Traceable Measurements of Water Vapour Transmission Rate Using Cavity Ring-down Spectroscopy

Paul Brewer

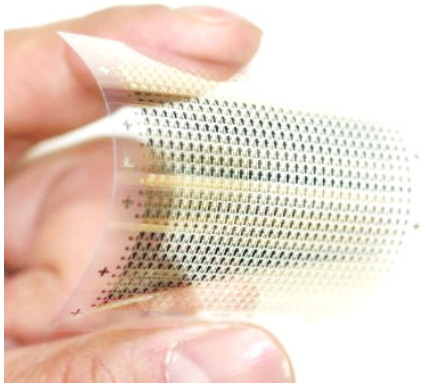
Durability of Thin Film Solar Cells: Status and Assessment

4TH April 2012

Water Vapour Transmission Rate

NPL National Physical Laboratory

- A major obstacle to introducing flexible electronics into the commercial market is their limited lifetime when exposed to water and oxygen
- Barrier layers are used to encapsulate and reduce water and oxygen ingress
- Transport is governed by the water vapour transmission rate (WVTR)
- WVTR = mass transfer rate of water vapour per unit area ($g/m^2/day$)



What Level of Barrier Layer is Required?

National Physical Laboratory

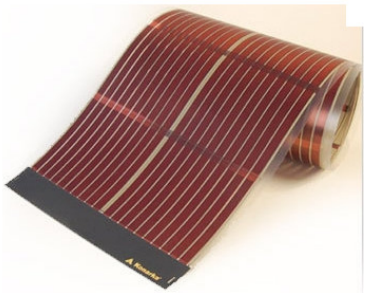
$10^{-0} – 10^{2}$ Sensitive food and pharmaceutical packaging

$10^{-2} – 10^{0}$ Thin film inorganics e.g. LCD, LED

$10^{-4} – 10^{-2}$ Other PV systems, OFETS and VIPs

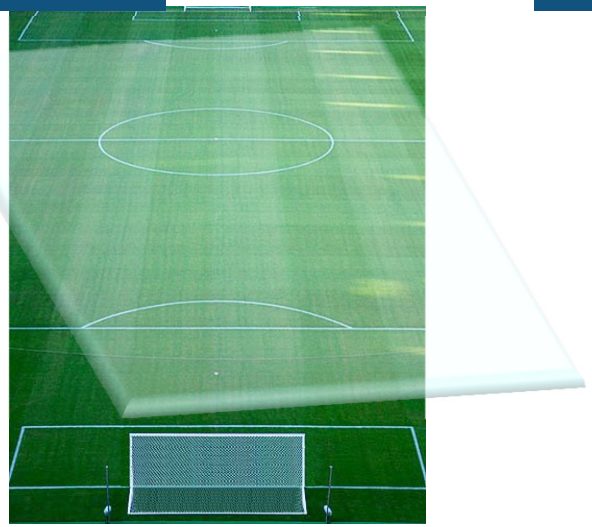
$10^{-5} – 10^{-4}$ OPV systems

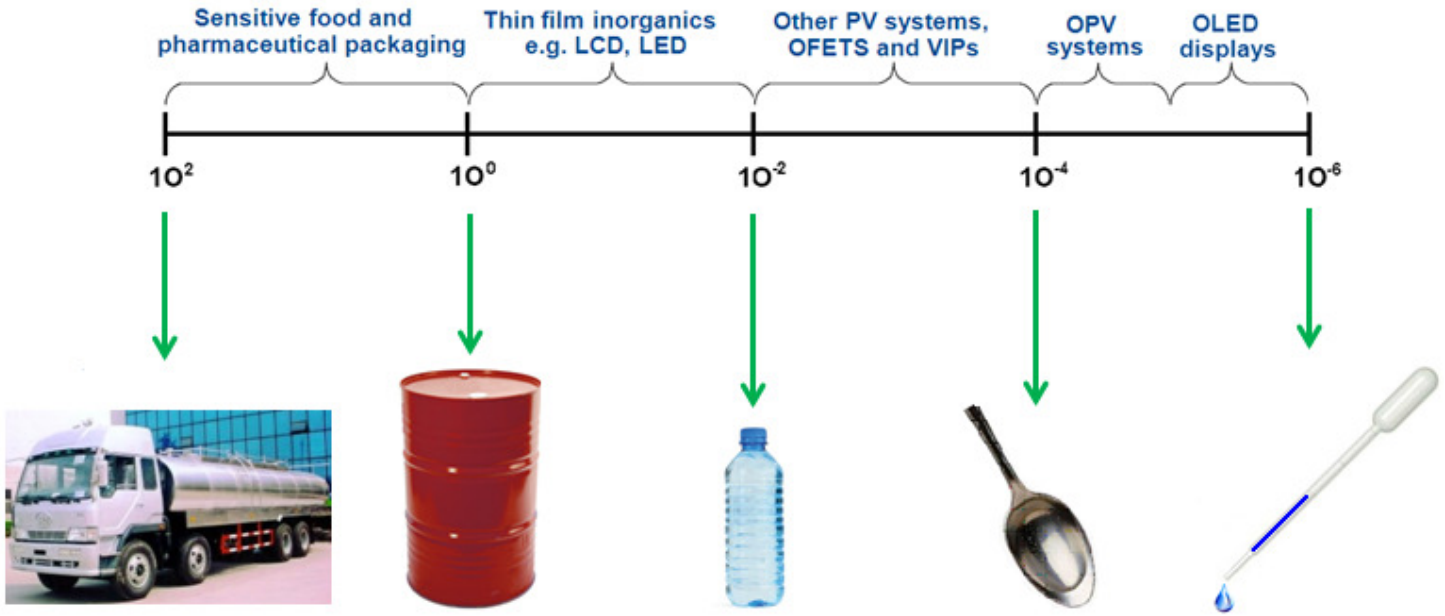
$10^{-7} – 10^{-6}$ OLED displays



What Does This Mean?

- Imagine a barrier layer the size of a football pitch (~100 x 50 m).
- How much water would pass through in one month at the various performance levels?



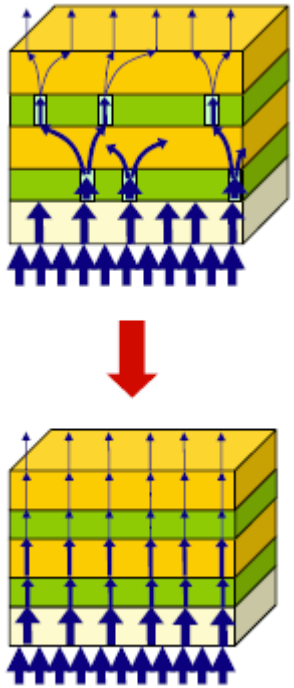


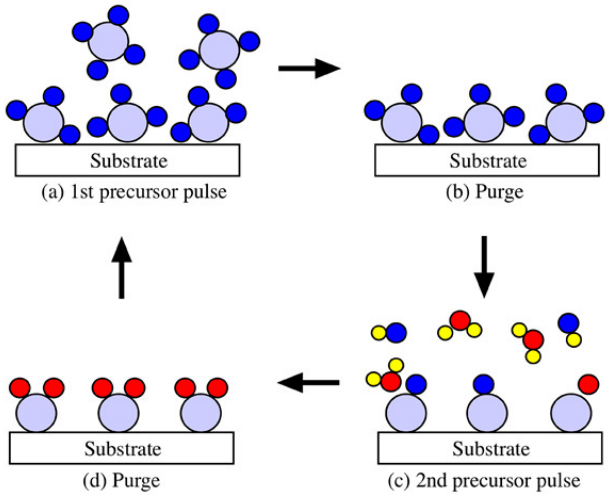
How are High Performance Barrier Layers Produced?

NPL National Physical Laboratory

Several approaches:

- Sputter deposition
- Atomic layer deposition
- Single layer and composite barriers



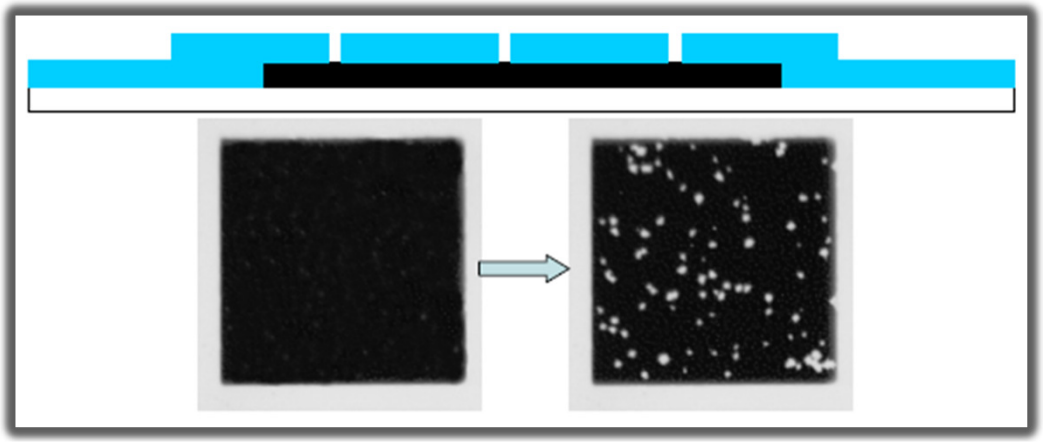


To assess efficacy of these barrier layers measurements of WVTR are required

Current State of the Art

NPL National Physical Laboratory

- Calcium test
- Coulometric methods such as the MOCON test
- Mass spectrometry
- Radioactive methods with tritiated water

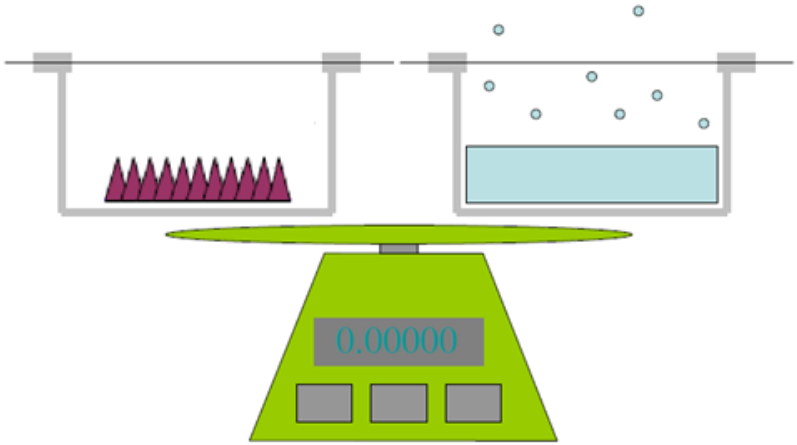


WVTR Standards

NPL National Physical Laboratory

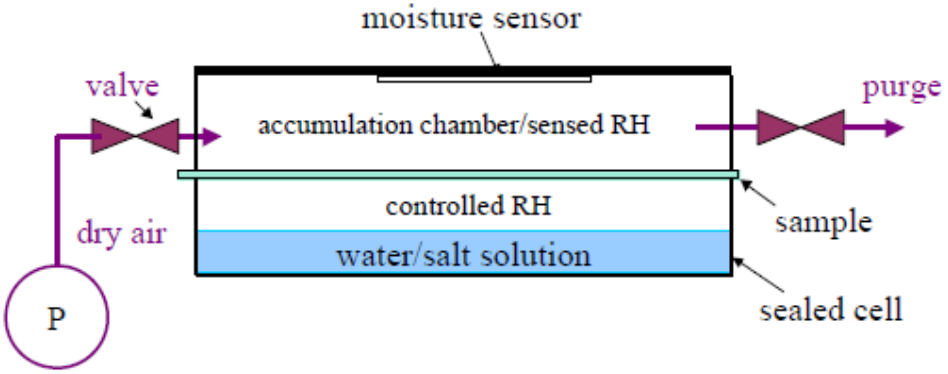
Gravimetric techniques

ASTM D1653
ASTM E96 (>10 g/m^2/day)
EN ISO 7783-1
ISO 2528



Accumulation techniques

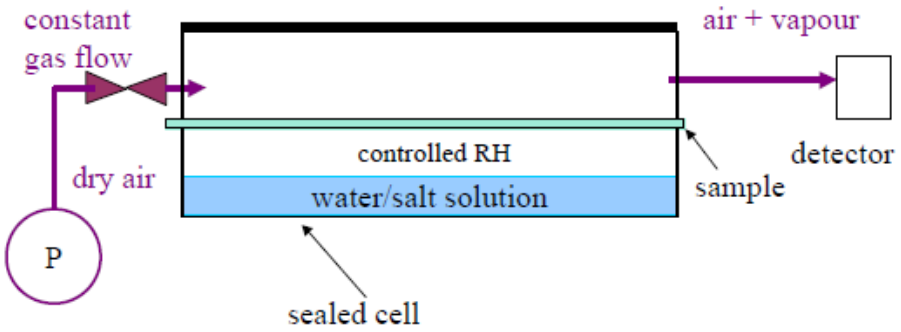
ASTM E398 (>0.01 g/m^2/day)



NPL National Physical Laboratory

Isostatic techniques

ASTM F 1249-01 (>5 x 10^{-3} g/m^2/day)
ISO 15106-2



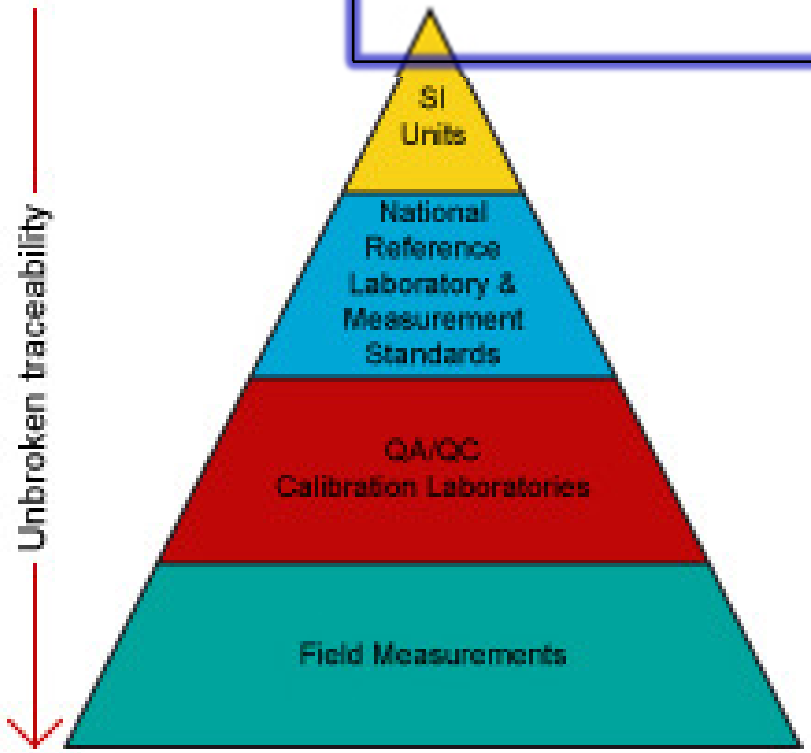
Different conditions of temperature and humidity employed in each

To enable flexible organic electronics with barriers 10^{-4} - 10^{-6} g/m^2/day, **accurate** and **traceable** measurements are required

What is Traceability?

NPL National Physical Laboratory

> *"Property of a result of a measurement (or the value of a standard) whereby it can be related to stated references, (usually national or international standards), through an unbroken chain of comparisons all having stated uncertainties"*
>
> *International Vocabulary for Metrology*

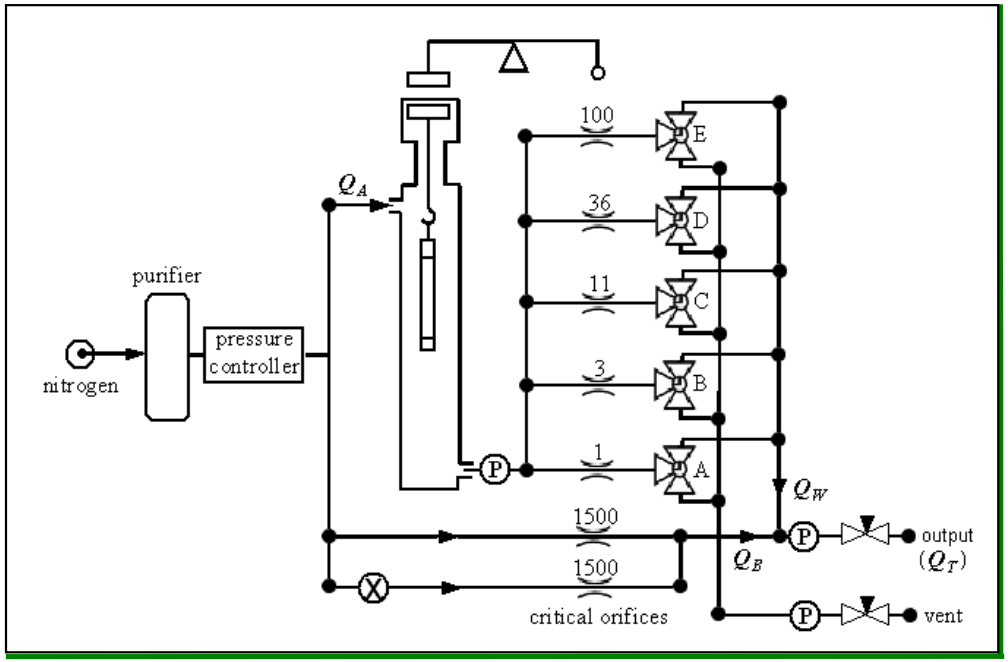


Traceability is a means to provide:

- Comparability
- Stability
- National and International recognition

Primary Trace Water Vapour Facility

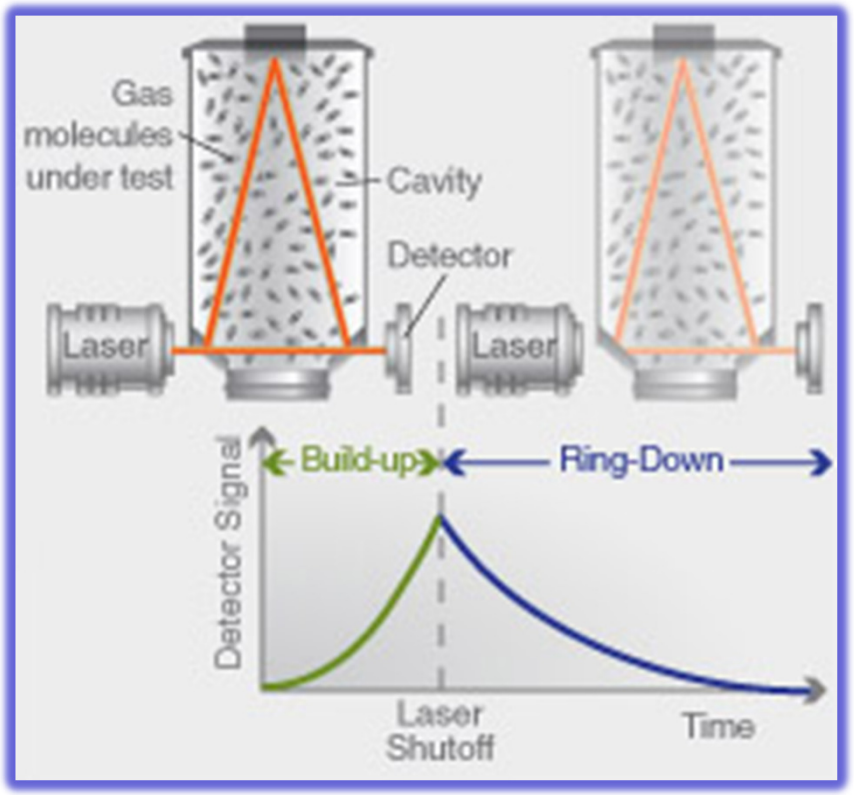
- To underpin process gas and microelectronics industries
- NPL internationally leading in trace water vapour analysis
- 2% uncertainty k=2

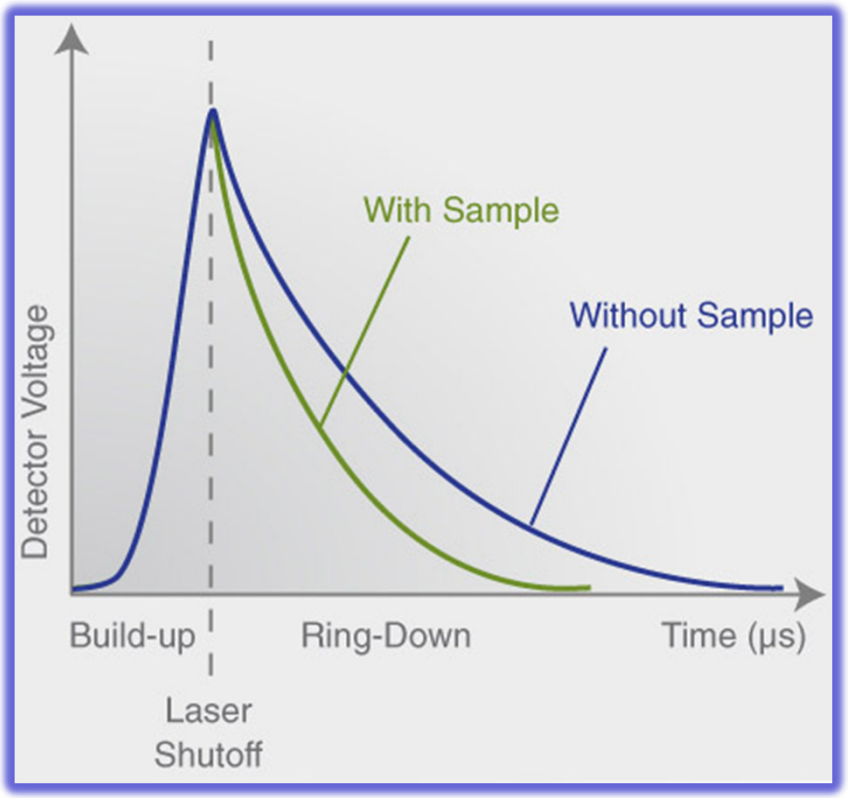


- Permeation device coupled to a novel dilution system
- H_2O standards from single figure ppb to ppm amount fractions.

P. J. Brewer, B. A. Goody, P. T. Woods, M. J. T. Milton, *Rev. Sci,. Inst.*, 82, 105102, (2011).

Cavity Ring Down Spectroscopy

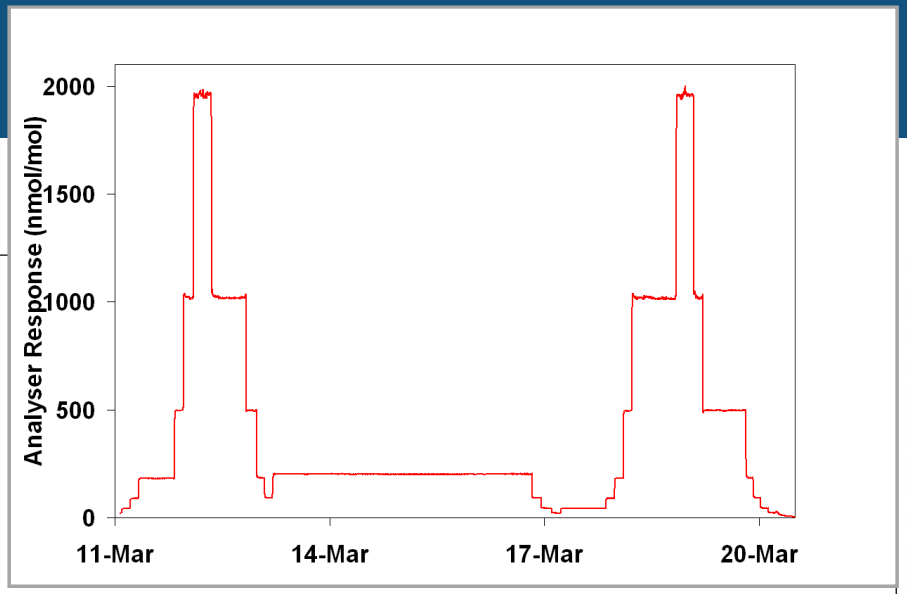


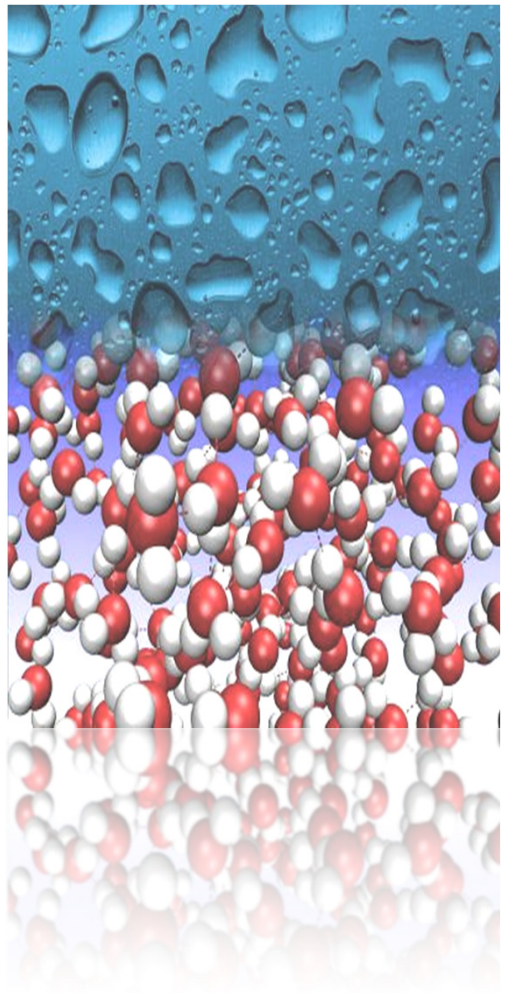


- H_2O has a unique near-infrared absorption spectrum (sharp lines at a characteristic wavelength)
- Effective path-length of several kilometres
- High sensitivity for detection at ppb levels in seconds

EURAMET 1002

NPL National Physical Laboratory





Rel. Degree of Equivalence (%)

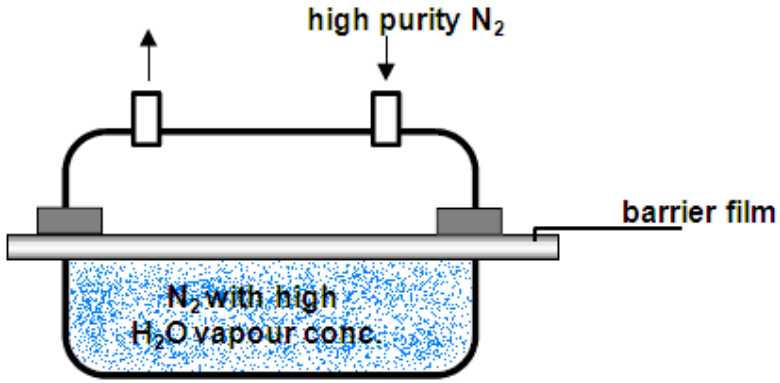
20 15 10 5 0 -5 -10 -15

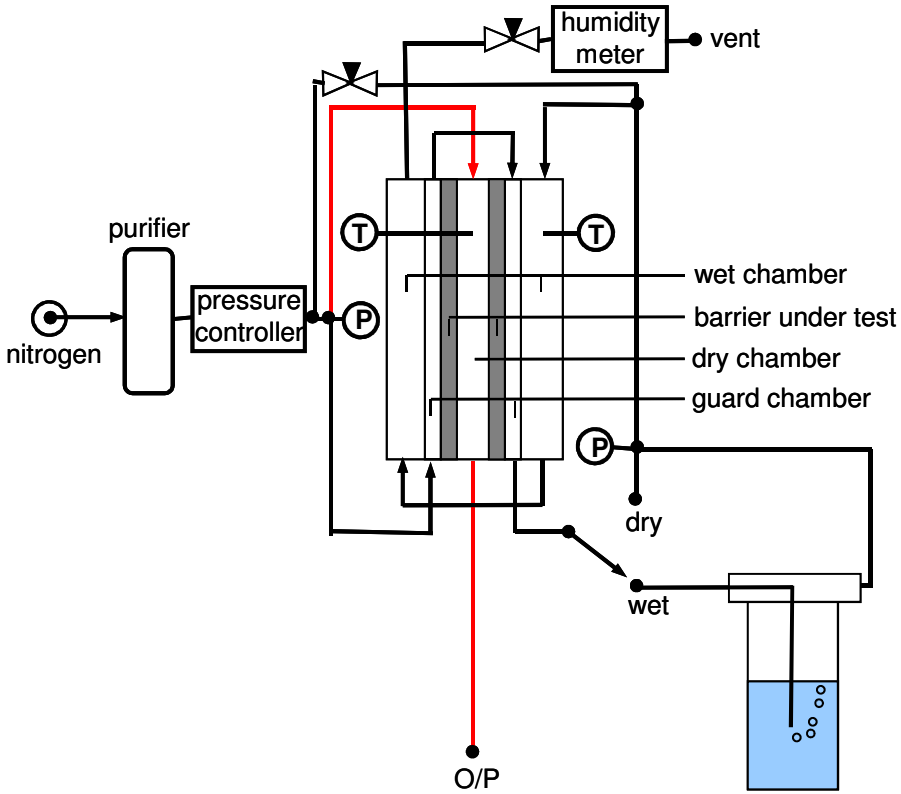
10 20 50 100 200 500 1000 2000

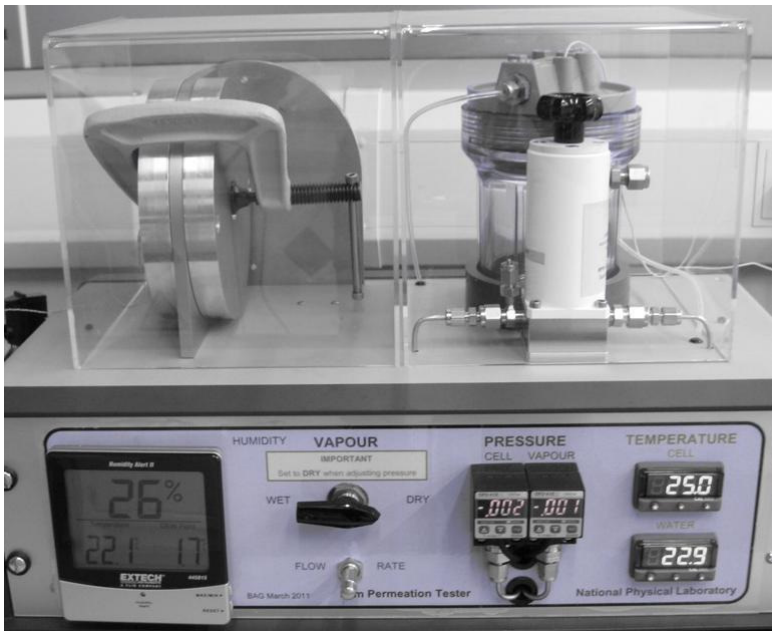
Nominal Amount Fraction (nmol/mol)

NIST, NPL, NMIJ & PTB

NPL WVTR Facility



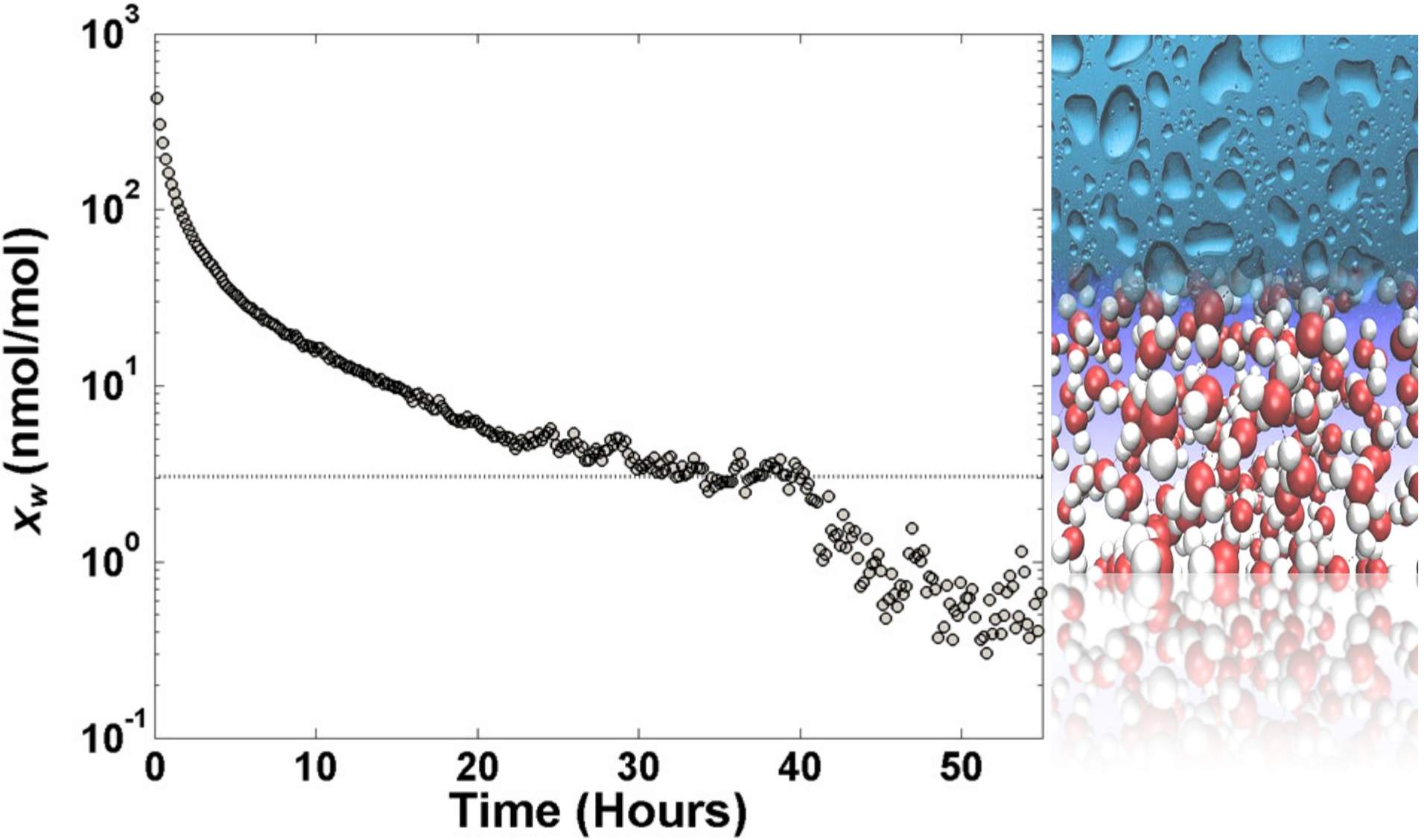




Underpinned by:

traceable H_20 standards

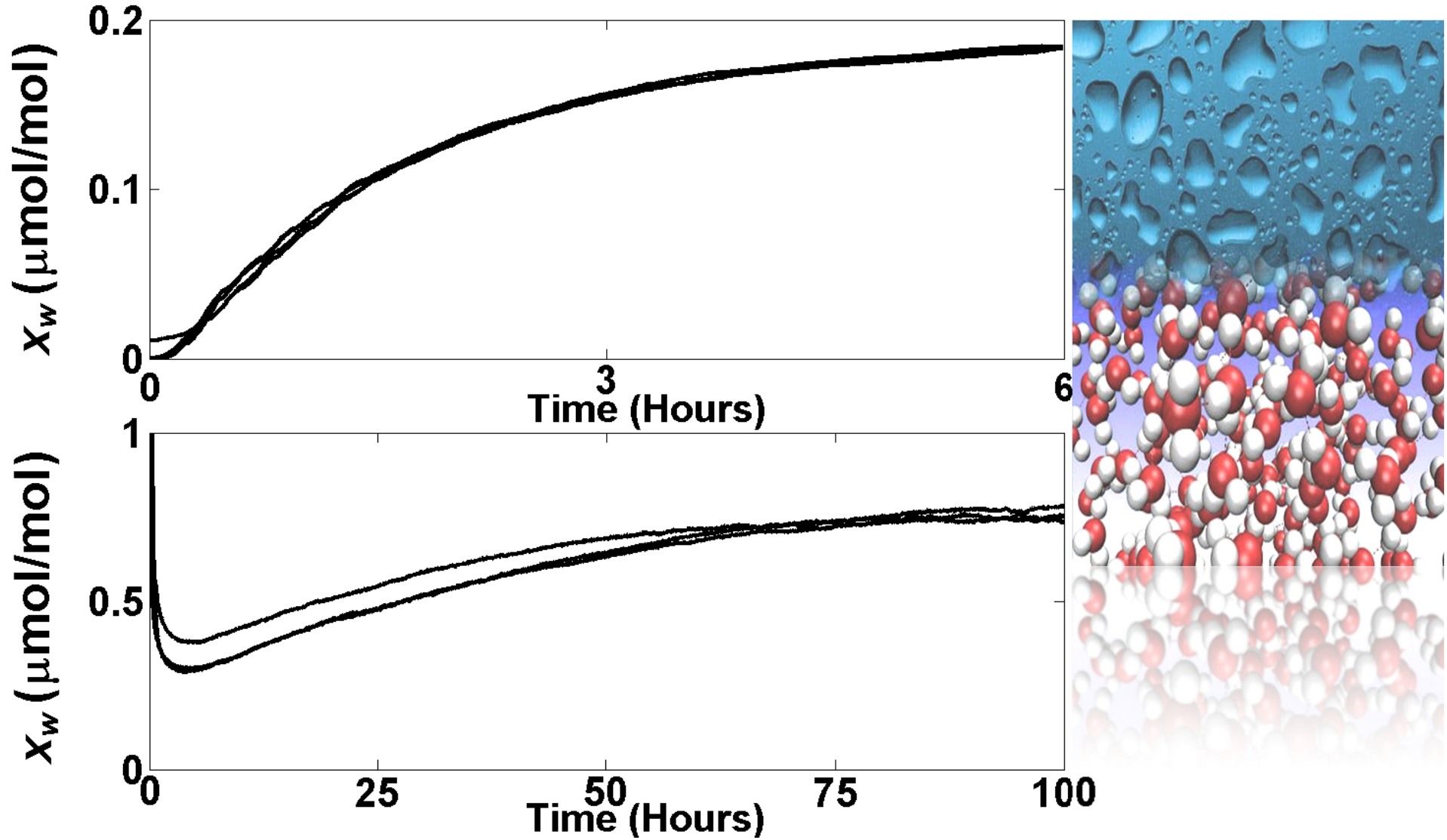
Detection Limit



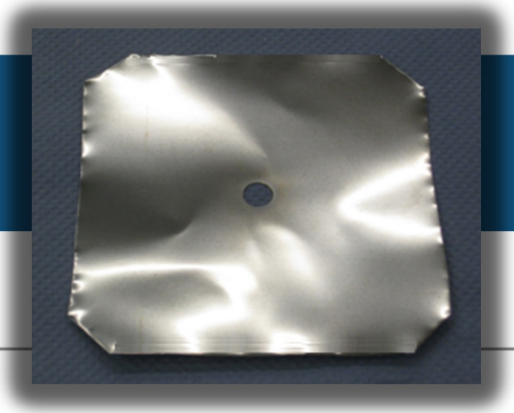
Repeatability

NPL National Physical Laboratory

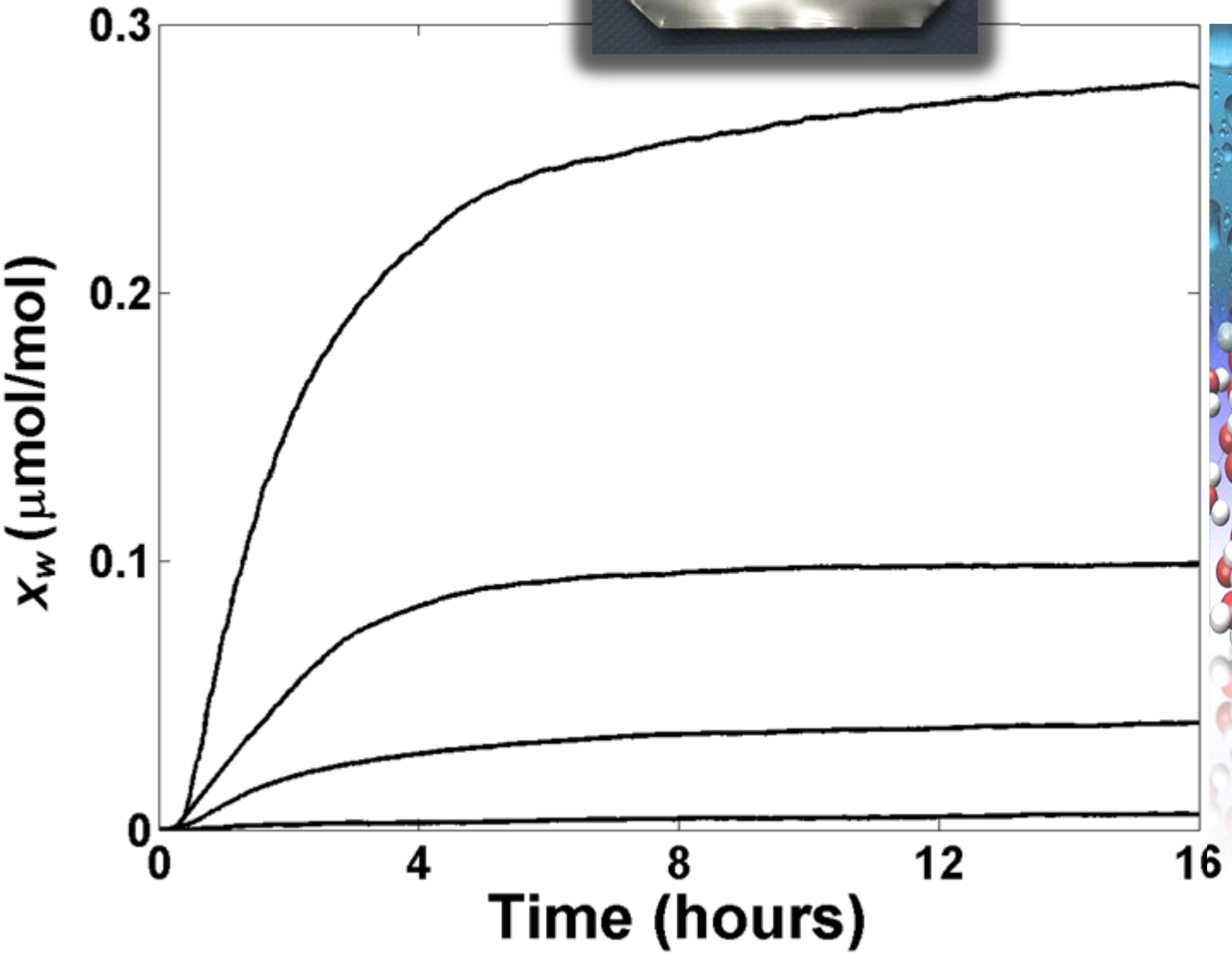
Figure 3

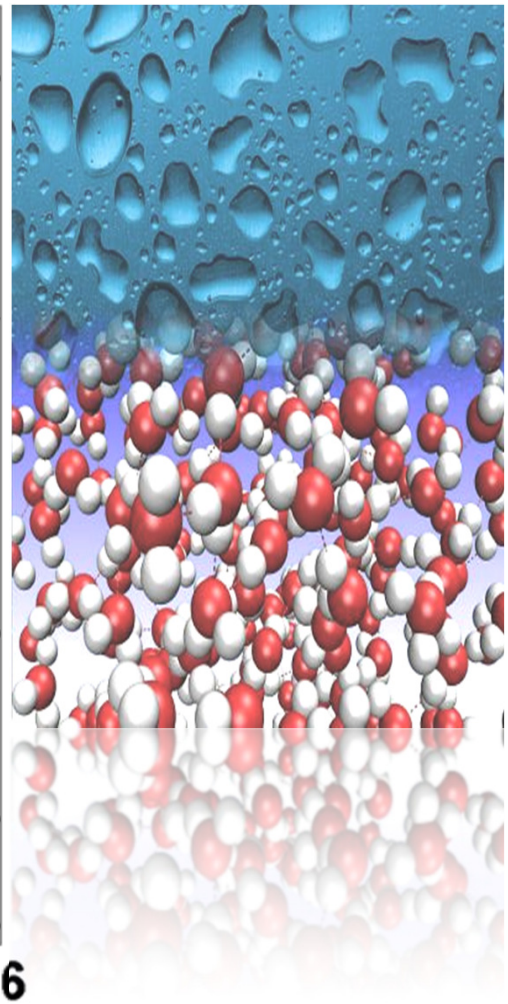


Reference Films

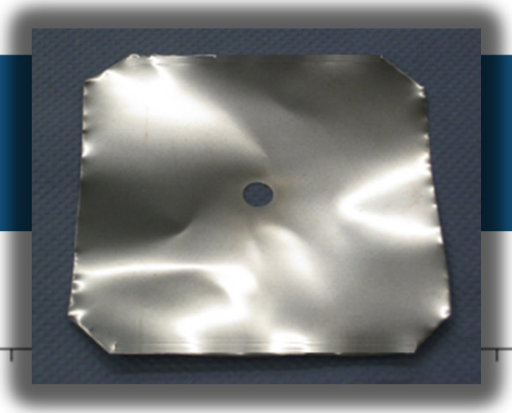


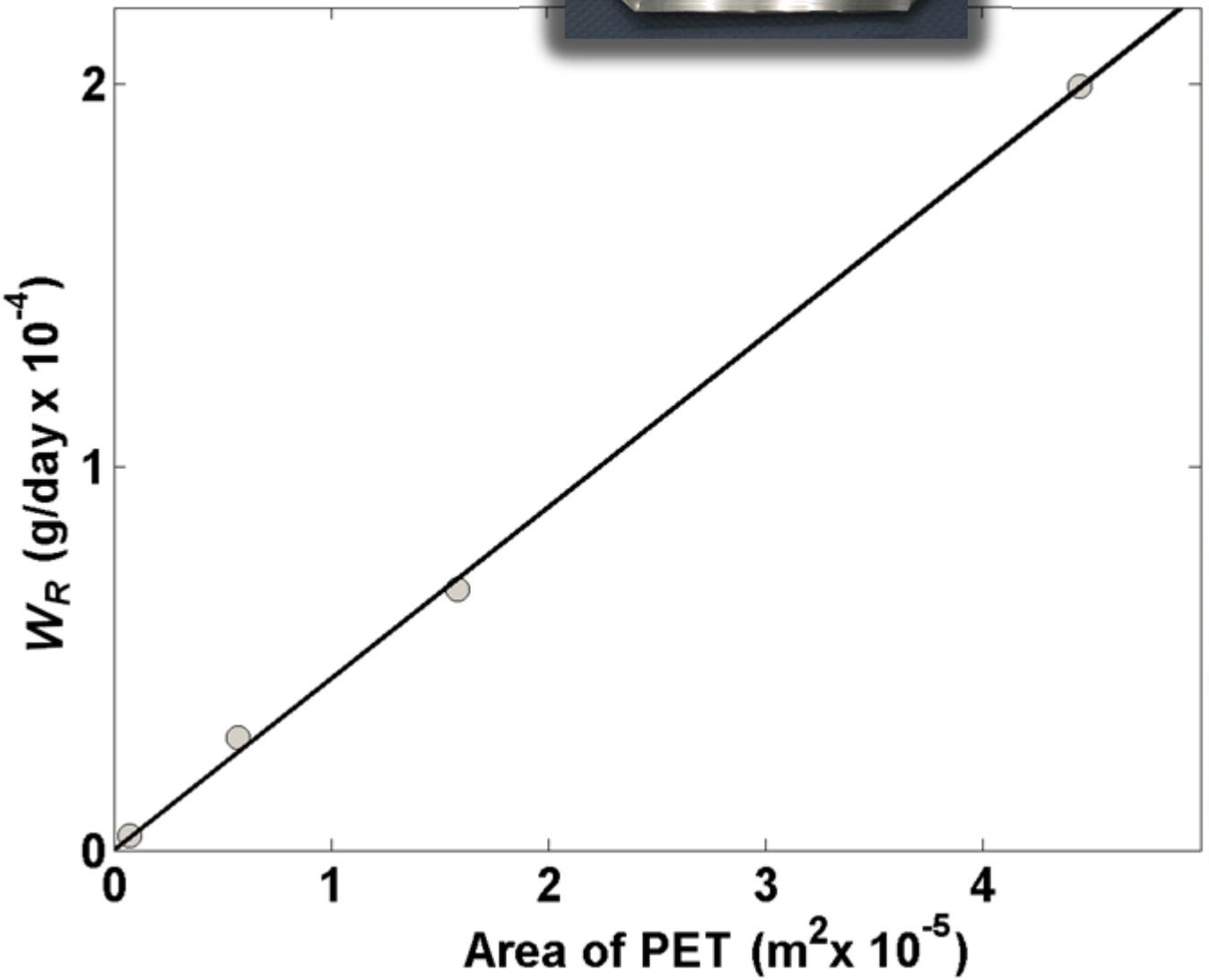
NPL
National Physical Laboratory

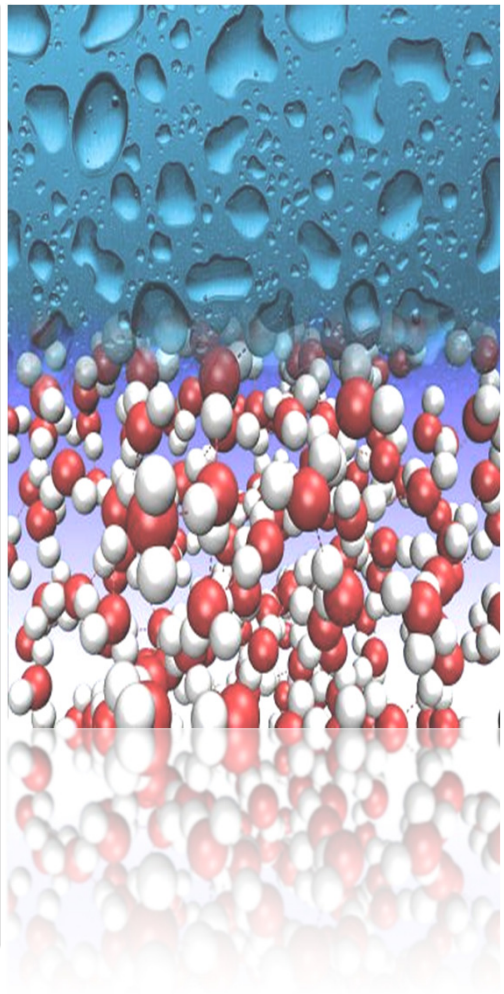




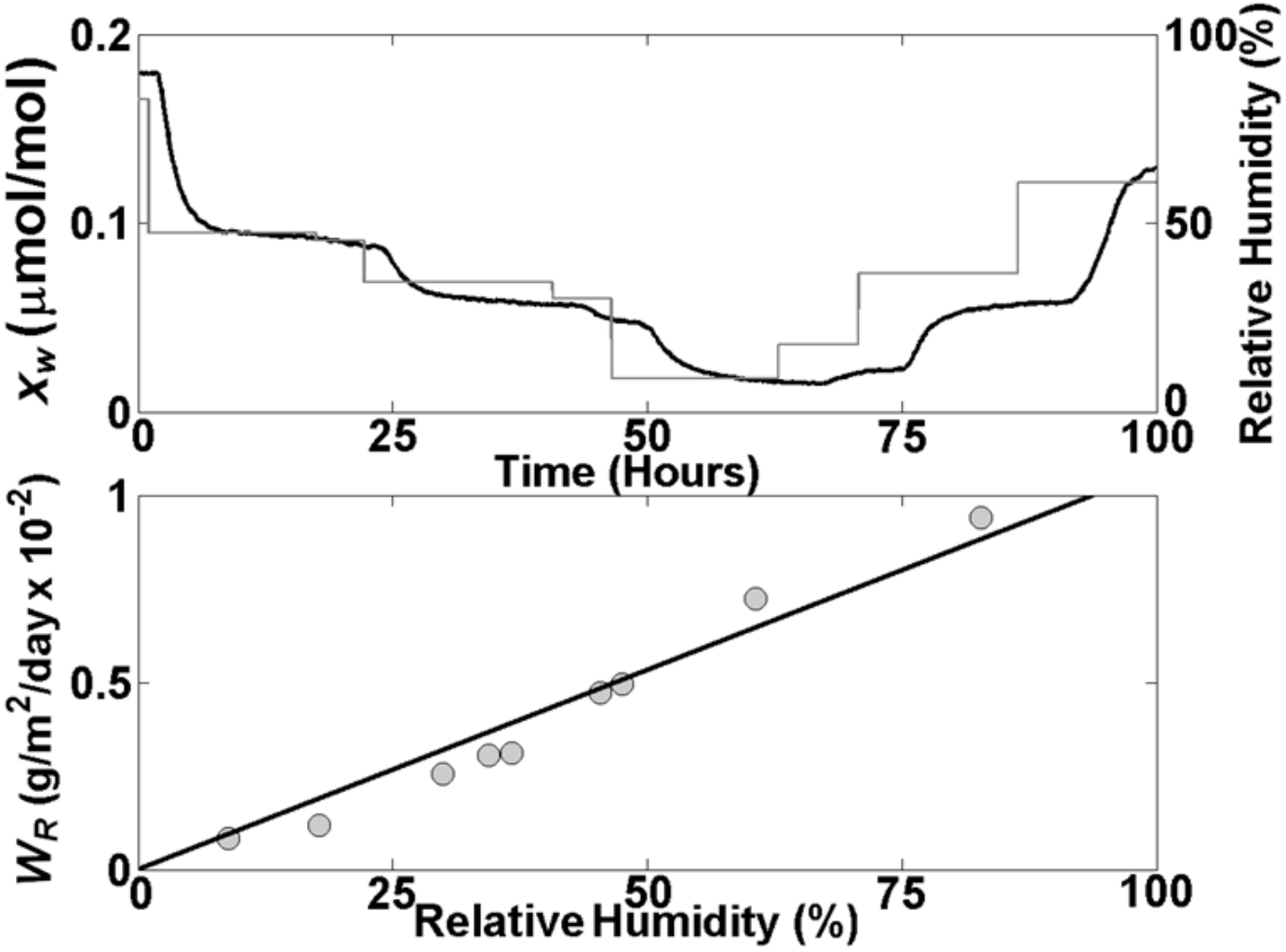
WVTR v Area of PET

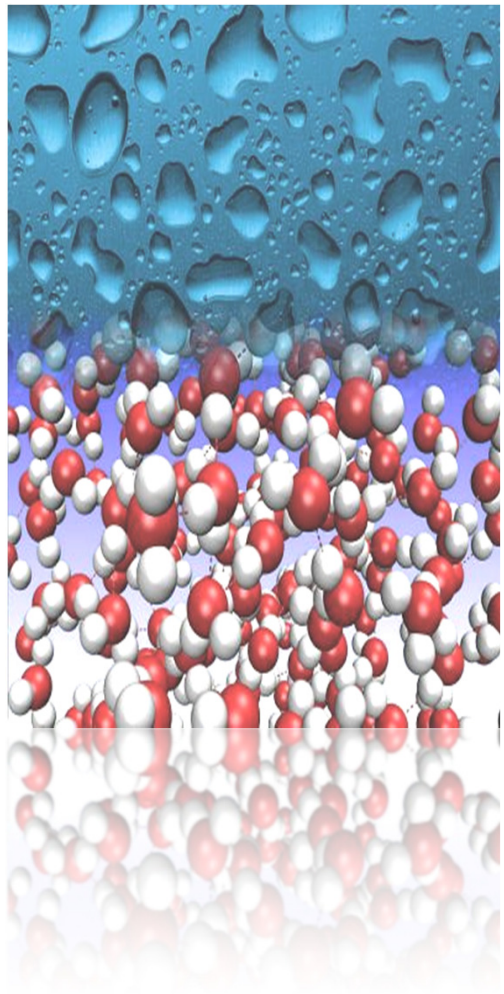




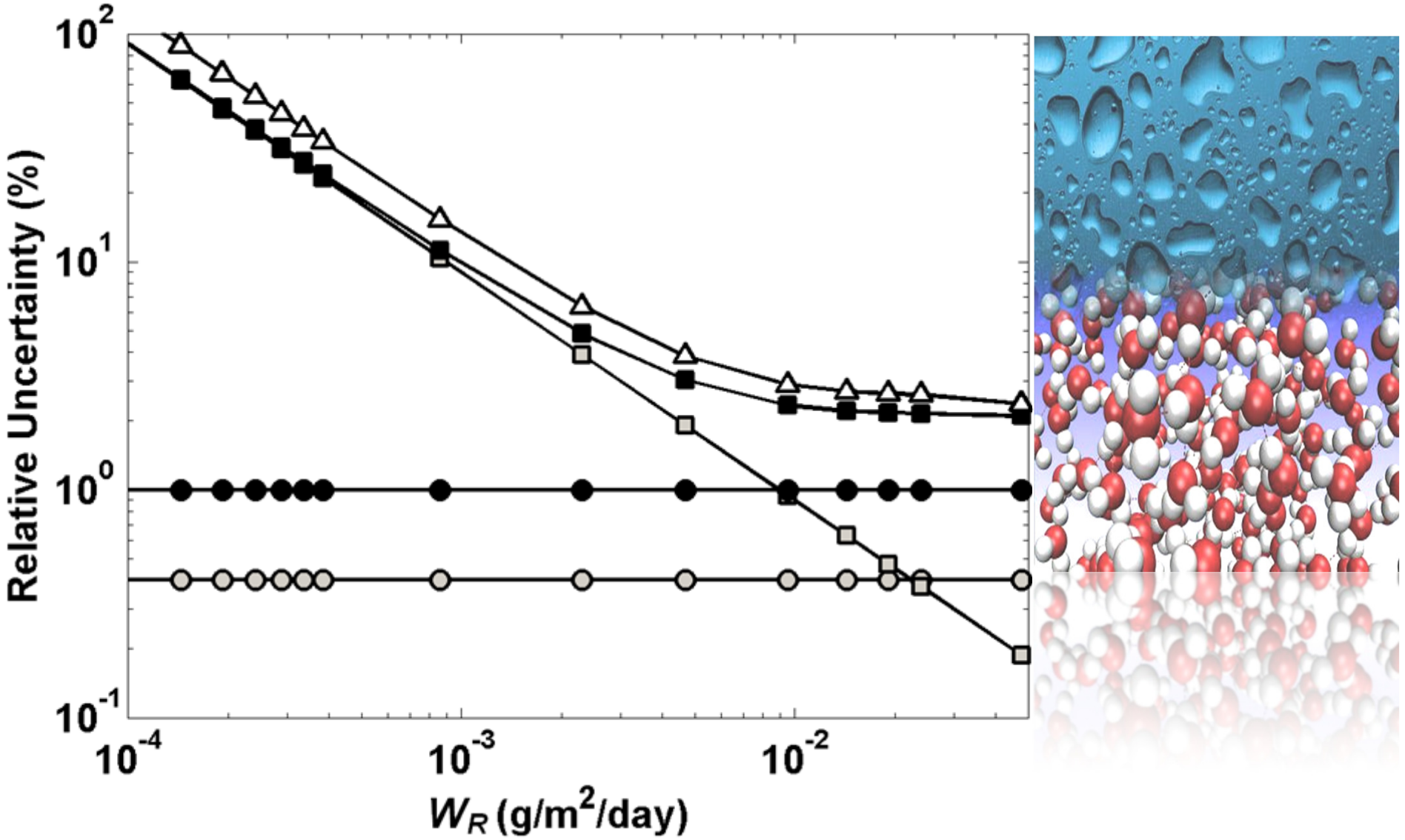


WVTR v Relative Humidity

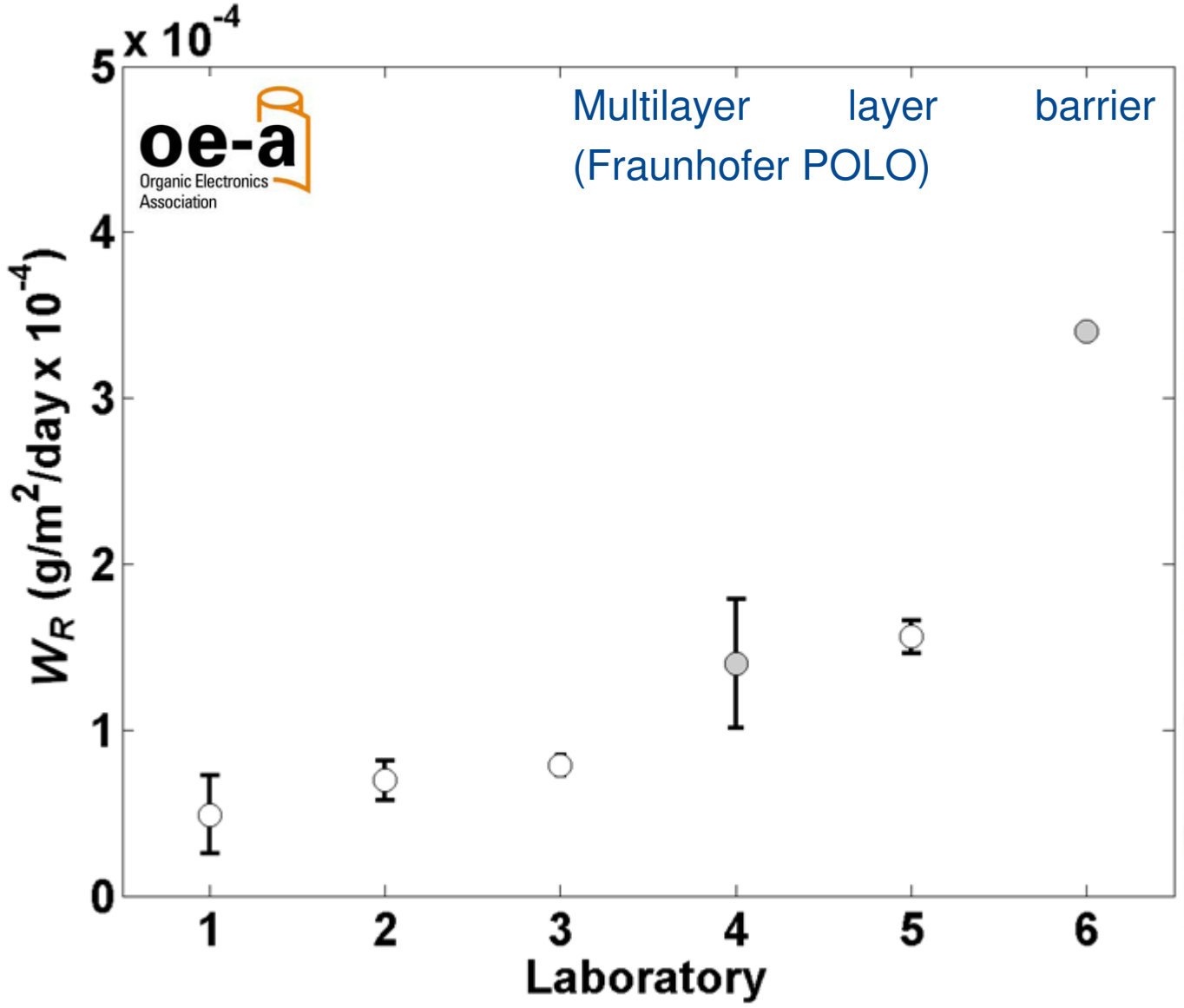


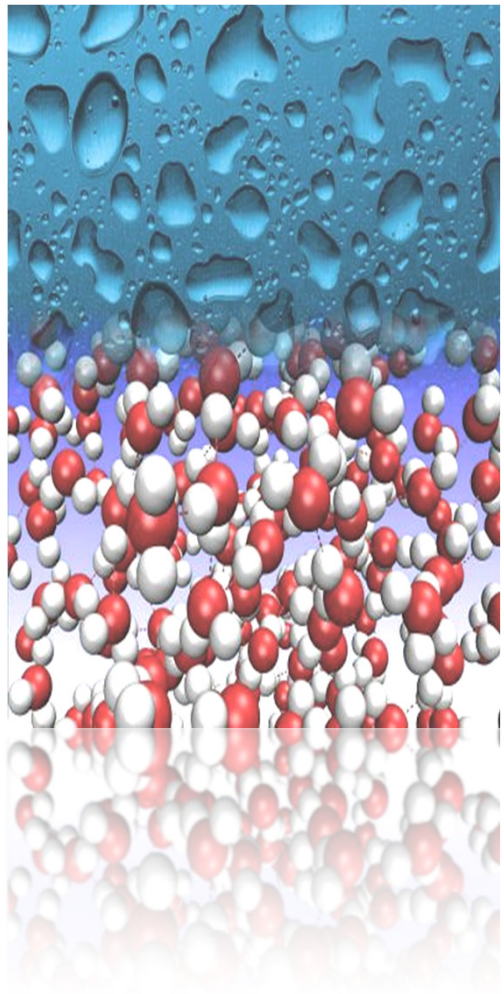


Uncertainty



Comparison


x 10^-4
5
4
3
2
1
0
W_R (g/m^2/day x 10^-4)
1
2
3
4
5
6
Laboratory
oe-a
Organic Electronics Association
Multilayer layer barrier
(Fraunhofer POLO)



Conclusions

NPL National Physical Laboratory

- Infrastructure developed to provide accurate and traceable measurements of WVTR
- Detection limit below 1 x 10^{-4} g/m^2/day to meet industrial requirements
- Good comparability demonstrated through international comparisons

Acknowledgements

- Brian Goody, Martin Milton, Yarshini Kumar, Fernando Castro and Craig Murphy (NPL)
- Organics Electronics Association
- Fraunhofer POLO
- Plasma Quest Ltd