Methods for Measuring Moisture Ingress

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Moisture Barrier Requirements

Next generation PV will require barriers with WVTR < 1× 10^{-6} g/m^2/day using materials with low defect density; Diffusivity <1× 10^{-18} cm^2/s

Material Goal: flexible & transparent material with these properties

General Strategy: alternating inorganic/organic thin films estimated ~12 dyads required

Measurement Goal: quantifiable means to evaluate, compare and improve materials

Device testing: demonstrates applicability but is not the best means to quantify moisture ingress

Lewis *et. al., IEEE Journal of Selected Topics in Quantum Electronics, vol.10 p. 44, 2004.*
G.L. Graff *et. al., Journal of Applied Physics*, vol. 96, pp. 1840, 2004.

How much water?

WVTR = 1 $g/m^2/day$

In a day:

X = ~1 um

In 20 years:

X = ~7.3 mm

~7.3 L collected

WVTR = 1×10^{-4} $g/m^2/day$

In 20 years:

X = ~1 μm

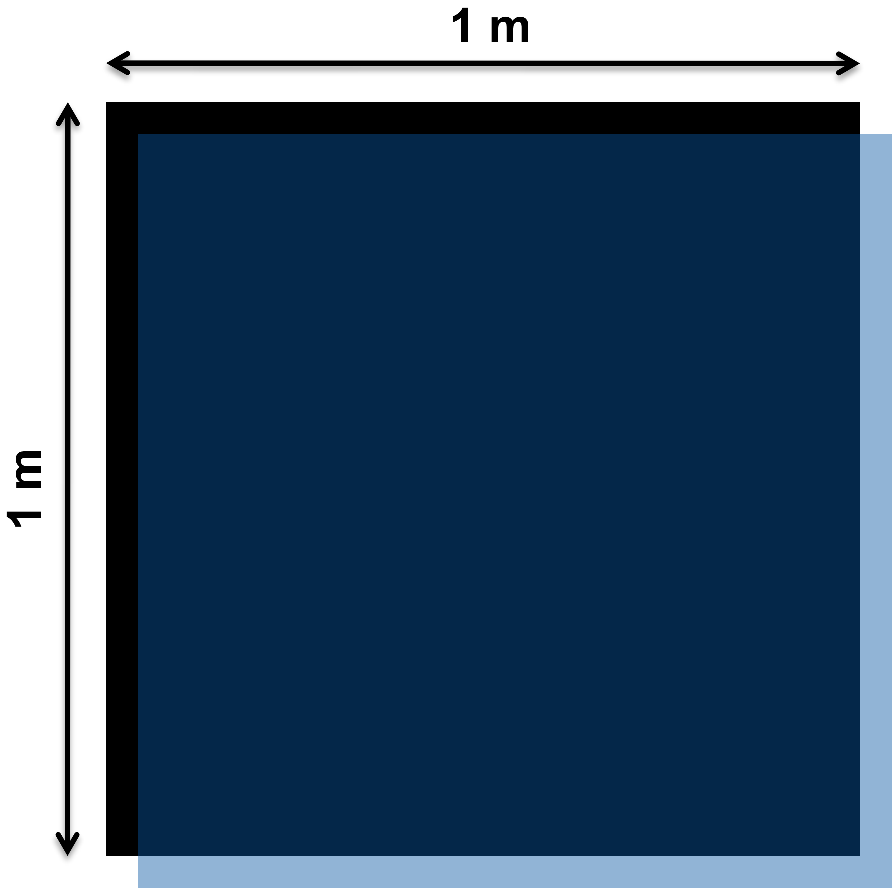
~7.3 mL collected

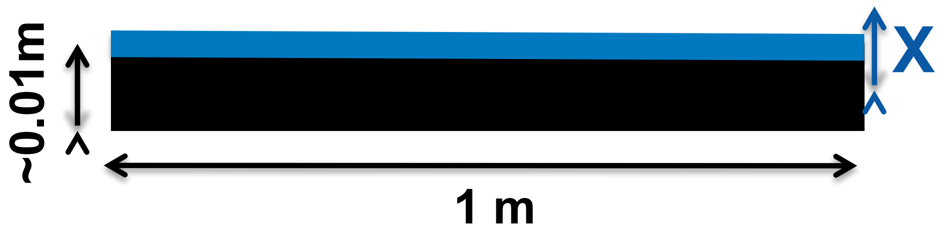
WVTR = 1×10^{-6} $g/m^2/day$

In 20 years:

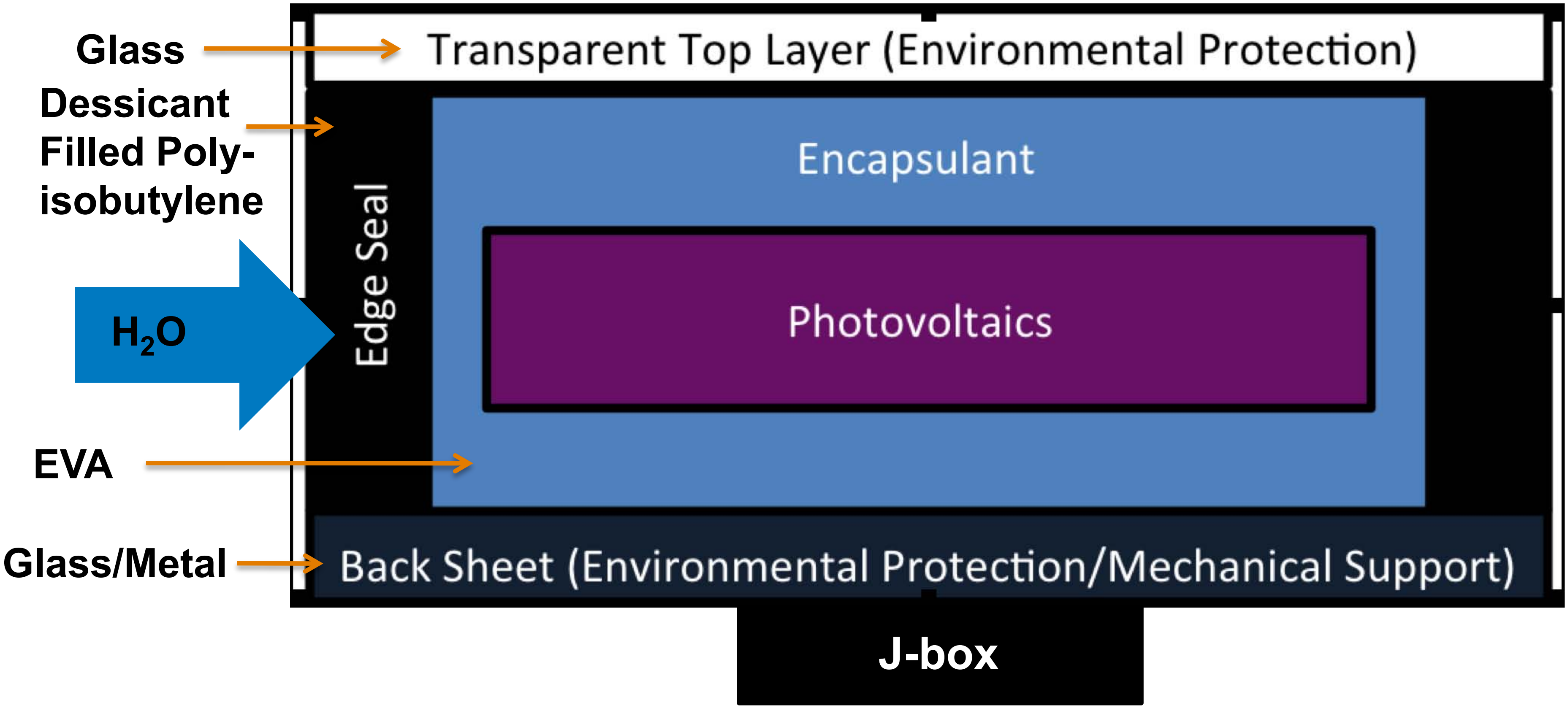
X = ~10 nm

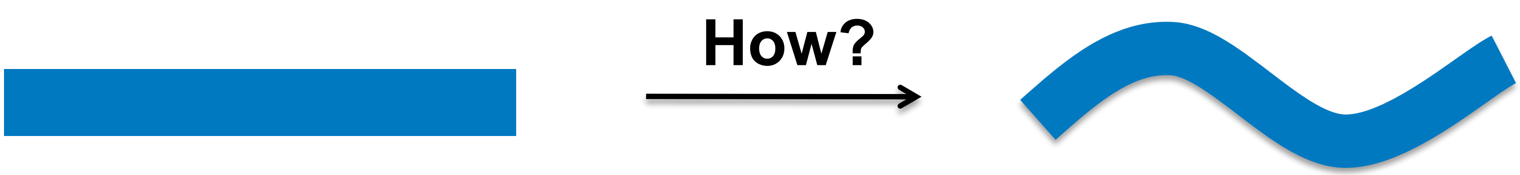
~7.3 μL collected





Entry Points





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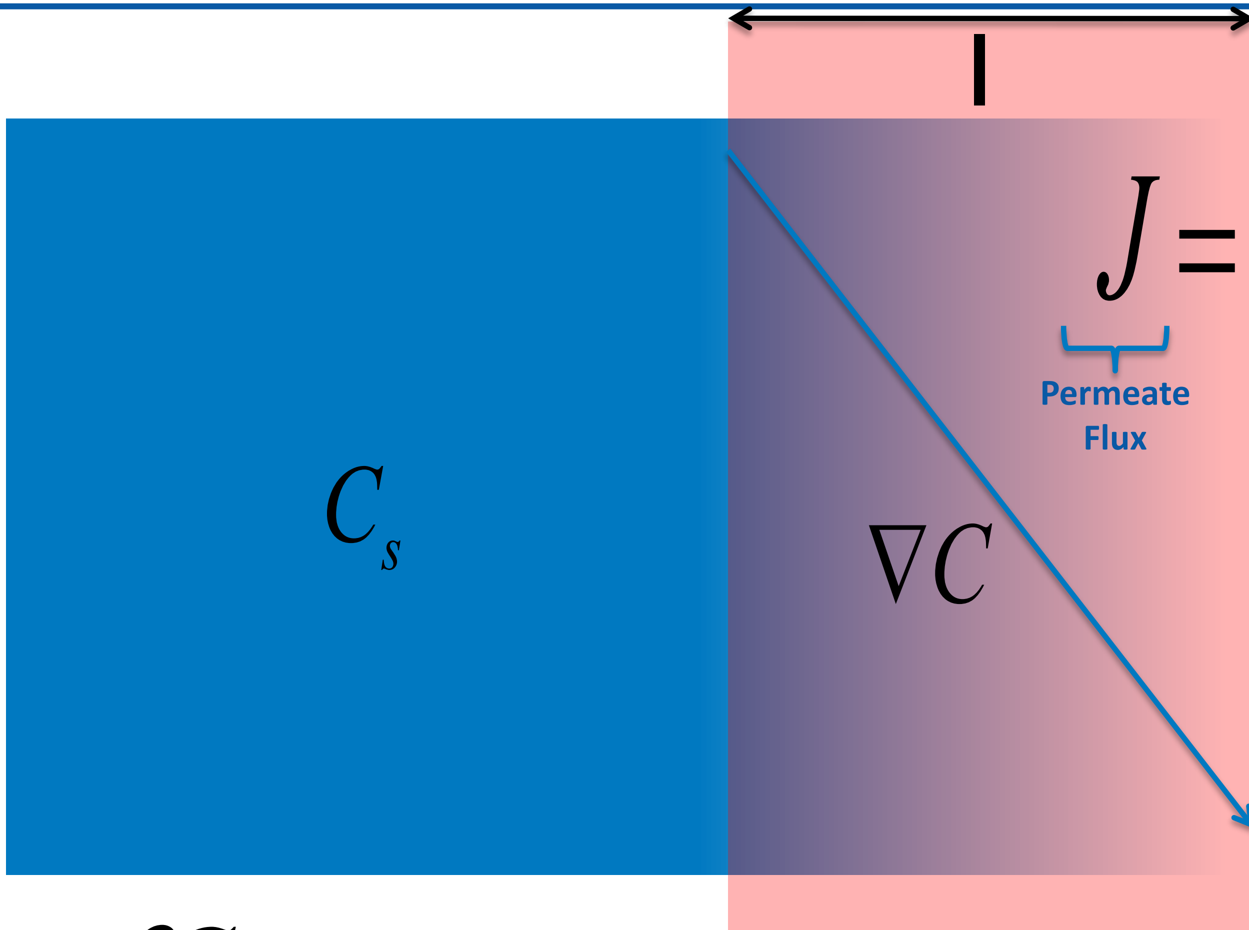
Diffusion



$$J = -D\nabla C$$

Permeate Flux (J) · Diffusivity (D) · Concentration Gradient (∇C)

$$\frac{\partial C}{\partial t} = -\nabla J = \nabla (D \nabla C) \longrightarrow D\nabla^2 C$$

Assume Fickian: material has no dependence on concentration

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WVTR, Permeability, Diffusivity and Solubility

$$WVTR(t) = \frac{DC_s}{\ell}\left[1 + 2\sum_{n=1}^{\infty}(-1)^n \exp\left(-\frac{Dn^2\pi^2 t}{\ell^2}\right)\right]$$

Thickness

$$Q(t) = \int_0^t WVTR(t)\,dt = \frac{DC_s}{\ell}t - \frac{\ell C_s}{6} - \frac{2\ell C_s}{\pi^2}\sum_{n=1}^{\infty}\frac{(-1)^n}{n^2}\exp\left(-\frac{Dn^2\pi^2 t}{\ell^2}\right)$$

Permeability

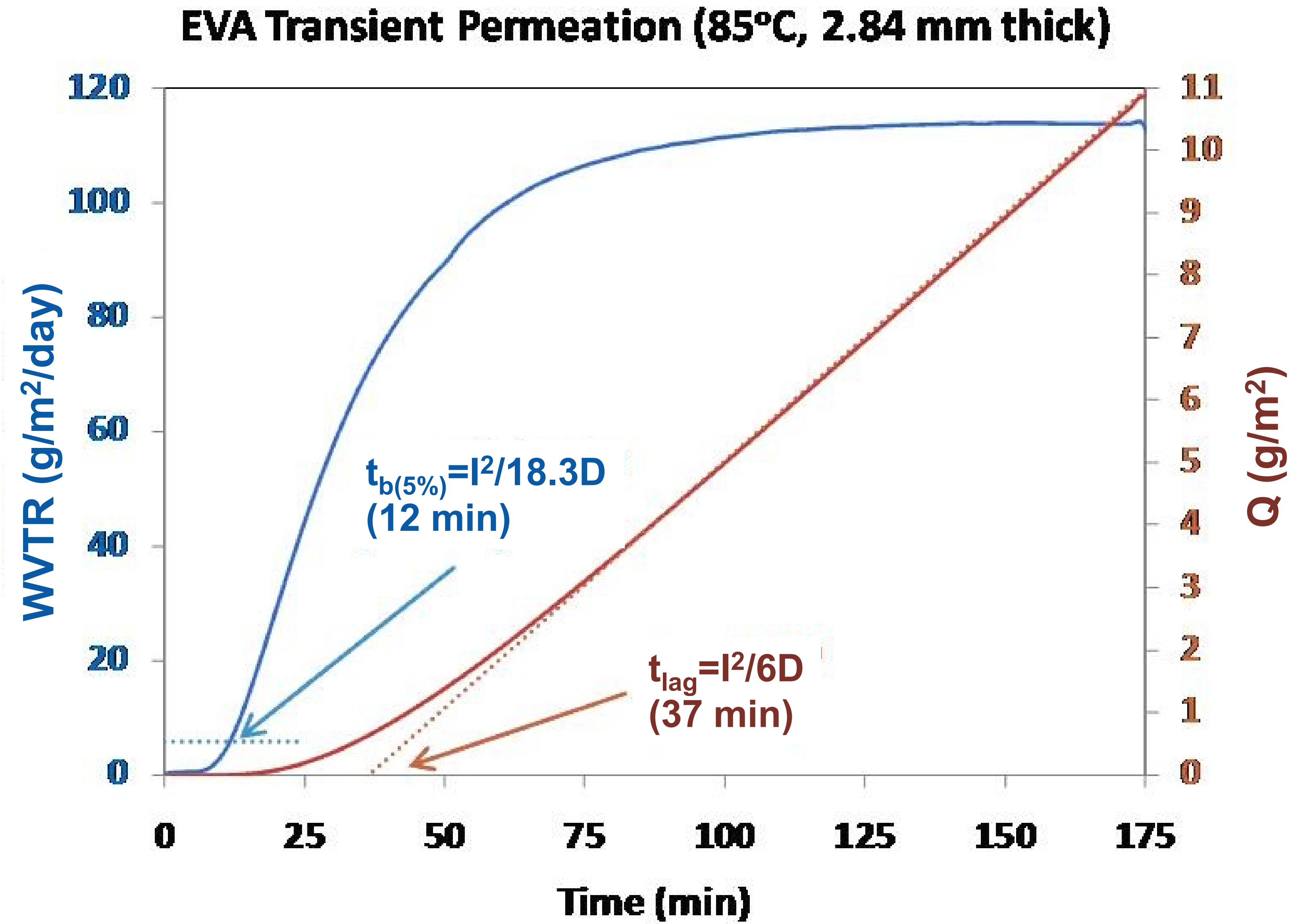
$$P = \frac{Q\ell}{At\Delta p} = DS$$

Solubility

Area

Partial Pressure

WVTR and Q



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Barrier Testing Method Types

Scavenger Methods

a material that reacts with or absorbs water is used for quantification

Q(t)

Calcium Test

Radioactive Tracer Test

Gravimetric Cup test

Diffusion Cell Methods

water is directly quantified

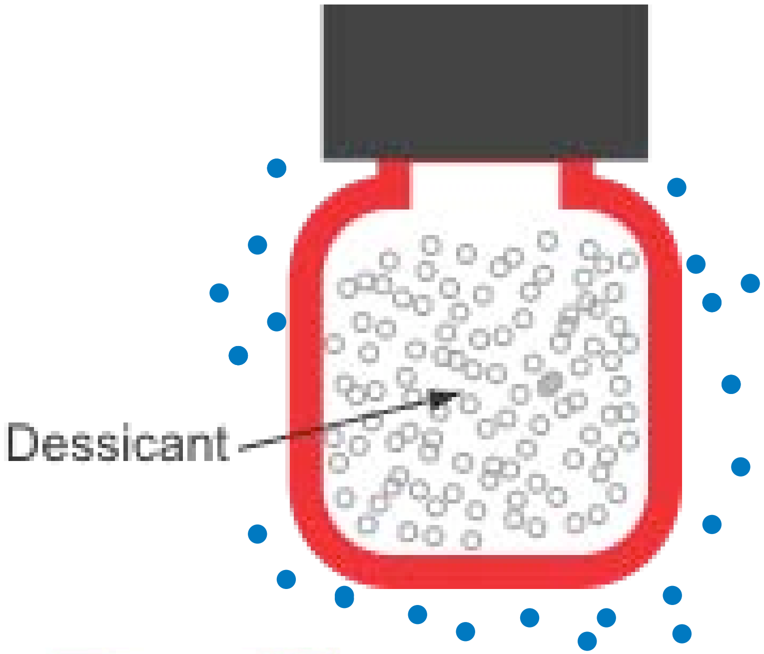
WVTR(t)

Isostatic Test (MOCON)

Radioactive Tracer Test

Mass Spectrometry

Gravimetric Cup Test



Standard: ASTM E96

Measurement:

$$\frac{\partial m}{\partial t} \Rightarrow WVTR(t)$$

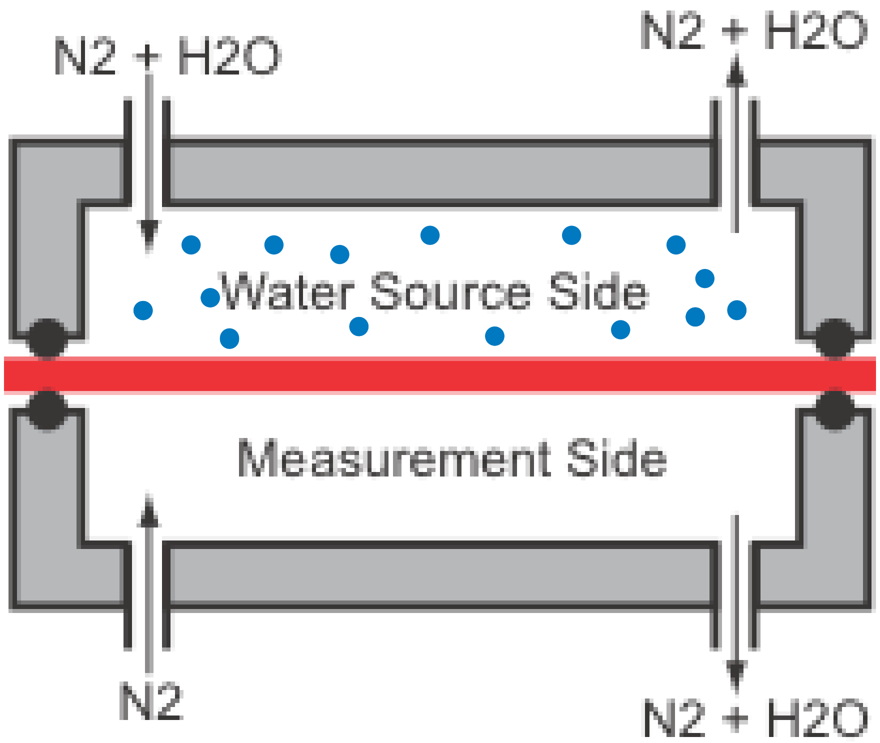
(assuming constant WVTR for Δt)

Sensitivity Limits:
>>1000 g/m^2/day – 0.1g/m^2/day

Pros: simplicity; throughput; controlled temp/humidity

Cons: sensitivity; no transients

Isostatic method (MOCON)



Standard: ASTM F1249

Measurement:

Infrared:

$$A_{\lambda}(t) \Rightarrow [H_2O]_{test}(t)$$

$$\frac{[H_2O]_{test}}{[H_2O]_{standard}}(t) \Rightarrow WVTR_{test}(t)$$

Coulometric:

$$\Delta R(t) \Rightarrow \Delta mol H_2O(t) \Rightarrow WVTR(t)$$

Sensitivity Limits:

100 g/m^2/day – 5×10^{-4} g/m^2/day

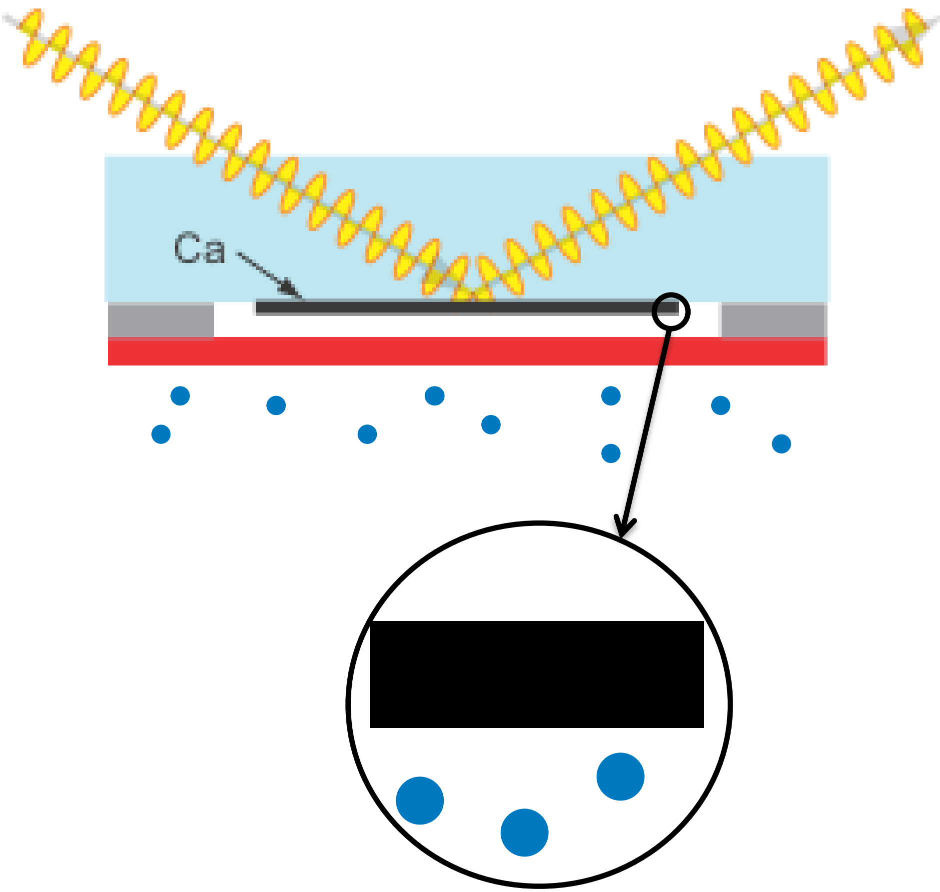
Pros: commercial availability

Cons: sensitivity; throughput; limited temp/humidity

Optical Ca Test

$Ca + H_2O \rightarrow CaO + H_2$

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



Standard: None

Measurement:

$$A_\lambda(t) \Rightarrow molCa(t)$$

$$molH_2O(t) \Rightarrow WVTR(t)$$

Sensitivity Limits:

1×10^{-2} g/m^2/day – 1×10^{-6} g/m^2/day

Pros: sensitivity; visual evaluation

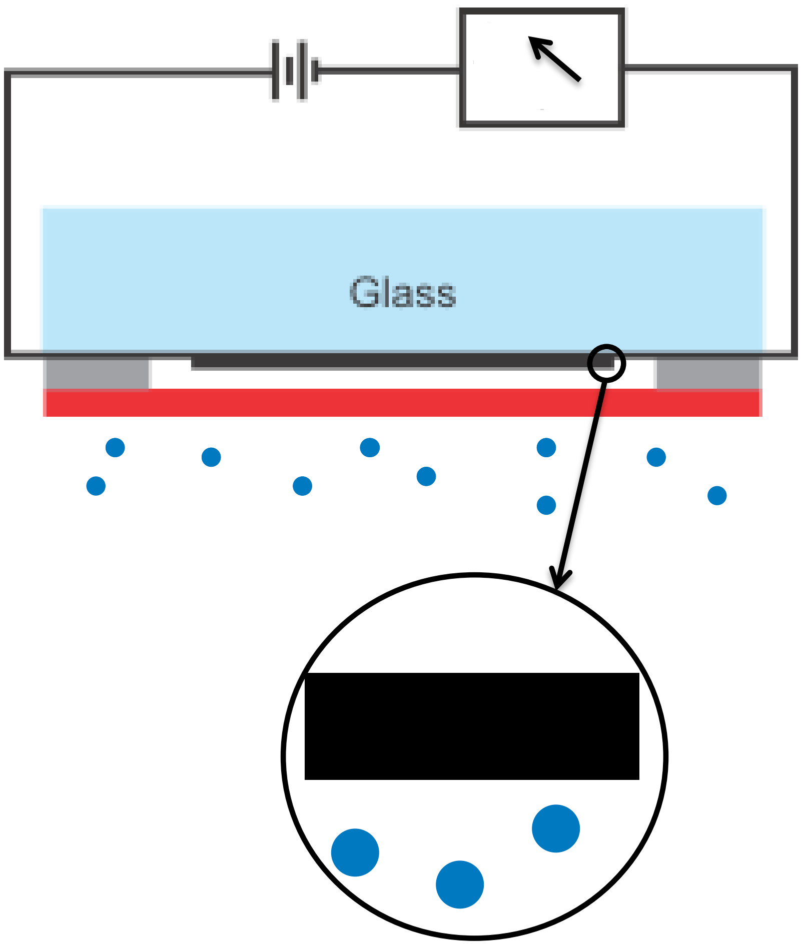
Cons: cost

G.Nisato *et al.*, *Proceedings Asia Display/IDW* pp. 1435, 2001.

Electrical Ca Test

$Ca + H_2O \rightarrow CaO + H_2$

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



Standard: None

Measurement:

$$R(t) \Rightarrow molCa(t) \Rightarrow$$
$$molH_2O(t) \Rightarrow WVTR(t)$$

Sensitivity Limits:

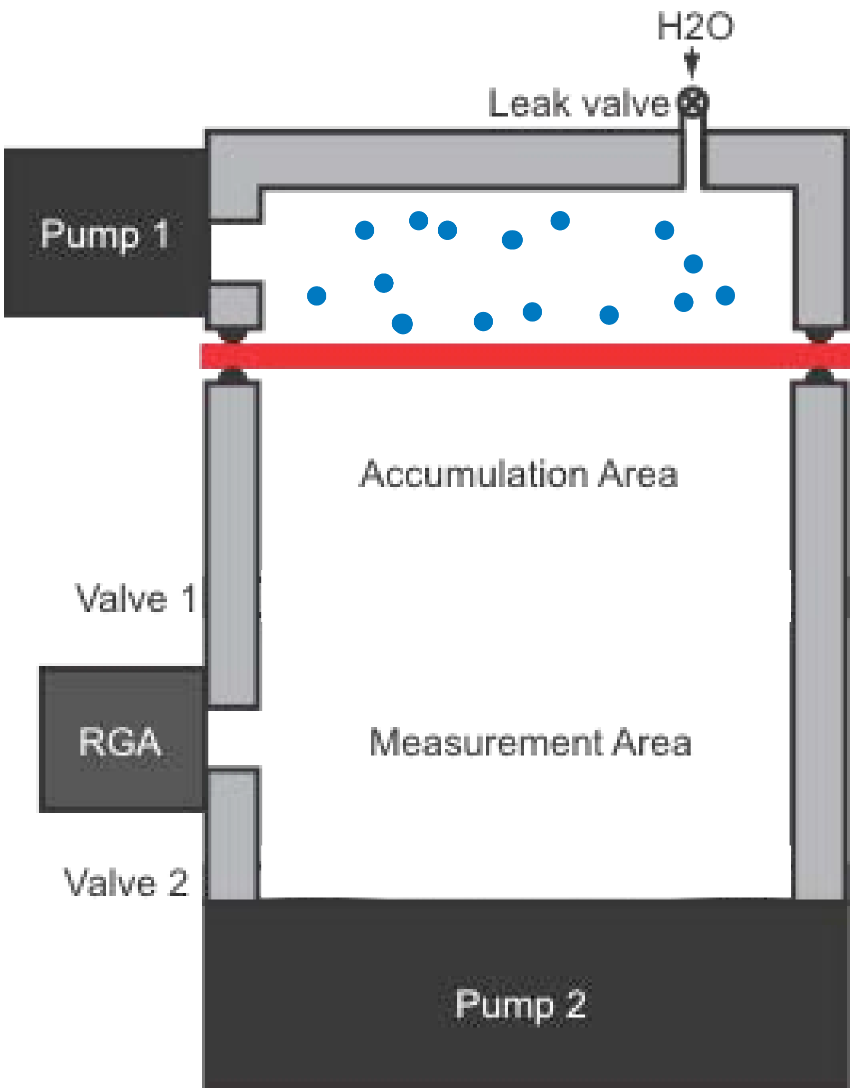
1 $g/m^2/day$ – 1×10^{-6} $g/m^2/day$

Pros: sensitivity; throughput; controlled temp/humidity

Cons: cost

R. Paetzold *et al.*, *Review of Scientific Instruments*, vol. 74, pp. 5147, 2003.

Mass Spectrometry



Standard: None

Measurement:

$$\frac{\partial P_{H_2O}}{\partial t} \Rightarrow molH_2O(t) \Rightarrow WVTR(t)$$

Sensitivity Limits:

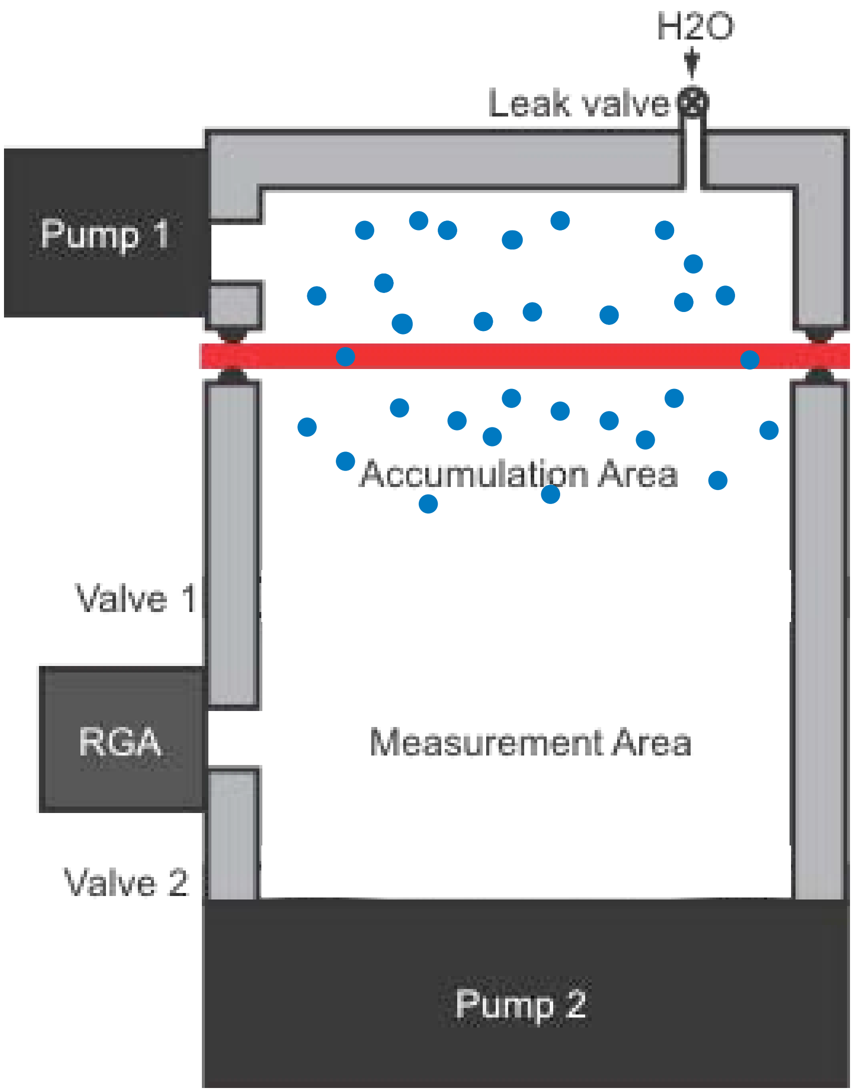
1 g/m^2/day – 1×10^{-7} g/m^2/day

Pros: sensitivity

Cons: cost; throughput; limited temp/humidity

A. Ranade *et al.*, Review of Scientific Instruments vol. 76, p. 013902, 2005.

Mass Spectrometry



Standard: None

Measurement:

$$\frac{\partial P_{H_2O}}{\partial t} \Rightarrow molH_2O(t) \Rightarrow WVTR$$

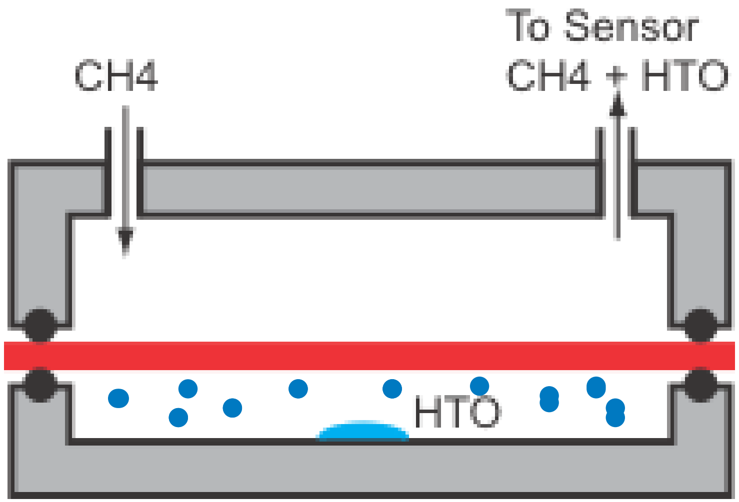
Sensitivity Limits:

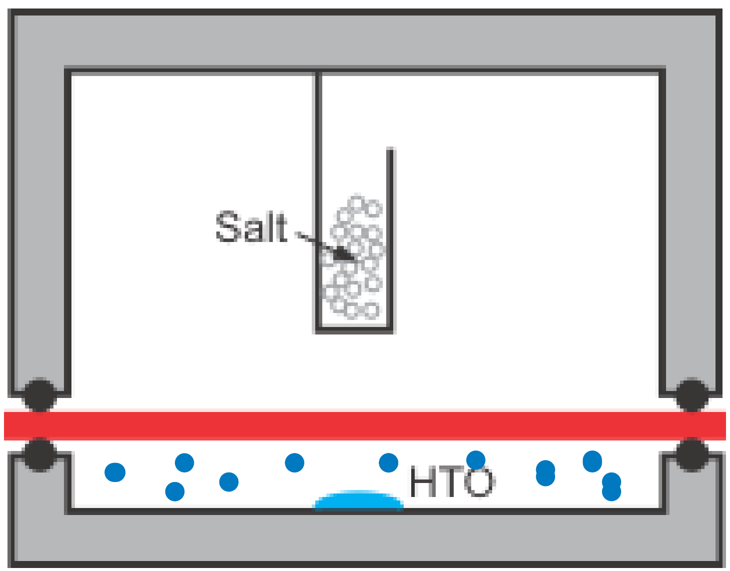
1 g/m^2/day – 1×10^{-7} g/m^2/day

Pros: sensitivity, multiple permeates simultaneously

Cons: cost; throughput; limited temp/humidity

Radioactive Tracer





Standard: None

Measurement:

Ionization: $i(t) \Rightarrow molT(t) \Rightarrow molH_2O(t) \Rightarrow WVTR$

Scintillation: $I(t) \Rightarrow molT(t) \Rightarrow molH_2O(t) \Rightarrow WVTR$

Sensitivity Limits:

1 g/m^2/day – 1×10^{-6} g/m^2/day

Pros: sensitivity; throughput

Cons: radioactivity; 100% RH

R. Dunkel *et al.*, *Proceedings of the IEEE, vol. 93, p. 1478, 2005.*

NREL's Ca Test

Electrical Ca Test (0.1 mΩ resolution)

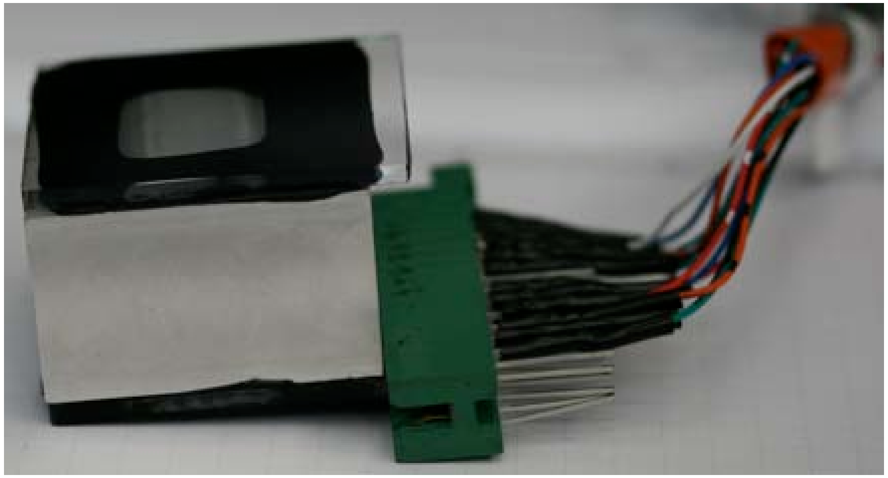
Easily configurable test card & edge connector assembly

•fast setup

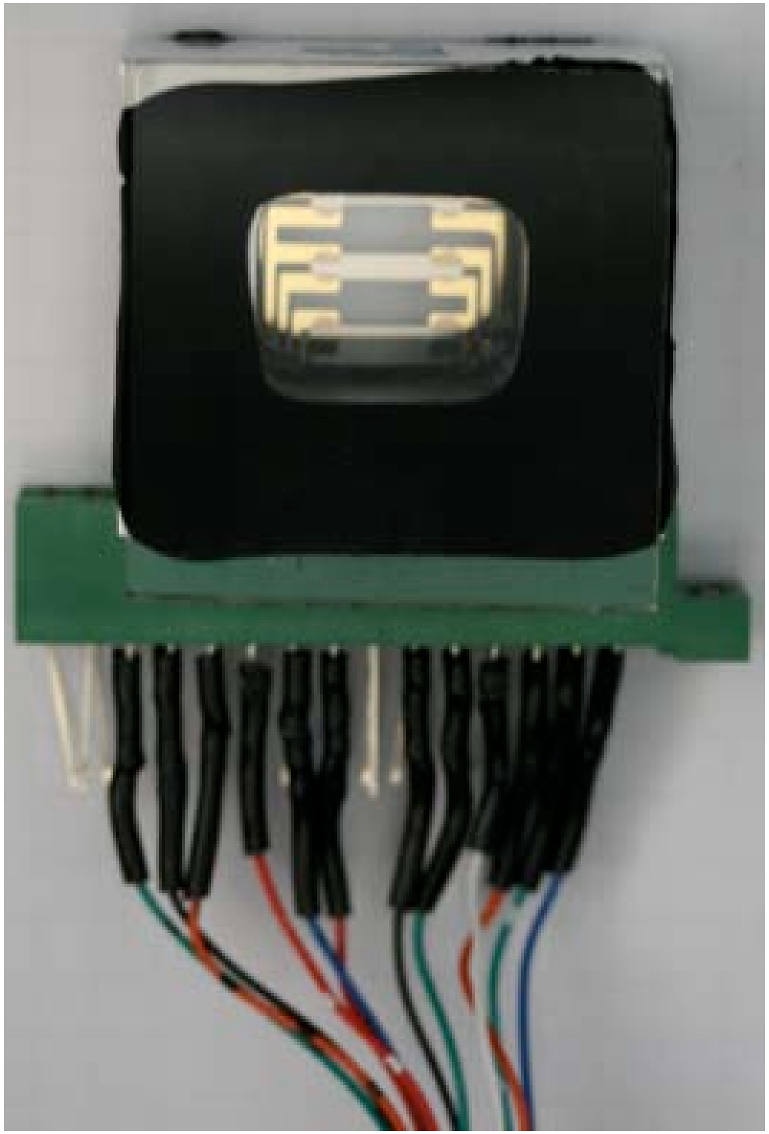
•no need to deposit Ca onto the barriers

16 tests simultaneously = high throughput

Variety of environmental conditions possible



Limits/Sensitivity



Sensitivity Limits

Theoretical:

$\sim 1\times10^{-7}$ g/m^2/day

Current Experimental:

$\sim 1\times10^{-5}$ g/m^2/day

Can measure WVTR @ 1×10^{-5} g/m^2/day within 24 h (post lag time)

Test Setup

Each test card:

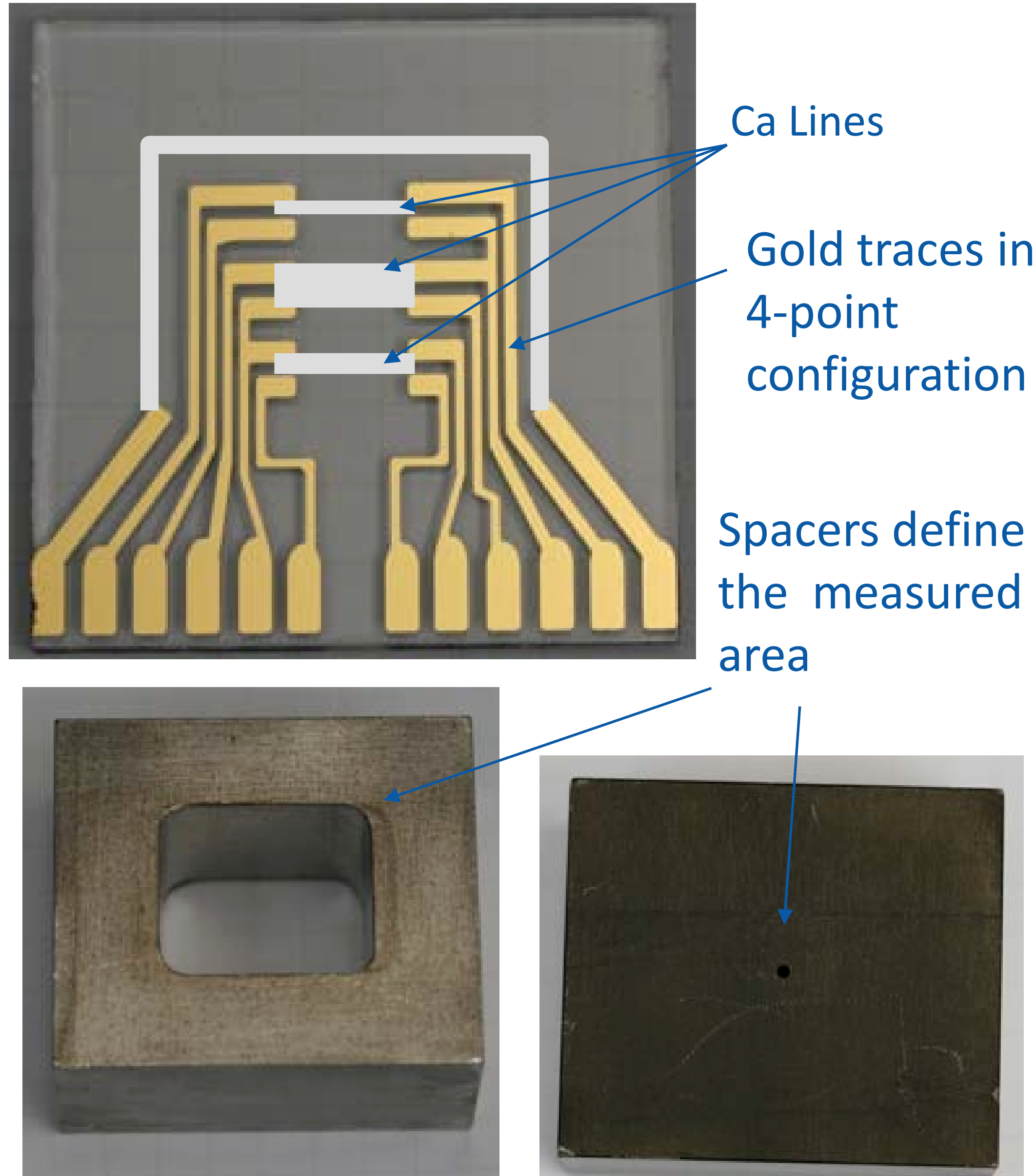
•Three 4-pt configured experimental traces

•1 edge seal witness

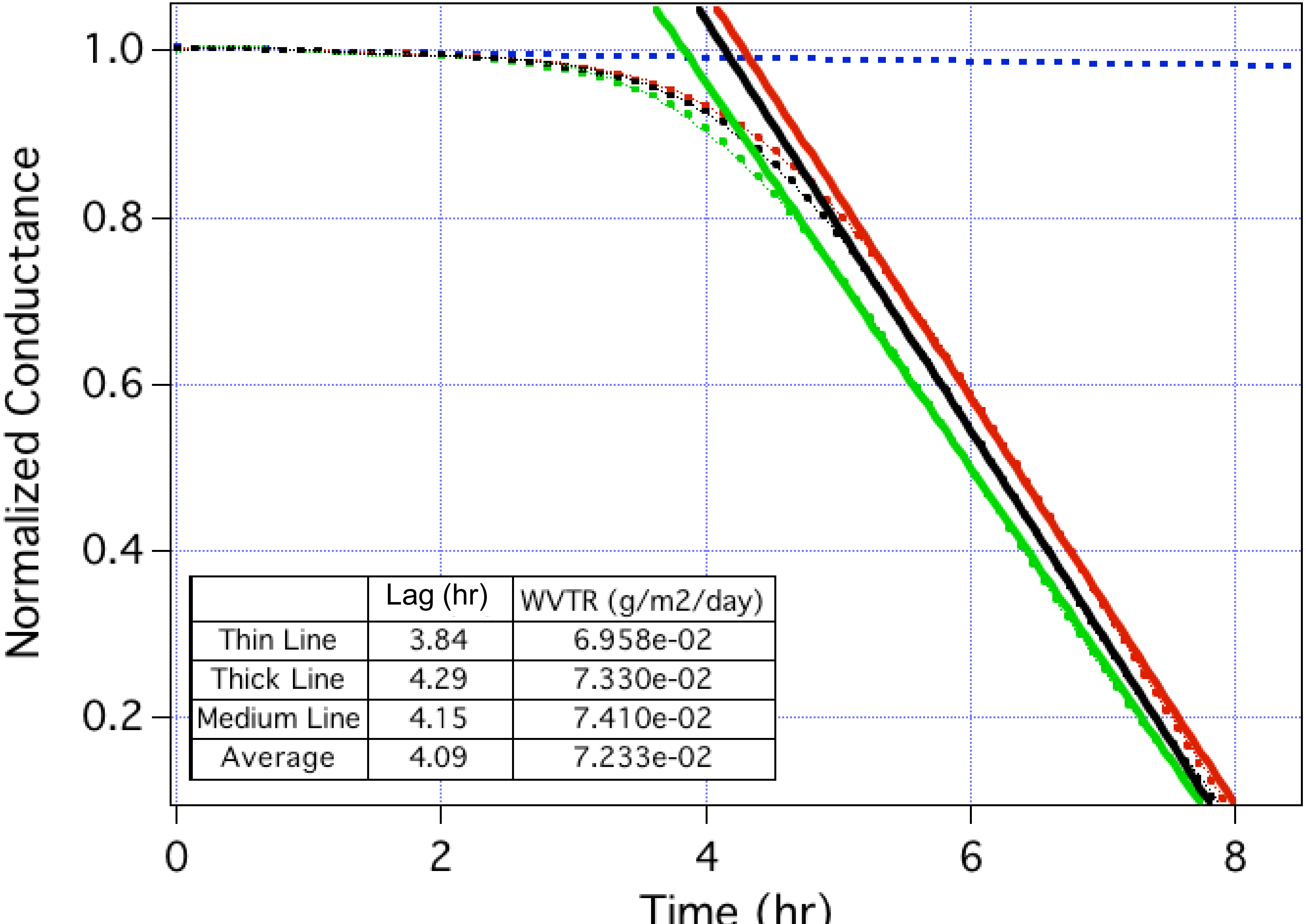
Spacers:

•Variety of test areas possible

•Incorporated diffusion length to average out pinhole/defect effects



Example: 70 nm IZO on PEN 40C/85%RH



	Lag (hr)	WVTR (g/m2/day)
Thin Line	3.84	6.958e-02
Thick Line	4.29	7.330e-02
Medium Line	4.15	7.410e-02
Average	4.09	7.233e-02

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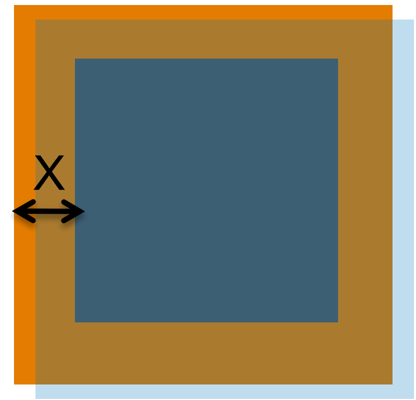
Edge Seal Testing

Using the Ca Test concept as an effective means of evaluating edge seal materials similar to applied environment

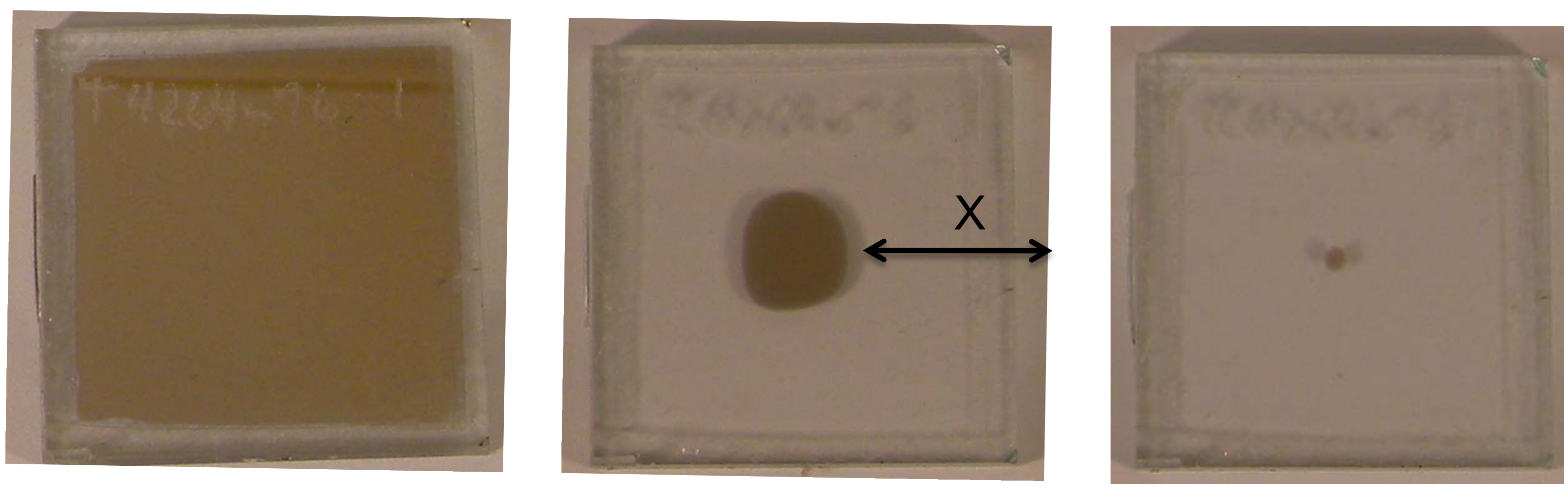
$$X = K\sqrt{t}$$



Ca turns transparent appearing to shrink toward center

PDMS

85C/ 85 %RH



0 h **3 h** **4.5 h**

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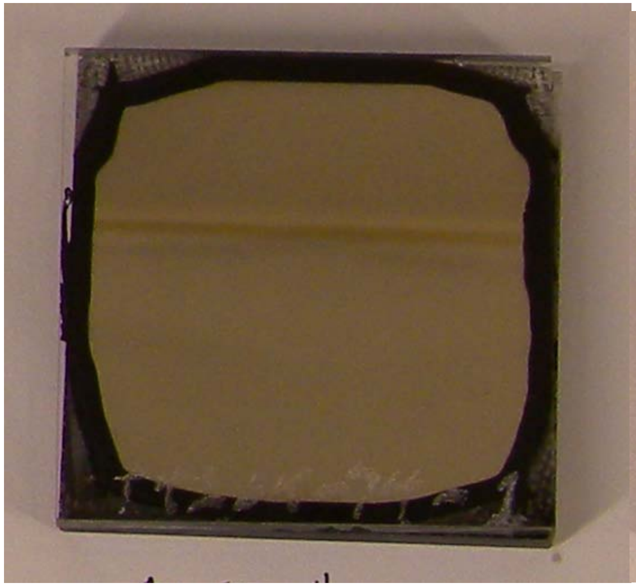
Polyisobutylene 1

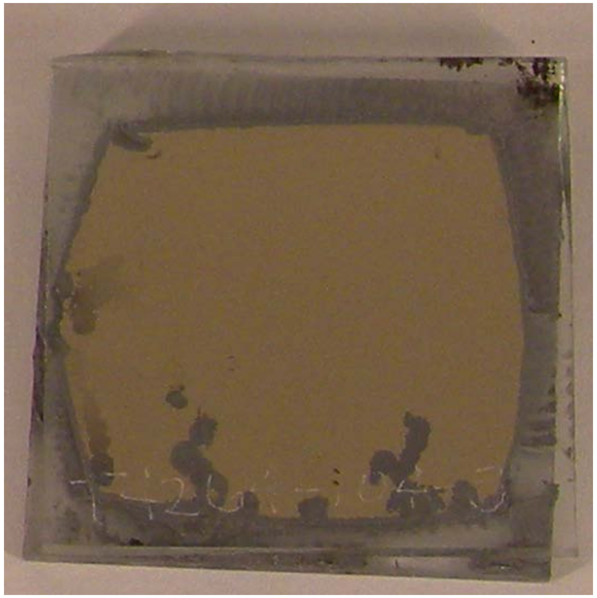


115 h

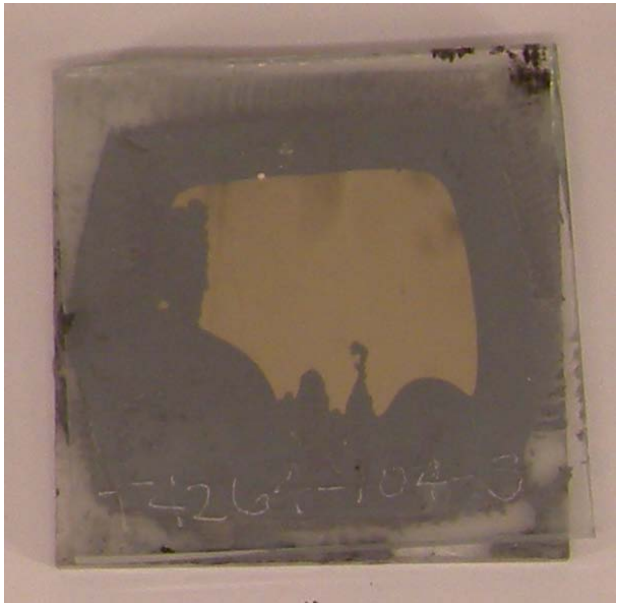
1488 h

3509 h

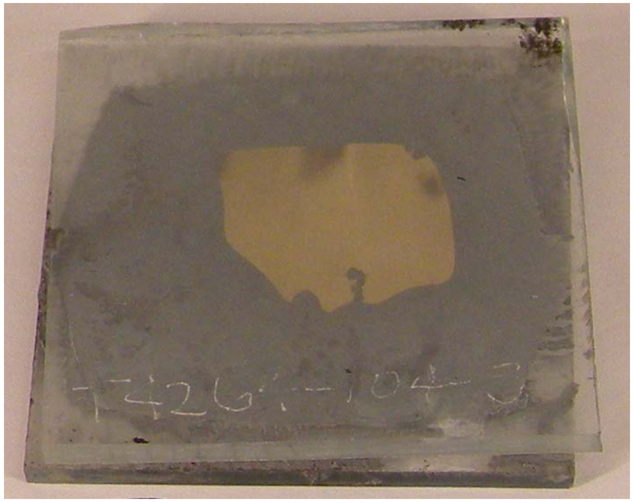
Polyisobutylene 2



3 h



652 h



1227 h

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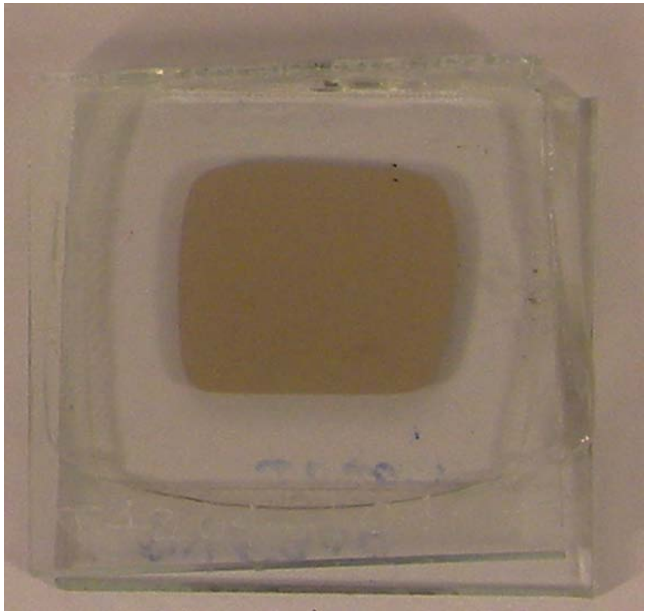
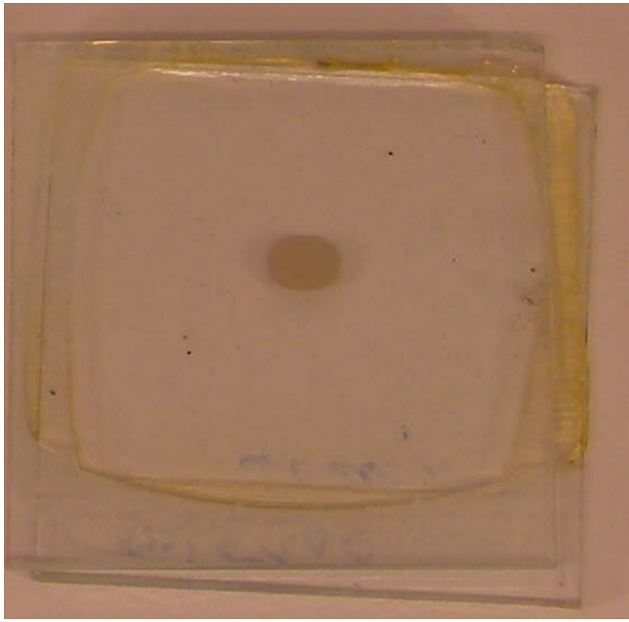
Ionomer

3 h



162 h



652 h

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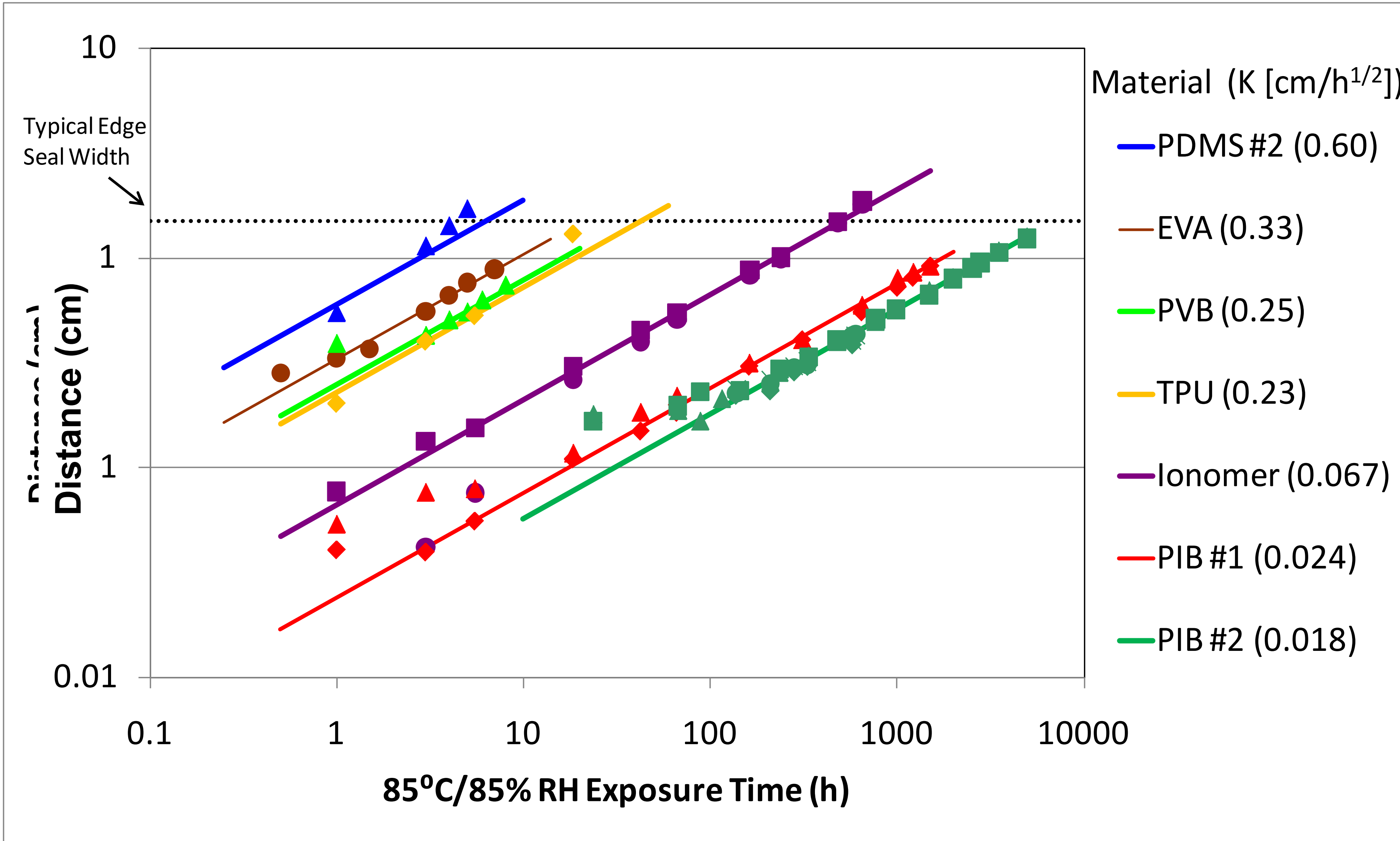
Relative Comparison of Materials



Conclusions

There are several techniques to quantifiably measure moisture permeation through thin films

At NREL we are working to improve existing barrier evaluation methods and developing routes to evaluate and compare edge seal materials in their applied environment