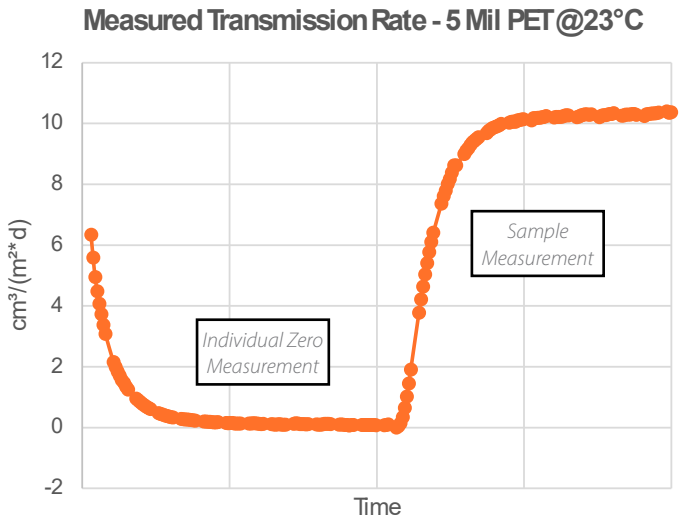
ZERO-SUM GAME

Understanding and Optimizing Individual Zero for Accurate Permeation Measurement

Background

Permeation instruments use a function called "Individual Zero" to ensure accurate transmission rate results for each sample. The purpose of the Individual Zero measurement is to account for and eliminate any leakage or background permeation bias that could affect the transmission rate (TR). To determine the true transmission rate of the sample being tested, the instrument software subtracts the Individual Zero value from the measured transmission rate result (see, below equation).

$$\frac{\text{Measured TR} - \text{Individual Zero TR}}{\text{TR of sample}}$$



Example of Individual Zero run at the beginning of a test.

INDIVIDUAL ZERO

Obtaining Individual Zero

In a permeation test, an Individual Zero is obtained by measuring a film placed in a diffusion cell and analyzed with carrier gas applied to both sides of the sample.

For an OTR test, an Individual Zero will have the N_2/H_2 carrier gas sweeping both the Test Gas side and the Carrier Gas side of the film. The COULOX® sensor in this case would be measuring the system bias for that specific sample as it is mounted in the instrument.

For WVTR, the Individual Zero for a precise RH is run in a similar way using dry N_2 on both sides of the film. For 100% RH WVTR testing, individual zero is conducted with a high-quality dense aluminum foil in place of the film.

When testing full/complete packages, samples are replaced with non-transmissive materials to simulate a Perfect Package and measure the leak rate. For instance, a glass cup or copper tubing can be used. The goal is to maintain the test setup as closely as possible, ensuring all connections have minimal leaks.

The key questions to ask are:

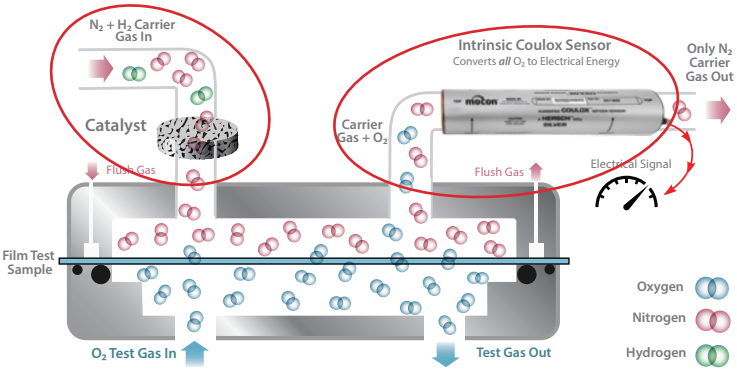
- Where could such leakage or bias occur?
- Can this effect be minimized?
- When should Individual Zero be utilized?

Bias leakage/permeation occurs in three areas

1. Leakage/permeation in the apparatus.

With room air (20.9% O_2) surrounding the instrument, there's a potential for leakage into the apparatus that would add to the measured OTR value. To minimize this effect, a catalyst is utilized just prior to the test cell that scrubs all oxygen from the carrier gas line, before it sweeps into the test cell.

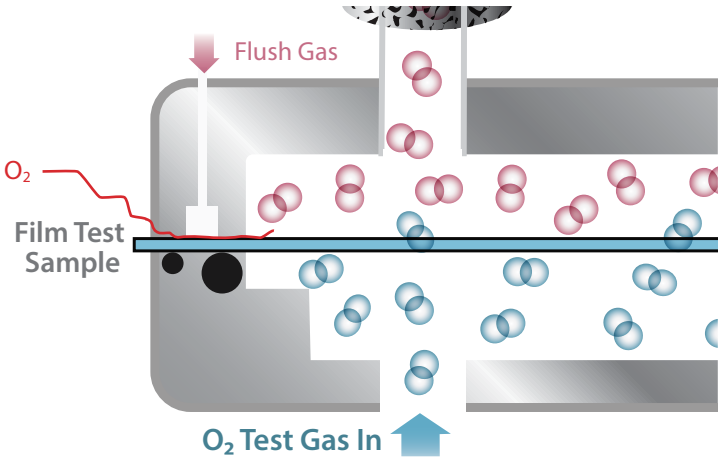
Note: The catalyst is crucial because even with pure N_2 carrier gas, unavoidable air leakage at the instrument's connections and humidification reservoirs will introduce oxygen. As the carrier gas leaves the test cell, AMETEK MOCON utilizes special gas tight connections to minimize leakage.



2. Leakage/permeation at the sealing interface between the sample and the carrier gas side of the diffusion cell. Samples with wrinkles or irregular and varying thickness are prone to leak at this interface.

To minimize leakage at the interface, AMETEK MOCON standardized the use of a special smooth diffusion cell "finish" along with a high molecular weight Apiezon grease.

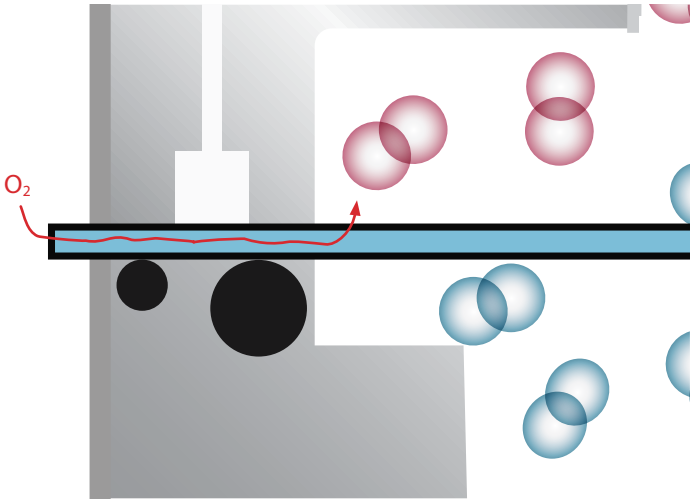
Additionally, with the development of AMETEK MOCON TruSeal®, a Flush Gas sweeps around the sample to further minimize oxygen transmitting from the environment and into the test cell.



3. Permeation through the edge of the sample.

For edge permeation, this is a challenge to minimize. Samples that exhibit this issue include coated papers (where oxygen permeates rampantly through the paper substrate) and relatively thick samples, including some with barrier coatings.

To minimize this, one can use an Edge Effect Cell, apply special sample masking or rely on a relatively high Individual Zero test to subtract this bias to the measurement.



Since leak areas two and three vary for each sample mounted, the individual zero is typically taken and subtracted as part of every test.

When to utilize Individual Zero?

When measuring typical flat polymer films, the AMETEK MOCON instrument cell design and features inherently yield low instrument bias. In some cases, depending on the transmission rate (TR) level, the final TR result can rely on the previously measured Individual Zero value. Within the AMETEK MOCON instrument software, this previously measured Individual Zero is known as "Use Last Individual Zero."

It is recommended to perform an Individual Zero if the expected transmission rate value is less than 50 times higher than the expected Individual Zero. In other words, if the Individual Zero level is below two percent of the expected TR of the sample, then an Individual Zero will be an insignificant percentage of the results and is not needed. This helps to reduce the overall testing time. In this case, the instrument can be set to use the last Individual zero rate for that test.

	Individual Zero	Expected Sample Transmission Rate	Individual Zero / TR Ratio	Individual Zero Recommended
Sample 1	0.02 $cm^3/(m^2 \cdot d)$	5.0 $cm^3/(m^2 \cdot d)$	0.4%	No
Sample 2	0.02 $cm^3/(m^2 \cdot d)$	2.0 $cm^3/(m^2 \cdot d)$	1%	No
Sample 3	0.02 $cm^3/m^2 \cdot d)$	1.0 $cm^3/(m^2 \cdot d)$	2%	Yes
Sample 4	0.02 $cm^3/m^2 \cdot d)$	0.5 $cm^3/m^2 \cdot d)$	4%	Yes

INDIVIDUAL ZERO

Should an Individual Zero be performed before or after the transmission measurement?

Both scenarios have their unique advantages:

1. Conducting the measurement at the beginning allows for the quick identification of any incorrectly inserted samples. The Individual Zero test typically takes as long as the subsequent test.
2. Doing the Individual Zero test at the end of the measurement often allows for a faster stabilization of the value.

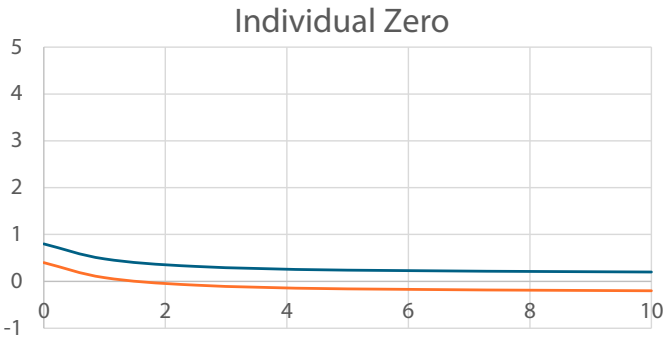
A helpful tip to bear in mind: If the measurement begins without an Individual Zero and it becomes apparent that it would make sense, the test method can still be adjusted to add an Individual Zero at the end, without needing restart the measurement.

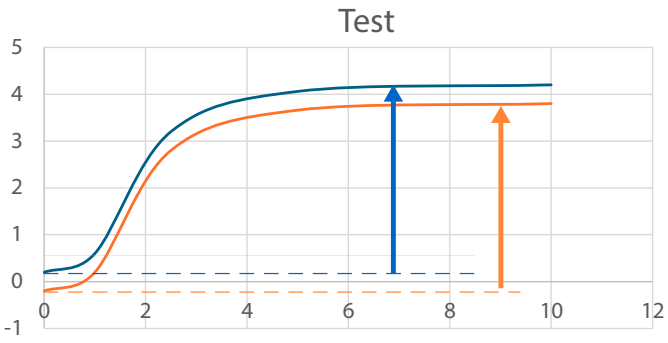
Re-Zero (Global Zero)

In addition to Individual Zero, MOCON instruments also utilize a Re-Zero function. In this process, fresh carrier gas is fed to the sensor via a separate valve without contacting the sample. Consequently, the Re-Zero reflects the carrier gas of the supply line through the device.

Fluctuations in the purity of the carrier gas, barometric pressure changes or the smallest leaks can thus be detected and eliminated by the Re-Zero measurement.

Thanks to the separate gas path, the Re-Zero can be performed both during an Individual Zero and during the test. This is why it is called Re-Zero (derived from the word “repeat”).





In very tight systems with low Re-Zero values, sometimes a negative value may be recorded for the Individual Zero. This is not necessarily an error but reflects changes during the test duration. The compensation still functions to provide the most accurate test result for that sample and test on that day.

In Summary

Using an active Individual Zero during permeation testing enhances both the consistency and accuracy of the results. By effectively eliminating any background leakage or bias, it ensures that the measurement reflects the true transmission rate of the sample itself, rather than being influenced by external factors or environmental interference. This precision is crucial to obtaining reliable data at low transmission rate levels, particularly in applications where slight differences in transmission rates can have a significant impact.

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