



An Anéolia white paper

# Nothing's tight !

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About packaging integrity

2025

## PEOPLE WE LEARN FROM



### **DENIS TREACY**

#### PACKAGING EXPERT

*As an executive leader with 40 years of FMCG industry experience, I strongly believe that delivering across any agenda requires a combination of Policies, Organisation, Performance & Culture*



### **KEVIN FERRIS**

#### PRINCIPAL ENGINEER MONDELEZ

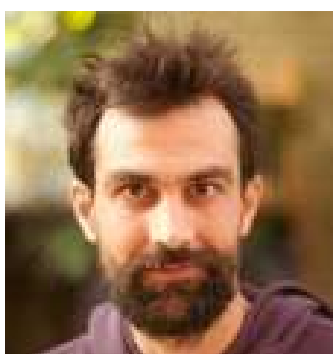
*In my position as Principal Engineer (RDQI Packaging) I am responsible for discovery and development of new concepts and technology for packaging machinery*



### **ERIC SCHALLER**

#### CEO ANEOLIA

*By founding Anéolia, I wanted to bring my passion for measurement accuracy and scientific analysis to the packaging industry. Today we analyze to better protect our customers and consumers.*



### **GUILLAUME POVIE PHD**

#### RESEARCHER

*Former researcher in physical and organic chemistry, specialist in questions of hermeticity and autoxidation – main causes of the aging of organic materials. Today, I am committed to sharing my knowledge and scientific methodologies*

# Summary

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BY ERIC SCHALLER – CEO ANEOLIA

There are sometimes discoveries that make us pioneers. Through extraordinary inventiveness combined with diligent work, these discoveries and inventions are imbued with an energy that transforms us and contributes to collective progress when they are communicated and implemented.



They can sometimes surprise the inventors themselves in their applications and performances. It was a great astonishment to see state services interested in the precision of our micro-leak measurements and to use this resolution to enable other variations of our instrumentation.

For Anéolia, inventiveness arises from collective work to provide its clientele with reference measuring instruments. In this approach, we equip our clients with analytical tools to better protect their brands, their products, their customers, and ultimately the consumers we all are.

Sometimes we are surprised to see performance claims or publications based on partial or biased metrological data. For example, claiming performance by stating an increasingly smaller detectability threshold expressed in micrometers without providing the context of time or stress applied to the packaging.

However, in the eyes of an uninformed user, there is nothing more reassuring than a detection or leak measurement instrument that detects nothing or announces a value close to zero because it frees them from having to justify an insufficiency. By avoiding rigorous analysis, this ultimately leads to inaction.

It then seemed necessary for us to provide users with a better understanding and practice of their technical means. By contributing to standardization efforts in Europe for testing the hermeticity and resistance of flexible packaging, we enable equipment buyers to learn about the actual capabilities of these means to better protect themselves against industrial, commercial, and health risks: more broadly, helping to strengthen their brand's reputation.

BY ERIC SCHALLER – CEO ANEOLIA

**Without accurate measurement, there is no analysis.  
Without analysis, there is no protection!  
Yet everything leaks!**

It seems essential to us to have a proper hierarchy of testing means by decreasing detectability thresholds (from the coarsest means to the finest means). And at each detectability threshold, it is also necessary to associate the discrimination capability of the instrumentation, that is, the measurability of any technical change in the packaging formation process.

At a time when the packaging industry must transform both its production means and the materials it employs, it becomes crucial to acquire instruments that allow for objective comparisons of different industrial choices, thus revealing the best implementation solution.

Analyzing for better protection also means assessing the production means and packaging solutions implemented. The commitment to this approach aims to help agro-industrial players make necessary strategic decisions.

This white paper also aims to help you determine the means to implement for better product preservation and waste reduction.

It is also a fantastic educational tool for teams so that they can ensure homogeneous production so that the provided foods meet their promises and live up to expectations regarding taste, product longevity, and consumer safety.

When packaging must fulfill the promises of the product!



## Why packaging?

It's an infamous principle of life –and thermodynamics– that things keep mixing up. Yet in most enterprises we work hard to keep these very things as pristine as possible. Food for example is something we like to see as fresh as intended to be, not mixed up with the whole world it has crossed on its way to the kitchen.

But hold on a moment, what exactly are we trying to keep out? dirt, obviously; pathogens, definitely; atmospheric gases, even better. Whether being moisture or oxygen sensitive, a variety of products will suffer quality losses when exposed to the surrounding atmosphere: the inner atmosphere of the packaging is key to the product shelf-life.

With a tight barrier all around, everything should be under control. But what is meant by tight? Diffusion is a tough contender: given enough time, gas molecules can pass through virtually any barrier. Tightness becomes thus a hopeless ambition, which has to be traded for a more practical yet more complex question: for how long will a barrier be tight enough for my purpose? – and at which cost?

## What's a packaging?

A packaging is an enclosing barrier designed to slow down the exchanges between its inner and outer parts.

Two main characteristics determine its performance:

1) *The intrinsic barrier properties of the material*, which relates to permeation phenomena.

- Nothing is tight, but certain materials let some gas slower than others through. This is measured by the permeability of the packaging for a given gas.

2) *The integrity of the packaging*, that is, the level of physical defects of the enclosure.

- Compared to permeation, which describes the slow diffusion of gas through solid materials, gas exchange through holes, cracks, or any opening of the packaging is a much faster process.
- Depending on the conditions, the loss of barrier properties caused even by a small defect can be sufficient to compromise the product life cycle.
- Whereas the permeability properties of the material are generally well controlled, the presence of physical defects in the barrier is a complex hurdle. By definition defects are unforeseen, and thus more difficult to systematically analyze.

# About Packaging

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## Which packaging?

First set what do you want to keep in (or out), and for how long, then choose the packaging materials accordingly. A meat product under a CO<sub>2</sub> / O<sub>2</sub> (30:70) atmosphere and with a shelf-life of about a week will not require the same type of packaging than a biscuit than can stay months on the shelf, but dreads moisture. The latter will do with a poor oxygen barrier material such as polypropylene, as long as moisture is kept away. The former however will require an excellent oxygen barrier material, such as EVOH, which in contrast let moisture diffuse quite easily through.

Various materials and film laminates are possible, offering different trade-off between properties and costs. The recent move towards non-plastic packaging has brought a new layer of complexity to the packaging process, which is more than ever in need of proper analytical tools to evaluate new designs.

## Is it well packaged?

Got the right material, designed the right packaging, everything should be fine; in theory at least. But things like to mix up, and always do so more than they should. Filling, folding, sealing, stacking... so much can go wrong along the line that the best initial design is in no way a guarantee for the best product quality.

Packaging processes are difficult to optimize and even more to maintain so: a wearing sealing jaw, a capricious new batch of film, and what used to be a clockwork packaging line starts scattering defects everywhere, until one notices. In practice it means product return to the manufacturer, and swift refunds that start cutting in the margin.

But after how many returns will you notice? and how to find the problem? One needs to test. Two things can be done to probe an individual packaging quality:

- For products with modified atmosphere packaging (MAP), once can measure the gas concentrations in the packaging headspace. Is it what it was intended to be? how long does it remain as intended?
- Check the integrity of the packaging. This is generally done through leakage rate measurements. The creation of a pressure difference between the inner and outer parts of the packaging provokes a flow of gas through the packaging defects, if present. Measurement of the leakage rate gives an accurate picture of the integrity of the packaging.

Various standardized methods exist to perform such test.

- Mass flow at constant pressure
- Pressure decay
- Vacuum decay
- Water bath
- Dye method
- Tracer gas

Is it always well packaged?

Yet, testing one packaging says few. The goal is to ensure that every product gets out of the line tight enough for the intended purpose. So, what's needed?

- A rigorous assessment of the maximum acceptable leak size.
- A standardized leak testing method that allows to monitor the leak size distribution in the packaging population.

Let's develop on the latter. First, one should go back to process development and optimization, where the leak size distribution (mean, spread, bimodal...) is analyzed in function of the process parameters. This step allows to determine the main causes of leakage (bad sealing, abrasion of the film, poor quality of the packaging material).

Then, monitoring: a well-optimized process should remain so, and only methodical sampling and testing of the output products can give a clear picture of the performance of the line.

Whereas a variety of methods are available to verify the integrity of individual packaging, few remains when the goal is to achieved reproducible analytical measurements. A metrological method is needed, that is, an instrument that measures the leakage rate under precise conditions to unambiguously assess the level of defect and its outcome on the product life cycle.

Arguably the most direct, reliable and versatile method –thereby also the simplest– is mass flow measurement at constant pressure. With a minimal set of parameters, a high level of sensitivity, and a direct measurement of the leakage rate, this standardized method (DIN55508-1) ensures that the measurement outcome is a true picture of the packaging integrity, not an artefact from a wrong set of measurement parameters.

Realizing that your product remains intact throughout its entire life cycle despite 100  $\mu\text{m}$  defects can be good news: you might be able to replace your expensive high-barrier film laminate with a much cheaper PE film!

Depending on the requirements of the product, packaging can represent a significant fraction of production costs. So, why invest in high-quality packaging materials if their protective effect is compromised by unnoticed defects? Only a quantitative measurement of the packaging integrity can ensure that the designed packaging fully plays its protective role.

This approach not only assures quality during production, but also helps reduce material costs during the development phase: assessing packaging performance through statistical analysis of packaging integrity is a powerful method for selecting the most suitable materials, thereby enhancing the quality-to-price ratio.

Whether for public health or industrial practices, investing in precautionary tools and regulations always leads to large savings in the long run. The packaging industry is no exception. While instrumentation and methods may initially seem like hurdles, systematic and quantitative leak testing is essential to ensuring consistent quality and preventing accidents that could ruin a brand – or even cost lives.

## 1 Crackers[1]

Crackers are dried products with a relatively long shelf-life (3 to 6 months), which allows large distribution circuits. But despite their apparent robustness to ageing, taken out of their packaging crackers quickly absorb atmospheric moisture and lose their crispiness within hours.

To prevent water intake, crackers are packaged with films having good water barrier properties at moderate cost, typically BOPP based laminates.



Take a 100 g of saltines crackers flow-packed in BOPP film (30  $\mu\text{m}$  thick, 15 cm x 6 cm x 6 cm, for a surface area = 0.043  $\text{m}^2$ ) with good water barrier properties ( $\text{WVTR} \approx 1 \text{ g}\cdot\text{m}^{-2}\cdot\text{day}^{-1}$ , 38 °C, 90% RH)

To lose crispiness, this pack of crackers require the absorption of about 5 grams of water.[1] In a pristine package, this would take about 120 days under tropical conditions (38 °C, 90% RH), and 500 days under temperate conditions (20 °C, 79% RH).

A 100  $\mu\text{m}$  hole is sufficient is already sufficient to divide those values by two, with only about 70 days and 300 days before failure under tropical and temperate conditions, respectively. With a 200  $\mu\text{m}$  defect, the shelf-lives drop to 30 and 120 days.

## 2 Puffed corn curl

Puffed snacks are sensitive to moisture but also to oxygen because of fat rancidification. They are typically found in metallized laminates with high gas barrier properties (see chips below). As such, a pristine packaging can possess a shelf-life of several years.

Take the loss of crispness, which occurs for puffed corn curl after only 2.4 g water uptake for 100 g product. With a 100-gram pack, dimensions 15 cm x 25 cm, a 100  $\mu\text{m}$  hole would reduce the shelf- life to 100 and 340 days under tropical and temperate conditions, respectively; 25 and 100 days with a 200  $\mu\text{m}$  defect.

| Water activity and moisture content of dry food products. <sup>1</sup> | Initial values |               | Critical values (loss of crispiness) |               |
|------------------------------------------------------------------------|----------------|---------------|--------------------------------------|---------------|
|                                                                        | $a_w$          | $mc$ (g/100g) | $a_w$                                | $mc$ (g/100g) |
| Saltine                                                                | 0.074          | 1.42          | 0.37                                 | 6.7           |
| Chips                                                                  | 0.076          | 0.65          | 0.47                                 | 5.2           |
| Puffed corn curl                                                       | 0.082          | 1.83          | 0.36                                 | 4.2           |
| Pop corn                                                               | 0.062          | 1.70          | 0.49                                 | 6.1           |



1] Katz, E. E., and Ted P. Labuza. "Effect of water activity on the sensory crispness and mechanical deformation of snack food products." Journal of Food Science 46.2 (1981): 403-409

### 3 Chips



In addition to moisture, fried snacks such as potato chips also fear rancidity. Chips packaging have generally a large headspace volume. If the product is packed under air, the oxygen content is sufficient to permit an  $\text{O}_2$  uptake in excess of  $3 \text{ mL O}_2 (\text{STP}) \cdot \text{g}^{-1}$ , with a resulting peroxide content (an index for rancidity) beyond acceptability.

Thus, potato chips are typically packed under nitrogen, with film laminated incorporating a metallized film for high gas barrier properties.

Consider a 150 g potato chips in a 30 cm x 20 cm flow pack made of a 50  $\mu\text{m}$  thick BOPP/MetPET/LDPE film laminate (the BOPP layer here ensures the printability of the exterior, whereas the LDPE is the sealable layer. The Metallized Polyester (MetPET) is the actual barrier (typical WVTR:  $0.1 - 0.5 \text{ g} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ , typical OTR:  $0.1 - 1 \text{ mL} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ ).

Assuming the packaging contains 2.5 L of gas, it would take years for the oxygen level to vary in the packaging only due to the film permeability.

In contrast, with a 50  $\mu\text{m}$  hole, the oxygen level would have reached 3% within a week, and 21%, the ambient level, within 4 months. With a 100  $\mu\text{m}$  hole, it will take only 32 days for the protective atmosphere to be completely lost.

## 4 Red meat

### A Under MAP

Red meat is a sensitive product, which generally requires modified atmosphere packaging (MAP). Such packaging is typically filled with a mixture of dioxygen ( $O_2$ ), to keep the red color of the meat, and carbon dioxide ( $CO_2$ ) to inhibit microbial growth.

A packaging with good oxygen barrier properties is required: typically a polystyrene tray coated and lidded with a 50  $\mu m$  thick EVOH film laminate (OTRs of trays and lidding films can be expected to be less than 0.42 and less than 4.2  $mL m^{-2} day^{-1}$ , respectively).

Take a steak twin pack in a 20 cm x 13 cm x 2 cm tray containing about 300 g of meat and a 0.2 L gas headspace. With a High  $O_2$  MAP ( $O_2 / CO_2$ ; 70:30), the meat can retain its bright cherry color up to 8 days.

But a single pinhole 50  $\mu m$  diameter allows enough exchange with air for the level of  $O_2$  to drop to 40%, and that of  $CO_2$  to 15% in 3 days. With a 100  $\mu m$  hole, the gas content of the packaging will be essentially air after 3 days.

Not only in both cases the desired red color of the meat would have already darkened, but much worse, the loss of  $CO_2$  now leaves room for bacterial growth with the associated health risks.



### B Under vacuum

Conversely, meat in vacuum pack can be stored for extended period of time (3 - 4 months) at 0 °C, provided the packaging material can ensure a strict oxygen barrier[2]. Although large defects are generally detected during the vacuum process, a 20  $\mu m$  hole will be barely noticeable, yet will bring about 1.5 mL/day of oxygen in the packaging, thereby completely compromising the expected shelf-life.

[2] Newton, K. G., and W. J. Rigg. "The effect of film permeability on the storage life and microbiology of vacuum-packed meat." *Journal of Applied Bacteriology* 47.3 (1979): 433-441.

## 5 Cheese

The usefulness of CO<sub>2</sub> MAP to inhibit microbial growth is well exemplified with cheese. Sliced mozzarella packed under 100% CO<sub>2</sub> can have a shelf-life up to 2 months, compared to 2 weeks under air.[3]

Consider a 170 g sliced Mozzarella packaged in oxygen and carbon dioxide high barrier film laminate (for example, 60 µm PET / EVOH / LDPE). Initially the headspace consists of about 0.3 L of pure CO<sub>2</sub>.

Although the film barrier properties are sufficient to preserve this atmosphere for the whole shelf-life, a mere 25 µm diameter pinhole in the packaging will already let the oxygen content reach 9% after 2 weeks. Take a 50 µm hole, and in 2 weeks the CO<sub>2</sub> fraction will have dropped to 40%, whereas the oxygen content will have almost reached the atmospheric level (19%).



[3] For the shelf-life of mozzarella in MAP, see : Alves, Rosa Maria Vercelino, et al. "Stability of sliced mozzarella cheese in modified-atmosphere packaging." *Journal of Food Protection* 59.8 (1996): 838-844

6 Bakery

The same applies to a variety of bakery products (Sliced bread, rice-bread, pita bread, cakes, croissant, crumpets, bun, brioche, crêpes, dough, pastries, sandwiches...), which can have a 3 to 4-fold longer shelf-life under CO<sub>2</sub>/N<sub>2</sub> MAP. Slice bread, buns, cake, crêpe, croissant, crumpets, fresh dough and even sandwiches, which shelf-life is often barely a week under air, can be stored up to 3 to 4 weeks with MAP... provided no leakage is there to ruin the gas mixture.

As a general example: for a 100 mL headspace, a 50 µm pinhole will be enough to lose essentially all the protective atmosphere within a week.

| Product<br>(storage temperature)                                      | Gas mixture                           | Typical Shelf-life |         |
|-----------------------------------------------------------------------|---------------------------------------|--------------------|---------|
|                                                                       |                                       | air                | MAP     |
| Pre-baked bread (20 °C)                                               | CO <sub>2</sub> 100                   | 5 days             | 20 days |
| Pastries (20 °C)                                                      | CO <sub>2</sub> /N <sub>2</sub> 30:70 | 7 days             | 21 days |
| Fresh dough (5 °C)                                                    | CO <sub>2</sub> /N <sub>2</sub> 50:50 | 3 days             | 18 days |
| Sandwiches (4 °C)                                                     | CO <sub>2</sub> /N <sub>2</sub> 30:70 | 5 days             | 20 days |
| Cream cakes (4 °C)                                                    | CO <sub>2</sub> /N <sub>2</sub> 30:70 | 3 days             | 21 days |
| The packaging contains about 50 – 100 ml of gas for 100 g of product. |                                       |                    |         |

**“It is not what they make in the factory but what they deliver to the consumer that matters.”**

Denis Treacy, packaging expert



## BY DENIS TREACY – PACKAGING EXPERT

**It is crucial for our industries to discuss importantly, the ways packaging can be validated, tested and assessed to ensure the material, the means of sealing and the capability all match with the product contained, the supply chain and all other influencing factors that might impact the ability of the packaging to meet its expectations.**

How could we define the fundamental purpose of packaging?

- An enterprise develops a product to meet a need, or to create a want.
- This is manifested at a marketing stage as a sensorial consumer experience - Visual, odour, taste, texture.
- The packaging is part of the delivery mechanism for that sensorial consumer experience, so must PROTECT and SECURE the product until it arrives at the consumer and can be enjoyed.
- Primary (first contact) packaging needs to protect from contamination, to maintain structure and integrity, to preserve chemical structure odour, taste, texture, to perhaps allow the development & maturity of the product.
- Secondary packaging projects the promise of the product, the sensorial consumer experience, its brand values and necessary nutritional or regulatory compliances.

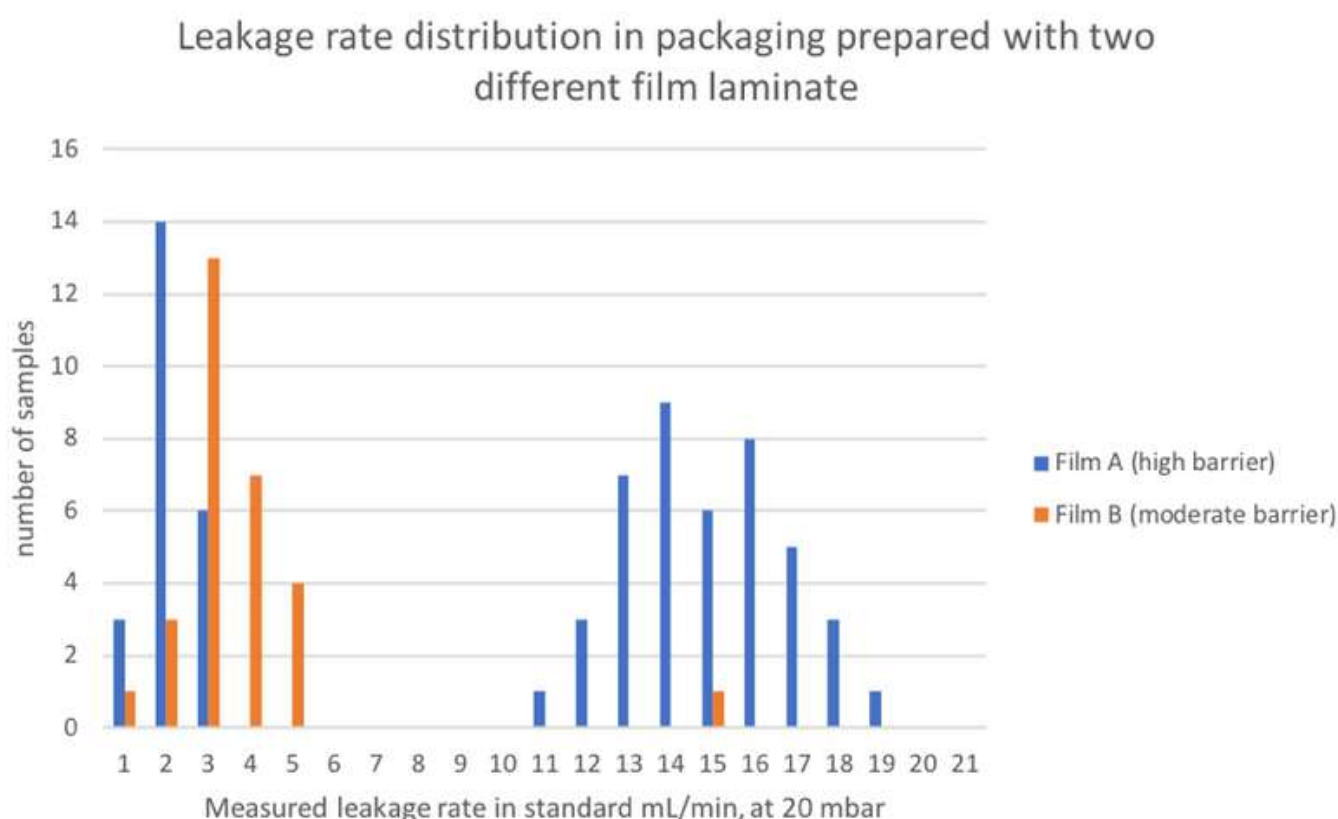
Food businesses are more commercially successful if they create something more people want, or they create something better than their competition. So how can a better packaging can help?

- Packaging delivers the consumer promise made by the brand.
- Packaging enables the efficiency of the supply chain.
- Packaging allows a product to deliver its sensorial consumer experience across wider markets, international opportunities.
- Packaging supports the cost effectiveness of the raw materials used to make the product which delivers the sensorial consumer experience.
- The better the data, its analysis, the information that results, the better the choices an enterprise makes and the more successfully it delivers sensorial consumer experience.



# 1 Statistical Analysis

The ability to analyse the leakage rate of a sample population is crucial in packaging development. The following chart shows the results of such analysis for a product packaged with two different types of film laminates.



Laminate A is an excellent barrier material, yet issues associated with the packaging process (fragility of the film laminate, sealing problems) result in an unacceptable proportion of leaky packages. In contrast, Laminate B, which has a less sophisticated barrier structure, performs well on the production line and consistently results in tightly sealed packages. For this particular example, it turned out that the cheaper Laminate B was also the best choice to ensure the desired shelf life.

The same type of analysis used for monitoring the production line can provide early signals of degradation in any part of the process, allowing for rapid actions to minimize production losses.

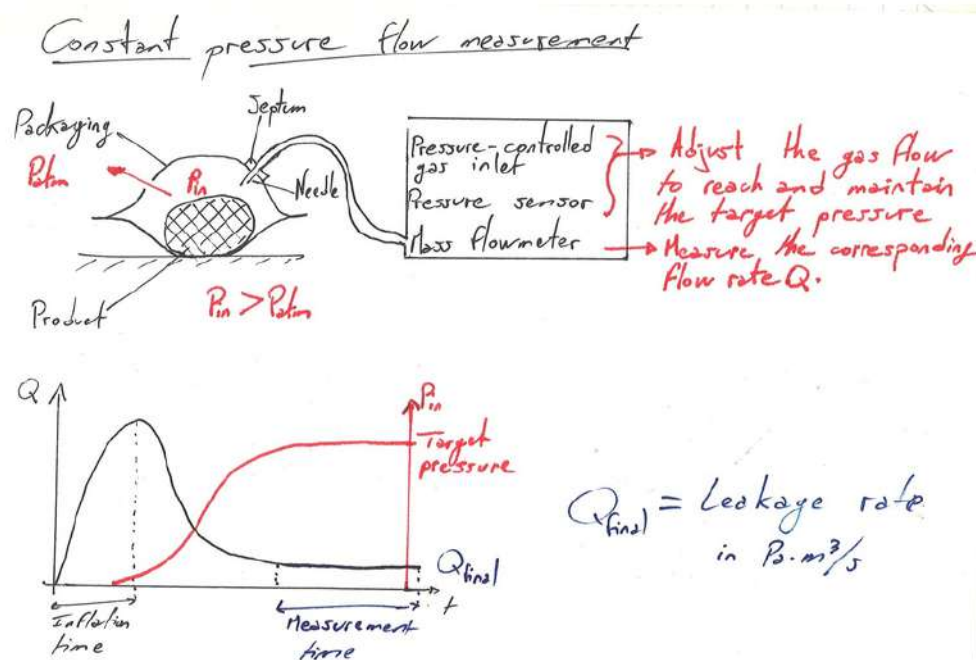
# 2 Leak testing methods

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## 2.1 - Mass flow at constant pressure

**Principle.** The closed packaging is punctured with a needle and inflated to a given gauge pressure. Measurement of the gas (typically air) flow required to maintain the target pressure directly yields the leakage rate without further manipulation of the measurement data. In packaging development, various tooling also exists to non-destructively test bags before sealing, non-perforated trays, or any other opened packaging piece.

Based on a straightforward set-up and a minimal number of measurement parameters, this method is particularly reliable and easy to implement on a large variety of packaging with a single apparatus. Pinholes as small as 5  $\mu\text{m}$  diameter can be identified.



### Measurement

A pressure controller maintains the target pressure, whereas a mass flowmeter measures the mass flow of air ( $Q_m = dm/dt$ ) required to maintain constant pressure.

## Determination of the leakage rate.

Differentiating the ideal gas law over time at constant temperature directly yields the expression of the leakage rate  $Q_{pV}$  in terms of mass flow ( $dm/dt$ ), which are simply related by a proportionality factor  $RT/M$ :

$$Q_{pV} = \frac{d(pV)}{dt} = \frac{RT}{M} \frac{dm}{dt}$$

with  $R$  the ideal gas constant,  $T$  the measurement temperature, and  $M$  the average molar mass of the injected gas, typically  $M_{air} = 29 \text{ g}\cdot\text{mol}^{-1}$ .

## Advantages.

Metrological method with high sensitivity (down to  $10^{-4} \text{ Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$ ), precision and reliability. Straightforward implementation with few measurement parameters. The calibration is stable and not application dependent.

## Drawbacks.

The method is destructive for sealed packaging. Localization of the leak requires additional implementations.

## Applications areas.

Packaging material development, optimization and monitoring of packaging processes, in-line product quality control.

Published standard(s): **DIN55508-1**

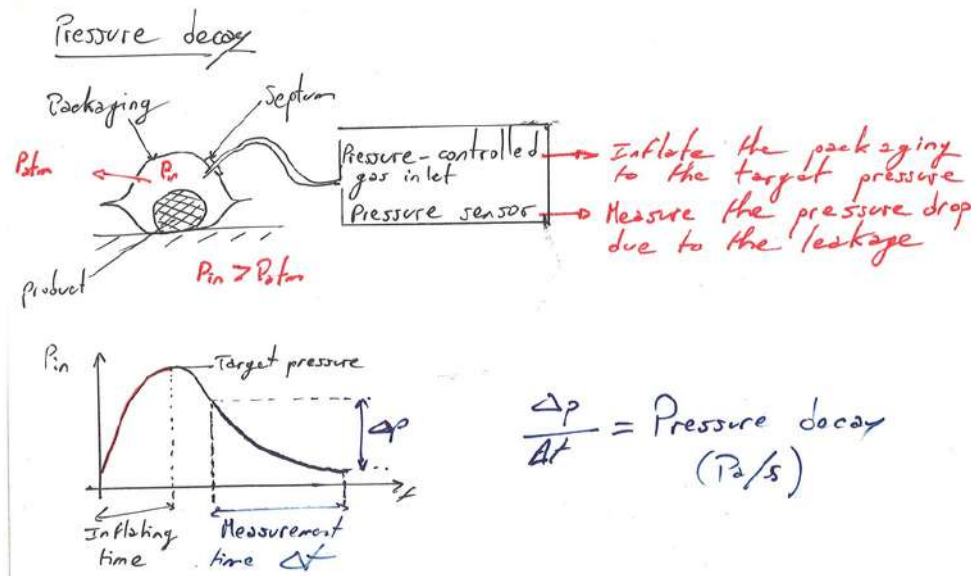
## 2.2 - Pressure decay

### Principle

The packaging is punctured with a needle and inflated to a given gauge pressure. After the target pressure is attained, an equilibration time is respected before measuring the variation of pressure in the sample ( $p_s$ ) in function of time. The presence of a leak leads to a pressure drop, which rate is measured by the instrument. The measured pressure decay rate can then be used to calculate the leakage rate if the sample volume is known.

# 2 Leak testing methods

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## Measurement

The apparatus is equipped with a time resolved pressure sensor, which outputs the rate of pressure decay, that is, the pressure variation inside the packaging per unit of time at constant volume and temperature:  $dP/dt$  (in  $\text{Pa}\cdot\text{s}^{-1}$ ).

## Determination of the leakage rate

The leakage rate can be obtained from the rate of pressure decay and the sample headspace volume ( $V_s$ ). An independent calibration method is thus generally required for each application.

$$Q_{pV} = \frac{d(P_s V_s)}{dt} = V_s \frac{d(P_s)}{dt}$$

## Advantages

Straightforward implementation with few measurement parameters.

## Drawbacks

Moderate sensitivity ( $10^{-2} \text{ Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$ ) according to the DIN standard, though, as the ASTM standard points, higher sensitivity can always be achieved with longer measurement time.

The method is destructive. Calibration is required for each application.

## Applications areas

In-line product quality control.

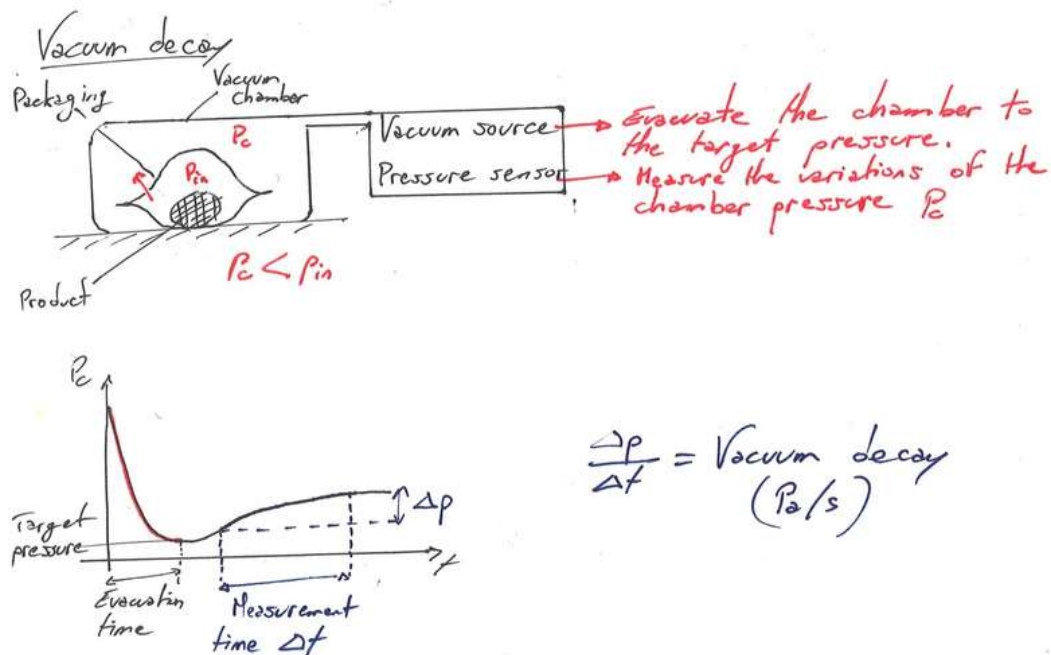
Standard(s):

DIN55508-3, ASTM F2095-01

## 2.3 - Vacuum decay

### Principle

Though similar to in principle to pressure decay, the difference of pressure is created here by placing the packaging in a sealed chamber, which is then evacuated down to the target pressure. After an equilibration time, the increase in pressure in the chamber ( $p_c$ ) in function of time is recorded. The leakage rate can be calculated if the chamber net volume during the measurement is known. The apparatus can be equipped with rigid or flexible chambers, which offer different advantages depending on the application.



### Measurement

The apparatus is equipped with a time resolved pressure sensor, which outputs the rate of vacuum decay in the chamber ( $p_c$ ), that is, the pressure variation per unit of time at constant volume and temperature:  $dp_c/dt$  (in  $\text{Pa}\cdot\text{s}^{-1}$ ).

### Determination of the leakage rate

The leakage rate can be obtained from the rate of vacuum decay and the chamber net volume of gas during measurement ( $V_c$ ). An independent calibration method is thus generally required for each application.

$$Q_{pv} = \frac{d(pV_c)}{dt} = V_c \frac{d(p_c)}{dt}$$

# 2 Leak testing methods

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## Advantages

Non-destructive, good sensitivity, can be automatized.

## Drawbacks

Multi-parameters method, adjustment of the testing conditions and calibration is required for each application.

## Applications areas

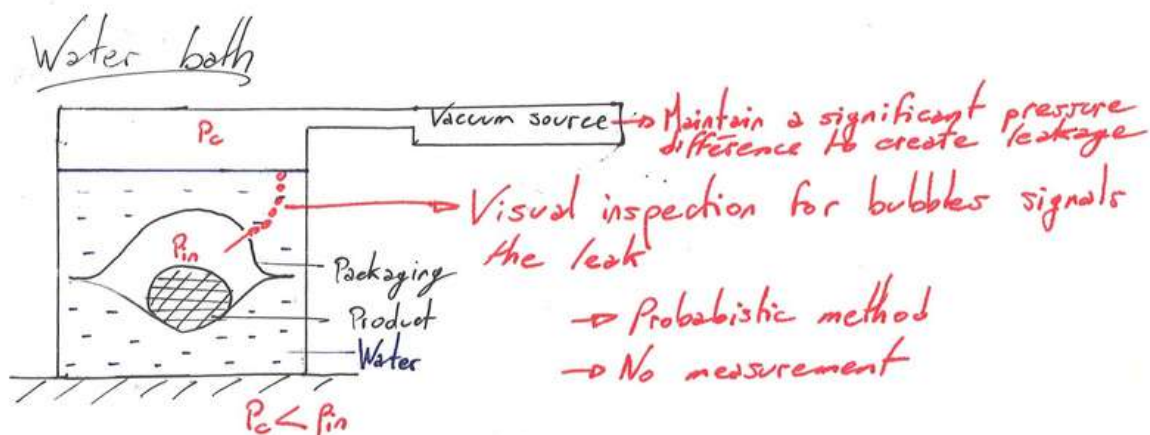
In-line product quality control.

Standard: **ASTMF2338-05**

## 2.4 - The water bath method (bubble test)

### Principle

Historically a prominent method, the water bath test involves the immersion of a pressurized packaging in a water bath. Visual detection of gas bubbles allows for identification and localization of the leak. The probabilistic aspect of this method is inherent to the visual detection: trapped air bubbles may appear as a leak where there is none, whereas some real defects may fail to cause detectable bubble formation.



### Measurement

This is not a metrological method. The test result is visually determined by the operator

### Advantages

Low cost; easy implementation; the leak can be localized.

## Drawbacks

Destructive method, low sensitivity, no leakage rate measurement, probabilistic outcome, hygienic issues associated with the use of water

## Applications areas.

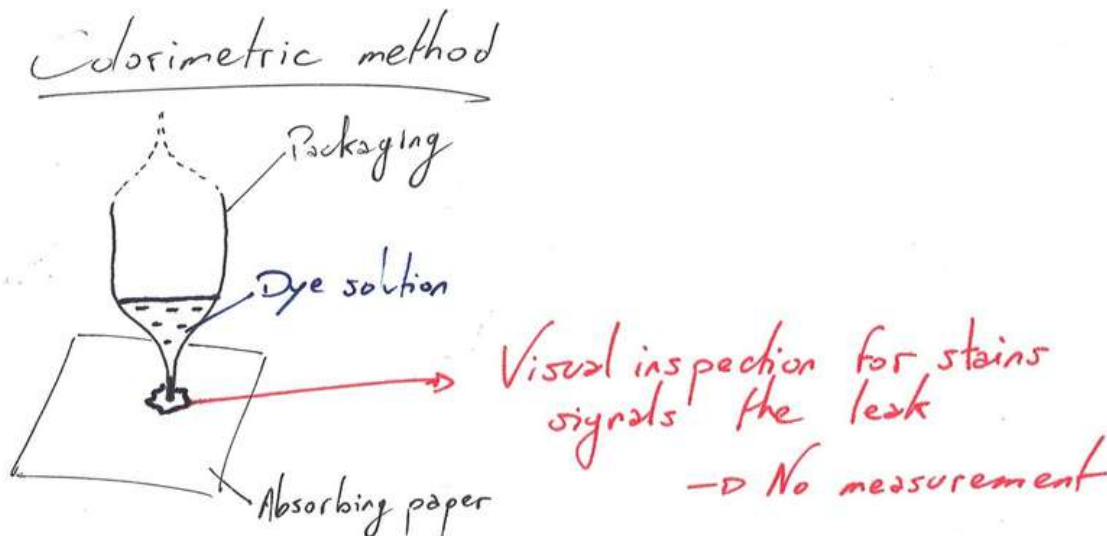
In-line product quality control.

Standards: DIN55508-5, ASTM F2096-11, ASTM D3078-02

### 2.5 - Colorimetric method (dye leak test)

#### Principle

This method relies on the capillary flow of a dye solution through the defects of the packaging. A dye solution in ethanol is injected in packaging to be tested. The outer part of the packaging is then held in contact with an absorbing paper (tissue paper, filter paper...). Visual detection of colored stains on the paper allows for localization of leaking areas.



#### Measurement

This is not a metrological method. The test result is visually determined by the operator.

#### Advantages

The method can achieve a good sensitivity. The leak is localized. The method does not require sophisticated apparatus.

## 2 Leak testing methods

## Drawbacks

The method is destructive. Long testing times. Only qualitative information on the leak size are obtained (stain size); the leakage rate is not measured.

## Applications areas

Packaging material development, optimization and monitoring of packaging processes.

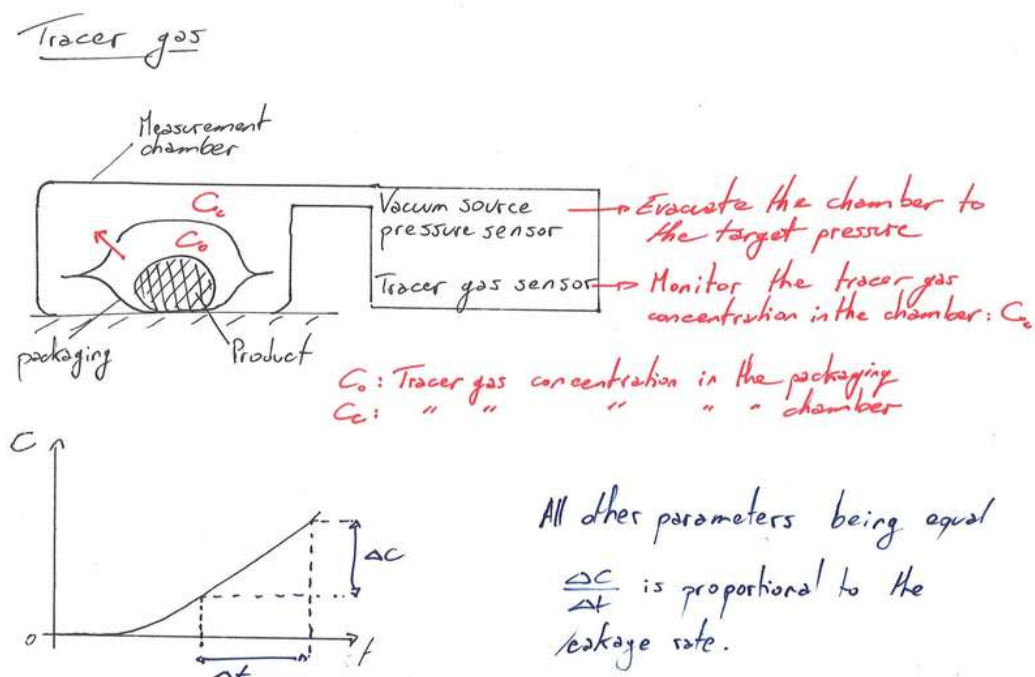
Standard: **DIN55508-2, ASTM F1929-12**

## 2.6 - Tracer gas method

## Principle

The packaging headspace should contain the tracer gas in known concentrations. It is then placed in a measurement chamber submitted to a partial vacuum. Under these conditions, a defect packaging leaks its gas content into the chamber. A detector monitors the evolution of the tracer gas concentration in the chamber, from which the leak rate can be calculated. In many applications, the tracer gas is helium, though CO<sub>2</sub> can also be used.

This method extends far beyond packaging leak testing. Numerous standards have been published, each with different purposes and applications. It is beyond the scope of this list to cover them in details.



## Advantages

Non-destructive method; very high sensitivity ( $10^{-8} \text{ Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$ ); the leak can be localized.

## Drawbacks

Expensive and delicate equipment. The packaging should be filled with the tracer gas in the first place.

## Applications areas

Leak testing of any kind of containers, from pipeline to electronic devices. It can also be used in packaging material development, optimization and monitoring of packaging processes.

*Standards:* **DIN 55533, EN 13185, ISO 20485:2017**



**“We know that the  
pack must protect the  
product in all required  
ways”**

Kevin Ferris Principal Engineer  
Mondelez



BY KEVIN FERRIS – PRINCIPAL ENGINEER – MONDELEZ

**Working for a large company and world-famous brands gives Kevin Ferris a keen insight into our industry and the expectations around packaging to better protect the products we can all buy and eat. We asked Kevin a few questions about the challenges of packaging.**

**What technical criteria must packaging meet to better protect food products ?**

From an environmental point of view, we know the CO<sub>2</sub> footprint of farming, producing, and delivering food products is high, much higher than that of the packaging. For this reason, it is vital to ensure products reach the consumer with a good shelf life remaining to avoid food waste and wasted CO<sub>2</sub> footprint.

In addition, we know that the pack must protect the product in all required ways as well as retaining a great appearance. The least attractive pack on the shelf will be the one left unsold.

**What is the acceptance threshold for the hermeticity of the packaging to be applied or represented ?**

For products such as snacks and biscuits which have relatively low protection requirements because degradation does not cause a food safety problem it is not studied in detail. Levels of protection are based on existing or Historic packs formats. These are known to provide sufficient protection for supply chain needs.

Biscuits sit on a borderline, the moisture uptake is important for quality and consumer liking, but it does not represent a food risk, apart from in extreme cases.

Sometimes manufacturing standards for seal integrity are set based on known good machine performance and applied across all pack formats rather than being specific to each application.

**How does the new regulation invite innovation in terms of food safety ?**

For products such as snacks and biscuits headspace regulations don't have a direct impact on product protection. In pillow pack applications for fragile products the headspace gives a mechanical protection against compression during transit, for block bottom packs headspace gives no protection. Flow wrapped packs rarely have any functional headspace, tightly wrapped packs are generally preferred.



A standard test method establishes the requirements, performances and limitations of a given measurement technique. Standards aimed at normalizing the most appropriate methods to harmonize processes and improve quality for both industry and consumers. As such, they do not only lay down good practices, but also set the state of the art in a field. The following list details the most relevant standards for the packaging leak testing industry.

In practice, a few standardization agencies play an important role in the field.

## 3.1 DIN standards

Through a focus on quality, safety and minimum expectations, the standards published by the century old *Deutsches Institut für Normung* (DIN, the German Institute for Standardization) aim at rationalizing production methods to minimize costs and reduce risks. The still growing DIN 55508 series of standards – Packaging test: Leakage test on flexible packagings – follows this trend by inventorying the state-of-the-art leak detection methods to offer a practical summary of this complex metrological field.

### 3.1.1 DIN55508-1

Constant pressure method. To comply with this standard, instruments should be equipped with a mass flow meter with a 0.1 mL/min minimum resolution, and a pressure sensor with 10 Pa resolution, providing a leakage rate detection limit at  $10^{-4} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$ . The target relative pressure can be set between 10 and 500 mbar, but should never exceed 20% of the burst pressure. Further details on the measurement protocol and data report are also provided. In particular, the use of a septum at the puncture position is crucial to avoid additional leakage around the needle.

### 3.1.2 DIN55508-2

Colorimetric method (dye leak test). The DIN standard set the requirements of the injection method. The test sample should be an empty, dry and clean packaging. A sufficient amount of a methylene blue solution in ethanol ( $2 \pm 1 \text{ g/L}$ ; at least 1 mL of solution for 10 cm seal length) should be used. Other dyes may be used, though safety regulations must then be adapted depending on the toxicity of the dye compound.

This method can detect leaks of at least 20  $\mu\text{m}$  diameter at the seals, and 10  $\mu\text{m}$  diameter on the foil surface. Depending on which element of the packaging is tested, the duration of the test is critical to achieve good sensitivity: about 1 min per millimeter of seal width, and 20 s per 100  $\mu\text{m}$  of foil thickness.

# 3 Leak testing standards

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## 3.1.3 DIN55508-3

Pressure decay. To comply with this standard, instruments should be equipped with a pressure sensor with 1 Pa resolution, and a clock of at least 1 s resolution. The relative pressures used can range from 10 to 1000 mbar, but should never exceed 20% of the burst pressure. Although packaging filled with products may be tested, the packaging should be punctured at a clean and dry place.

## 3.1.4 DIN55508-5

Water bath. In this method, the packaging should be immersed in the bath at least 5 cm below the water surface. The pressure differential between the packaging headspace and the chamber is created either by puncturing the packaging with a needle, through a gas is injected to inflate the packaging to a target pressure, or by placing the bath inside a vacuum chamber. Visual inspection by the operator to locate a flow of bubbles from a leak sets the outcome of the test.

## 3.1.5 DIN 55533

Tracer gas method. Describes the use of a flexible chamber which minimize the constrains of the packaging.

## 3.2 ASTM. The American Society for Testing and Materials (today ASTM international)

The ASTM is one of the oldest standard publishing organizations. Its standards offer a comprehensive overview of the described method, as well as experimental assessments of the test results accuracy.

### 3.2.1 ASTM F1929-12

Colorimetric method. The ASTM standard details other uses of the technic such as the edge dip method, where leakage of the dye solution from the outer edge towards the inner edge of the package seal is probed. Here, a test duration of 5 to 20 s is recommended, for the detection of a leak equal to or greater than a channel formed by a 50 µm wire in package edge seals.

## 3.2.2 ASTM F2095-01

### Pressure decay

Here two variants of the method are described: with or without restraining plates to limit the inflation of the packaging. The sensitivity of the method is directly related to the sample volume, smaller volumes leading to higher sensitivity. A guide to determining test parameters is provided.

## 3.2.3 ASTM F2338-05

### Vacuum decay

This extensive standard describes the theoretical principle, the instrumentation and the measurement and calibration procedures for a rigid chamber set-up. An assessment of laboratory results is presented, demonstrating a sensitivity about  $10^{-3} - 10^{-4} \text{ Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$ . A guide for establishing the numerous test parameters and for verifying the sensitivity is also provided.

## 3.2.4 ASTM F2096-11

### Water bath

Similar in essence to the DIN55508-5 method, this comprehensive description of the method also sets the probability of detecting a leak under specified test conditions. A 250  $\mu\text{m}$  hole displays an 81% probability of being detected, whereas this value drops to 41% for a 125  $\mu\text{m}$  hole. In addition, there is 11% chance for a package with no defect to appear as leaking.

## 3.2.5 ASTM D3078-02

### Water bath

This standard describes an alternative method where the packaging does not need to be punctured. After immersion in the water bath, the chamber is set under vacuum to create a pressure differential with the packaging headspace. Visual inspection for bubbles from a leak sets the outcome of the test.

# 3 Leak testing standards

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## 3.3 International standard publishing organizations

Besides national standard publishing organizations, international ones such as the European Committee for Standardization (CEN) and the International Organisation for Standardization (ISO) have also published general standards in the field, in an effort to harmonize terminology and method selection. These organizations are generally composed of representatives from the standards organizations of member countries.

### 3.3.1 ISO 20485:2017

Tracer gas method. It affords a comprehensive overview of tracer gas methods in terms of protocols and detection systems.

### 3.3.2 EN 13185 and EN 13625

Tracer gas method. The EN 13185 standard also describes exhaustively the method whereas the EN 13625 offers a guide to choose the proper instrumentation.

### 3.3.3 Other general standards

DIN EN ISO 20484:2017-07 – Terminology

EN 1779 Non-destructive testing – Leak testing – Criteria for method and technique selection

ISO 20486:2017-12 – Non-destructive testing – Leak testing – Calibration of reference leaks for gases

# 4 Leakage rate and leak testing

## 4.1 - Mass transport phenomena (convection)

The transfer of matter from or into a container can arise from two distinct phenomena:

- Diffusion

Molecular diffusion is the intrinsic capacity of molecules to disperse throughout space driven by their thermal motion. This stochastic movement results in the overall transport of matter from high to low concentration regions. The time evolution of a species concentration (C) can be computed using the diffusion equation, sometimes referred to as the second Fick's law.

$$\frac{\delta c}{\delta t} = D\Delta C = D\left[\frac{\delta^2 C}{\delta x^2} + \frac{\delta^2 C}{\delta y^2} + \frac{\delta^2 C}{\delta z^2}\right]$$

where D is the diffusion coefficient of the species in the studied medium.

- Free diffusion

Free diffusion in the gas phase is the main cause of gas exchange through the defects of a container (e.g. holes in a packaging). Diffusion also occurs from gas (or liquid) to solid phases and vice-versa, allowing the transfer of gaseous content across solid membranes. Such permeation processes are influenced by the permeability and thickness of the membrane material, dictating the rate of mass transfer. Permeation is not exclusive to solid membranes, but also encompasses the behavior of porous materials where diffusion also occurs freely through the gaseous content of the pores.

- Advection

Advection involves mass transport resulting from the bulk velocity of a fluid induced by body forces, typically a pressure difference. Many leak-testing methods rely on generating such flow through the container defects and measuring it to evaluate the leak's size.

## 4.2 - Leakage rate

The leakage rate of a fluid is defined as the variation of the "pressure times volume" term (p·V) over time due to the leak. In SI units it is expressed in Pa·m<sup>3</sup>·s<sup>-3</sup>, which is a Watt (W = kg·m<sup>2</sup>·s<sup>-3</sup>). In that sense a leakage is an energy loss.

$$Q_{pv} = \frac{dpV}{dt} = p\frac{dV}{dt} + V\frac{dp}{dt}$$

Note. Standard cm<sup>3</sup> (Scc) and Normo cubic meter (Nm<sup>3</sup>) are units of gas quantity in equivalent volume at standard pressure and temperature (1 atm = 1013 mbar, 0 °C, DIN 1343). They give rise to alternative units for the leakage rate: the standard cubic centimeter per minute (Sccm, Scc.min<sup>-1</sup>) and the Normo cubic meter per hour (Nm<sup>3</sup>.h<sup>-1</sup>):

# 4 Leakage rate and leak testing

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1 Pa·m<sup>3</sup>·s<sup>-1</sup> is equivalent to 592 Sccm

1 Pa·m<sup>3</sup>·s<sup>-1</sup> is equivalent to 0.0355 Nm<sup>3</sup>·h<sup>-1</sup>

1 Pa·m<sup>3</sup>·s<sup>-1</sup> is equivalent to 10 mbar·L·s<sup>-1</sup>

The cc unit is sometimes replaced by the equivalent milliliter (mL), with or without explicit reference to the 1 atm, 0 °C standard.

## 4.3 - Ideal gas law

An ideal gas is a simple model where particle size as well as interparticle interactions are neglected. Despite these apparently bold hypotheses, this model accurately describes the behavior of many real gas (O<sub>2</sub>, N<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O...) and gas mixture (air) under standard pressure and temperature conditions. For example, between 0 and 100 °C, and at pressures from 0 up to 2 bars (absolute), the behavior of air deviates from the ideal gas law by less than 0.1%. This value increases to about 1% for CO<sub>2</sub> under similar conditions.

An ideal gas obeys the ideal gas law:

$$pV = nRT$$
$$pV = \frac{mRT}{M}$$

where:

p is the absolute pressure of the fluid (Pa = J·m<sup>-3</sup>)

V is the volume of the gas or of the container (m<sup>3</sup>)

n is the number of mole of gas (mol)

R is the ideal gas constant (8.314 J·K<sup>-1</sup>·mol<sup>-1</sup>)

T is the temperature (K)

m is the mass of gas (kg)

M is the molar mass of gas (kg·mol<sup>-1</sup>)

## 4.4. Mass flow rate

As long as the ideal gas law can be assumed, the leakage rate is proportional to the mass flow rate (SI: kg·s<sup>-1</sup>).

$$Q_m = \frac{dm}{dt}$$
$$Q_{pv} = \frac{RT}{M} \cdot Q_m$$

# 4 Leakage rate and leak testing

## 4.5 - Volumetric flow rate

At constant pressure  $p$ , the volumetric flow rate (SI:  $\text{m}^3 \cdot \text{s}^{-1}$ ) is proportional to the leakage rate.

$$Q_v = \frac{dV}{dt}$$

$$Q_{pv} = p \cdot Q_v$$

## 4.6 - Rate of pressure change

Similarly, at constant volume  $V$ , the rate of pressure change (in  $\text{Pa} \cdot \text{s}^{-1}$ ) can be used to compute the leakage rate. The rate of pressure decay and the rate of vacuum decay refer to the same physical quantity up to the sign.

$$Q_p = \frac{dp}{dt}$$

$$Q_{pv} = V \cdot Q_p$$

## 4.7 - Leakage rate and container integrity

Since by definition no leakage should be observed from a pristine sealed packaging, the leakage rate is a key measurement to evaluate the defect level of a container. Yet, the leakage rate alone is not enough, the driving pressure gradient ( $\Delta p$ ) must also be known. For example, submitting a small defect to a large pressure difference can produce a much higher leakage rate than a larger defect under a lower pressure.

When both the relative pressure ( $\Delta p$ ) and the leakage rate ( $Q_{pv}$ ) are known, the Hagen-Poiseuille equation or the Bernoulli's law can be used to evaluate the size of the defects. The results of such computations should not be taken as actual leak dimensions – generally only the diameter and length of an ideal cylindrical channel are considered – but rather as an indicative scale of the defect-level.

Alternatively, calibrated leaks are available to calibrate measurement devices or to study the effects of a given leak size on the product shelf-life.

# 4 Leakage rate and leak testing

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## 4.8 - Hagen-Poiseuille Equation

The Hagen-Poiseuille equation relates a fluid flow rate through a channel to the difference of pressure at both ends and the viscosity of the fluid. It applies for sufficiently long channels where a laminar flow can develop, and as long as the fluid density can be considered constant (incompressible fluid). The latter point is often loosely assumed for ideal gases submitted to small pressure differences, and an average mass density  $\rho_m = (M/RT)(p_1+p_2)/2$  is used.

Under these conditions the volumetric flow rate is given by:

$$Q_V = \frac{\pi d^4}{128\mu L}(p_1 - p_2)$$

and the leakage rate is given by:

$$Q_{PV} = \frac{\pi d^4}{128\mu L}(p_1^2 - p_2^2)$$

Where:

d is the diameter of the channel

L is the length of the channel

$\mu$  is the dynamic viscosity of the fluid (1.86 Pa·s for air at 25 °C)

$p_1$  and  $p_2$  the pressures at both end of the channel

The equation fails for too short pipes, where the flow is bounded by Bernoulli's law.

## 4.9 - Bernoulli's Law

Bernoulli's principle can be applied for short channels (pinholes) to relate the leak rate to the pressure difference. In its form for incompressible fluids (using the average mass density of the gas  $\rho_m = (M/RT)(p_1+p_2)/2$ , the volumetric flow rate is given by:

$$Q_V = \frac{\pi d^2}{4} \sqrt{\frac{2}{\rho_m}(p_1 - p_2)}$$

and the leak rate by:

$$Q_{PV} = \frac{\pi d^2}{4} \sqrt{\frac{RT}{M}(p_1^2 - p_2^2)}$$

Where:

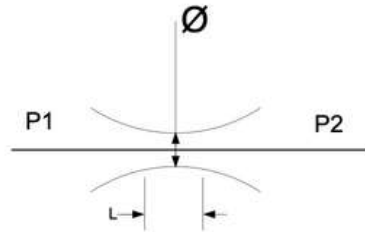
d is the diameter of the hole

$p_1$  and  $p_2$  the pressures on both sides of the hole

This expression yields an upper boundary for the leakage rate, as all pressure drops due to frictions effects or turbulences are neglected.

# 4 Leakage rate and leak testing

## 4.10 - Calibrated leak



A calibrated leak is an opening in a material through which the flow rate has been accurately measured using a certified method for one or more pressure differences ( $p_1 - p_2$ ). This calibrated leak can then be utilized to calibrate an instrument or a testing method, ensuring the accuracy and reliability of measurements. The performance characteristics of different calibrated leaks are outlined in the EN 13192 standard.

## 1 - Flexible container

Container defined by a variable volume limited by a closed foil that can expand in the three directions of space. Expansion is limited by the elasticity of the film. The ideal model is a perfect spherical balloon.

## 2 - Rigid container

By opposition to soft containers, rigid containers are not significantly expanding under internal pressure.

## 3 - Semi-rigid container

Containers inside which the effect of building-up pressure significantly affects one axis. The ideal model is a cylinder where a closing end is soft, or a cubic form with an upper soft cover.

## 4 - Headspace

The head space refers to the gaseous content of a packaging.

## 5 - Burst pressure

The burst pressure is the maximal relative pressure a flexible package can withstand before the seals or parts of the material break.

## 6 - Standard cubic centimetre and normo cubic meter

Standard  $\text{cm}^3$  (Scc) and Normo cubic meter ( $\text{Nm}^3$ ) are units of gas quantity in equivalent volume at standard pressure and temperature (1 atm = 1013 mbar, 0 °C), cf. DIN 1343.

They give rise to alternative units for the leakage rate: the standard cubic centimeter per minute (Sccm,  $\text{Scc} \cdot \text{min}^{-1}$ ) and the Normo cubic meter per hour ( $\text{Nm}^3 \cdot \text{h}^{-1}$ ):

$1 \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$  is equivalent to 592 Sccm

$1 \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$  is equivalent to  $0.0355 \text{ Nm}^3 \cdot \text{h}^{-1}$

In practice, the Scc unit is often replaced by the equivalent milliliter (mL) without explicit reference to the 1 atm, 0 °C standard.

# 5 Packaging's defects and product shelf-life

## 5.1 - Packaging material

Packaging can be rigid, semi-rigid or flexible, depending on their ability to expand in space under constraints. Though not exclusively, the packaging industry relies essentially on polymer materials, and a variety of laminates has been developed to build trays, films and pouches of all sorts. Most of these materials undergo a plastic deformation under constraints – hence their nick-name – but their expansion is often limited by their poor elasticity.

A variety of multilayer laminates are made combining different polymer films according to their respective features such as sealability, gas barrier properties, printability. Polyethylene (PE) and polypropylene (PP) are typically used for sealing, while polyethylene vinyl alcohol (EVOH) offers very good oxygen barrier properties. Biaxially-oriented polypropylene (BOPP) is a popular choice as a printable material with good moisture barrier properties. Polyvinylidene chloride (PVDC), metallized polyester (met-PET), and aluminum foil, in increasing order of barrier effectiveness, offer excellent protections against both oxygen and moisture, and materials are commonly used in packaging for sensitive products, such as snacks or processed foods.

Oil-based polymers still dominate the industry; however, there is a growing shift toward new materials, such as paper-based and biopolymers like coated polylactic acid (PLA), which are beginning to offer comparable barrier properties and meet the general demand for sustainable options.

## 5.2 - Seal and burst

After being filled with the product, a packaging is sealed, typically by heat welding of two PE layers together. Seals are crucial, because they are the weakest point of the packaging – sometimes designed to be weak enough for the customer!

Which pressure a seal can resist is often a good indication on the general solidity of the packaging and the tightness of the seals.

The burst pressure is thus an important measurement for the quality management.

## 5.3 - Defects

Defects of the packaging allowing gas exchange can be divided in two groups:

**Seal defects**, which leaves open channels between the two layers of the film. These channels can be tortuous (often longer than the width of the seal), of varying diameter, and sometimes interconnected. Only a leakage rate measurement can accurately assess the impact of apparent seal irregularities on the overall tightness of the packaging.

Because such leak paths are much longer than wide, the flow rate through channels are best modeled using the Poiseuille equation.

**Film defects.** Be it during its fabrication, transportation or processing, there is a significant chance that a laminate undergoes microdamage. Though often invisible, with dimensions from 10 to 100  $\mu\text{m}$ , direct holes through the thin film lead to much faster gas exchange compared to longer channels of similar section. These defects may consist of scratches, tears, et micro-cuts, yet they are commonly modelled by circular pinholes of a certain diameter. In the theoretical limit of zero film thickness and according to Bernoulli principle, only the defect surface area determines the leakage rate at a given pressure.

## 5.4 Product shelf-life

The shelf-life of a product is a key parameter of the food supply chain. For example, supermarkets will generally not accept products into their distribution centres unless at least 75% of their shelf life remains.

The shelf-life of a product can be defined by the time needed after packaging for the apparition of a first index of failure, that is, a property indicating that the food product is no more acceptable for the consumer.

A variety of physical, chemical, biochemical and biological reactions can lead to deterioration of the product and the apparition of an index of failure:

- Moisture intake, drying, staling or lipid oxidation can deteriorate organoleptic properties (loss of crispness, apparition of rancid flavours)
- Enzymatic reactions or exposure to light can lead to changes in color and appearances.
  - Microbial growth generally results in unacceptable appearance and organoleptic properties, but more importantly can pose severe health risks.

All these degradations reactions are highly dependent on the nature and composition of the product as well as on its environment. For a given product, two important factors can be underlined:

- The water activity of the product has a critical effect on the rate of these reactions
- The gaseous atmosphere surrounding the product, which has a strong influence on bacterial growth, as well as on chemical oxidation processes.

## 5.5 Water activity ( $a_w$ )

The water activity of a product is the ratio of the water vapor pressure of a food to the vapor pressure of pure water at the same temperature. In other words, it is the relative humidity of the atmosphere with which the product would be in equilibrium in terms of water exchange. The water activity of a food product dictates the nature of the degradation reactions that can take place:

- $a_w = 0.2 - 0.3$ : monolayer moisture (mo) optimal moisture content region where dehydrated foods have a maximum shelf-life.

- $a_w = 0.35 - 0.45$ : physical state changes begin, loss of crispiness, stickiness of powders and hard candies, caking of powders.
- $a_w = 0.4 - 0.5$ : soft materials (e.g., raisins) become hard as they dry out.
- $a_w = 0.6$  (critical point): potential growth of microbes. In the 0.6 to 0.75 range, mold dominates.
- $a_w = 0.6 - 0.8$ : the rates of food deteriorating chemical reactions in the aqueous phase reach a maximum.
- $a_w = 0.85$  (critical point) bacterial pathogens and spoilage bacteria begin to grow.

## 5.6 Moisture content (mc)

The moisture content is a measure of the quantity of water contained in a product. It corresponds to the ratio of the mass of water on the overall mass of the product (sometimes on the mass of the dried product), and is often given as a percentage. It should not be confused with the water activity, The latter will increase with an increase in moisture content, and conversely, but there is no standard relation between the two: water sorption isotherm need to be determined for each individual product to evaluate the relation between moisture content and water activity.

## 5.7 Relative humidity (RH)

The relative humidity of an atmosphere is the ratio of the water vapor pressure in the atmosphere to the vapor pressure of pure water at the same temperature, and is often given as a percentage. It corresponds to "how saturated" in water vapor is the atmosphere. The absolute amount of water in saturated air (100% RH) is about:

4.7 g/m<sup>3</sup> at 0 °C  
9.4 g/m<sup>3</sup> at 10 °C  
18 g/m<sup>3</sup> at 20 °C  
29 g/m<sup>3</sup> at 30 °C  
51 g/m<sup>3</sup> at 38 °C

## 5.8 Leakage (advection)

Advection, that is, mass transfer driven by a pressure difference, normally contributes less than diffusion to the gas exchange of a leaky packaging with its environment. However, handling of the product results in forces applied to the packaging that do accelerate gas exchange through the leak. Shelf-lives predicted from diffusion processes alone are thus always upper bounds.

An important case is represented by pressure variations in the environment of a packaging that cannot adjust its volume (a rigid packaging, or a flexible or semi-rigid packaging that has met its expansion limits):

- A decrease in the external pressure ( $p_1 < p_0$ ) and/or increase in temperature ( $T_1 > T_0$ ) create an overpressure in the packaging, which starts leaking its gas content to the environment.
- Conversely, an increase in the external pressure ( $p_1 > p_0$ ) and/or a decrease in temperature ( $T_1 < T_0$ ) leads to a flow of air into the packaging, diluting its atmosphere.

The succession of lower and higher-pressure conditions, for example during transport, can lead to rapid dilution of the packaging atmosphere with air. For example, after exposure to a pressure of 700 mbar, a rigid package is returned to 1000 mbar. Its internal atmosphere has been diluted with air, meaning, for a headspace initially composed of 50% CO<sub>2</sub>, that only 35% will remain after one cycle.

## 5.9 Diffusion through the container's defects

Free diffusion in the gas phase is the main cause of gas exchange through the defects of a container. Although the diffusion equation provides an accurate description of the diffusion kinetics, it requires dedicated computational tools to solve for real applications. To model molecular transport through a channel between a container and its environment, the steady state approximation (SSA) can be used to considerably simplify the computation. This approximation relies on the assumption that the time scale of the overall mass transfer is much greater than the timescale of steady state equilibration. In practice, three main hypotheses are made:

1. The concentration  $C$  varies instantaneously across the whole container, so that only an average inner concentration is considered (i.e. the homogenization of the concentration inside the container is much faster than its overall variation ).[3]
2. The external concentration  $C_{ext}$  is taken as constant.[4]
3. The concentration gradient profile along the channel instantaneously adjusts to the variations of the concentration in the container.

[3] The average distance travelled by an air molecule by diffusion is about 3–5 cm/min under standard conditions, which allows homogenization of the internal concentration to be faster than the overall concentration change due to the leak in most relevant cases.

[4] For the same reason, dilution of the escaping gas in the environment is sufficiently rapid compared to the leak rate to make this assumption reasonable.

# Packaging's defects and product shelf-life

Using Fick's first law and the SSA, the flux  $J$  (here in  $\text{mol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$ ) through the channel is given by:

$$J(t) = -D \frac{\Delta C(t)}{l}$$

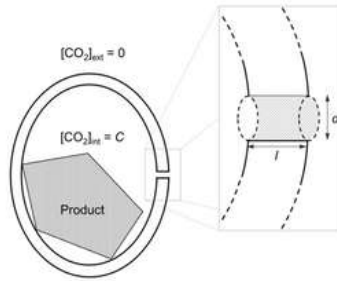
with  $\Delta C(t) = C(t) - C_{\text{ext}}$ , the difference of concentrations in the species of interest,

$D$  is the diffusion coefficient of the gas of interest in air,

and  $l$  is the length of the channel.

Multiplying the flux by the channel section (in  $\text{m}^2$ ) yields the overall rate of transport in  $\text{mol}\cdot\text{s}^{-1}$ .

This one-dimensional equation is well-suited to study the effect of a spherical hole in a packaging on the evolution of gas concentration.



Let's take as an example the evolution of the  $\text{CO}_2$  concentration inside a packaging. A variety of food products are packaged under a  $\text{CO}_2$  atmosphere to ensure aseptic conditions. Taking this initial concentration as  $C_0$ , and neglecting the usual concentration of  $\text{CO}_2$  in the atmosphere (0.04%), so that  $C_{\text{ext}} = 0$ , the rate of evolution of the  $\text{CO}_2$  concentration  $C(t)$  in a packaging of volume  $V$ , through a leak of section  $A = \pi \cdot d^2/4$ , and length  $l$ , is given by:

$$\frac{dC(t)}{dt} = \frac{A}{V} J = -D \frac{A}{V} \frac{C(t)}{l}$$

Solving this equation for the initial conditions yields:

$$C(t) = C_0 \exp\left(-\frac{AD}{Vl} t\right)$$

Where  $D$  represents the diffusion coefficient of  $\text{CO}_2$  in the air at the temperature considered. For all practical purposes, the gas can be considered ideal, allowing the equation to be written in terms of volume fraction,  $X$ , or partial pressure,  $p$ :

$$x(t) = x_0 \exp\left(-\frac{AD}{Vl}t\right)$$

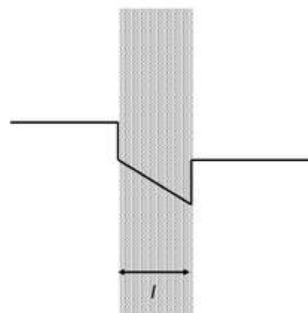
$$p(t) = p_0 \exp\left(-\frac{AD}{Vl}t\right)$$

## Note on the effects of temperature and pressure variations

- Unlike the permeability coefficient of polymer films, gas diffusion coefficients are not very sensitive to temperature variations. In the range of our concern (say 0 – 40 °C), these variations are negligible.
- in the case of a flexible packaging, variations in pressure and temperature conditions only affect the gas density according to the ideal gas law. As long as the packaging expansion limits are not met, the plastic deformation of the packaging almost instantly balances its volume with the density of the contained gas. No leakage rate (in the sense of  $dPV/dt$ ) will be observed, and the effect on the diffusion rate is reflected in equations (3) and (4) by a change in the internal gas volume  $V$ : at low temperature and/or high pressure, the smaller volume ( $V = nRT/p$ ) will result in a greater net flow.
- Conversely, in the case of a rigid packaging (or a semi-flexible packaging that has reached its deformation limits), variations in temperature and pressure conditions lead to a change in gas density inside the package. Unable to compensate through volume changes, advection will occur through the defect to rebalance internal and external pressures.

## 5.10 Permeability: diffusion through a solid membrane

In contrast to the free diffusion through a leak channel, which only occurs in the gas phase, diffusion through a solid membrane implies phase transfer. Thus, knowing the diffusion coefficient ( $D$ ) of the gas of interest in the studied material no longer allows to determine the rate of transfer: the solubility ( $S$ ) of the gas in that material should also be considered. In practice, both parameters are measured together, and are given on the form of a permeability coefficient:  $P = D.S$ .



Example of a concentration gradient profile along a barrier film of thickness  $l$  separating two mixtures of different concentration in the gas of interest

# Packaging's defects and product shelf-life

The flow is generally expressed in terms of the partial pressure of the gas of interest on both side of the membranes, though modifying the equation to set it in terms of concentrations or volume fractions is easily done using the ideal gas law. The steady state approximation and Fick's first law yield an expression for the flux  $J$ :

$$J(t) = -P \frac{\Delta p}{l} \quad (2)$$

Where  $\Delta p = p - p_{ext}$  is the difference of the gas partial pressures on each side of the film,  $l$  is the thickness of the film (in mm, or SI: m), and  $P$  is the permeability coefficient (in  $\text{Nm}^3 \cdot \text{mm} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{atm}^{-1}$ , or SI:  $\text{mol} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ ). There a large variety of units in use for permeability coefficients, and one should take great care at the homogeneity of units when carrying actual computations.

## 5.11 Choice of the packaging material

The permeability coefficients are generally dependent on the conditions of temperature and humidity. Thus, rather than a permeability coefficient, packaging film producers generally provide the oxygen transmission rate (OTR, typically in  $\text{mL} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ ) or the water vapor transmission rate (WVTR, typically in  $\text{g} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ ) for a particular laminate material and thickness under controlled humidity and temperature conditions. These values can be used to calculate the rate of oxygen or water intake for a given packaging under given conditions.

Such computations, based on the characteristics of the product, its sensitivity to oxygen and moisture, and the desired shelf-life will direct the choice of a given type of laminate for a given application.

## OUR FUTURE BY LYES IRID CTO ANEOLIA

**Imagine! Imagine being able to enjoy every bite without a single worry.  
That's what we offer you through our research.**

By combining our passion for science and our industrial expertise, we develop intelligent packaging that protects your food from contaminants and preserves its freshness. Our goal? To offer you healthy and safe food, while reducing food waste. Because we believe that quality food is a right for all.

Defective packaging can compromise the shelf life of a product, allow contamination and lead to health risks. So we work to refine our testing devices and methods to detect even microscopic leaks.

**We push the limits of what is possible in terms of precision.**

But beyond the technical aspect, there is another motivation: as a consumer myself, this effort is also personal. We are not only looking to achieve industrial targets, but to ensure that the products we consume are safe, that packaging fully plays its role as a barrier against contaminants and that testing methods are compliant. Our commitment goes beyond simple compliance with standards. We work to offer our customers a truly scientific and reliable solution, capable of proving the compliance of packaging, and therefore protecting consumers from risks.

At the same time, we are committed to reducing food waste and minimizing the use of materials, in an eco-responsible approach. The rigorous testing methods that we implement allow us to choose suitable packaging, while avoiding unnecessary waste of resources. This approach is part of a future where hygiene, waste reduction and environmental preservation are essential.

Thus, our collective effort is not simply a contribution to the industry, but an act of conscience for a healthier future, for our children and future generations. Packaging integrity, which we seek to improve every day, is the key not only to guaranteeing the quality of the products we consume, but also to reducing our ecological footprint. Together, we must recognize that this responsibility falls on all of us, because we are all concerned with the quality and safety of the products we consume every day.



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