

**Standard Operating Procedure
(SOP)**

Gas Permeability

Test ID # LTE-P-8

Rev 2

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Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
04/16/2019	All	First Release
09/19/2019	All	Edits and clarifications

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3. Procedures

a. Scope and Applicability – This SOP provides a standard procedure for the measurement of gas permeability of an AEM. An alkaline water electrolyzer decomposes water at the cathode to hydrogen and HO^- , and the latter migrates through the AEM, liberating oxygen at the anode. During the course of the electrochemical reaction, low gas permeation of hydrogen and oxygen through the membrane is crucial to run the process efficiently. Hydrogen and oxygen are the gases of the interest for the purpose.

b. Summary of Method – The gas permeation rate can be measured using (A) pressure permeation cell,¹ (B) electrochemical monitoring cell,² (C) microelectrode³ and (D) *in situ* measurement in MEA.⁴ The process relies on a chronoamperometric technique to accurately measure the gas permeation rate across the membrane.

c. Definitions –

AEM – Anion exchange membrane

EMT – Electrochemical monitoring techniques

MEA – Membrane electrode assembly

PPE – Personal protective equipment

d. Health & Safety Warning – During the measurement, wear appropriate PPE, including protective eyewear, gloves and lab coats, in the laboratory.

e. Cautions – Avoid inhalation of vapor or mist. Ensure adequate ventilation. Remove all sources of ignition, including open flame, smoking or electrostatic charge. Oxygen cylinders must be separated from hydrogen or combustible materials. Keep cylinders tightly closed in a dry and well-ventilated place. Avoid heat, open flames, and sparks.

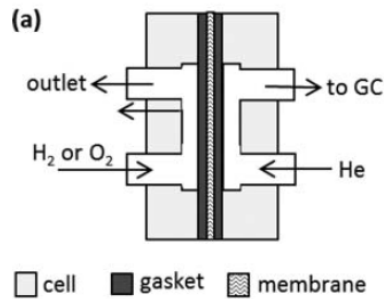
f. Interferences – Not applicable for this SOP

g. Personnel Qualifications / Responsibilities – The user should be properly trained in standard laboratory procedures. The user must also be trained to operate the instruments and must be able to run the analysis to collect data used for permeability coefficient calculations.

h. Equipment and Supplies – A pressure permeation cell, electrochemical monitoring cell, microelectrode and MEA set up will be required to conduct gas permeability measurements. In addition, one or more of these set ups should be equipped with data collection capability.

i. Step by Step⁵

(A) Pressure permeation cell⁵

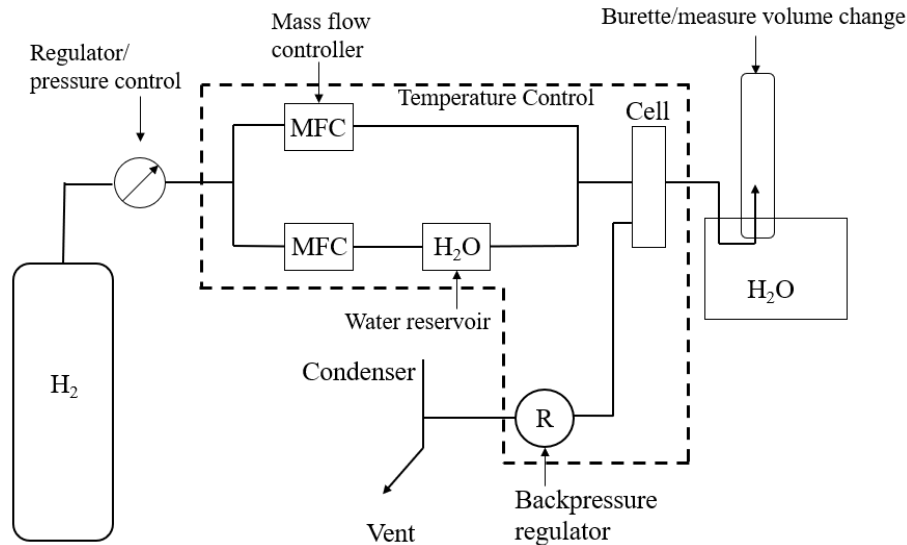


- Instrument or Method Calibration and Standardization – The pressure permeation cell is used with controlled humidified gas flow and gas chromatography.
- Sample Preparation and Analysis –
- The cell is constructed as follows: the membrane is sandwiched between two stainless steel plates, each having a gas inlet and outlet as well as gas chambers facing the membrane. Oxygen or hydrogen is supplied to the left chamber and inert gas to the right chamber of the cell. If necessary, use the same flow rate, ca. 100 sccm to remove the possible effects of pressure gradient. One of the cell outlets leads to a Gas Chromatograph (GC), the other one is immersed in the beaker with distilled water or is controlled with a valve to generate backpressure, but continual flow is maintained so as to not generate condensation on the membrane.

For ambient pressure conditions, ensure that the flow rate of the sweep gas is the same as the feed gas (ca. 100 sccm) or at least a sweep flow rate such that a pressure gradient isn't formed. For pressurized upstream feed gas, it is recommended that the pressure be similar to those utilized in an operating fuel cell (less than 2 atm). For other applications, 10 atm is useful for making comparisons to the Robeson Upper Bound and may be a good reference point for analysis of membranes utilized in electrolyzers. The temperature range of interest is R.T.-80°C, however two temperatures may be most useful. The lower temperature is 35°C which is a common reference temperature in the gas permeation literature, while a temperature of 60 °C is useful for the analysis of membranes for fuel cell or electrolyzer applications. At each value of temperature, pressure, and relative humidity, ensure that steady state permeation conditions are reached before recording data.

- Dry Conditions: In the permeation cell, the membrane and apparatus should be degassed under vacuum overnight (≥ 12 h). Dry gas should be introduced upstream and a sweep gas should be introduced downstream, after which the downstream should be analyzed by GC.
- Humidified Conditions: Follow the same general procedure in the “Dry Conditions” case above, except that there is no need to degas the apparatus or membrane. Instead, provide water-soaked membrane pieces (equilibrated in ASTM Type I deionized water for 1 hour at 25 ± 5 °C) with the known dimension including thickness. The RH of the gases may be controlled by the temperature difference between the humidifiers and the thermostatic cell or via other methods depending on the system.

A less costly, but less accurate gas permeation system is shown below as an alternative testing apparatus. The relative humidity is controlled by the two mass flow controllers, where one feed gas stream is at 100% RH and can be mixed in any amount with the other dry gas stream. Also, for testing dry conditions, it is recommended that the below system (besides the water containers) be placed under vacuum (for ≥ 12 h) to degas the apparatus and the membrane prior to permeation testing. (the vacuum control is not shown in the below schematic) The same operational conditions and sample preparation recommendations as in the above permeation testing system apply here. However, this system cannot be used to determine the diffusion or solubility coefficients and will have some loss of permeate gas due to solubility in the water/burette apparatus. Furthermore, without a sweep gas, some condensation of water is possible on the permeate side of the membrane, which could influence gas transport. Nevertheless, approximate permeability values may be obtained via calculation of the monitored volume change in the burette due to filling by the permeate gas. Data should not be recorded until steady state gas permeation is obtained.



(B) EMT

- Instrument or Method Calibration and Standardization – EMT measures the limiting current of the hydrogen or oxygen reaction through the membrane using an electrochemical monitoring cell that performs either an oxidation or reduction reaction on the permeating specie.
- Sample Preparation and Analysis – Typically, one side of the membrane with a working electrode at the surface is exposed to an acid solution with a counter electrode. The current is generated as all hydrogen (or oxygen) gas that passes through the membrane is oxidized (or reduced) under mass-transfer limiting conditions.

(C) Chronoamperometric technique

- Instrument or Method Calibration and Standardization – Microelectrode is used for hydrogen or oxygen permeability.
- Sample Preparation and Analysis – a thin film of polymer electrolyte is recast on the Pt micro disk electrode; the microelectrode is connected to the other sides of the glass body of the electrode by ion conductive ribbon, e.g., Nafion, which acts as an electrolytic bridge between the external reference and working electrode. Subsequently, the mini cell is connected to the reference electrode and placed in a humidity-controlled chamber. Oxygen permeability can be determined from the voltammetric limiting currents, assuming that the oxygen reduction kinetics is a four-electron reaction and the linear diffusion in a diffusion field limited by the film thickness is a dominant factor for oxygen transport.

- Data Acquisition, Calculations & Data Reduction Requirements – The corresponding limiting current is described by the following equation:

$$I = 4FCD\delta^1$$

where F is the Faraday constant (s A/mol), A is the geometric surface area of the electrode (cm^2), C is the oxygen solubility (mol/cm^3), D is the oxygen diffusion coefficient (cm^2/s), and δ is the film thickness (cm).

(D) *In-situ* measurement in the MEA

- Instrument or Method Calibration and Standardization – MEA configurations for fuel cell testing is used for hydrogen or oxygen permeability measurement via *in situ* measurement.
- Sample Preparation and Analysis – For hydrogen permeability, hydrogen flows on one side of a cell membrane, and the corresponding electrode acts as the reference and counter electrode. The reactant crosses over to the other side of the membrane and becomes oxidized at the other electrode (working electrode) by the application of a voltage, and the resulting current is measured. For oxygen permeability measurement, the concentration of the oxygen that is transported may be directly measured by spectroscopic techniques while inert gas is supplied at the anode because of slower oxygen reduction reaction.
- Data Acquisition, Calculations & Data Reduction Requirements – The gas permeation coefficient, K_i of the PEM can be expressed as:

$$K_i = (i_{H2}/nF) \times (th/p_{H2})$$

where i_{H2} is the crossover current density of hydrogen, p_{H2} feed partial pressure of hydrogen, th is the thickness of the AEM, F is the Faraday constant, and n is the number of electrons.

j. Data and Records Management – All experimental data must be recorded in the lab notebook.

4. Quality Control and Quality Assurance Section

TBD

5. Reference Section

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