technologies

Part 7-1: Test methods — Single cell performance tests for polymer electrolyte fuel cells (PEFC)

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Fuel cell technologies –
Part 7-1: Test methods – Single cell performance tests for polymer electrolyte fuel cells (PEFC)

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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FUEL CELL TECHNOLOGIES –

Part 7-1: Test methods – Single cell performance tests for polymer electrolyte fuel cells (PEFC)

FOREWORD

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- the subject is still under technical development or where, for any other reason, there is the future but no immediate possibility of an agreement on an International Standard.

Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62282-7-1, which is a Technical Specification, has been prepared by IEC technical committee 105: Fuel cell technologies.

This second edition cancels and replaces the first edition published in 2010. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) addition of new tests, mainly regarding transportation applications; and,
b) restructuring of the format: basic and applied performance test methods.

The text of this Technical Specification is based on the following documents:

Enquiry draft	Report on voting
105/568/DTS	105/621/RVC

Full information on the voting for the approval of this technical specification can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts of the IEC 62282 series, published under the general title: *Fuel cell technologies*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- transformed into an International standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

This part of IEC 62282 describes standard single-cell test methods for polymer electrolyte fuel cells (PEFCs). This document provides consistent and repeatable methods to test the performance of single cells. This document should be used by component manufacturers or stack manufacturers who assemble components in order to evaluate the performance of cell components, including membrane-electrode assemblies (MEAs) and flow plates. This document is also available for fuel suppliers to determine the maximum allowable impurities in fuels.

Users of this document can selectively execute test items suitable for their purposes from those described in this document. This document is not intended to exclude any other methods.

FUEL CELL TECHNOLOGIES –

Part 7-1: Test methods – Single cell performance tests for polymer electrolyte fuel cells (PEFC)

1 Scope

This document covers cell assemblies, test station setup, measuring instruments and measuring methods, performance test methods, and test reports for PEFC single cells.

This document is used for evaluating:

a) the performance of membrane electrode assemblies (MEAs) for PEFCs in a single cell configuration;
b) materials or structures of PEFCs in a single cell configuration; or,
c) the influence of impurities in fuel and/or in air on the fuel cell performance.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14687-2, *Hydrogen fuel – Product specification – Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
anode
electrode (3.8) at which the oxidation of fuel (3.11) takes place

3.2
catalyst
substance that accelerates (increases the rate of) a reaction without being consumed itself

Note 1 to entry: The catalyst lowers the activation energy of the reaction, allowing for an increase in the reaction rate.

3.3
catalyst-coated membrane
CCM
<in a PEFC (3.24)> membrane whose surfaces are coated with a catalyst layer (3.4) to form the reaction zone of the electrode (3.8)

Note 1 to entry: See also membrane electrode assembly (MEA) (3.19).

3.4
catalyst layer
porous region adjacent to either side of the membrane containing the catalyst (3.2), typically with ionic and electronic conductivity

Note 1 to entry: The catalyst layer comprises the spatial region where the electrochemical reactions may take place.

3.5
cathode
electrode (3.8) at which oxidant (3.22) reduction takes place

3.6
clamping plate
pressure plate
frame used to compress the cell components together to maintain electrical conductivity and sealing

3.7
current collector
conductive material in a fuel cell (3.12) that collects electrons from the anode (3.1) side or conducts electrons to the cathode (3.5) side

3.8
electrode
electronic conductor (or semi-conductor) through which an electric current enters or leaves the electrochemical cell as the result of an electrochemical reaction

Note 1 to entry: An electrode may be either an anode (3.1) or a cathode (3.5).

[SOURCE: IEC TS 62282-1:2013, 3.33]

3.9
electrolyte
liquid or solid substance containing mobile ions that render it ionically conductive

Note 1 to entry: The electrolyte is the main distinctive feature of the different fuel cell technologies (e.g. a liquid, polymer, molten salt, solid oxide) and determines the usable operating temperature range.

[SOURCE: IEC 60050-482:2004, 482-02-29, modified — the note has been modified]

3.10
flow plate
conductive plate made of metal, a material such as graphite, or a conductive polymer that may be a carbon-filled composite, which is incorporated with flow channels for fuel (3.11) or an oxidant (3.22) gas feed and has an electrical contact with an electrode (3.8)

3.11
fuel
hydrogen or hydrogen-containing gas that reacts at the anode (3.1)

3.12
fuel cell
electrochemical device that converts the chemical energy of a fuel (3.11) and an oxidant (3.22) to electrical energy (DC power), heat and reaction products

Note 1 to entry: The fuel and oxidant are typically stored outside of the fuel cell and transferred into the fuel cell as they are consumed.

[SOURCE: IEC/TS 62282-1:2013, 3.43]

3.13
gas diffusion electrode
GDE
component on the anode (3.1) or cathode (3.5) side comprising all electronic conductive elements of the electrode (3.8), i.e. gas diffusion layer (3.14) and catalyst layer (3.4)

3.14
gas diffusion layer
GDL
porous substrate placed between the catalyst layer (3.4) and the flow plate (3.10) to serve as electric contact and allow the access of reactants to the catalyst layer and the removal of reaction products

Note 1 to entry: The gas diffusion layer is also called a porous transport layer (PTL).

[SOURCE: IEC TS 62282-1:2013, 3.57, modified — "flow plate" replaces "bipolar plate" and note modified.]

3.15
gasket
sealing component which prevents the reactant gas from leaking out of a cell

3.16
internal resistance
ohmic resistance inside a fuel cell (3.12), measured between current collectors (3.7), caused by the electronic and ionic resistances of the different components (electrodes (3.8), electrolyte (3.9), flow plates (3.10) and current collectors)

Note 1 to entry: The term ohmic refers to the fact that the relation between voltage drop and current is linear and obeys Ohm's Law.

[SOURCE: IEC TS 62282-1:2013, 3.66, modified — "flow plates" replaces "bipolar plates"]

3.17
limiting current density
maximum current density that can be attained by the cell under a given set of test conditions where the cell voltage sharply decreases to near zero

3.18
maximum current density
highest current density allowed for a short time as specified by the manufacturer

3.19
membrane electrode assembly
MEA
component of a fuel cell (3.12), usually PEFC (3.24), consisting of an electrolyte membrane with gas diffusion electrodes (3.13) on either side

[SOURCE: IEC TS 62282-1:2013, 3.73, modified — "DMFC" deleted]

3.20
minimum cell voltage
lowest permitted cell voltage specified by the manufacturer

3.21
open circuit voltage
OCV
voltage across the terminals of a fuel cell (3.12) with fuel (3.11) and an oxidant (3.22) present and in the absence of external current flow

Note 1 to entry: The open circuit voltage is expressed in V.

Note 2 to entry: Also known as "no-load voltage".

[SOURCE: IEC TS 62282-1:2013, 3.117.2]

3.22
oxidant
oxygen or oxygen-containing gas (e.g. air) that reacts at the cathode (3.5)

3.23
polymer electrolyte
polymer material containing mobile ions that render it ionically conductive

3.24
polymer electrolyte fuel cell
PEFC
fuel cell (3.12) that employs a polymer with ionic exchange capability as the electrolyte (3.9)

Note 1 to entry: The polymer electrolyte fuel cell is also called a proton exchange membrane fuel cell (PEMFC) and solid polymer fuel cell (SPFC).

[SOURCE: IEC TS 62282-1:2013, 3.43.7]

3.25
power
energy per unit time, calculated from the voltage multiplied by the current

3.26
power density
measure calculated by dividing the power by the geometric electrode area

Note 1 to entry: Power density is expressed in W/cm^2.

3.27
rated current density
maximum current density specified by the manufacturer of the MEA (3.19) or single cell (3.29) for continuous operation

3.28
rated voltage
minimum cell voltage specified by the manufacturer of the MEA (3.19) or single cell (3.29) for continuous operation

3.29
single cell
cell typically consisting of an anode flow plate (3.10), MEA (3.19), cathode flow plate (3.10) and sealing gaskets (3.15)

Note 1 to entry: See Annex B for additional information.

3.30
single cell test
test of the fuel cell (3.12) performance based on a single cell (3.29)

[SOURCE: IEC TS 62282-1:2013, 3.112.5]

3.31
stoichiometry
molar ratio of the fuel (3.11) or oxidant (3.22) gas flow rate supplied to the cell to that required by the chemical reaction, as calculated from the current

Note 1 to entry: This is the inverse value of fuel (or oxidant) utilization as defined in IEC TS 62282-1:2013.

4 General safety considerations

An operating fuel cell uses oxidizing and reducing gases. Typically, these gases are stored in high-pressure containers. The fuel cell itself may or may not be operated at pressures greater than atmospheric pressure.

Those who carry out single cell testing should be trained and experienced in the operation of single cell test systems and specifically in safety procedures involving electrical equipment and reactive, compressed gases. Safely operating a single cell test station requires appropriate technical training and experience as well as safe facilities and equipment, all of which are outside the scope of this document.

5 Cell components

5.1 General

The following components are typically used:

a) an MEA,

b) gaskets,

c) an anode-side flow plate and a cathode-side flow plate,

d) an anode-side current collector and a cathode-side current collector,

e) an anode-side clamping plate and a cathode-side clamping plate,

f) electrically insulating sheets,

g) clamping or axial load hardware which may include bolts, washers, springs, etc., and,

h) temperature control devices.

5.2 Membrane electrode assembly (MEA)

The electrode area shall be as large as needed to measure desired parameters. A suggested electrode size should be approximately 25 cm^2, though cells having larger electrodes may give more relevant data for practical applications. The active electrode area shall be recorded. The approximate uncertainty in the area measurement shall also be recorded.

NOTE For a larger active area, heterogeneities in parameters such as temperature, flow rate, and/or compression can become significant.

5.3 Gasket

The gasket material shall be compatible with fuel cell reactants, components, reaction products and cell operating temperature. It shall minimize gas leakage.

5.4 Flow plate

Flow plates shall be made of materials that have negligible gas permeability and high electrical conductivity. Resin-impregnated, high-density, synthetic graphite, polymer/carbon composites, or corrosion-resistant metals, such as titanium or stainless steel, are recommended. If metal is used, the plate surface may be coated/plated (e.g. with gold) in order to reduce contact resistance. The flow plate should be corrosion-resistant and provide a suitable seal.

A serpentine flow channel is suggested. Further information about a suggested design is given in Annex A. The flow field configuration shall be documented in the test report.

The flow plates for testing shall allow the accurate measurement of cell operating temperature. For example, flow plates may have a small hole on an edgewise face in order to accommodate a temperature sensor. In this case, the hole shall reach the centre of the flow plate.

If the objective of testing is to evaluate the design of a particular flow channel, it is not necessary to use the suggested flow plate design.

5.5 Current collector

Current collectors shall be made of materials that have high electrical conductivity, such as metal. Metal collectors may be plated with contact resistance-reducing materials, such as gold or silver. However, care should be taken in choosing the coating material. It shall be compatible with the cell components and reactants and products.

Current collectors shall be thick enough to minimize voltage drop over their surface area. They should provide an output terminal for wire connection.

If metal flow plates act as current collectors, independent current collectors are not required.

5.6 Clamping plate (or pressure plate)

Clamping plates (or pressure plates) shall be flat and smooth-surfaced, with their mechanical properties strong enough to withstand the bending force being applied when clamped with bolts.

If the clamping plates are conductive, they shall be insulated from the current collectors in order to prevent short-circuiting.

5.7 Clamping hardware

Clamping hardware shall have high mechanical strength in order to withstand the stresses generated during installation and operation. Washers and springs may be used to maintain constant, uniform pressure on the single cell. A calibrated torque wrench or other measuring device shall be used to set exact pressure on the cell.

The clamping pressure should be tested and noted before and after each experiment. The spatial variation should not be higher than 10 %. If possible, the clamping pressure (magnitude, distribution) should be controlled continuously.

It is recommended to electrically insulate the clamping hardware.

5.8 Temperature-control device

The single cell shall be provided with a temperature-control device (for heating/cooling) in order to maintain it at a targeted temperature or temperature profile along the flow plate and across the cell. The temperature-control device may be programmable to follow a fixed temperature profile. The temperature-control device shall have a means to prevent over-temperature.

There are multiple ways of achieving this requirement.

One simple way is to convectively cool and/or electrically heat the clamping (pressure) plates. The heating can be achieved by attaching a skin resistance heater to the external surface of the plate. An alternative method is to insert a cartridge heater into a hole in the plate.

In either case, care is required to maintain insulation for electrical safety.

6 Cell assembly

6.1 Assembly procedure

Cell assembly procedures have a large impact on the repeatability of fuel cell data. Specific procedures shall be documented for the following assembly operations:

a) CCM alignment, including identification of anode and cathode sides,

b) diffusion media (i.e., GDL) alignment, including identification of anode and cathode parts, as well as the sides to be placed facing the membrane and flow field,

c) gasket/seal placement,

d) alignment fixtures or jigs to be used, if any,

e) compression procedures and specifications, such as diffusion media compression values, bolt tightening order, compression springs, and final torque specifications.

Pressure may be checked by pressure-sensitive paper/film.

Typical alignment of cell components is shown in Annex B.

After assembly, the isolation between the clamping plates and current collectors shall be checked.

6.2 Cell orientation and gas connections

A cell shall be operated in an orientation which facilitates product water removal. The cell orientation shall be documented.

6.3 Leak check

The cell shall have minimal external and internal leakage. Examples of leak-check procedures are given in Annex C. In principle, the leak-check procedures consist of injecting an inert or test gas into both the anode and cathode sides. By using a suitable pressure difference, the nature and direction of the leak can be ascertained. The maximum pressures, the nature of the test gas and leakage rates shall be documented. If a leak is detected, other tests, such as a bubble test, may be performed to further delineate the type and nature of the leak.

The differential pressure on the membrane is critical. The maximum differential pressure specified by the manufacturer should not be exceeded.

7 Test station setup

7.1 Minimum equipment requirement

A fuel cell test station is required to conduct the testing of a single cell. The minimum test equipment functionality in order to meet the intention of the single cell test procedure includes the following test parameters:

a) reactant gas flow rate control to meter and control the flow rates of fuel and oxidant gases to the fuel cell at a desired stoichiometric ratio;

b) reactant gas humidification control to humidify the reactant gases to a specified dew point prior to delivery to the fuel cell. The recommended water resistivity is at least 0,1 $M\Omega \cdot cm$ (or at most a conductivity of 10^{-6} $S \cdot cm^{-1}$). The gas transfer lines between the humidifiers and the cell should be heated to at least 5 °C above the dew point temperature to minimize condensation. The lines should be insulated to minimize heat loss. For some MEAs, humidification may not be required;

c) reactant gas pressure control to regulate the reactant gas pressure within the fuel cell;

d) load control: load bank to draw a specified current from the cell. It should be capable of operating in either constant current or constant voltage mode;

e) cell heating/cooling control to heat or cool the single cell to the desired operating temperature;

f) cell voltage monitoring and data acquisition instrumentation to measure and record the cell voltage throughout the test;

g) test station control: test station capable of controlling the above parameters;

h) automatic shutdown safety features are recommended when testing large active area cells. They are to allow the testing system to recognize undesirable operating conditions and respond by shutting down to prevent any damage to the fuel cell and/or stop any potentially unsafe event.

7.2 Schematic diagram

Figure 1 is a schematic block representation of the major sub-systems required in a test station to conduct fuel cell testing.

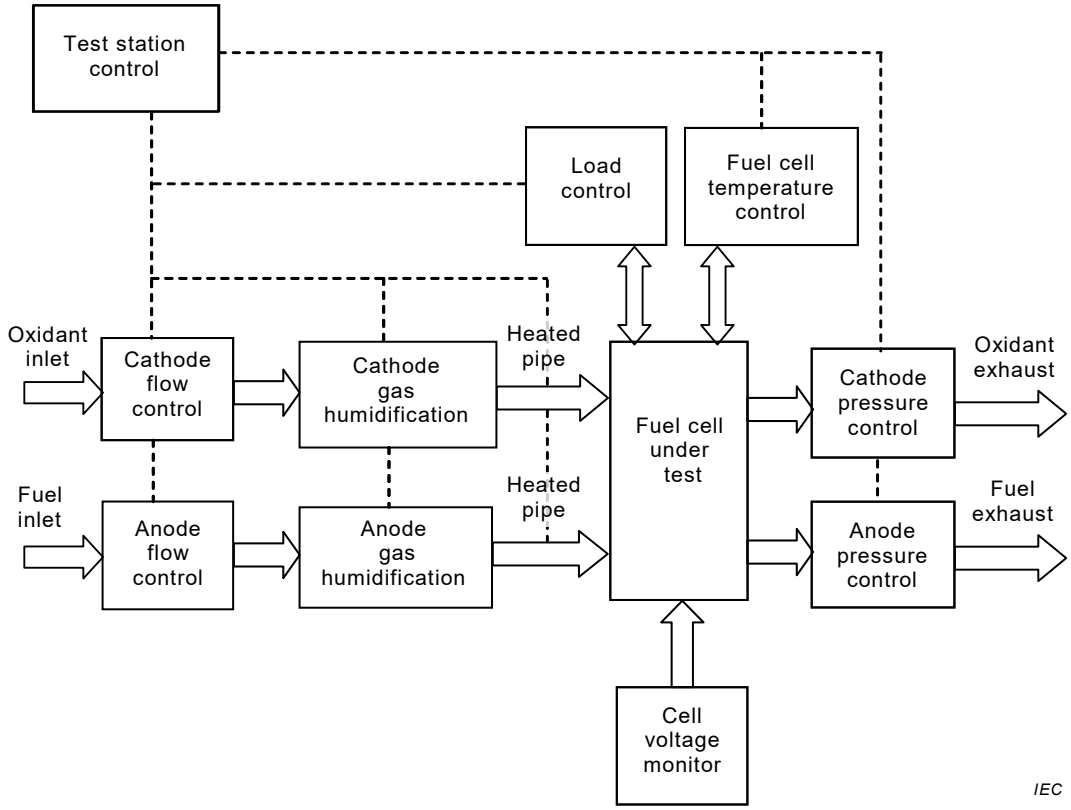


Figure 1 – Test station schematic diagram for single cell testing

Materials used for any component that will be in contact with humidified gas or humidifier water shall be compatible with the gas or water to prevent the extraction of impurities from the material. Example materials include stainless steel and fluoro-plastics.

Beware of leaching of metal cations from stainless steel in pure water at elevated temperature and the need to drain humidifiers periodically if not in constant use.

In the case of impurity testing, the gas humidification system shall be designed to avoid removing the test impurities from the gas stream prior to the gas entering the cell.

NOTE Impurities are given in ISO 14687-2.

If this test is not to be executed, a bubbler saturator can be used for fuel humidification.

Variations to this configuration are acceptable provided that the functional requirements of this document are met.

7.3 Maximum variation in test station controls (inputs to test)

The fuel cell test station shall have the following maximum variation in its controls:

a) current control ±1 % relative to set point;

b) voltage control ±1 % relative to set point;

c) cell temperature control ±1 °C at set point (in steady state);

d) humidity dew point control ±2 °C at set point (in steady state), if applicable;

e) flow rate control ±5 % relative to set point;

f) pressure control ±3 % relative to set point.

8 Measuring instruments

8.1 Instrument uncertainty

The maximum instrument uncertainty for measurements (test outputs) in the tests shall be as follows:

a) current ±1 % of maximum expected value;

b) voltage ±0,5 % of maximum expected value;

c) temperature ±1 °C;

d) dew point ±2 °C;

e) flow rate ±2 % of maximum expected value; and

f) pressure ±3 % of maximum expected value.

NOTE At low current, voltage and flow rates, the uncertainties can be very large with respect to the measured values.

8.2 Measuring instruments and measuring methods

8.2.1 General

Measuring instruments shall be selected in accordance with the range of values to be measured. The instruments shall be calibrated regularly in order to maintain the level of accuracy described in 8.1. All measuring devices shall be calibrated according to the manufacturer’s instructions or relevant national or international standards.

8.2.2 Voltage

A voltage meter shall be connected to the anode and cathode flow plates or current collectors, minimizing the influence of electrical contact resistances. The electrical contact resistances between the connections of the voltage meter, either anode and cathode flow plates or output terminals of anode and cathode current collectors shall be measured and recorded, if not negligible.

8.2.3 Current

A current measuring device shall be located in the current-carrying circuit of the cell. The current-measuring device may consist of a low-impedance ammeter or a calibrated shunt resistor, which develops a precisely known voltage in proportion to the current flowing. The current may also be measured using the features of an electronic load.

8.2.4 Internal resistance (IR)

Recommended IR measuring methods are the current-interrupt method (see 11.6.2.1) and the electrochemical impedance spectroscopy (EIS) method (see 11.7.5). An AC resistance method at a fixed frequency (typically 1 kHz), such as the use of an AC milliohm meter, is also acceptable (see 11.6.2.2). Care should be taken in the selection of a frequency to ensure the impedance contains no imaginary component. The frequency of the milliohm meter should be recorded.

Care should be taken when using an external AC signal in parallel with the load in order to avoid interference.

Plus/minus sense leads of these measuring instruments shall be connected to the output terminals of the cathode and anode current collectors, respectively.

8.2.5 Fuel and oxidant flow rates

Fuel and oxidant flow rates shall be measured by means of a volumetric meter, a mass flow meter, or a turbine-type flow meter. If such a method is not practical, flow measurement by a nozzle, orifices or Venturi meter is recommended. The location of a flow meter shall be upstream of the humidifier.

If the flow meter requires pressure compensation, a static pressure measuring port shall be located immediately upstream of the flow meter to be corrected.

8.2.6 Fuel and oxidant temperature

The recommended sensor for direct temperature measurement is a thermocouple, a resistance thermometer with a transducer or a thermistor.

The temperature sensor shall be located immediately upstream of the single cell. It is recommended to position another sensor immediately downstream of the single cell.

If the fuel and/or oxidant flow meter requires temperature compensation, the sensor for such correction shall be located immediately upstream of the flow meter.

8.2.7 Cell temperature

The recommended sensor for direct temperature measurement is a thermocouple, a resistance thermometer with a transducer or a thermistor

The temperature sensor should be located as close as possible to the centre of the cathode active area. Ideally, it should be at the centre of both anode and cathode flow plates (see 5.4 and Annex A for more details).

8.2.8 Fuel and oxidant pressures

For measuring fuel and oxidant pressures, calibrated pressure transducers are the preferred method. Other acceptable methods include calibrated manometers, dead-weight gauges, bourdon tubes or other elastic-type gauges.

Static pressure-measuring ports shall be located immediately upstream or immediately downstream of the single cell. Fuel and oxidant pressures may be controlled either upstream of the cell (inlet pressure mode) or downstream of the cell (back pressure mode).

Connecting piping shall be checked to verify that it is leak-free under working conditions in advance of the performance tests. Liquid water in the piping should be avoided.

If pressure fluctuations occur, a suitable means of damping shall be installed in an effective position.

Pressures shall be measured as static pressures with the effect of velocity considered and eliminated.

8.2.9 Fuel and oxidant humidity

To measure fuel and oxidant humidity, a chilled mirror, aluminum oxide, bulk polymer resistive or capacitance-type hygrometer can be used to obtain humidity values, depending on the fuel and oxidant temperatures.

Humidity shall be expressed as a dew-point temperature or as relative humidity in % calculated at the cell temperature.

A humidity measuring port shall be located upstream of the single cell, or the humidity sensor can be in the reactant gas before testing commences. In the case of using ambient or synthetic air as oxidant, the dew point shall be measured and recorded.

8.2.10 Ambient conditions

It is recommended that the ambient temperature, pressure and humidity be measured and recorded.

For the direct measurement of ambient temperature, thermocouples with a transducer or a resistance thermometer with a transducer is recommended.

For the direct measurement of ambient pressure, a barometer is recommended.

For direct measurement of ambient humidity, a hygrometer is recommended.

8.3 Measurement units

Table 1 identifies the parameters and their measurement units for the tests.

Table 1 – Parameters and units

Parameter	Unit
Temperature	°C
Fuel and oxidant pressures	kPa [c]
Dew points of fuel and oxidant	°C
Fuel and oxidant flow rates (STP [a])	$cm^3\ min^{-1}$, $cm^3\ s^{-1}$
Fuel and oxidant stoichiometries	--
Current	A
Current density	$A\ cm^{-2}$
Voltage	V
Output power	W
Power density	$W\ cm^{-2}$
Area-specific cell resistance	$\Omega\ cm^2$
Fuel composition [b]	$(mol)\ mol^{-1}$
Oxidant composition [b]	$(mol)\ mol^{-1}$
Time: hour, minute, second	h, min, s
Electric charge	C

[a] STP = standard temperature and pressure: 0 °C and 101,325 kPa (absolute). Unless stated otherwise, STP is used for the flow rate.

[b] Impurities are expressed as $(\mu mol)\ mol^{-1}$.

[c] ISO recommends using absolute pressure (kPa) if possible. If gauge pressure is used, it should be noted as such and be given in kPaG.

9 Gas composition

9.1 Fuel composition

9.1.1 Hydrogen

According to ISO 14687-2, hydrogen fuel index (minimum mole fraction) shall be 99,97 %.

9.1.2 Reformed gases

The simulated reformed gas may be specified by the cell or component manufacturer. The purity and composition of the reformed gas shall be determined by chemical analysis. The results of the chemical analysis shall be recorded.

9.2 Oxidant composition

If air is used as an oxidant, then either atmospheric or synthetic air may be used. In the case of atmospheric air, it is recommended that the air be oil- and particulate-free. The oxidant composition, including the concentration of impurities, shall be recorded.

10 Test preparation

10.1 Standard test conditions

The following shall be specified by the cell and/or cell component manufacturer as the standard test conditions before commencing a test. If no specifications are given, the conditions will depend on the study to be performed. These conditions shall be recorded:

a) cell temperature (recommended location: centre of cathode flow plate);
b) fuel operating pressure;
c) oxidant operating pressure;
d) fuel humidity at fuel cell operating temperature;
e) oxidant humidity at fuel cell operating temperature;
f) fuel composition;
g) oxidant composition;
h) fuel stoichiometry;
i) oxidant stoichiometry;
j) rated current density;
k) rated voltage;
l) maximum current density;
m) minimum cell voltage.

Performance tests shall be carried out under standard testing conditions unless otherwise specified in the description of the respective test methods. A typical testing flowchart is given in Figure 2.

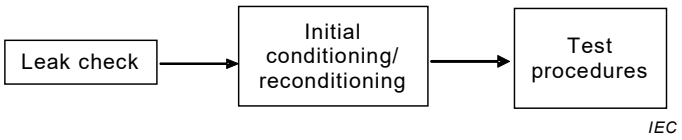


Figure 2 – Typical testing flowchart

10.2 Ambient conditions

For each test run, it is recommended that the following ambient conditions be measured:

a) temperature,
b) absolute pressure; and
c) relative humidity.

NOTE Altitude can have an influence on the air composition.

10.3 Data sampling rate

The recommended data sampling rate is 1 Hz. If the system is at a steady state, it is appropriate to take one measurement value as an average value over 1 min (i.e. mean value of 60 single measurements).

10.4 Repeatability and reproducibility

It is recommended to verify the repeatability and reproducibility of the measurements at an appropriate interval in accordance with ISO/IEC Guide 98-3.

10.5 Number of test samples

Tests can be conducted either in sequence with one sample or in parallel with multiple samples. The repeatability and reproducibility specifications are limited to polarization curves only. The measurements shall be performed consecutively. Between measurements, the fuel cell shall be shut down and re-conditioned from ambient startup conditions.

10.6 Leak check of gas circuit with inert or test gas

All materials used for leak checking the gas circuit shall be compatible with the gas piping and cell components. After mounting the single cell in the stand, check for gas leaks using inert or test gas and using a liquid leak detector test on all connections.

10.7 Initial conditioning and stable state check

Initial conditioning shall be applied to a newly assembled cell. The main purpose of initial conditioning is to bring the cell to a stable state in performance for subsequent testing.

Initial conditioning shall be:

a) carried out in accordance with the MEA or component manufacturer's instruction;

b) as proposed by the manufacturer of the test object; or

c) one that is common practice at the testing organization.

Representative initial conditioning procedures are provided for information in Annex D.

A baseline polarization curve shall be established by measuring it three times under the reference conditions and the average cell voltage values shall be calculated at each current density. The stability of performance is verified by a maximum allowable deviation of 5 % among the three polarization curves.

10.8 Shutdown

The shutdown procedure shall be carried out to bring a cell to a storage state (cold state). The general purpose of the shutdown procedure is to cool the cell to ambient temperature while avoiding leaving liquid water in the cell at ambient temperature.

The shutdown procedure shall be carried out in accordance with the MEA or component manufacturer's instruction.

A representative shutdown procedure is provided for information in Annex E.

10.9 Reconditioning

Reconditioning procedures shall be used upon restarting a cell after shutdown to ensure that the MEA is properly humidified.

Reconditioning shall be carried out in accordance with the MEA or the component manufacturer's instruction.

Representative reconditioning procedures are provided for reference in Annex F.

11 Basic performance test methods

11.1 General

The following tests or measurements provide the fundamental techniques for testing PEFCs using single cells:

- polarization curve tests (11.2);
- steady-state test (11.3);
- long-term operation test (11.4);
- voltammetry (11.5);

- internal resistance (IR) measurement (11.6);
- electrochemical impedance spectroscopy (EIS) (11.7).

11.2 Polarization curve tests

11.2.1 General

The objective of this test is to measure polarization curves under defined cell conditions in the tests given in Table 2.

NOTE Polarization curves are sometimes referred to as I-V or V-I curves.

For the measurement of polarization curves, there are two methods: measurement at constant gas stoichiometries and measurement at constant gas flow rates.

For the constant gas stoichiometry method, the fuel and oxidant stoichiometries are held constant in the current range of 0 to I_{max}, and both fuel and oxidant flow rates are varied as the current changes except for values below I_{min}. This is similar to the actual conditions of fuel cell operation. For the minimum current density, a constant minimum flow rate specified by the cell manufacturer is usually applied in order to avoid unstable cell voltages. If not specified, I_{min} can be assumed to be 10 % of I_{max}.

NOTE Due to a different flow rate at each current level, local heat and water balances are changed as the current varies. It requires a substantial amount of time to reach a new steady-state point after each current change. Specifically, at low current values including 0 A, the cell voltage becomes unstable due to low gas flow rates.

For the constant gas flow rate method, the fuel and oxidant flow rates are constant in the current range of 0 to I_{max}, and both fuel and oxidant stoichiometries will change as the current changes. Although this may be different from the actual conditions of fuel cell operation, cell conditions such as temperature, pressure and humidity are maintained stable even under variable current at a constant flow rate.

The test methods of polarization curves at constant gas stoichiometries and at constant gas flow rates are provided in 11.2.2 and 11.2.3 respectively.

11.2.2 Polarization curves at constant gas stoichiometries

11.2.2.1 General

The objective of this test is to determine the evolution of cell voltage (and, in parallel, of power density) when varying the current density under constant gas stoichiometry conditions.

11.2.2.2 Test method

Set the fuel and oxidant flow rates to correspond to the stoichiometries at maximum current I_{max} that is specified by the cell manufacturer.

Set current I_{max} and maintain the current until the cell voltage is stabilized within ±5 mV for 15 min.

Obtain polarization curves of the cell in the range of 0 to I_{max} by varying the current between 0 and I_{max} at suitable intervals, while maintaining the fuel and oxidant stoichiometries constant at each current level above I_{min} to be specified. The direction of current change shall be recorded.

The minimum requirement for cell voltage stabilization is ±5 mV for 5 min at each current density value. Typical current density increments are given in Annex G.

This test can also be performed by varying the cell voltage in the range of OCV to minimum cell voltage and allowing the current to stabilize to within ±2 % for 5 min.

It is recommended that internal resistance measurement (11.6) is carried out during this polarization curve test.

As the current is increased, the change in gas flow rate should precede the change in current. As the current is decreased, the change in gas flow rate should follow the change in current.

At low stoichiometry, care should be taken that the equipment is capable of controlling the desired gas flow rate within the limit specified in 7.3e) and I_{min} should be chosen accordingly.

11.2.3 Polarization curves at constant flow rate

11.2.3.1 General

The objective of this test is to determine the evolution of cell voltage (and, in parallel, of power density) when varying the current density under constant gas flow rate conditions.

11.2.3.2 Test method

Set the fuel and oxidant flow rates to correspond to the standard stoichiometries at the maximum current density, I_{max}.

Set current I_{max} and maintain the current until the cell voltage is stabilized within ±5 mV for 15 min.

Obtain polarization curves of the cell by varying the current in the current range of 0 to I_{max} at a suitable interval, while maintaining the fuel and oxidant flow rates constant.

The minimum requirement for cell voltage stabilization is ±5 mV for 5 min at each current density value. The exact procedure shall be recorded. Typical current density increments are given in Annex G.

This test can also be performed by varying the cell voltage in the range of OCV to minimum cell voltage and allowing the current to stabilize to within ±2 % for 5 min.

It is recommended that internal resistance measurement (11.6) is carried out during this polarization curve test.

11.3 Steady-state test

11.3.1 General

The objective of this test is to verify the output from the cell under constant-current conditions with all test input parameters set to desired values. This test can also be carried out under constant-voltage conditions.

11.3.2 Test methods

There are two similar methods to perform this test.

a) Constant-current method

   Set all test input parameters to the desired values.

   Set the current at a level corresponding to the rated current density (I_{st}) and maintain it for the duration of the test until the cell voltage is stabilized within ±5 mV for an allocated time based on the objective of the test.

   Record the cell voltage (V_{st}).

   Power output under the standard test conditions (P_{st}) is calculated from the test results.

b) Constant-voltage method

Set all test input parameters to the desired values.

Set the cell voltage (V_{st}) corresponding to the desired value and maintain it until the cell current is stabilized within ±2 % for an allocated time.

Record the cell current (I_{st}).

Power output under the standard test conditions (P_{st}) is calculated from the test results.

11.4 Long-term operation test

11.4.1 General

The objective of this test is to determine the evolution of voltage of a cell operating for a long period of time under the specified constant-current conditions.

The cell should be subjected to diagnostics before and after the test, also during the test if desired, and to a teardown analysis after the test, although this is out of the scope of this document.

The diagnostics may include the following:

- electrochemical surface area (ECA) measurement for the anode and the cathode (11.5.3),
- hydrogen crossover (11.5.2),
- polarization curves (11.2),
- IR measurement (11.6),
- electrochemical impedance spectroscopy (11.7).

Comparison of the results of these diagnostics is useful to investigate the cause of any performance degradation.

11.4.2 Test method

Perform steady-state test (11.3) and, if desired, additional diagnostic tests.

Then run the cell at the current specified by the manufacturer for an extended period of time, a suggested minimum of 1 000 h, under the standard test conditions in accordance with the allowable operating time specified by the cell manufacturer.

Monitor the cell voltage during operation. If desired, perform diagnostics. The suggested number of these diagnostic tests is between 1 and 10 every 1 000 h.

At the end of the test period, perform steady-state test (11.3), and if desired, perform additional diagnostic tests.

The performance decay, expressed in µV/h, is evaluated by the voltage drop between two steady-state tests (11.3) performed at the beginning and end of the test period divided by the operation time between the two steady-state tests (11.3).

Decoupling of temporary and permanent degradation can be achieved by implementing appropriate recovery procedures before steady-state tests and other diagnostics.

11.5 Voltammetry

11.5.1 General

Voltammetry may be used as a diagnostic technique to quantify the rate of hydrogen crossover and the electrochemical surface area (ECA) of the platinum catalyst on each electrode at any stage in the lifetime of the MEA. It can also be used to determine surface

coverage by the adsorption of contaminants. A potentiostat shall be used to control the applied potential during the measurement.

11.5.2 Hydrogen crossover test

11.5.2.1 General

The objective of this test is to measure the permeation rate of hydrogen from the anode to the cathode through the membrane (crossover rate).

An increased hydrogen crossover rate leads to a decrease in performance. It is caused by the deterioration of the membrane and can lead to additional deterioration of the MEA, including the membrane and particularly the cathode active layer materials.

Hydrogen permeability depends not only on the physical properties of the membrane, but also on the humidity, temperature and pressure of gas. It is recommended to carry out the test under a range of operating conditions.

11.5.2.2 Test method

Condition the cell and keep the cell in the conditions suggested by the manufacturer for this test.

With the cell fed on the anode side with hydrogen (2 ml/min/cm^2 to 4 ml/min/cm^2 active area), and on the cathode side with nitrogen (2 ml/min/cm^2 to 20 ml/min/cm^2 active area), perform a linear potential sweep from 0,1 V to 0,5 V versus RHE at a sweep rate of no more than 5 mV/s.

Hydrogen evolution should be avoided. If it appears, the minimum potential should be raised.

For the voltammetry, connect cell terminals to a potentiostat with the counter and reference electrode leads connected to the anode and the working electrode leads connected to the cathode.

The hydrogen crossover rate, ν_{co}, (mol·s^{-1}·cm^{-2}) can be obtained from the crossover current density, I_{co} (interpolated current density at zero potential):

$$\nu_{co} = I_{co} / 2F$$

where

F is Faraday constant (96 485 C mol^{-1}).

See Figure 3 for illustration.

This test also provides the resistance of a short circuit between the anode and cathode sides, from the slope of the linear portion of Figure 3. A cell shall be conditioned for the test at the relevant temperature and pressure.

To compare results, the temperature shall be controlled at a fixed value. The test conditions shall be recorded.

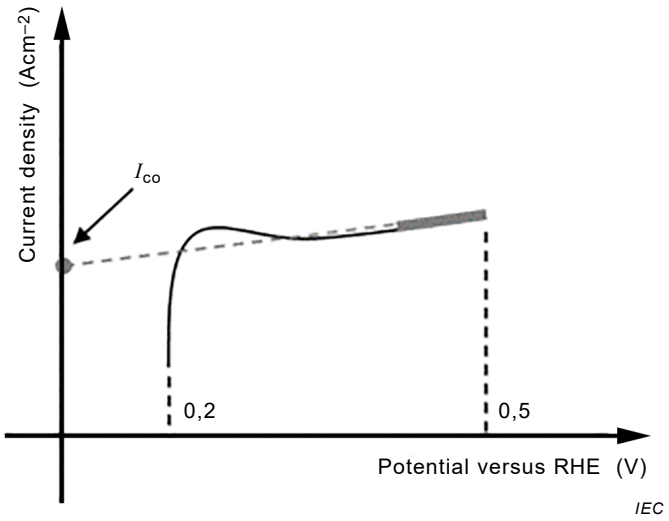


Figure 3 – Hydrogen crossover test

11.5.3 Electrochemical surface area (ECA) measurement

11.5.3.1 General

The objective of this test is to determine the active electrochemical surface area (ECA) of the anode and cathode electrodes. The test is specific to a platinum catalyst; it determines the electrochemical area of platinum.

Both cathode and anode electrochemical areas can be measured. In electrochemical voltammetry methods, the test electrode will be the working electrode, whereas the other electrode will be the reference and counter electrode.

Connect the cell terminals to a potentiostat: connect the working electrode leads to the test electrode, and the counter and the reference electrode leads to the other electrode.

11.5.3.2 Test methods

Two procedures may be used.

a) Measuring hydrogen underpotential adsorption (desorption) charge

Condition and keep the cell in the conditions suggested by the manufacturer for this test. If none are available, the suggested conditions are 10 °C to 80 °C at a pressure between atmospheric pressure and 150 kPa, and 100 % gas humidification. To compare results, all conditions shall be controlled at a fixed value. The test conditions shall be recorded.

Run a nitrogen stream (4 ml/min/cm^2 active area) through both electrodes for 30 min in advance of the test to purge oxygen from the cell.

Run a flow of nitrogen at 2 ml/min/cm^2 to 20 ml/min/cm^2 active area through the working electrode for 15 min.

Run a flow of hydrogen at 2 ml/min/cm^2 to 4 ml/min/cm^2 active area to the reference/counter electrode for 15 min.

Perform voltammetry with nitrogen at the selected flow rate or no flow, varying potential between approximately 0,05 V and at least 0,6 V at different sweep rates and take the maximum ECA value.

Potential limits should be chosen to minimize the effect of hydrogen evolution and material oxidation.

Obtain cyclic voltammograms repeatedly until they become stable and use the last waveform for calculating the ECA.

From the voltammetry, determine the hydrogen desorption or adsorption charge (q_h) by integrating the relevant area under the curve as shown in the example in Figure 4. Either the adsorption or desorption peak may be used. Use the most accepted equivalent factor to calculate ECA:

$$A_{e\text{-}Pt} = q_h / (\sigma_H \times A), \text{ or}$$

$$A_g = q_h / (\sigma_H \times L)$$

where

$A_{e\text{-}Pt}$ is the ECA of platinum catalyst per unit electrode area (m^2/cm^2);

q_h is the hydrogen desorption or adsorption charge (C: coulomb);

σ_H is the hydrogen desorption or adsorption charge per unit active area of platinum (2,1 $\mu C/m^2$);

A is the geometric area of the electrode (cm^2);

A_g is the electrochemical surface area per 1 g of platinum catalyst (m^2/g);

L is the platinum loading (g).

It shall be reported if the desorption charge, or adsorption charge, or the mean value of both, is used.

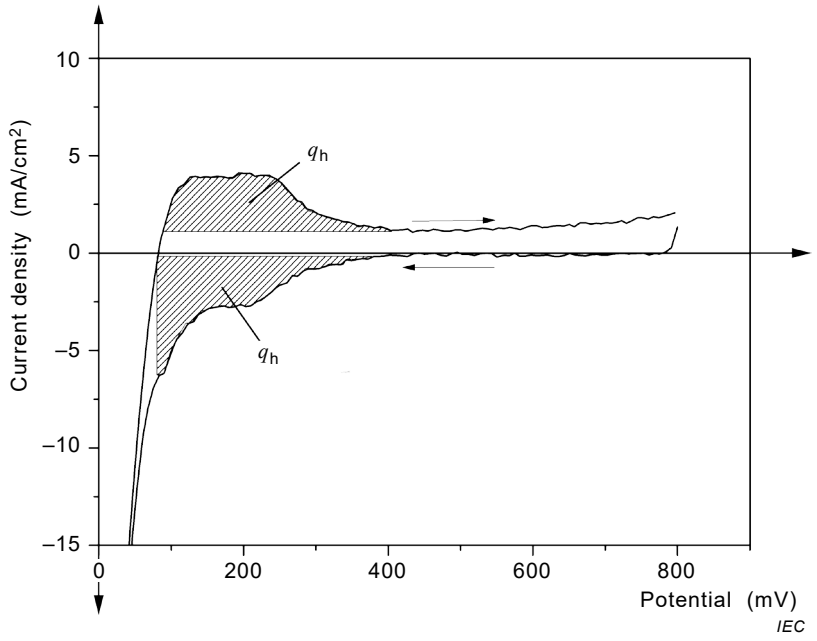


Figure 4 – Determination of adsorption/desorption charge (q_h)

b) Carbon monoxide stripping voltammetry

Condition and keep the cell in the conditions suggested by the manufacturer for this test. If no conditions are specified, suggested conditions are 10 °C to 80 °C at a pressure between atmospheric pressure and 150 kPa, and 100 % gas humidification. To compare results, all conditions shall be controlled at a fixed value. The test conditions shall be recorded.

Run a nitrogen stream (4 ml/min/cm^2 active area) through both electrodes for 30 min in advance of the test to purge the oxygen from the cell.

Run a flow of nitrogen (99 %) and carbon monoxide (1 %) at 2 ml/min/cm^2 to 20 ml/min/cm^2 active area through the working electrode for 15 min.

Run a flow of hydrogen at 2 ml/min/cm^2 to 4 ml/min/cm^2 active area to the reference/counter electrode.

Perform voltammetry with nitrogen at the selected flow rate or no flow, varying the potential from approximately 0,05 V to 1,0 V, at a sweep rate from 10 mV/s to 50 mV/s.

When it is necessary to minimize the effect of these measurements on the ECA, the maximum potential should be limited to a lower value in order to avoid excessive oxidation of the materials composing the active layer.

Measure the carbon monoxide desorption charge (q_{co}) by integrating the relevant area under the curve as shown in the example in Figure 5. Use the most accepted equivalent factor to calculate the ECA:

$$A_{e\text{-}Pt} = q_{co} / (\sigma_{co} \times A), \text{ or}$$

$$A_g = q_{co} / (\sigma_{co} \times L)$$

where

$A_{e\text{-}Pt}$ is the ECA of platinum catalyst per unit electrode area (m^2/cm^2);

q_{co} is the CO desorption charge (C: coulomb);

σ_{co} is the CO desorption charge per unit active area of platinum (4,2 $\mu C/m^2$);

A is the geometric area of the electrode (cm^2);

A_g is the electrochemical surface area per 1 g of platinum catalyst (m^2/g);

L is the platinum loading (g).

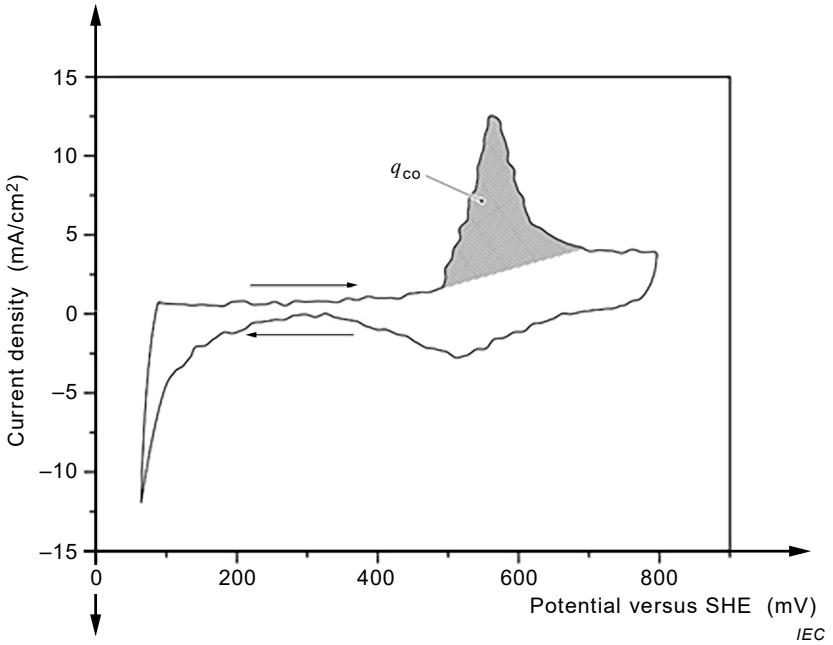


Figure 5 – Determination of CO desorption charge (q_{co})

11.6 Internal resistance (IR) measurement

11.6.1 General

The objective of this test is to determine the internal resistance, R_{int}, of the cell at different current densities in order to facilitate IR correction of potential measurements. The measuring techniques described in 8.2.4 shall be used for internal resistance measurement. If internal resistance measurements are conducted in parallel with polarization curve measurements, the measuring technique should not disturb the steady state. If the measurement disturbs the steady state, record the cell voltage and current density values then perform the internal resistance measurement. Polarization curve measurements shall be made according to 11.2.

11.6.2 Test methods

11.6.2.1 Current-interrupt technique

The principle behind this technique is that the instantaneous change in cell voltage when the current is switched off, ΔV_{CI}, is a function of the internal resistance of the cell.

Set the current density to the desired value and allow the cell voltage to reach a steady value (minimum requirement ±5 mV for 5 min).

Switch the cell current to zero and monitor the evolution of the cell voltage over time using an oscilloscope or similar measuring instrument, as shown in Figure 6. ΔV_{CI} is the instantaneous change of cell voltage upon current-interrupt as illustrated in Figure 6. ΔV_{CI} is obtained by extrapolation of the cell voltage before and after current-interrupt.

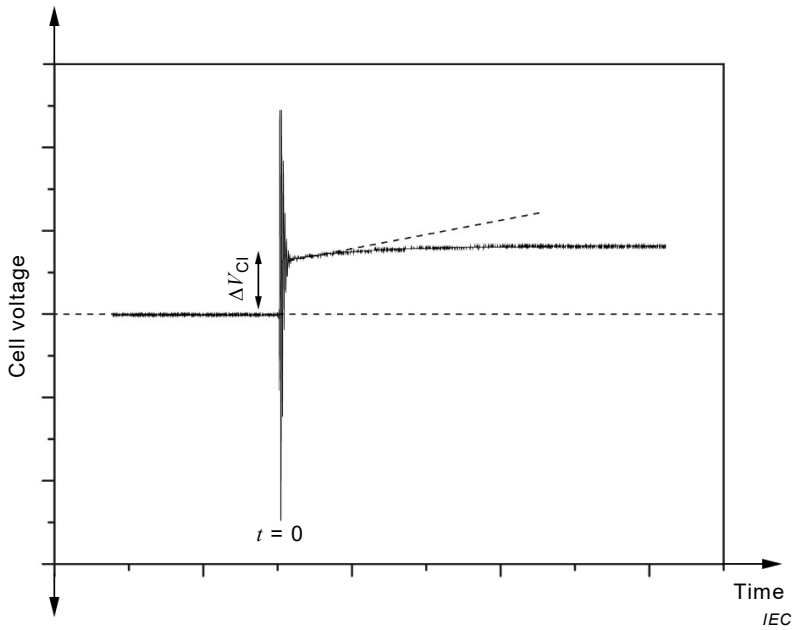


Figure 6 – Measurement of ΔV_{CI}

Cell resistance, R_{int} is calculated as follows:

$$R_{int} = \Delta V_{CI} / I$$

where

R_{int} is the cell resistance (Ω);

ΔV_{CI} is the instantaneous change of cell voltage upon current-interrupt (V);

I is the cell current (A).

Area-specific resistance, R_{ASR}, is calculated by the following equation:

$$R_{ASR} = R_{int} \times A$$

where

R_{ASR} is the area-specific resistance (Ω cm^2);

R_{int} is the cell resistance (Ω);

A is the geometric area of the electrode (cm^2).

11.6.2.2 Single-frequency AC resistance measurement

Set the current density to the desired value and allow the cell voltage to reach a steady value (minimum requirement ±5 mV for 5 min).

Connect an AC resistance meter to the cell and record the reading which is cell resistance R_{int} (Ω).

Measuring conditions shall be indicated, including AC amplitude and frequency of the measurement, current density, cell voltage, cell temperature, and gas feed conditions in the anode and cathode (composition, humidity, temperature, pressure, and flow rates).

The area-specific resistance R_{ASR} (Ω cm^2) shall be calculated by the equation in 11.6.2.1.

11.7 Electrochemical impedance spectroscopy (EIS)

11.7.1 General

EIS is an electrochemical technique that allows the impedance spectrum of a cell to be recorded as a function of the frequency of applied AC signals, and the spectrum thus obtained is to be analysed by transfer function analysis.

11.7.2 Test conditions

The following test conditions shall be determined in advance by conducting preliminary tests:

a) measuring range of frequencies,

b) number of measurement points,

c) galvanostatic or potentiostatic method,

d) amplitude of applied AC signal,

e) fed gas (H_2/H_2, H_2/N_2, H_2/Air, H_2/O_2, etc.), and

f) operating conditions (temperature, pressure, humidity etc.).

Concerning the number of measurement points, it is preferable to plot five to twenty points per decade of frequency in order to distribute them evenly as logarithms so that the geometry of impedance plots is clearly identified, and also to avoid the fundamental electrical grid frequency and its harmonics.

11.7.3 Test method

The test shall be conducted as follows:

a) establish the test conditions,

b) verify that the stable state of the cell has been reached,

c) superimpose AC sinusoidal waves on DC current or voltage and start measurements, and

d) sweep the AC sinusoidal waves within the prescribed frequency range and measure the impedance at each frequency.

The amplitude of the AC signal for the measurement shall be small enough not to perturb the operation of the cell.

The validity of the impedance measurement shall be verified, for example, by using the Kramers-Kronig (KK) relationships.

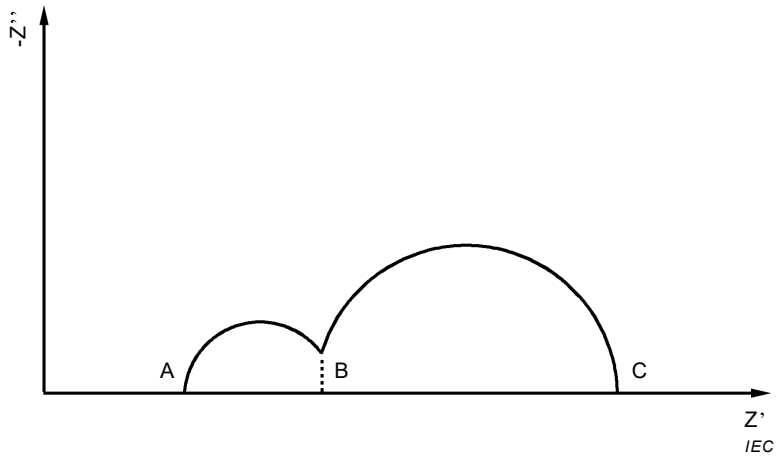
11.7.4 Analysis of EIS data

The test results shall be expressed as a complex impedance (Nyquist/Cole-Cole) plot or a Bode plot (a plot of impedance against the logarithm of measured frequency). The impedance per unit active area shall be plotted. A typical complex impedance plot is shown in Figure 7. The intercept A gives the IR drop in the cell. Other parameters may be extracted via the construction of an equivalent circuit for the cell, consisting of circuit elements (e.g. resistors, capacitors, constant phase elements) representing different charge transfer and mass transfer processes. Caution should be taken in the use of such equivalent circuits and complementary characterization techniques shall be applied to establish their validity.

11.7.5 IR measurement by EIS

Figure 7 shows a typical diagram of complex impedance plotting.

The intercept of the real impedance axis, generally occuring between1 kHz and 20 kHz, identifies point A. The impedance measured or interpolated at point A represents the IR, consisting of a purely resistive impedance.



Key

A	High frequency limit impedance
C	Low frequency limit impedance
A-B	High frequency arc impedance
B-C	Low frequency arc impedance
Z'	Real part of impedance
Z''	Imaginary part of impedance

Figure 7 – Typical diagram of a complex impedance plot

12 Applied performance test methods

The tests and measurements described in Clause 11 are basic performance test methods that can be applied to a variety of tests for specific purposes.

Applied performance tests based on the basic performance test methods described in 11.2 to 11.7 are summarized in Table 2 and described in Annex H.

The applied performance tests described in Table 2 can be selectively executed depending on the objectives of the users of this document.

Table 2 – Applied performance tests

	Test name		Objective	Applicable basic test methods	Clause/ subclause
1	**Gain tests**			Pol curve	H.1
	1	Hydrogen gain test	Evaluate the diffusion capability of the anode.	Steady	H.1.1
	2	Oxygen gain test	Evaluate the diffusion capability of the cathode.		H.1.2
2	**Gas stoichiometry tests**			Pol curve	H.2
	1	Fuel stoichiometry test	Evaluate the diffusion capability of the anode.	Steady	H.2.1
	2	Oxidant stoichiometry test	Evaluate the diffusion capability of the cathode.		H.2.2
3	Temperature effect test		Measure the effect of cell temperature on cell performance.	Pol curve Steady	H.3
4	Pressure effect test		Measure the effect of fuel and oxidant pressures on cell performance.	Pol curve Steady	H.4
5	**Humidity effect tests**			Pol curve	H.5
	1	Fuel humidity effect test	Measure the effect of varying humidity of the fuel on cell performance.	Steady	H.5.1
	2	Oxidant humidity effect test	Measure the effect of varying humidity of the oxidant on cell performance.		H.5.2
6	Limiting current test		Identify mass transport limitations within an MEA.	Pol curve Steady	H.6
7	Overload test		Evaluate the electrical overload performance of a cell.	Pol curve Steady	H.7
8	Subzero storage test		Investigate the effect of storage at subzero temperature on performance.	Pol curve Steady Long-term	H.8
9	Subzero start test		Evaluate the cold start capability of a cell in a subzero temperature environment.	Pol curve Steady Long-term	H.9
10	Membrane swelling test (humidity cycle test)		Evaluate the mechanical strength of a membrane for dry/wet cycling.	Long-term Voltammetry	H.10
11	Open circuit voltage (OCV) test		Evaluate the chemical stability of an MEA under OCV conditions.	Pol curve Steady	H.11
12	Oxygen reduction reaction (ORR) activity test		Evaluate the ORR activity of a catalyst using an MEA.	Pol curve Steady	H.12
13	Fuel composition test		Measure the effect of the composition of reformed gas on cell performance.	Pol curve Steady Long-term Voltammetry IR EIS	H.13
14	**Cycling tests**			Pol curve	H.14
	1	Start/stop cycling test	Determine the evolution of the performance of a cell operating under the specified conditions as a function of start/stop profile count.	Voltammetry IR EIS	H.14.1
	2	Load cycling test	Determine the evolution of the voltage of a fuel cell operating under the specified conditions as a function of current density following a dynamic profile versus time.		H.14.2

	Test name		Objective	Applicable basic test methods	Clause/ subclause
	3	Potential cycle test (start/stop durability)	Evaluate the durability of a catalyst against startup and shutdown cycling by cycling the potential under accelerated conditions.		H.14.3
	4	Potential cycle test (load cycle durability)	Evaluate the durability of a catalyst against load cycling by cycling the potential under accelerated conditions.		H.14.4
15	**Impurity influence tests**			Pol curve	H.15
	1	Influence at rated current density	Determine the influence of impurities in fuel or air on cell performance and the degree of recovery of the cell performance from damage at rated current density.	Steady Long-term Voltammetry	H.15.1
	2	Influence on polarization curves	Determine the influence of impurities in fuel or air on the polarization curve of a cell.	IR	H.15.2
	3	Long-term impurity influence test	Determine the influence of impurities in fuel or air on long-term fuel cell operation.	EIS	H.15.3

Key

Pol curve:	polarization curve test (11.2)
Steady:	steady-state test (11.3)
Long-term:	long-term operation test (11.4)
Voltammetry:	voltammetry (11.5)
IR:	internal resistance (IR) measurement (11.6)
EIS:	electrochemical impedance spectroscopy (EIS) (11.7)

13 Test report

13.1 General

Test reports shall accurately, clearly and generally present sufficient information to demonstrate that all the objectives of the tests have been attained. A suggested template for the test report for the polarization curve test is given in Annex I.

13.2 Report items

The report shall present at least the following information:

a) title of the report,
b) authors of the report,
c) entity conducting the test(s),
d) date of the report,
e) standard number/test procedure number,
f) location of the test(s), and
g) test data (see 13.3 for details).

13.3 Test data description

Test data shall include the following information:

a) title of the test(s),
b) measurement conditions (see 13.4 for details),
c) measurement data,
d) date and time when the test(s) was conducted,

e) ambient conditions,
f) name and qualifications of person(s) conducting the test(s), and
g) test cell parameters (see 13.5 for details).

13.4 Description of measurement conditions

The measurement condition description shall include at least the following information:

a) cell temperature,
b) fuel and oxidant pressures,
c) dew points of fuel and oxidant,
d) fuel and oxidant compositions,
e) fuel and oxidant stoichiometries, and
f) flow rates of fuel and oxidant.

13.5 Test cell parameter description

Test cell parameters should include the following information:

a) active electrode area,
b) product name and brand name of MEA (optional),
c) type and thickness of membrane (optional),
d) type and amount of anode and cathode catalysts (optional),
e) type of gas flow path (optional),
f) type of GDL material (optional), and
g) clamping pressure (optional).

Annex A
(informative)

Flow plate

Figure A.1 shows an example design for the flow plate according to this document. The example design is intended for an active area of 25 cm^2. Both anode and cathode flow plates have a horizontal serpentine single-groove as the gas flow channel on the faces that contact the MEA. The recommended channel configuration is as follows:

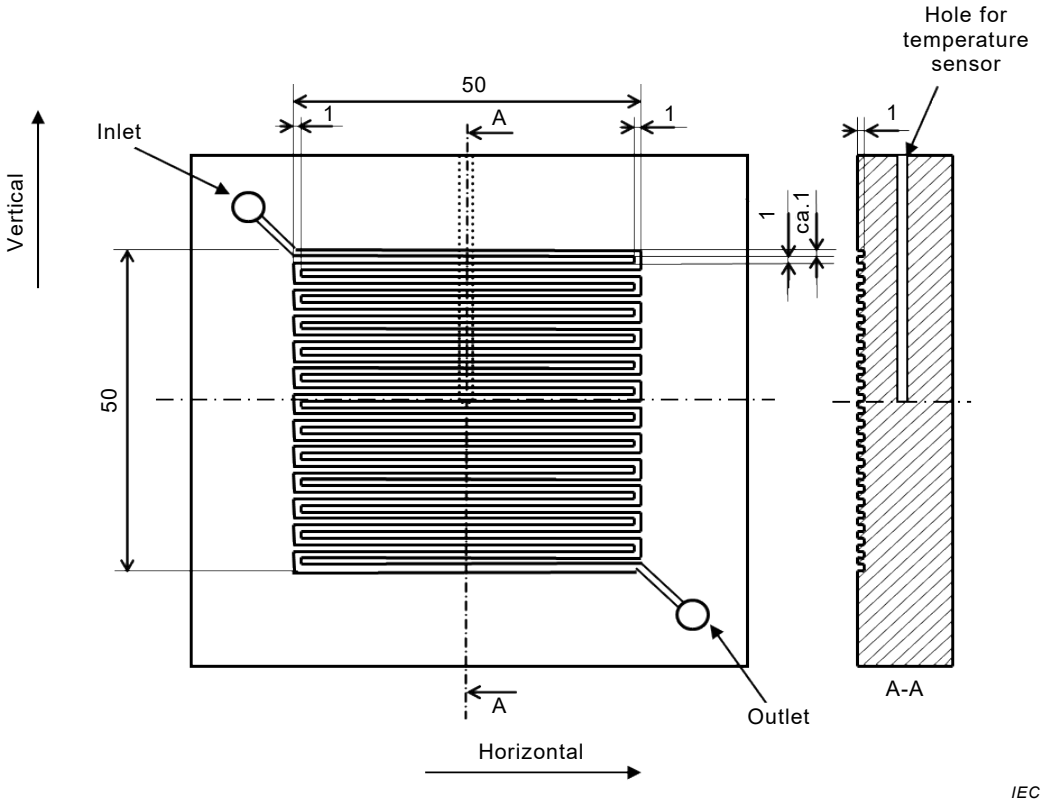
- width: 1,0 mm,
- depth: 1,0 mm,
- interval: ~1,0 mm.

The area covered with flow field channels should be slightly smaller than the active area of the electrodes to prevent the membrane from being damaged by the edge of the channel. Use an assembly procedure that avoids direct contact of the membrane and the edge of the channel.

The anode and cathode flow plates normally have the same channel configuration but their orientation can differ in the cell. Different possibilities may be used in this respect:

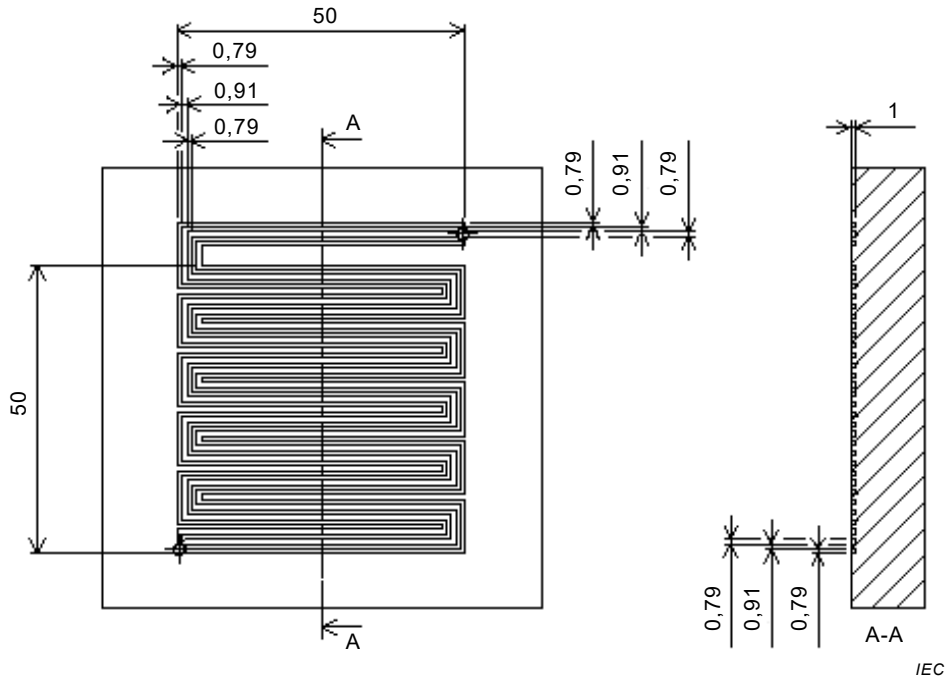
- co-flow: when the anode and the cathode gas paths are parallel;
- counter-flow: when the anode and cathode gas paths are parallel but the flow is in opposite directions;
- cross-flow: when the anode and cathode gas paths are at a certain angle, normally 90°.

Figure A.2 shows another example design for the flow plate which is also used in this document. The example design is intended for an active area of 25 cm^2. The difference between Figure A.1 and Figure A.2 is that Figure A.1 has a single-serpentine flow channel and Figure A. 2 has a triple-serpentine flow channel.



NOTE All dimensions in mm.

Figure A.1 – Design for flow plate (single-serpentine flow channel)



NOTE All dimensions in mm.

Figure A.2 – Design for flow plate (triple-serpentine flow channel)

Annex B
(informative)

Cell component alignment

Figure B.1 shows a single cell assembly using typical components; these are compressed together with nuts and bolts. If necessary, spring washers or spring disks can be placed on the bolts in order to prevent loosening. Alignment of components may be ensured by using a guide rod or positioning pins.

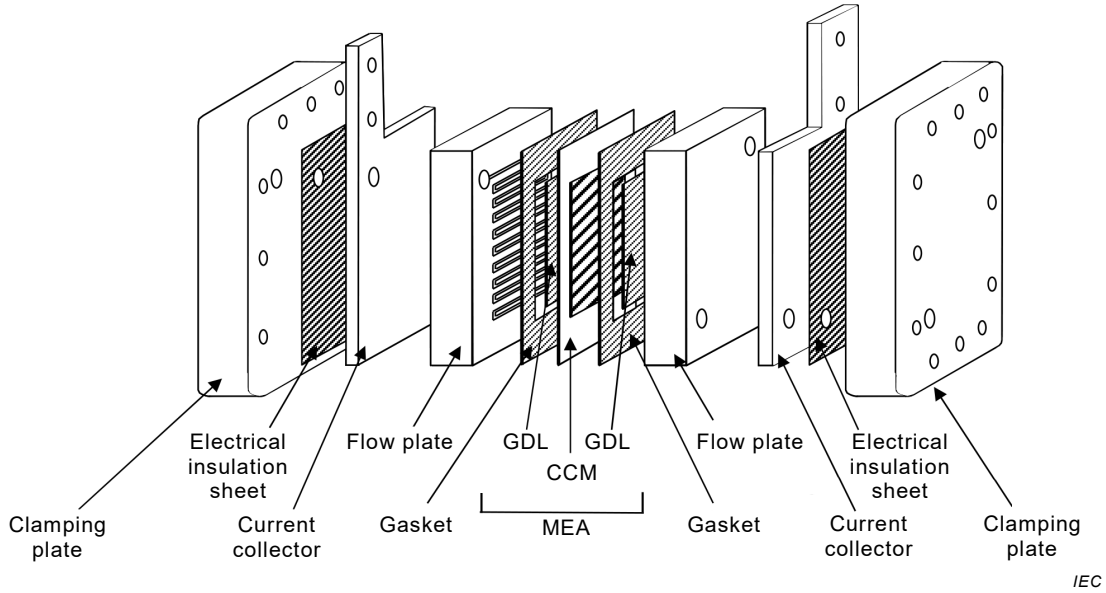


Figure B.1 – Single cell assembly using typical components

Annex C
(informative)

Leak test

C.1 Purpose

The purpose of leak testing is to quantify the leakage rate of the gas, and if applicable the coolant loop, to determine the serviceability of the fuel cell prior to operating or testing. It is generally recommended that leak-testing of the gas circuit be carried out using an inert gas, preferably helium, at room temperature.

C.2 Test procedures

Typical leak-test protocols are described below.

a) Procedure 1

Inject gas into both the anode and cathode sides. Set the back pressure to nearly 0 kPaG, then shut off the exit valves of the anode and cathode gases. First, pressurize the anode side to 50 kPaG (or 150 % of the maximum operating pressure) and the cathode side to 30 kPaG (or 125 % of the maximum operating pressure) simultaneously. Shut off the inlet valves of the anode and cathode gases to seal the gas in the cell. Keep the cell in this condition for 10 min while monitoring the pressures on the anode and cathode sides, respectively.

Secondly, pressurize the cathode side to 50 kPaG (or 150 % of the maximum operating pressure) and the anode side to 30 kPaG (or 125 % of the maximum operating pressure) simultaneously. Shut off the inlet valves of the anode and cathode gases to seal the gas in the cell. Keep the cell in this condition for 10 min while monitoring the pressures in the anode and cathode sides, respectively.

In the above procedures, the pressure change after 10 min should be less than 5 kPaG on both the anode and cathode sides.

In the first step, if the pressure in the anode side falls and that in the cathode side rises, gas crossover through the membrane has occurred. In the second step, if the cathode pressure drops and the anode pressure rises, gas crossover in the other direction has occurred. If either pressure drops independently of the other, then an overboard leak has occurred. If both pressures drop, external leaks are suspected.

b) Procedure 2

Inject nitrogen into both the anode and cathode sides. Set the back pressure to nearly 0 kPaG, then shut off the exit valves of the anode and cathode gases. First, pressurize both the anode and cathode sides to 30 kPaG simultaneously. Shut off the inlet valves of the anode and the cathode gases to seal the gas in the cell. Keep the cell in this condition for 10 min while monitoring the pressures in the anode and cathode sides, respectively. Document any leakage out of the cell.

Secondly, pressurize the anode side to 30 kPaG and the cathode side to 0 kPaG simultaneously. Shut off the inlet valves of the anode and the cathode gases to seal the gas in the cell. Keep the cell in this condition for 10 min while monitoring the pressures in the anode and cathode sides, respectively. Document any crossover from the anode to the cathode side.

Thirdly, pressurize the cathode side to 30 kPaG and the anode side to 0 kPaG simultaneously. Shut off the inlet valves of the anode and the cathode gases to seal the gas in the cell. Keep the cell in this condition for 10 min while monitoring the pressures in the anode and cathode sides, respectively. Document any crossover from the cathode to the anode side.

In the above procedures, the pressure drop after 10 min should be less than 5 kPaG on both the anode and the cathode sides.

NOTE If the cell operates at higher pressures, the test pressure is normally roughly equivalent to the operating pressure of the cell.

c) Procedure 3

The gas outlets are closed and the gases are fed to the cell such that the pressure is slowly increasing. Upon reaching a pressure level of, for example, 300 kPa (or 150 % of the maximum operating pressure) on both sides, the gas flow is reduced as long as the pressure is still increasing. The mass flow rate at which the pressure is not changing is defined as the external leakage rate. The pressure at which the external leakage rate is measured shall be ±10 kPa of the adjusted pressure level.

Subsequently, the pressure in one gas compartment is reduced to atmospheric pressure. The outlet of one gas compartment is closed while the other compartment remains open. A gas flow is fed to the closed gas compartment such that the pressure is slowly increasing. When reaching a pressure level of, for example, 130 kPa, the gas flow is reduced as long as the pressure is still increasing. The mass flow rate at which the pressure is no longer increasing is defined as the internal leakage rate. The pressure at which the external leakage rate is measured shall be ±5 kPa.

Annex D
(informative)

Initial conditioning

As an alternative to the MEA supplier's initial conditioning procedure, four examples of conditioning protocols are described below. Users of this document can selectively follow these procedures.

a) Procedure A

1) Set up the test station to operate the cell.
2) Purge both anode and cathode with an inert (e.g. nitrogen) gas. The flow rates should be similar to those used in the subsequent tests and will be used until the cell is purged completely.
3) Heat up the cell to 80 °C using a cell heater or other appropriate heating method. Introduce fully-humidified N_2 gas (100 % RH). The flow rates should be similar to those used in the subsequent test. During warm-up, maintain the cell temperature and inlet and outlet gas piping temperatures higher than the gas dew point at all times in order to prevent water condensation in the system.
4) Wait until the cell temperature and gas humidity are stabilized. Introduce fully-humidified reactant gases at appropriate stoichiometries, for example 1,4 for hydrogen and 2,5 for air, with respect to 1 000 mA/cm^2. Increase the load gradually while keeping the cell voltage above 0,4 V until the current density reaches 1000 mA cm^{-2}.
5) Maintain the load (the current density) at 1000 mA cm^{-2} with stoichiometry of 1,4 for H_2 and 2,5 for air until the cell voltage variability settles to less than 5 mV over 5 h. Fulfilling this criterion indicates the completion of cell break-in.

b) Procedure B

Under the standard operating conditions to be used during the subsequent test, run the cell on pure hydrogen at open circuit voltage (OCV) for 15 min, run at 600 mV for 75 min, then run three cycles consisting of holding at 850 mV for 20 min, followed by 600 mV for 30 min. The total time for conditioning is approximately 4 h.

c) Procedure C

Set up the test station to operate the cell with the standard operating conditions to be used during the subsequent test. In potentiostatic mode, hold the cell at 500 mV for 5 min and run potential cycles at constant gas flow rates from 800 mV to 300 mV in 50 mV steps for 10 s at each step, and then back from 300 mV to 800 mV at the same rate. Hold the cell at 500 mV for 5 min. Run the cycle until the variations of current density at 500 mV are below ±10 mA cm^{-2} in 5 min and the deviation of current density in 3 subsequent cycles is below ±10 mA cm^{-2}.

d) Procedure D

Operate the cell in galvanostatic mode at the selected operating temperature and at the gas conditions given in the standard test conditions. Increase the current density in steps of 100 mA cm^{-2} or at a rate of not greater than 10 mA cm^{-2} sec^{-1}, while keeping the cell voltage higher than 500 mV until reaching the current density identified for the conditioning. The current density for the conditioning of the cell will correspond either to the maximum current possible at 500 mV in the selected conditions or to a current density defined by the specific objective of the test.

Annex E
(informative)

Shutdown

A typical shutdown protocol is described below.

a) Decrease the electrical load to values corresponding to the steps in the polarization curve in the reverse sequence (that is, decrease current density from I_{max} to 0 while keeping the gases flowing). Disengage the electrical load.

b) Purge the anode and the cathode with nitrogen at flow rates corresponding to the lowest polarization curve load step until the cell cools to ambient temperature (e.g. at 4 ml/min/cm^2 active area). During cool-down, maintain the cell temperature and gas piping temperature higher than the gas dew point at all times in order to prevent water condensation in the system.

c) After the cell has reached ambient temperature, switch to dry nitrogen flows on the anode and cathode for 5 min to 10 min (this step is optional, depending on the final level of membrane hydration desired).

d) Disconnect the cell and tightly cap the anode and cathode inlets and outlets.

Annex F
(informative)

Reconditioning protocols

Typical reconditioning protocols are described below.

a) Procedure A:

   1) Repeat steps 1) to 5) of Procedure A in Annex D. One hour should be enough for step 5) for stabilization of the cell conditions.

   2) Specify the test condition(s) appropriate for the test to be carried out and maintain the condition(s) until stabilized.

b) Procedure B:

   1) Supply fully-humidified gases (100 % RH) with a hydrogen stoichiometry of 1,4 and an oxidant stoichiometry of 2,5 through the cell for one hour while heating to 80 °C, using a current density of 400 mA cm^{-2}.

   2) Operate the cell under these conditions for 4 h; the cell is reconditioned when the cell voltage is equilibrated.

Annex G
(informative)

Polarization curve test supplement

Typical current density increments used for the polarization curve test are given below.

If the expected maximum current density is known (e.g. indicated by the manufacturer or from previous measurements), select the current steps as given in Table G.1.

Table G.1 – Current density increments if maximum current density is known

Step	Percentage of expected maximum current density
0	0 (OCV)
1	2 %
2	5 %
3	10 %
4	20 %
5	30 %
6	50 %
7	70 %
8	90 %
9	100 %

More points may be necessary at low current if Tafel slope analysis is of interest.

If the maximum current density is unknown, use the current density increments given in Table G.2.

Table G.2 – Current density increments if maximum current density is unknown

Step	Current density ($A\ cm^{-2}$)	Recommended dwell time (s)
0	0 (OCV)	30
1	0,02	60
2	0,03	60
3	0,04	120
4	0,06	120
5	0,08	120
6	0,1	120
7	0,2	120
8	0,4	120
9	0,6	120
10	0,8	120
11	1,0	120
12	1,2	120
13	1,4	120
14	1,6	120
15	1,8	120
16	2,0	120
NOTE The test is typically terminated when the maximum current density is reached or if the cell voltage goes below 0,3 V or reaches the minimum value recommended by the manufacturer (in order to avoid irreversible damage to the cell components).		

Annex H
(normative)

Applied performance tests

H.1 Gain tests

H.1.1 Hydrogen gain test

H.1.1.1 General

The objective of this test is to evaluate the diffusion capabilities of anodes. A hydrogen gain test is conducted to evaluate MEAs which use reformed gas (a mixture of hydrogen, carbon dioxide, nitrogen and other impurities) for fuel in actual use.

This test can be performed using one of two methods: constant gas stoichiometries or constant gas flow rates. These are described in 11.2.2 and 11.2.3, respectively. Once a method is selected, it shall be used throughout the entire test. The test shall be conducted as set out below.

H.1.1.2 Test method

First, use hydrogen as the fuel on the anode side of the cell at the desired flow rate, humidification and pressure. The CO concentration in the hydrogen gas should be at or below that given in ISO 14687-2.

Measure polarization curves with hydrogen and oxidant (air or oxygen) using the selected method.

Next, use a hydrogen and nitrogen gas mixture as fuel. Here, nitrogen represents the non-hydrogen constituents in the reformed-gas mixture, the composition of which is specified by the manufacturer.

Obtain polarization curves with the hydrogen-nitrogen mixture and the oxidant (air or oxygen) using the selected method.

Compare the polarization curve of hydrogen to that of the gas mixture.

NOTE The reason nitrogen is used instead of carbon dioxide is that carbon dioxide can produce trace amounts of carbon monoxide that are formed by the reaction with hydrogen, which poisons the anode. This test purely evaluates the diffusion capability of the anode.

H.1.2 Oxygen gain test

H.1.2.1 General

The objective of this test is to evaluate the diffusion capabilities of cathodes. An oxygen gain test is conducted to evaluate MEAs which use air for oxidant in actual use.

This test can be performed using one of two methods: constant gas stoichiometries or constant gas flow rates. These are described in 11.2.2 and 11.2.3, respectively. Once a method is selected, it shall be used throughout the entire test. The test shall be conducted as set out below.

H.1.2.2 Test method

Measure polarization curves with air using the selected method.

Then, use oxygen instead of air using the same molar flow rate of oxygen as air, humidification and pressure.

Measure polarization curves with oxygen under identical conditions using the selected method.

Compare the polarization curve of oxygen to that of air.

Crossover leakage using O_2 can result in a rapid exothermic event resulting in hardware damage and, potentially, personal injury.

The O_2 system needs to be designed and cleaned to special requirements.

A mixture of 79 % in mole fraction helium and 21 % in mole fraction oxygen (helox) may be used to decouple the effects of oxygen concentration on diffusion and kinetics. See Annex J for further information.

H.2 Gas stoichiometry tests

H.2.1 Fuel stoichiometry test

H.2.1.1 General

The objective of this test is to evaluate the diffusion capabilities of anodes, as in H.1.1 (Hydrogen gain test).

H.2.1.2 Test method

Set the current density as specified under the standard test conditions.

Set the oxidant stoichiometry to be 1 to 4 times that given in the standard test conditions.

Change the fuel flow rate within a certain range as specified by the cell manufacturer and record the cell voltage.

Care should be taken with this test since it intentionally stresses the anode by almost starving it. Starvation can cause irreparable damage to the anode.

H.2.2 Oxidant stoichiometry test

H.2.2.1 General

The objective of this test is to evaluate the diffusion capabilities of cathodes, as in H.1.2 (Oxygen gain test). The test shall be conducted as set out below.

H.2.2.2 Test method

Set the current density as specified under the standard test conditions.

Set the fuel stoichiometry to be 1 to 2 times that given in the standard test conditions.

Change the oxidant flow rate in a certain range as specified by the cell manufacturer and record the cell voltage.

Care should be taken with this test since it intentionally stresses the cathode by almost starving it.

H.3 Temperature effect test

H.3.1 General

The objective of this test is to measure the effect of cell temperature on cell performance. Temperature is expected to affect the electrode reaction rate and electrolyte conductivity.

H.3.2 Test method

Set the cell temperature at T_1, T_2,... T_n where the values of T_1, T_2,...T_n are specified by the cell manufacturer and T_n is the maximum operating temperature.

Additionally, keep anode and cathode relative humidities at the fuel cell operating temperature constant by increasing/decreasing the dew points and gas inlet temperatures.

At each temperature level, obtain the polarization curve of the cell. The procedure given in 11.2 may be used.

H.4 Pressure effect test

H.4.1 General

The objective of this test is to measure the effect of fuel and oxidant pressures on cell performance. Care should be taken to keep the pressure difference across the membrane constant. High pressure increases the density of reactant gases, which is expected to increase the electrode reaction rate.

H.4.2 Test method

Set the fuel or oxidant pressure at p_1, p_2, ... p_n, where the values of p_1, p_2, ... p_n are specified by the cell manufacturer and p_n is the maximum operating pressure.

Simultaneously, change the other pressure accordingly to keep the pressure differential constant.

At each pressure level, obtain the polarization curves of the cell. The procedure given in either 11.2.2 or 11.2.3 may be used.

Care should be taken so as not to unintentionally damage the cell or membrane. The testing personnel should be alert for indications of leaks through the membrane, such as pinholes, which may lead to internal ignition.

H.5 Humidity effect tests

H.5.1 Fuel humidity effect test

H.5.1.1 General

The objective of this test is to measure the effect of varying the humidity of fuel on cell performance. Humidity in fuel is expected to affect the electrolyte conductivity as well as gas diffusion into anodes.

This test can be performed using one of two methods: constant gas stoichiometries or constant gas flow rates. These are described in 11.2.2 and 11.2.3, respectively. Once a method is selected, it shall be used throughout the entire test. The test shall be conducted as set out below.

H.5.1.2 Test method

Set the oxidant humidity at the standard test conditions.

Set fuel humidity at several levels corresponding to the desired dew point temperatures and obtain polarization curves.

H.5.2 Oxidant humidity effect test

H.5.2.1 General

The objective of this test is to measure the effect of varying the humidity of the oxidant on cell performance. Humidity in the oxidant is expected to affect the electrolyte conductivity as well as gas diffusion into cathodes.

This test can be performed using one of two methods: constant gas stoichiometries or constant gas flow rates. These are described in 11.2.2 and 11.2.3 respectively. Once a method is selected, it shall be used throughout the entire test. The test shall be conducted as set out below.

H.5.2.2 Test method

Set the fuel humidity at the standard test conditions.

Set the oxidant humidity at several levels corresponding to the desired dew point temperatures and obtain polarization curves.

H.6 Limiting current test

H.6.1 General

The rate of gas diffusion through electrodes has a significant influence on the performance of the MEA. The magnitude of the limiting current density is an indication of the gas diffusion capability of the MEA.

The objective of this test is to identify the mass transport limitation within an MEA.

H.6.2 Test method

Set the fuel and oxidant flow rates to correspond to the standard stoichiometries at the rated current density specified by the cell manufacturer.

Increase the current step by step in small increments while maintaining the fuel and oxidant stoichiometries constant (that is to increase fuel and oxidant flow rates step by step).

Record the cell voltage at each step.

When the cell voltage sharply drops to near, but not to, 0 V, record the current, and decrease the current at once so as not to damage the MEA. Use the extrapolated value to 0 V as the limiting current.

For this method, caution should be exercised to prevent the cell voltage from reaching 0 V or below, which may cause irreversible degradation to the MEA. Also precautions should be taken to use load cables capable of handling the limiting current.

H.7 Overload test

H.7.1 General

Overload operation for a short duration may be required depending on the application of the fuel cell (e.g. vehicle application), and the overload durability (magnitude of overload and duration of time) is affected by the catalyst activity and gas diffusion performance of the electrodes.

The objective of this test is to evaluate the electrical overload durability of a cell.

H.7.2 Test method

Set the current at more than the rated and less than the limiting current, which are specified by the cell manufacturer.

Set the fuel and oxidant flow rates to correspond to the standard stoichiometries according to the set current.

Operate the cell for a period of time specified by the cell manufacturer or until the cell voltage drops to the value specified by the cell manufacturer.

Record the cell voltage during operation.

If necessary, repeat the above test procedure by changing the set current value.

The minimum cell voltage specified by the cell manufacturer should be a voltage that does not cause irreversible damage to the MEA.

H.8 Subzero storage test

H.8.1 General

The objective of this test is to investigate the effect of storage at subzero temperatures on performance degradation.

Since the residual water content in the MEA after shutdown has a significant impact on the performance degradation due to the freeze/thaw cycle, it is recommended to carry out this test with a shutdown procedure designed to simulate realistic operation in a fuel cell system.

H.8.2 Test method

Operate the cell under the standard test conditions specified by the manufacturer.

Measure the polarization curve and internal resistance according to 11.2 and 11.6, respectively.

Shut down the cell with the shutdown procedure specified by the manufacturer.

Place the fuel cell in a climate chamber for subzero storage. Set the temperature as the subzero storage protocol requires. Maintain the temperature for at least 12 h.

Bring the cell to the standard test conditions and maintain these for at least 12 h.

Operate the cell as required by the manufacturer under the standard test conditions and measure the polarization curve and internal resistance according to 11.2 and 11.6 respectively.

Repeat the procedure described above twice.

The performance decay can be determined by comparing the polarization curve and internal resistance before subzero storage with those after the test.

H.9 Subzero start test

H.9.1 General

The objective of this test is to evaluate the cold start capability of a cell in a subzero temperature environment.

Since the residual water content in the MEA, the GDLs and flow plates of a cell after shutdown affects the cell's cold start capability, it is recommended to perform the cold start test with different shutdown processes in order to identify an appropriate shutdown procedure for cold starts.

H.9.2 Test method

Operate the cell under the standard test conditions.

Shut down the cell with the shutdown procedure specified by the manufacturer. If one is not available, an example is provided in Annex E.

Place the cell in a climate chamber in which the cell is started up at a subzero temperature.

Set the chamber temperature as required by the subzero startup protocol, and maintain the temperature for at least 12 h.

Start up the cell in the climate chamber at the subzero temperature, as specified by the manufacturer.

It is necessary to control the output current so that the cell voltage does not fall below the voltage specified by the manufacturer or 0,3 V if not specified, to avoid unnecessary damage to the cell components.

Run the cell at the rated voltage specified by the manufacturer for 10 min at the subzero temperature.

Repeat the procedure described above twice.

Under the standard test conditions, test the cell as described in 11.2 and 11.6.

NOTE An example of the test report template for a subzero start test is given in Annex K.

H.10 Membrane swelling test (humidity cycle test)

H.10.1 General

The objective of this test is to evaluate the mechanical durability of a membrane during a humidity cycle test.

H.10.2 Test conditions

Test specimen size (permeable cross section): 10 cm^2 to 50 cm^2 (depending on cell geometry)

Sample setup:	single cell (MEA and flow plate)
Sample number:	N = 3
Temperature:	80 °C
Ambient gas:	air (nitrogen may also be used)
RH:	0 % to 150 %

NOTE Relative humidity levels over 100 % can be obtained by setting the humidifier temperature above the cell temperature. In this case liquid water will be formed in the cell by condensation.

Cycling: 4 min (2 min dry, followed by 2 min wet)

Flow rate: a typical flow rate such as 2 l/min (at normal conditions) for 25 cm^2 electrodes for both the anode and the cathode

Measurement frequency for crossover: initially every 67 h (1 000 cycles), and every 24 h (360 cycles) after increase of leak rate is detected.

H.10.3 Test method

Use equipment that allows for the dry and wet lines to be automatically switched, with a three-way valve at the joint of the dry and wet lines.

It is recommended to monitor the changes of relative humidity at the outlet.

Supply air (or nitrogen) with different humidity levels to both sides of the MEA periodically.

Replace one side with hydrogen every 24 h to measure the crossover. The level of crossover shall be measured by gas chromatography or a hydrogen crossover method (11.5.2).

Testing is completed when hydrogen crossover rate (crossover current) is more than ten times the initial value or when 20 000 cycles have been completed, whichever is earlier.

High-frequency resistance should be measured during the test.

H.11 Open circuit voltage (OCV) test

H.11.1 General

The objective of this test is to evaluate the chemical degradation of an MEA at OCV conditions.

H.11.2 Test conditions

Temperature:	90 °C
Gas pressure:	atmospheric pressure
Relative humidity (RH):	30 %
Anode:	H_2
Cathode:	air

H.11.3 Test method

Conduct a hydrogen crossover test in accordance with 11.5.2.

Start an OCV test at the test conditions above.

Continuously monitor the voltage during testing. Conduct diagnostic testing every 10 h to 100 h by measuring hydrogen crossover leakage (80 °C). Measure the fluoride concentration at the outlets of the anode and the cathode at the time of the interim measurements as required.

NOTE For measuring F ion concentration, suggested techniques include ion selective electrode (ISE), inductively coupled plasma - mass spectroscopy (ICP-MS), and liquid chromatography (LC) methods.

Terminate the test when the hydrogen crossover rate exceeds ten times its initial value or when 500 h has been reached, whichever is earlier.

After the test, the MEA may be subjected to the following analyses:

- membrane thickness measurement,
- observation of platinum bands within the membrane, and
- changes in the membrane molecular weight and molecule structure.

H.12 Oxygen reduction reaction (ORR) activity test

H.12.1 General

The objective of this test is to evaluate the ORR activity of a cathode using an MEA.

H.12.2 Test conditions

Temperature:	80 °C
Gas pressure:	150 kPa (absolute) (or atmospheric pressure)
RH:	100 %
Anode:	H_2
Cathode:	O_2

H.12.3 Test method

Conduct an ECA test and hydrogen crossover test to measure the electrochemical surface area and crossover current density in accordance with 11.5.3 and 11.5.2 respectively.

Measure the polarization curve and cell resistance (IR) according to the following procedures (see also Figure H.1):

1) before switching to oxygen, make sure that the cell voltage is near 0 V for more than 30 s to ensure repeatable platinum surface for the measurement;
2) replace nitrogen with oxygen and keep the cell at OCV state for 2 min;
3) measure the polarization curves and the IR, focusing on lower current densities.

Calculate the IR-free voltage at each current density measured with the following equation:

$$V_{\text{IR-free}} = V_{\text{measured}} + R_{\text{ASR}} \times I$$

where

$V_{\text{IR-free}}$ is the IR-free voltage (V);

$V_{measured}$ is the measured voltage (V);

R_{ASR} is the area-specific resistance measured ($\Omega \cdot cm^2$);

I is the applied current density (A/cm^2).

Calculate the total current density with the following equation:

$$I_{total} = I + I_{coc}$$

where

I_{total} is the sum of the applied and the crossover current densities (A/cm^2);

I_{coc} is the crossover current density (A/cm^2).

Create a Tafel plot, in which the horizontal axis is the total current density and the vertical axis is the IR-free voltage (see also Figure H.2).

Derive the total current density at 0,9 V (IR-free) by either interpolation or extrapolation of the Tafel plot.

NOTE This condition is selected to minimize the transport losses associated with protons and oxygen, and, at the same time, control the reproducibility of the effect of platinum oxide on ORR.

Finally, calculate the mass activity of 1 g of platinum catalyst (A/g) of the cathode with the following equation:

$$A_{mass} = I_{0,9V} / L_{pt}$$

where

A_{mass} is the mass activity of the cathode (A/g);

$I_{0,9V}$ is the total current density at 0,9 V (IR-free voltage) (A/cm^2);

L_{pt} is the platinum loading of the cathode (g/cm^2).

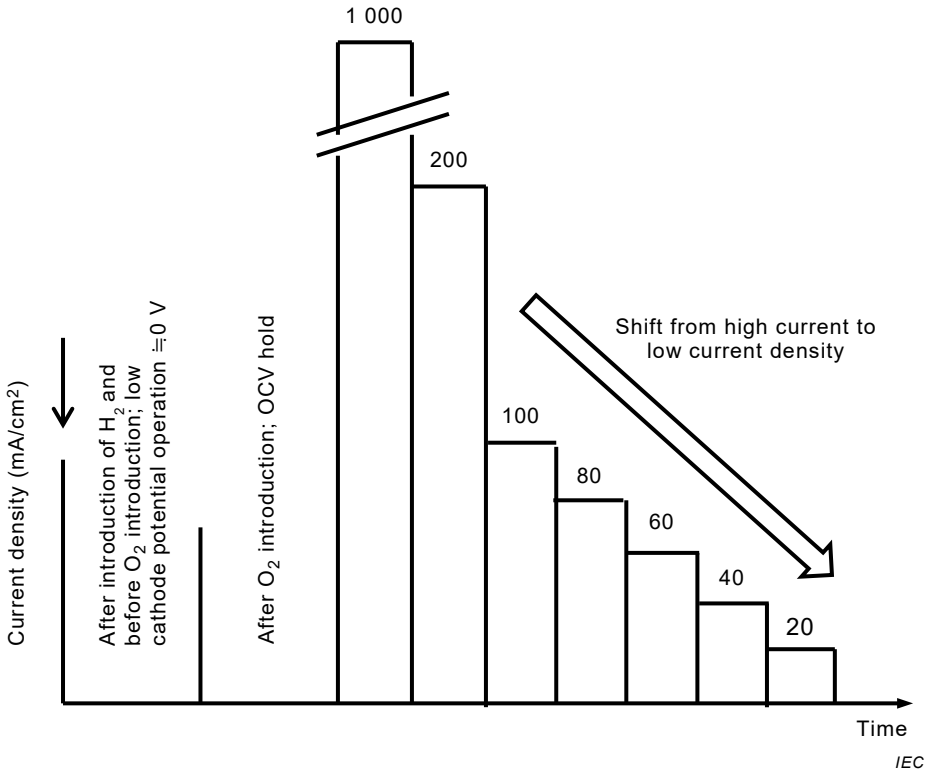


Figure H.1 – ORR activity test procedure

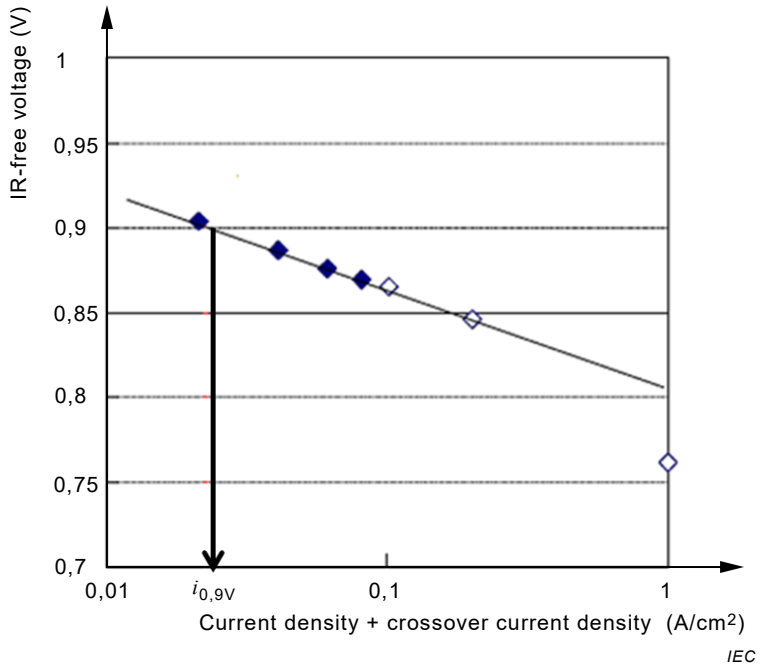


Figure H.2 – Example of Tafel plot

H.13 Fuel composition test

H.13.1 General

The objective of this test is to measure the effect of the composition of reformed gas on cell performance in order to check the electrode capability for different types of fuels. Reformed gas contains hydrogen, CO, CH_4 or traces of other raw fuels and inert gases such as CO_2 and/or N_2, the ratio of which depends on the difference in raw fuels and reforming methods. The inert gas is expected to affect the diffusion of hydrogen into the electrodes.

This test can be performed using one of two methods: constant gas stoichiometries or constant gas flow rates. These are described in 11.2.2 and 11.2.3, respectively. Once a method is selected, it shall be used throughout the entire test. The test shall be conducted as set out below.

H.13.2 Test method

Using the standard fuel, obtain polarization curves using the selected method.

Change the standard fuel to another fuel which has a different composition to the standard gas.

Obtain polarization curves using the selected method.

H.14 Cycling tests

H.14.1 Start/stop cycling test

H.14.1.1 General

The objective of the start/stop cycling test is to determine the evolution of the performance of a cell operating under specified conditions as a function of a start/stop profile count.

This may be used as a specific test to qualify the lifetime of MEAs for a specific application with adapted operating conditions.

Optionally, gas flow and temperature control can be part of this test.

Start/stop profiles and operating duration are specified by the cell manufacturer. A typical start/stop cycling profile and its operating duration are given in Annex L.

H.14.1.2 Test method

After operating the cell at 100 % load (rated current density) for a specified period of time, switch off the load (open circuit) for a specified period of time.

After that, switch on the load and operate at 100 % load.

Repeat the procedure while recording the cell voltage.

The performance decay per cycle can be calculated by dividing the voltage drop by the cycle number.

NOTE Operating a fuel cell at open circuit voltage for long periods of time can accelerate the degradation of the electrode materials.

H.14.2 Load cycling test

H.14.2.1 General

The objective of the load cycling test is to determine the evolution of the voltage of a fuel cell operating under specified conditions as a function of current density following a dynamic profile versus time.

This may be used as a specific test to qualify the lifetime of MEAs for a specific application with adapted operating conditions.

A typical load profile and its operating duration are given in Annex M.

H.14.2.2 Test method

After operating a cell at 100 % load (rated current density) for a defined period of time, switch the load from 100 % to a partial load with the gas stoichiometries maintained constant and operate for a defined period of time at a partial load. Load profile and operating duration are specified by the cell manufacturer.

After that, increase the load again to 100 %.

Repeat this procedure while recording the cell voltage.

The performance decay per cycle can be calculated by dividing the voltage drop by the cycle number.

H.14.3 Potential cycle test (start/stop durability)

H.14.3.1 General

The objective of the potential cycle test is to evaluate the durability of the catalyst against startup and shutdown cycles by cycling the potential under accelerated conditions.

H.14.3.2 Test conditions

Temperature: 80 °C

Gas pressure: atmospheric

RH: 100 %

Anode: H_2

Cathode: N_2

H.14.3.3 Test method

Conduct an ECA test to measure the electrochemical surface area in accordance with 11.5.3.

Once the voltammogram is reproducible, start a potential cycle test using the test conditions and protocol as illustrated in Figure H.3.

During the test, conduct an ORR activity test (H.12), polarization curve measurement (11.2) and ECA measurement (11.5.3) at appropriate intervals.

Continue to test until the electrochemical surface area has decreased to a predetermined value (e.g. 40 % of its initial value) or a predetermined number of cycles (e.g. 60 000 cycles) has been reached.

Measure CO_2 emissions at the outlet of the cathode as needed.

After the test, the MEA shall be subjected to such analyses as corrosion of the catalyst support, degree of elution/oxidation of the catalyst, and changes in the particle diameters of the catalyst.

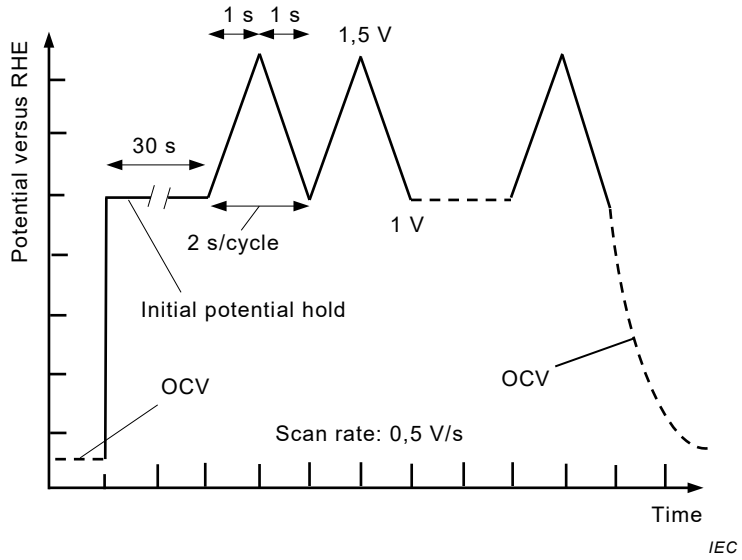


Figure H.3 – Potential cycle test (start/stop durability) procedure

H.14.4 Potential cycle test (load cycle durability)

H.14.4.1 General

The objective of this test is to evaluate catalyst durability against load cycling by conducting a potential cycling test under accelerated conditions.

H.14.4.2 Test condition

Temperature: 80 °C

Gas pressure: atmospheric

RH: 100 %

Anode: H_2

Cathode: N_2

H.14.4.3 Test method

Measure the ECA value in accordance with 11.5.3.

Once the voltammogram is reproducible, start a potential cycle test using the test conditions and protocol as illustrated in Figure H.4.

During the test, conduct an ORR activity test (H.12), polarization curve measurement and ECA measurement (11.5.3) at appropriate intervals.

Continue the test until the ECA comes down to 50 % of the initial value or a predetermined number of cycles.

After the test, the MEA shall be subjected to such analyses as corrosion of the catalyst support, amount of elution/oxidation of the catalyst metal and change in the particle diameters of the catalyst.

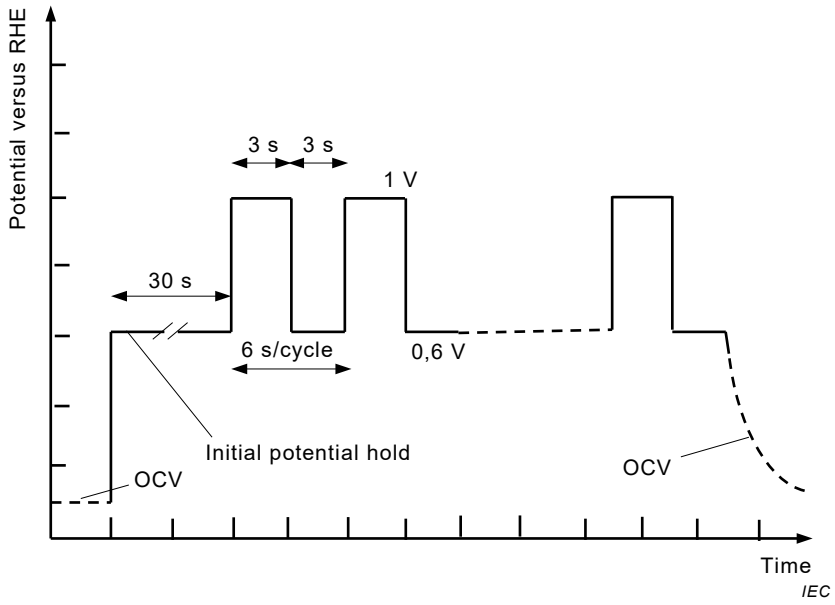


Figure H.4 – Potential cycle test (load cycle durability) procedure

H.15 Impurity influence tests

H.15.1 Influence at rated current density

H.15.1.1 General

The objective of this test is to determine the influence of impurities in the fuel or in the air on cell performance and the degree of recovery of the cell performance from damage at the rated current density. A cell is subjected to steady-state tests and operated with fuel or air containing some impurities.

The test shall be conducted at several levels of impurity in the fuel or in the air in order to identify how impurities affect the performance and to determine the highest impurity level that does not affect the cell performance.

ECA measurement (11.5.3) is recommended as an electrochemical diagnosis before and after this test.

NOTE Impurities in hydrogen are given in ISO 14687-2.

H.15.1.2 Test method

Operate the cell at the rated current density with high-purity fuel and clean air until the cell voltage is stabilized within ±5 mV for 15 min.

Change the high-purity fuel or clean air to an impurity-containing fuel or air with the impurity species and their quantities specified by the cell manufacturer, by the system specifications, or by the targeted application specifications.

Run the cell until the cell voltage is stabilized within ±5 mV for 15 min and record the cell voltage.

Then, change the impurity-containing fuel or air to the high-purity fuel or clean air.

Run the cell until the cell voltage is stabilized within ±5 mV for 15 min and record the cell voltage.

Tolerance can be evaluated based on the differences in cell voltage between clean and polluted gas operation, while reversibility can be determined from the differences between initial and final values under clean reactants.

Some impurities, such as H_2S, can react with the exposed surfaces of components of the test station (for example, gas supply piping and gaskets). Care should be taken to ensure that the test station materials are compatible with the impurities being used. The piping needs to be flushed with inert gas or pure hydrogen after the test in order to minimize the presence of impurities for further testing.

H.15.2 Influence on polarization curves

H.15.2.1 General

The objective of this test is to determine the influence of impurities in fuel or air on the polarization curve of a cell. The test shall be conducted at several levels of impurity in order to identify how impurities affect the performance and to determine the highest impurity level in fuel or air that does not affect the cell performance.

H.15.2.2 Test method

Measure the polarization curve using high-purity fuel and clean air at a constant gas stoichiometry or at a constant gas flow rate as in 11.2.2 or 11.2.3, respectively.

Change high-purity fuel or clean air to an impurity-containing fuel or air. Obtain the polarization curve of the cell using the same method as that used in the measurement with high-purity fuel and clean air.

H.15.3 Long-term impurity influence test

H.15.3.1 General

The objective of this test is to determine the influence of impurities in fuel or air on long-term fuel cell operation.

This test shall be carried out with fuel or air of the highest impurity level or the lowest level that does not affect the cell performance as determined by the rated current density in 11.3.2a) (steady-state test) or by 11.2 (polarization curve test) in order to study the cumulative effect of impurities during long-term operation.

H.15.3.2 Test method

Operate the cell at the rated current density with high-purity fuel and clean air until the cell voltage is stabilized within ±5 mV for 15 min.

Change the high-purity fuel or clean air to an impurity-containing fuel or air with the impurity species and their quantities specified by the cell manufacturer or by the system specifications, or by the targeted application specifications or system manufacturer, etc.

Run the cell for a minimum of 1 000 h for transportation applications and for 5 000 h for stationary applications.

Record the cell voltage during operation. If desired, obtain the polarization curve and internal resistance of the cell under the standard test conditions at regular intervals.

The basic procedure is given in either 11.2 for polarization curve measurement or in 11.6 for internal resistance measurement.

The average performance decay can be calculated as described in 11.4. It is recommended to add the number of cycles and duration of the test.

Annex I
(informative)

Test report for polarization curve tests

I.1 General

A suggested template for a report of polarization curve tests is given below.

I.2 General information

I.2.1 General information on the test report

Test report reference / identification	
Test report title	
Authors	

I.2.2 General information concerning the test

Test procedure number		Test date	
Test version		Company performing test	
Company requesting test		Test location	
Test request number		Test cell / equipment	

I.3 Introductory remarks

Here the authors should refer to:

- the procedure employed and, if relevant, the reasoning behind the choice of such procedure;
- the test plan between tester and customer which may also include acceptance criteria; and
- any other documentation used in the report or in the test (terminology document, symbol harmonization, etc.).

I.4 Objective and scope of the test

The objective is to determine the polarization curve of a PEFC single cell operating under specified operating conditions.

The cell performance is measured from open circuit voltage to the highest current density that has been determined by:

- the properties of the cell components;
- the specifications of the intended application;
- the measurement method.

The objectives of the test are to quantify or qualify:

- the generic performance of a single polymer electrolyte fuel cell;
- PEFC components such as MEAs or sub-components of MEAs and bipolar plate materials or design.

The operating conditions considered for this test correspond to:

- the standard conditions used by members of the scientific community;
- the intended application.

I.5 Description of cell components

Cell manufacturer	
Cell model	
Product or article tested	
Product number	
Test sample identity number	

Fuel cell: material of flow plates	
Fuel cell: flow field design	
Fuel cell: active area	
Gasket type	
Gasket thickness	
Current collector material	
Cell clamping force	
Heating/cooling system	
Orientation of the cell [a] (vertical versus horizontal)	
Gas flow direction (co-flow, counter flow, etc.)	
[a] If needed, include a drawing for better understanding.	

MEA assembly (Yes/No, 3 Layers, 5L, 7L)	
Electrodes	
Gas diffusion layers (thickness and type)	
Catalyst layers (loading and composition)	
Membrane (thickness and type)	

Lowest cell voltage allowed (V)	
Pressure difference allowed between anode and cathode (kPa)	
Manufacturer-recommended air stoichiometry	

Provide additional remarks or information from the manufacturer on the cell or the MEA.

I.6 Background

The author describes the testing history of the cell with a brief description of all diagnostic experiments, specific or baseline experiments and their respective identifiers in sequential order.

I.7 Description of the test setup

A detailed description of the test equipment and setup that were employed, including sensor types and locations and specific devices (for example heating/cooling and humidification sub-systems) should be given here in the test report to assist in the understanding of results.

I.8 Description of operating conditions, inputs and outputs

Table I.1 lists all the test inputs that are controlled during the test, while Table I.2 lists all the test outputs. Measurement uncertainties and sampling rates are to be entered as well. The testing personnel complete the input value column for each test.

Table I.1 – Test input parameters

Input	Description	Unit	Input value	Measurement uncertainty	Sampling rate Hz	Control accuracy
I	Current density (i = applied current / active geometric area)	A cm^{-2}				
T_{amb}	Ambient temperature	°C				
T_c	Cell temperature	°C				
X_{fuel}	Fuel composition	% H_2; % other gases			-	
X_{ox}	Oxidant composition	Air or O_2; % other gases			-	
p_{H2}	Fuel pressure at cell inlet or outlet [c] port	kPa				
p_{Air}	Oxidant pressure at cell inlet or outlet[c] port	kPa				
Q_{fuel}	Fuel flow rate [a] (at STP)	cm^3 min^{-1}				
Q_{ox}	Oxidant flow rate [a] (at STP)	cm^3 min^{-1}				
$Q_{fuel,min}$	Fuel minimum flow rate	cm^3 min^{-1}				
$Q_{ox,min}$	Oxidant minimum flow rate	cm^3 min^{-1}				
λ_{fuel}	Fuel stoichiometry	(dimensionless)		-	-	
λ_{ox}	Air stoichiometry	(dimensionless)		-	-	
RH_{fuel}	Fuel relative humidity [b]	%				
RH_{ox}	Oxidant relative humidity [b]	%				
T_{fuel}	Fuel dew point	°C				
T_{ox}	Oxidant dew point	°C				
Tb_{fuel}	Fuel bubbler temperature	°C				
Tb_{ox}	Oxidant bubbler temperature	°C				
Tl_{fuel}	Fuel line temperature	°C				
Tl_{ox}	Oxidant line temperature	°C				
P_{amb}	Ambient pressure	kPa				
RH_{amb}	Ambient relative humidity	%				

[a] $Q_{\lambda fuel}$ and $Q_{\lambda ox}$ are respectively the stoichiometry controlled volumetric flow rates of fuel and oxidant. The actual volumetric flow rates used during the test are the stoichiometry controlled flow rates, unless the values are smaller than the minimum flow rates: $Q_{fuel,min}$ and $Q_{ox,min}$.

[b] Relative humidity is calculated based on the cell temperature.

[c] State in the test report which option was selected and whether the cell inlet or outlet pressure was controlled to be constant.

Table I.2 – Test output parameters

Output	Description	Unit	Instrument uncertainty	Sample rate Hz
V	Cell voltage	V		
P	Cell power density	W cm^{-2}	Calculated	
NOTE In the case where the test is performed by varying the voltage in the range of OCV to minimum cell voltage, I will be the test output parameter instead of V.				

I.9 Test procedure and results

I.9.1 Description of startup and conditioning

The following information should be included (see Table I.3):

- detailed description of the setting up of the conditions;
- measurements (description, tables or figures showing the inputs and outputs during startup and conditioning).

Table I.3 – Cell performance during startup and conditioning

Dwell time min	Current density A cm^{-2}	Average cell voltage over the last XX min (V); Standard deviation (±V)	Average cell power density W cm^{-2}

Figures should include the primary test inputs and outputs during the measurement in order to facilitate the interpretation of the main results.

For the polarization curve: I, V, T_c, p_{fuel}, p_{oxl}, Q_{fuel}, Q_{ox}, RH_{fuel} and RH_{ox} (or relevant inputs related to RH), t (time).

Use average values and indicate standard deviations.

I.9.2 Description of shutdown (when relevant)

A complete description of the shutdown procedure should be included.

I.9.3 Description of measurement and results

The following information should be included (see Table I.4):

- setting up of the test conditions (initial test inputs) if an additional procedure is performed after the conditioning and before setting the conditions for OCV;
- measurements (description, tables or figures showing the inputs and outputs during the measurement) (e.g. a table of dwell time, and the current density, cell voltage and power for the polarization curve).

Table I.4 – Cell performance during test

Dwell time min	Current density A cm^{-2}	Average cell voltage V (within 2 standard deviations[a])	Average cell power output W cm^{-2} (within 2 standard deviations[a])

[a] See ISO/IEC Guide 98-3 for guidance to estimate the uncertainties.

Figures should append the following primary test inputs and outputs during measurement in order to facilitate interpretation of the main results:

T_c, p_{fuel}, p_{ox}, Q_{fuel}, Q_{ox}, RH_{fuel} and RH_{ox} (or relevant inputs related to RH), and t (time).

I.9.4 Deviation from test procedures

Deviation from any test procedures arising from the operator or equipment should be recorded.

I.10 Data post-processing

Procedures used to calculate any parameters from experimental data should be described.

I.11 Conclusion and acceptance criteria

Here the results of the test should be commented in consideration of the objective of the test and the acceptance criteria, if available.

Annex J
(informative)

Polarization curves in helox

Polarization curves obtained with helox (79 % helium, 21 % oxygen) at the cathode can provide useful diagnostic information on rates of mass transport in diffusion media. Since diffusion of oxygen in helium is significantly faster than it is in air, the onset of mass transport limitation occurs at higher current densities. Comparisons of performance in air and helox (such as the example in Figure J.1) can be used to identify the nature and location of sources of mass transport limitation, such as GDL or catalyst layer flooding.

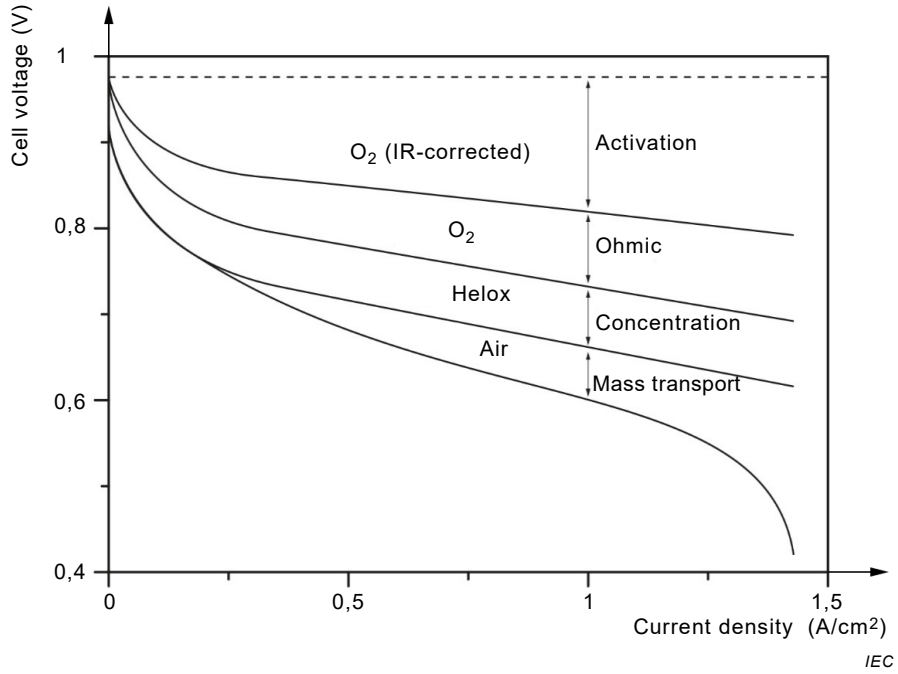


Figure J.1 – Illustration of losses identified by comparison of polarization curves in oxygen, helox and air

Annex K
(informative)

Test report for subzero start test

A suggested template for the test report of a subzero start test is given below (see Table K.1 and Table K.2).

Table K.1 – Energy consumption, gas consumption and heat balance data during subzero startup

Item	Parameter	Measured value
Subzero startup time	Startup time	
Energy consumption during subzero startup	External electric energy input	
	External heat input	
Gas consumption	Fuel flow rate	
	Oxidant flow rate	
	Temperature difference between input and output fuel	
	Temperature difference between input and output oxidant	

Table K.2 – Cell characteristics comparison before and after subzero testing

	Parameter	Before subzero	After subzero	Difference %
Safety characteristics				
1	Gas leakage			
2	Hydrogen crossover			
Operating characteristics				
1	Nominal power output			
2	Peak power output			

Annex L
(informative)

Start/stop cycling test supplement

A typical start/stop cycling profile and its operating duration are described below.

The cycling will follow the following profile:

a) “off” phase = 15 min at 0 A cm^{-2};
b) “on” phase = 15 min at I_{st} A cm^{-2} (I_{st}: rated current density).

It is recommended to increase the current density step by step in order to avoid significant voltage drops. The current should be increased from 0 to I_{st} as follows:

a) 25 % I_{st} A cm^{-2} for 10 s;
b) 50 % I_{st} A cm^{-2} for 10 s;
c) 75 % I_{st} A cm^{-2} for 10 s;
d) I_{st} A cm^{-2} for 14 min 30 s.

The duration of this step depends on the specific objective of the test and on the related specific “ending” criteria: a fixed time frame or fixed performance losses (such as power output, voltage on load or open circuit voltage).

Apart from the different possible ending criteria specifically defined, the test should be terminated (the gases, temperature controllers and load are to be turned off) if the cell voltage goes below 0,3 V or the minimum value recommended by the manufacturer (in order to avoid irreversible damage to the cell components).

Annex M
(informative)

Load cycling test supplement

Typical load cycling profiles and test durations are given below.

Two current density profiles that allow a cell to operate between two current density levels are proposed: one is more dynamic with the profile lasting 1 min, and the other less so with the profile lasting 1 h.

The current density is fixed at I_{st} (rated current density) for the first high-power phase in order to stabilize the operating conditions before starting the cycling phase. Then the load cycling should be performed with one of the two current density profiles shown in Figures M.1 and M.2

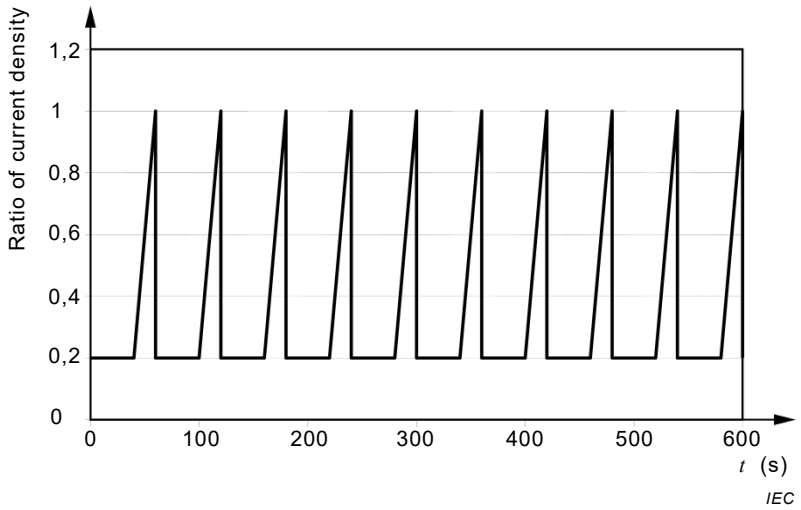


Figure M.1 – Dynamic load cycling profile.

The profile is divided into two sections: the low current density phase = 40 s at 0,2 I_{st} and the high current density phase = current ramp from 0,2 I_{st} to I_{st} for a duration of 20 s.

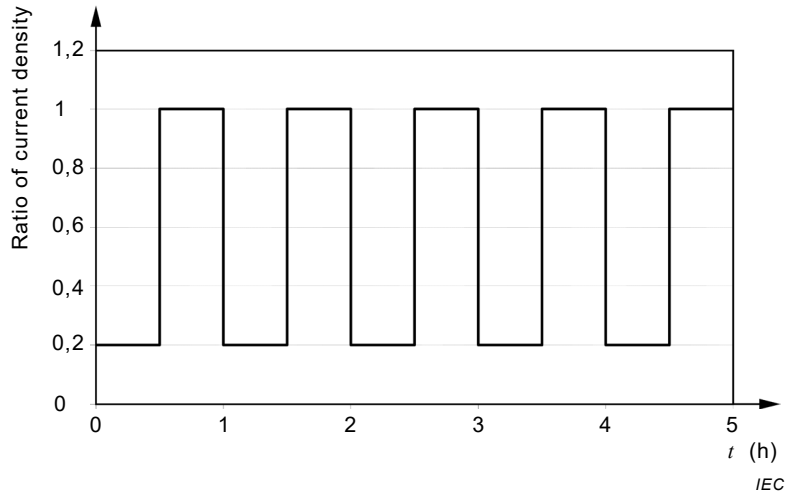


Figure M.2 – Second dynamic load cycling profile

The profile is divided into two sections: the low current density phase = 30 min at 0,2 I_{st} A cm^{-2} (0,2 I_{st}: 20 % current density of I_{st}); and the high current density phase = 30 min at I_{st} A cm^{-2}.

Operating time can be fixed between 500 h and 10 000 h depending on the operating conditions and on the specific application considered.

Apart from the fixed operating duration, other specific ending criteria can be specified depending on the objective of the test, such as acceptable fixed performance losses (including power output, voltage at low or high power, or open circuit voltage).

Apart from the specifically defined ending criteria, the test should be terminated (gases, temperatures and load are to be turned off) if the cell voltage goes below 0,3 V or the minimum voltage value recommended by the manufacturer (in order to avoid irreversible damage to the cell components).

In the case in which the test is performed by varying the voltage in the range of OCV to the minimum cell voltage, I will be test output parameter instead of V.

Another profile shown in Figure M.3 is based on the actual power demand of a road vehicle during city driving. The 100 % power point is to be determined based on the result of the initial power calibration curve and the pre-agreed limits of the cell and/or test stand designs. It is acceptable to determine the partial power points from this curve and then run the test using the corresponding current levels as the set points.

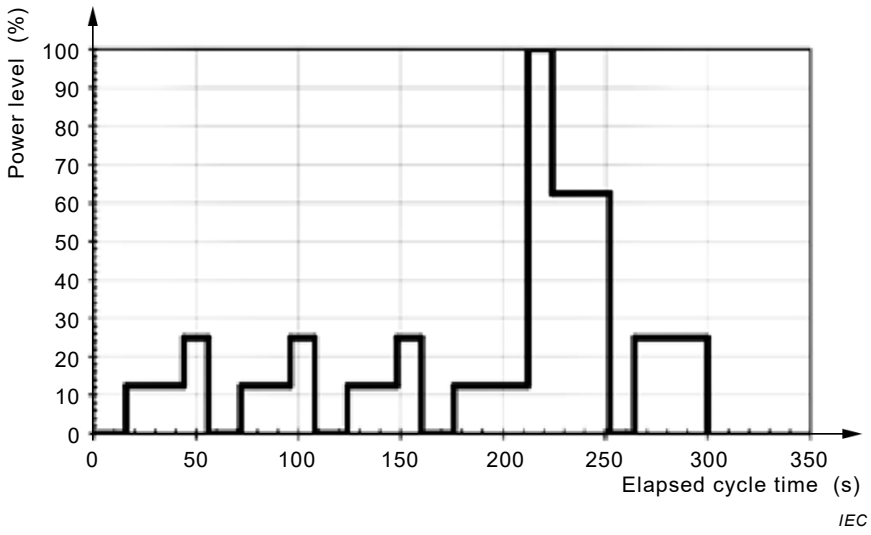


Figure M.3 – Dynamic load cycling based on road vehicle driving

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