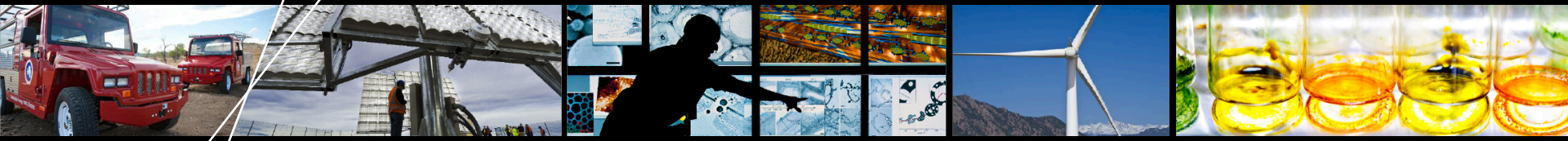
NREL
NATIONAL RENEWABLE ENERGY LABORATORY

NREL's e-Ca Test



Energy Innovation Portal Webinar

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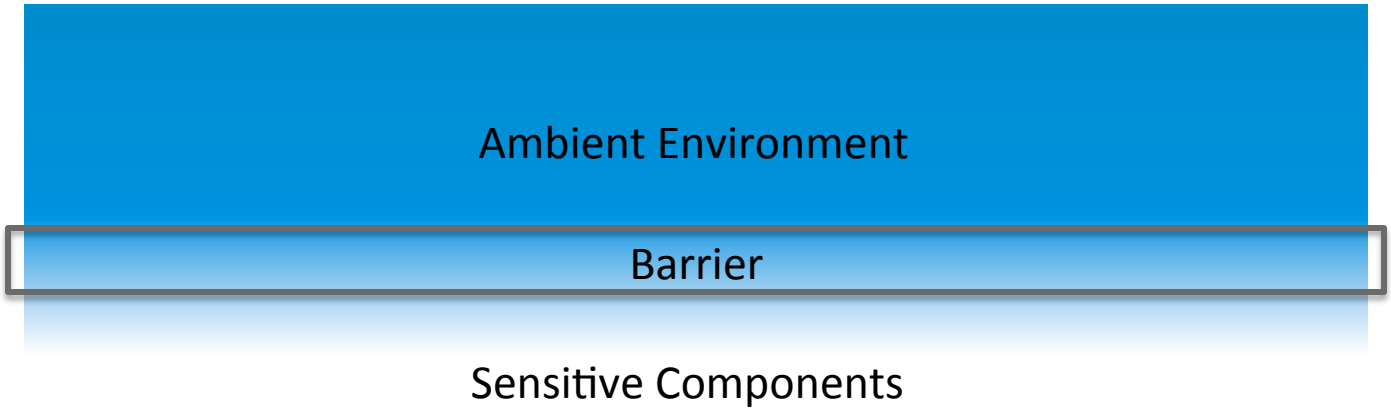
March 27, 2012

NREL is a national laboratory of the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, operated by the Alliance for Sustainable Energy, LLC.

Characterization of Water Permeation Barriers

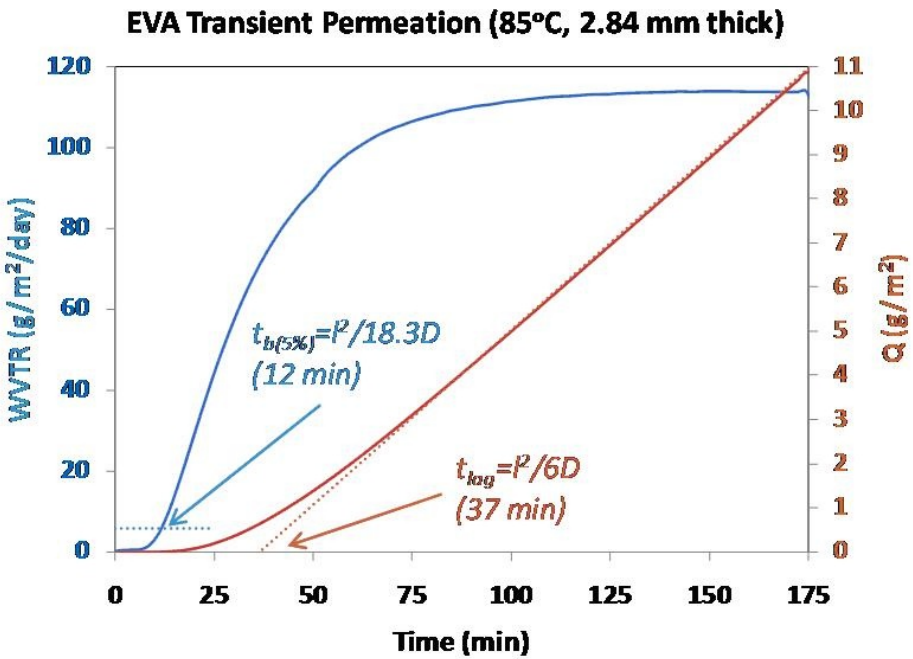


Vapor adsorbs and dissolves into barrier

Vapor diffuses

Vapor desorbs/ outgasses

- **Barrier manufacturers need a characterization method that is:**
  - Reproducible
  - Highly sensitive
  - Easy to use
  - Scalable



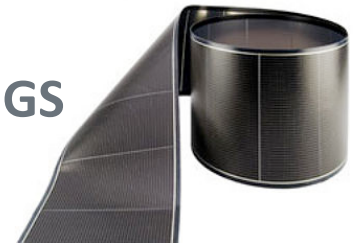
Application Specific Tolerable Water Permeation

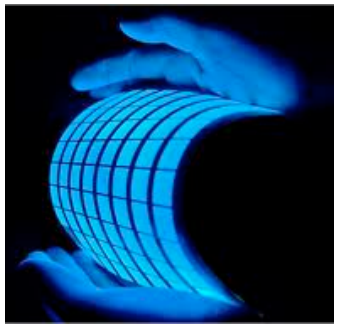
Volume in 1 m^2 after 20 yrs	WVTR ($g/m^2/day$)	
~7 L	1	Food Packaging
	0.1	LCDs
	0.01	
	10^{-3}	TFTs
		CdTe /Thin Film
~7 mL	10^{-4}	
	10^{-5}	CIGS
~7 uL	10^{-6}	OPV & OLEDs
	10^{-7}	

Gravimetric Test Limit
ASTM E96
(1×10^{-1})

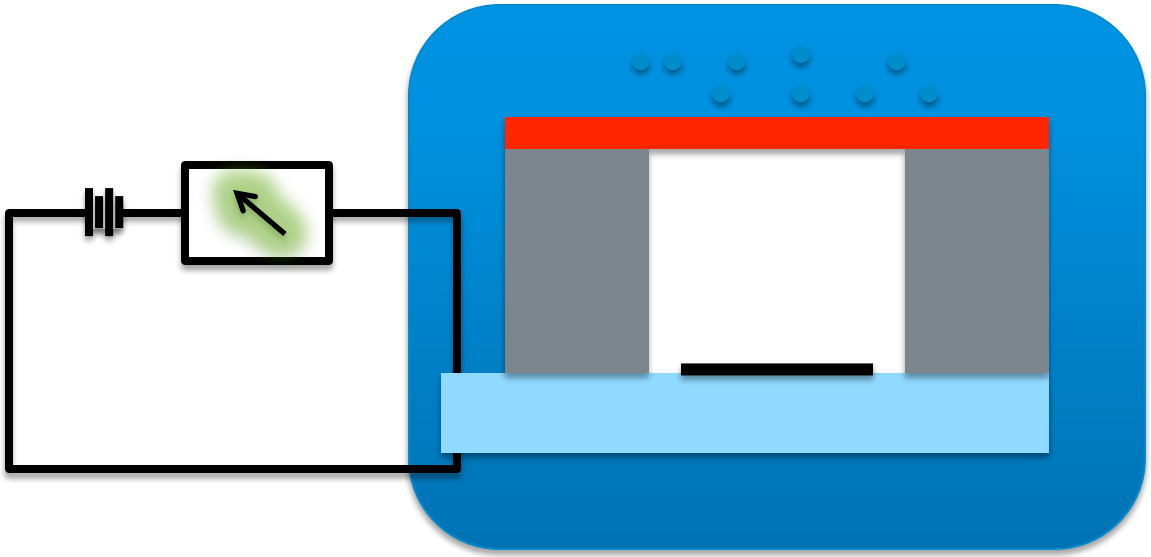
Isostatic Test Limit
ASTM F1249
(5×10^{-4})

Ca Test Limit
(5×10^{-7})



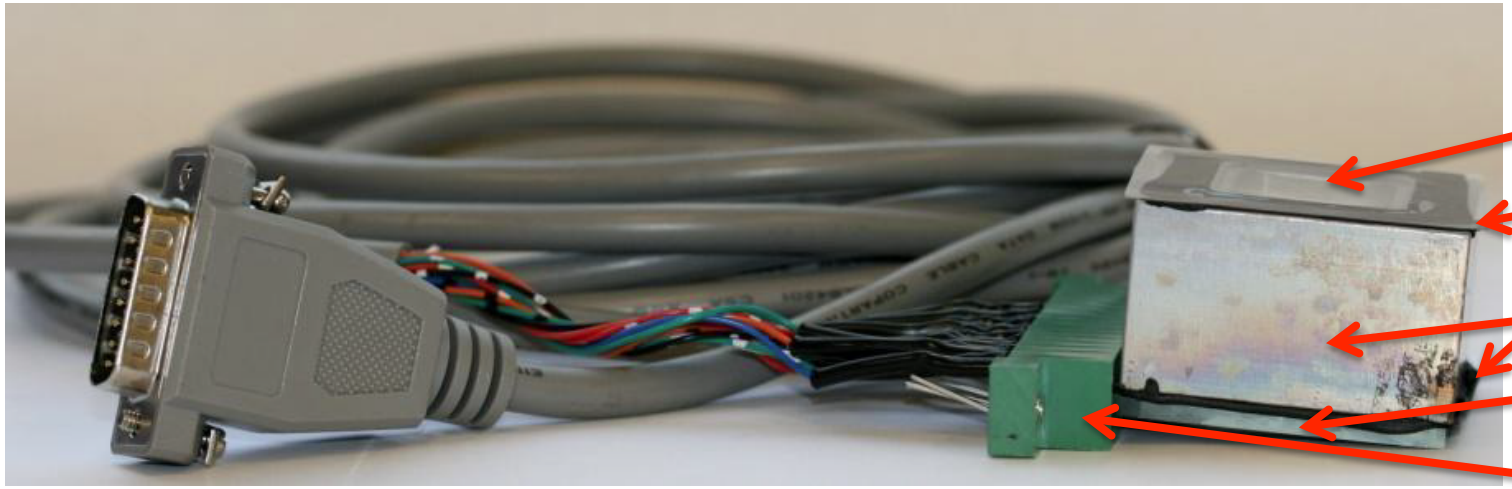


NREL's e-Ca Test



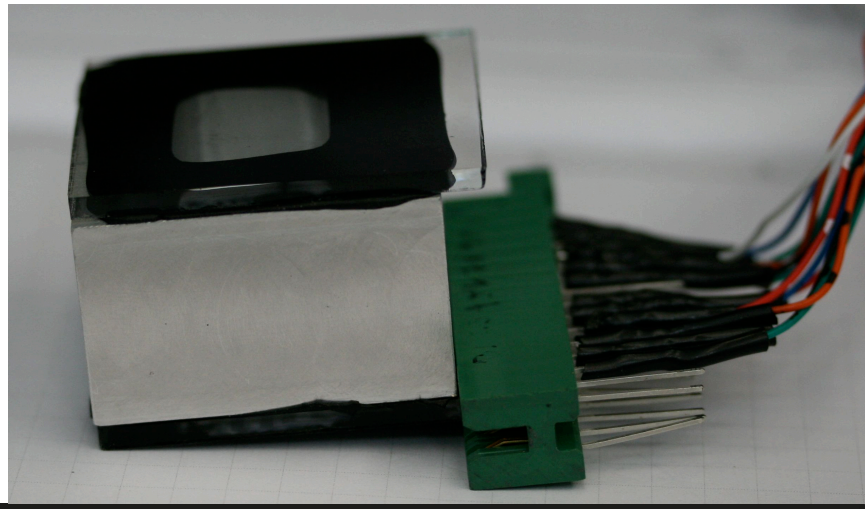
$$Ca + 2H_2O(g) \rightarrow Ca(OH)_2 + H_2$$

Ca transforms from opaque, conductive metal to transparent, resistive oxide upon exposure to water



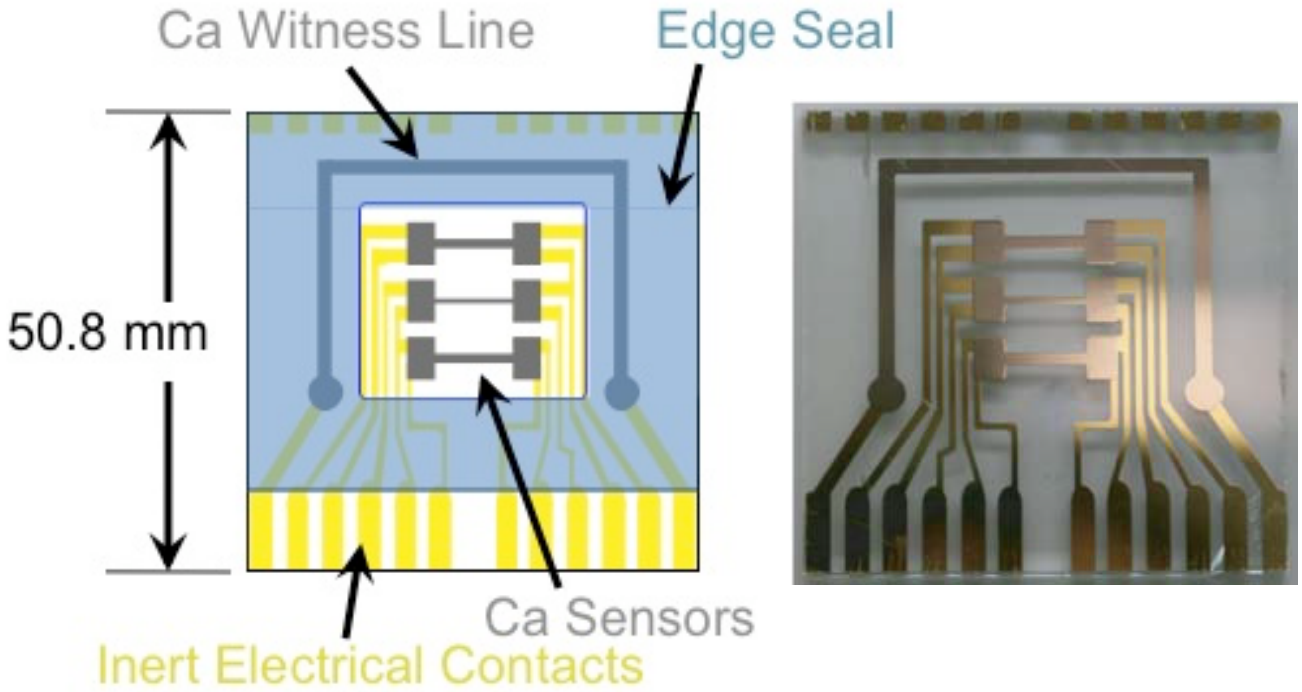
Ca Test Components

- **Computer for Data collection**
  - Custom software
    - Data collection real-time WVTR display and calculations
- **Commercial digital multimeter**
  - 6.5 digit accuracy
- **Multiplexing Distribution Hardware**
  - Combination of commercially available and custom assembled instrumentation
  - Current configuration can measure up to 128 samples
- **Ca Test Assembly**
  - Cables between electronics and Ca assembly
  - Commercial edgecard connector for easy/fast connect/disconnect
  - ***Custom Test Card with Ca and Au traces***
    - Evaporated contacts using shadow mask onto glass substrate
  - Commercial edgeseal (extensive testing done)
  - ***Custom diffusion spacer element***
    - Machined aluminum block
  - Barrier Film



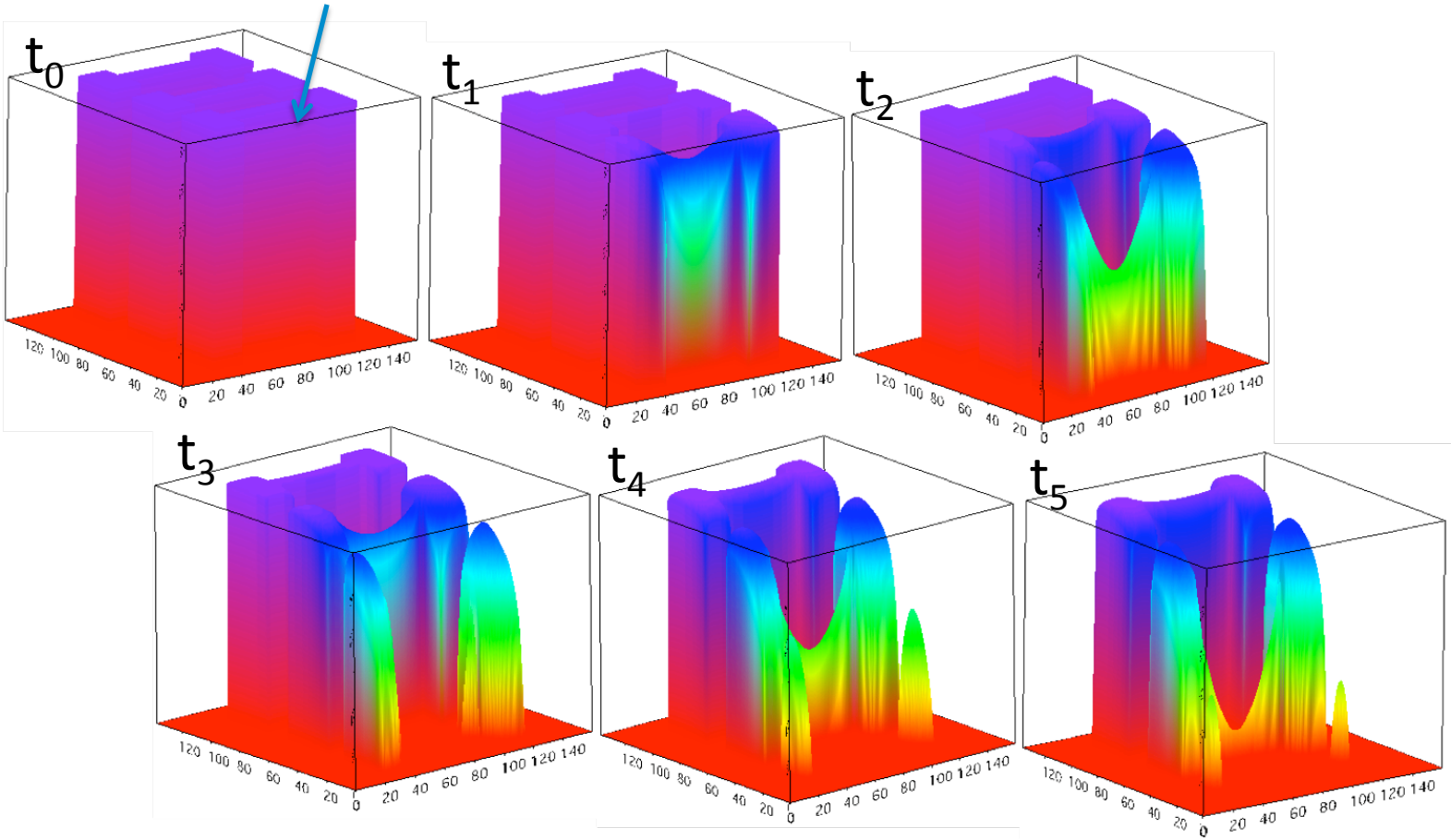
Test Card

- Ca Traces are fabricated separate from the barrier film
- 3 redundant 4-pt measurements of Ca resistance as a function of time
- Witness line monitors edgeseal integrity and provides internal standard of sensitivity limits
- Inert contacts pass signal to the outside of the assembly and fit into standard edgecard connector
- Test cards can be produced in bulk (and stored in inert environment) prior to obtaining barriers to be tested



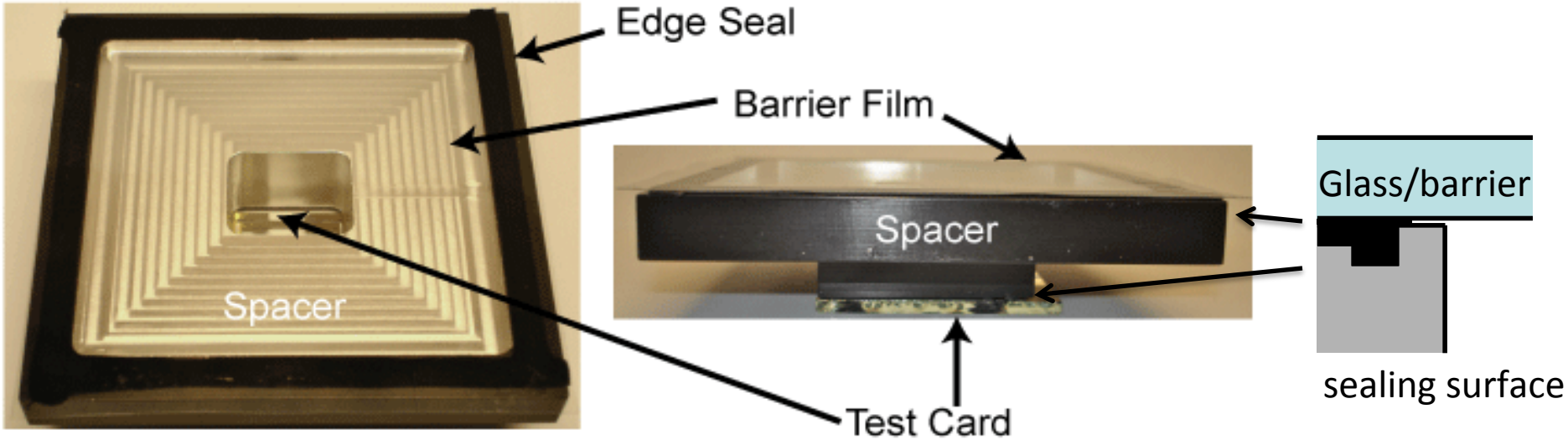
Why Incorporate a Diffusion Spacer?

- **Worst Case Scenario:** **Ca traces are deposited onto the barrier but the barrier has a pinhole defect:**



- **Resultant measurement is not reflective of the average WVTR!**
- **For Accurate Test: diffusion distance approx. equal to area of the Ca**

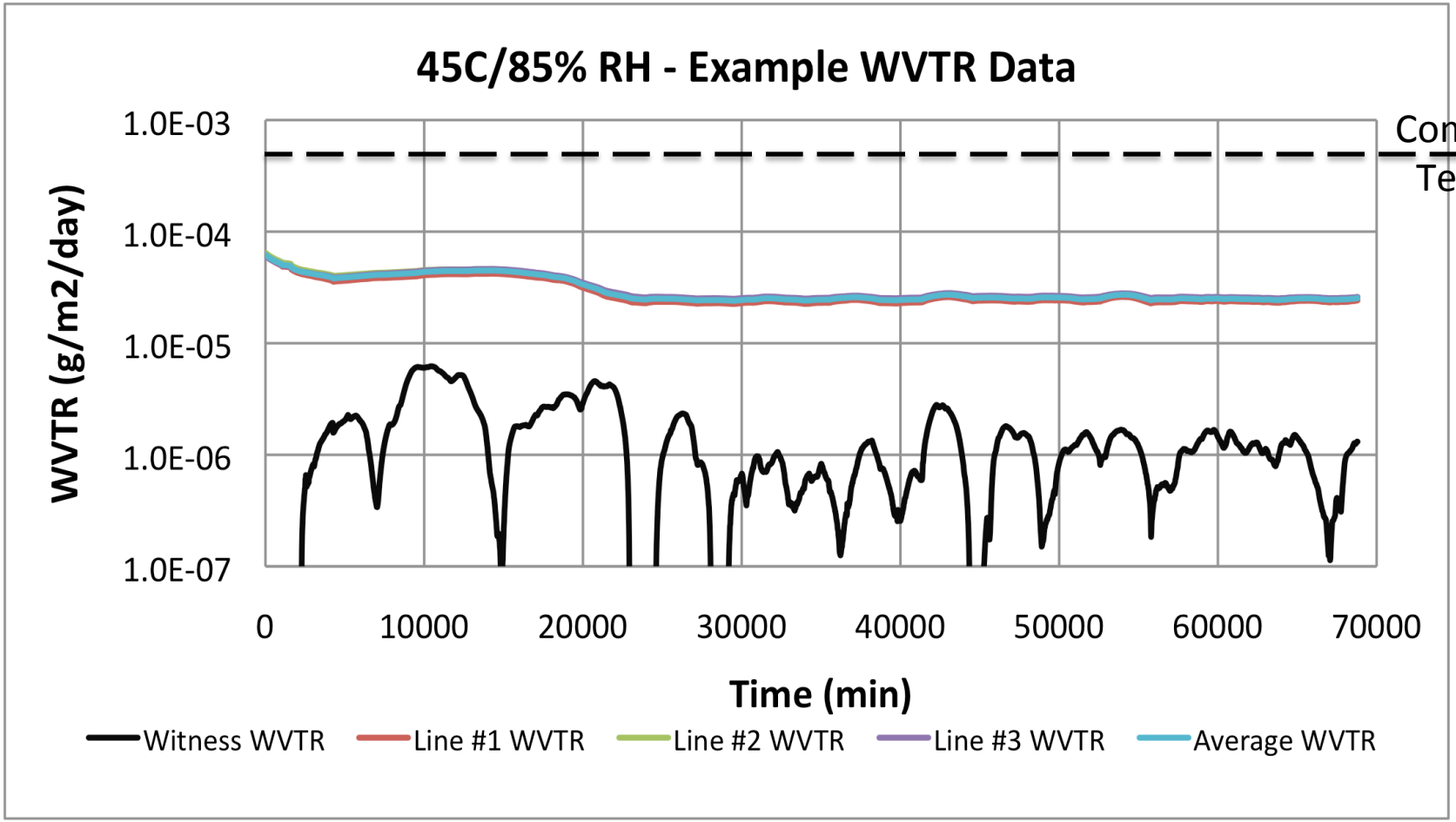
Spacer Advantages



- **The ratio of apertures controls the sensitivity range by adjusting the barrier area relative to the Ca area**
  - Some ratios that we can use presently are 9x, 30x, 60x, 700x
- **The sealing surface design allows for reproducible areas, and also limits the influence of the edgeseal materials on the measurement**
- **The spacer also allows the Ca Test to be assembled quickly with a mechanical press**

Measured Sensitivity of e-Ca Test

Actual measured data from a barrier film provided by a commercial partner shows steady state permeation at ~3 × 10^{-5} g/m^2/day.



This data is well above the sensitivity limits of NREL's e-Ca test and well below what can be measured using a commercially available test.

Possible Market Integration of e-Ca Test

- **Integration of capabilities at a test lab**
  - Provide testing services
  - NREL receives requests for testing constantly
    - Several ongoing commercial partners
- **Instrumentation/Laboratory Equipment**
  - Source the instrumentation and required deposition designs
    - Lab scale
    - Commercial quality control scale
  - Source the consumables
    - Test cards
    - spacers