

**University of Saskatchewan  
Department of Mechanical Engineering  
Standard Operating Procedure # HT0005**

**Procedure Title: Mocon Permatran-W 101K**

**Minimum Review Requirements:**    **Annually**

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**1. Version History**

Version #: 1.1  
Supersedes: 1.0

Handwritten amendments to the official procedures can be made by a single line through the text, along with the date, and initialed by the authorized individual making the correction. Changes are to be noted below. Formal changes to this SOP are made on the date of revision or sooner, where required.

Section	Changes made to official copy	Date	Initials
All	Minor housekeeping	July 2016	MTF

Reference Only

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**2. Introduction**

This procedure outlines the proper operation of the Mocon Permatran-W 101K Water Vapor Permeation Measurement System. This system is designed to analyze and measure the water vapor transmission rate (WVTR) of high transmitter barrier materials. Typical barrier materials are non-wovens, surgical dressings, polyester films and other flexible packaging materials. A film sample is mounted in one of the six test cells. The test cells are divided into two chambers. The lower chamber is filled with nitrogen and the upper chamber with water vapour at 100% RH. The water vapour transmission rate is determined by measuring the increase in the amount of water vapour present in the lower chamber.

**3. Definition**

CalC	Cell Calibration
Module	Permatran-W 101K machine
N <sub>2</sub>	Nitrogen gas
SOP	Standard Operating Procedure
WVTR	Water Vapor Transmission Rate

**4. Personnel**

Persons authorized to perform this SOP:

By signing this form I acknowledge that I have read and understand this SOP, as well as the applicable MSDS's and that I will conduct myself in accordance with this SOP and the general laboratory rules.

**NOTE: ALL SIGNATURES MUST BE PRESENT ON THE SOP LOCATED IN THE YELLOW BINDER IN ROOM 1B39.1.1 – the Heat Transfer Departmental Assistant's Office.** Digital copies of SOP's are made available for reference and convenience only. Printed SOP's are valid for 24 hours only, after that time their accuracy must be verified with the **OFFICIAL HARDCOPY VERSION**. A reference copy of this SOP can be found beside the machine in room 1B39.1.

Name (Print)	NSID	Dep't	Signature	Date

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**5. Safety**

- Gloves should be worn when changing the guard film to avoid damage from skin oils.
- The maximum input N<sub>2</sub> pressure to the machine should not exceed 20 psi, to avoid damage to the system.
- Do not turn the instrument off or interrupt gas flow at an elevated test temperature. Cool the instrument to ambient before turning off the module power or the gas flow.
- The module has an operating temperature range of 20°C to 50°C and a test range of 500 to 100,000 g/m<sup>2</sup>/day. Do not test outside of this range.

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## **6. Procedure**

### **6.1 Sample Preparation**

Samples should be prepared before testing begins to keep the time that the test head assembly is off to a minimum.

- To prepare your membrane, apply a thin coat of glue to one side of the film holder, and place the membrane to be tested on the film holder. Trim any excess if necessary.
- Use good samples. Wrinkles and creases contribute to false readings.
- When testing a multi-layered or laminate, install with the barrier coating or laminate toward the lower cell.
- After the test is completed wash the film holder with water to remove any glue so that it is ready for the next sample.

### **6.2 Pre-test Preparation**

- Open the nitrogen (N<sub>2</sub>) tank.
- Adjust the regulator on the N<sub>2</sub> tank until the pressure entering the machine reads ~15 psi. The pressure on the regulator should read ~20 psi. **The maximum input N<sub>2</sub> pressure should not exceed 20 psi or the system may be damaged.**
- Close the N<sub>2</sub> tank until ready to test.
- If the pressure in the N<sub>2</sub> tank is below 600 psi, there is probably not enough N<sub>2</sub> left to complete a full test, the tank should be changed.
- Turn on the computer and log-in.
  - Username: mocon
  - Password: mocon
- Turn on the Permatran-W 101K with the power button on the back, right hand side.
- Launch the Permeability System software from the desktop after the machine has been turned on.
- The *Home window* will appear. This shows the status of up to ten modules. We only have one module, so the rest will be grayed out.
- Open the following windows from the main menu bar at the top of the screen
  - Status -> Cell Status
  - Status -> Module Status
  - Control -> Control Tests
  - Setup -> New Test Method

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### **6.3 Calibration**

For the most accurate results, a calibration should be performed before each new test. The calibration (CalC) measures the WVTR through the water, guard film and N<sub>2</sub>. This value will be used to calculate the WVTR through the membrane. Calibration is required when you start up the machine, when you change temperature or when your system flow rates change.

#### **Preparing the test cells**

- Open the cover on the module
- Turn the clamping knob on the top of the test cell counter-clockwise and unlatch the two clamps. Lower the clamps away from the head assembly until they rest on the module case.
- Gently lift the test cell head off using the two handles attached to either side of the head.
- Place an empty metal film holder onto the block, with the small holes facing towards the bottom.
- Verify that the guard film is mounted to the bottom of the head assembly and that there are no pinholes or tears in the guard film.
- Replace the head assembly onto the test cell.
- Remove the top of the head assembly and check the water level in the wells. If the cells are dry, refill them (refer to Section 6.8 for instructions).
- Replace the top of the head assembly.
- Latch the test cell clamps onto the test cell head and secure by turning the knob on top of the head clockwise until snug.

#### **Performing the calibration**

To achieve the optimum repeatability and accuracy, the flow rates must be adjusted on each of the six cells. The flow should be adjusted to obtain a relative humidity value of 60% RH ± 1% RH during the CalC process.

- Use the *New Test Method* window to set the following parameters for the test:
  - Test Mode – Convergence by Cycle,
  - Exam Minutes – 5,
  - Period – 3 (use 2 if you have limited time to perform the test),
  - Area – 1 cm<sup>2</sup> (may be different if using a different film holder),
  - Thickness – 1 mil, and
  - CalC Mode – Active CalC.
- Click on **Assign to All Cells**

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Note: The convergence by cycle mode compares the current WVTR to the average value over the last x number of cycles, where x is the period chosen (typically 3). Convergence occurs when the current WVTR and the average WVTR agree within 1%. Another option is to use the continuous mode, which will run the program until stopped by the user.

- Open the N<sub>2</sub> tank and ensure the pressure entering the machine is ~15 psi.
- In the *Module Status* window set the Temperature setpoint to the desired test temperature (typically 23°C).
- Verify that the Flow Rate sensor is in Auto mode.
- In the *Control Test* window click **Start All Tests**. The program will run one cycle on Cell A, then one cycle on Cell B, and so on. After the cycle on Cell F is completed, the second cycle on Cell A will begin and the process will be repeated. Each cycle runs for the specified Exam Time (typically 5 min).
- In the *Module Status* window monitor the water vapour reading of the active cell.
- Adjust the flow rate knob on the machine for that cell until the water vapour field displays 60% RH ± 1% RH. Turn the knob clockwise to decrease the flow rate and increase the RH value or counter-clockwise to increase the flow rate and decrease the RH value. **Note: There is a lag of several seconds between the adjustment of the knob and the display of the new flow rate and water vapour rate on the *Module Status* window.** The flow rate is typically in the range of 20 – 22 sccm.
- After 5 minutes (or specified exam time) the module will move to the next cell.
- In the *Cell Status* window you can view the progress of the test. Click on a cell label in the upper right corner to view the progress for that cell. The active cell will have a blue light under the label. After each cycle the current WVTR will be displayed in this window.
- Once you are satisfied that all six cells are at acceptable flow rates, close the lid on the machine and allow the program to run until convergence is met.
- When a cell reaches convergence, the indicator light on the cell label will change to pink.
- Once all cell labels are pink the calibration is complete and the module is ready to test the membrane.
- **Do not change the flow or temperature settings after a CalC has been performed.** If you do, you must perform another CalC, readjusting the flows to achieve the desired RH for each cell in the process.

#### **6.4 Testing a Membrane**

- The membrane can be inserted without turning off the program or module.
- Open the cover on the machine.

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- Remove the test cell clamps on the test head assembly by turning the clamping knob counter-clockwise. Unlatch the test cell clamps and lower them away from the head assembly until they rest on the module case.
- Remove the test head assembly by gently lifting the head using the two handles attached to either side of the head.
- Remove the empty film holder and install the film holder with the membrane sample into the test cell with the small holes facing towards the bottom.
- Place the head assembly onto the test cell.
- Close and clamp the test cell head by latching the test cell clamps over the head assembly and turning the knob until snug.
- Click **Advance Test** in the *Control Test* window to begin the test on the membrane.
- Monitor the test using the *Cell Status* and the *Module Status* windows. Compare the current flow rate to the flow rate from the CalC portion of the test for each cell. If the difference in flow rates is  $> 1$  sccm the test results will not be accurate. If that is the case, you should start the whole test over.
- The WVTR reported is the total for the water, membrane, guard membrane and  $N_2$ . To obtain the WVTR for the membrane alone, you will need to calculate the difference between the reported WVTR and the CalC WVTR.

Note: The test may be stopped at any time by clicking the **Abort Test** button in the *Control Test* window. Each cell must be stopped individually.

### 6.5 Results

After the tests have been completed you should save the data to be used later.

- From the menu bar at the top of the main window, click on **Multi Test Summary Report** (the button looks like a spreadsheet).
- Click **Print Summary**. This will save a report for all six cells.
- From the menu bar at the top of the main window, click on **Detail Test Report** (beside the multi test summary report button).
- The important parameters in this window are the Transmission Rate, CalC Mode value, Flow Rate and CalC Flow Rate.
- In the bottom left corner select **Include Chart In Report**.
- Click **Print Report**. This must be done for each of the six cells separately.

### 6.6 Shutdown

- After testing is completed, remove the membrane sample from the test chamber and place the empty film holder used for calibrations back into the test chamber.



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Anytime the machine is not being used the empty film holder should be stored in the test chamber.

- Close the N<sub>2</sub> tank.
- Turn off the machine.
- Close the software and log out of the computer.

### **6.7 Nitrogen Tank Replacement**

Whenever a fresh tank of N<sub>2</sub> is installed, air is admitted into the regulator body and supply line. This air should be purged from the system to minimize background water vapour before admitting the nitrogen gas into the sensor. To purge the system:

- Send nitrogen gas through the system for 30-45 minutes with the sensor in *Standby mode*
- After installing a new tank, leak-test all gas connections using a soap and water solution to check for bubbles
- Tighten any leaky connections.

### **6.8 Water Reservoir Filling**

If at any point during testing there is not enough water in the reservoir, follow these steps to add more:

- From the main menu, select **Control -> Pause**. This will put the module into *Standby Mode*.
- Turn the clamping knob on the top of the test cell counter-clockwise and unlatch the two clamps. Lower the clamps away from the head assembly until they rest on the module case.
- Lift off the top of the head assembly to reveal the water wells.
- Fill the six water wells approximately 2/3 full with HPLC-grade water. Do not over fill. The water must not touch the top of the head assembly when closed.
- Replace the top of the head assembly. Latch the two clamps onto the head assembly and secure with by turning the knob clockwise.
- Resume testing by selecting **Control -> Resume** from the main menu.
- The software uses a timer to keep track of the status of the water reservoir (in the *Module Status* window). The default value is 72 hours. This is approximately the amount of time before the reservoir will run dry under normal operating conditions.
- If the timer expires, testing will pause and a *Reservoir Dry* dialog box will appear. Refill the reservoir and click the **Reservoir Filled** button to restart the timer.

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**7. Equipment or Materials Required**

- Membrane samples
- Film holders
- Glue stick
- HPLC-grade water
- Compressed Nitrogen tank

**8. References**

PERMATRAN-W Model 101K Operator's Manual, Revision B, Mocon, Minneapolis, MN, <http://www.mocon.com/distributor/pdfmanuals/Permatran%20101K%20-%20b.pdf>

US Patent Number 5,837,888, <http://www.patentstorm.us/patents/5837888.html>

Reference Only