Anéolia

Gas analysis and micro leak measurement

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TOPIC

Preservation of product: Methodology for the leak measurement and integrity control in order to optimize breathability of packages

24/7

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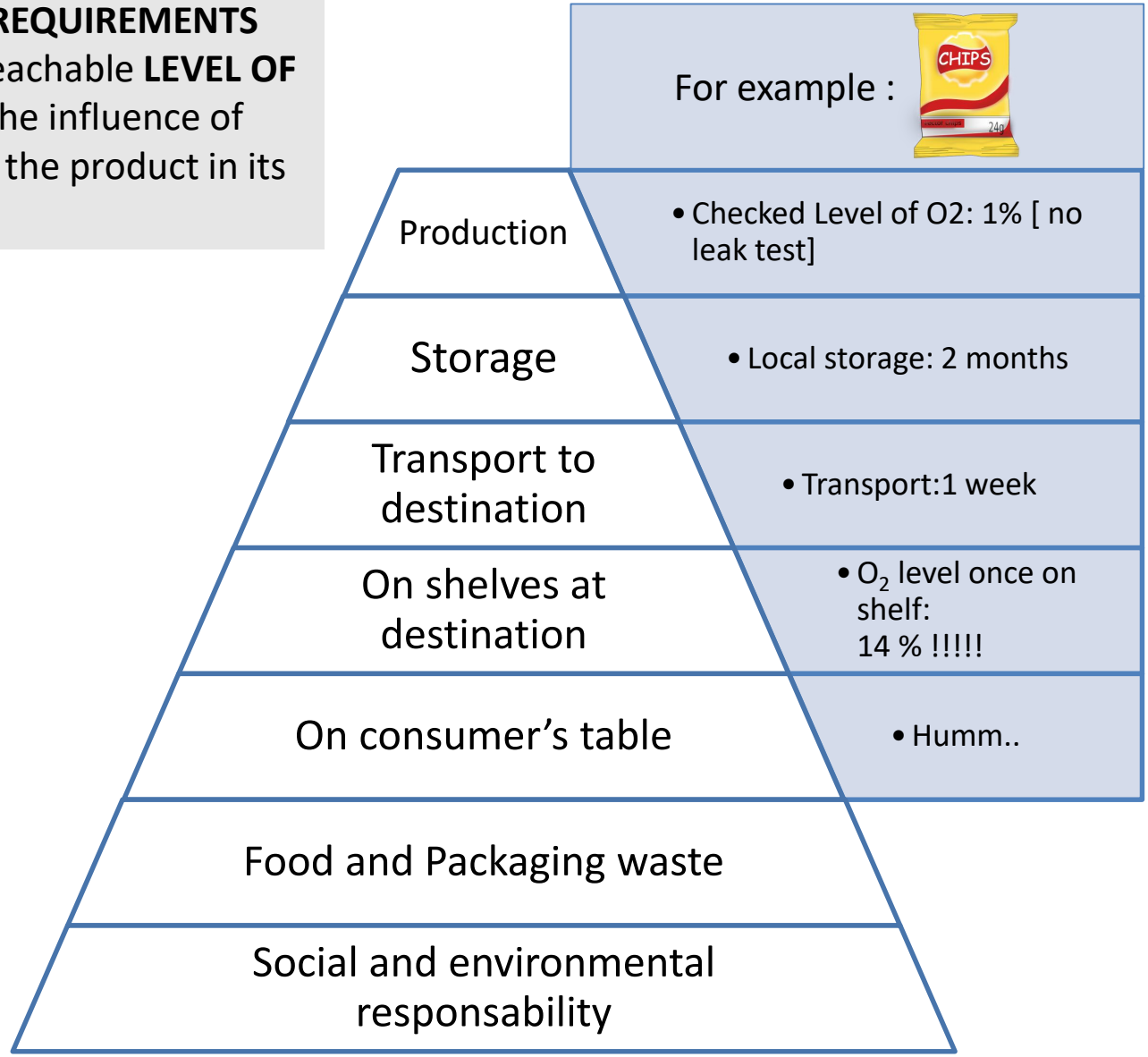
Each area in this triangle represents **THE REQUIREMENTS OF THE MANUFACTURER** as well as the reachable **LEVEL OF SATISFACTION** of the consumer towards the influence of technical and environmental variables for the product in its packaging

Environmental variables:

- ✓ Transport mode
- ✓ Destination
- ✓ Shelf life
- ✓ Acceptable reject rate

Technical variable:

- ✓ Sealing methods
- ✓ Line speed
- ✓ films, recipes
- ✓ Leak test



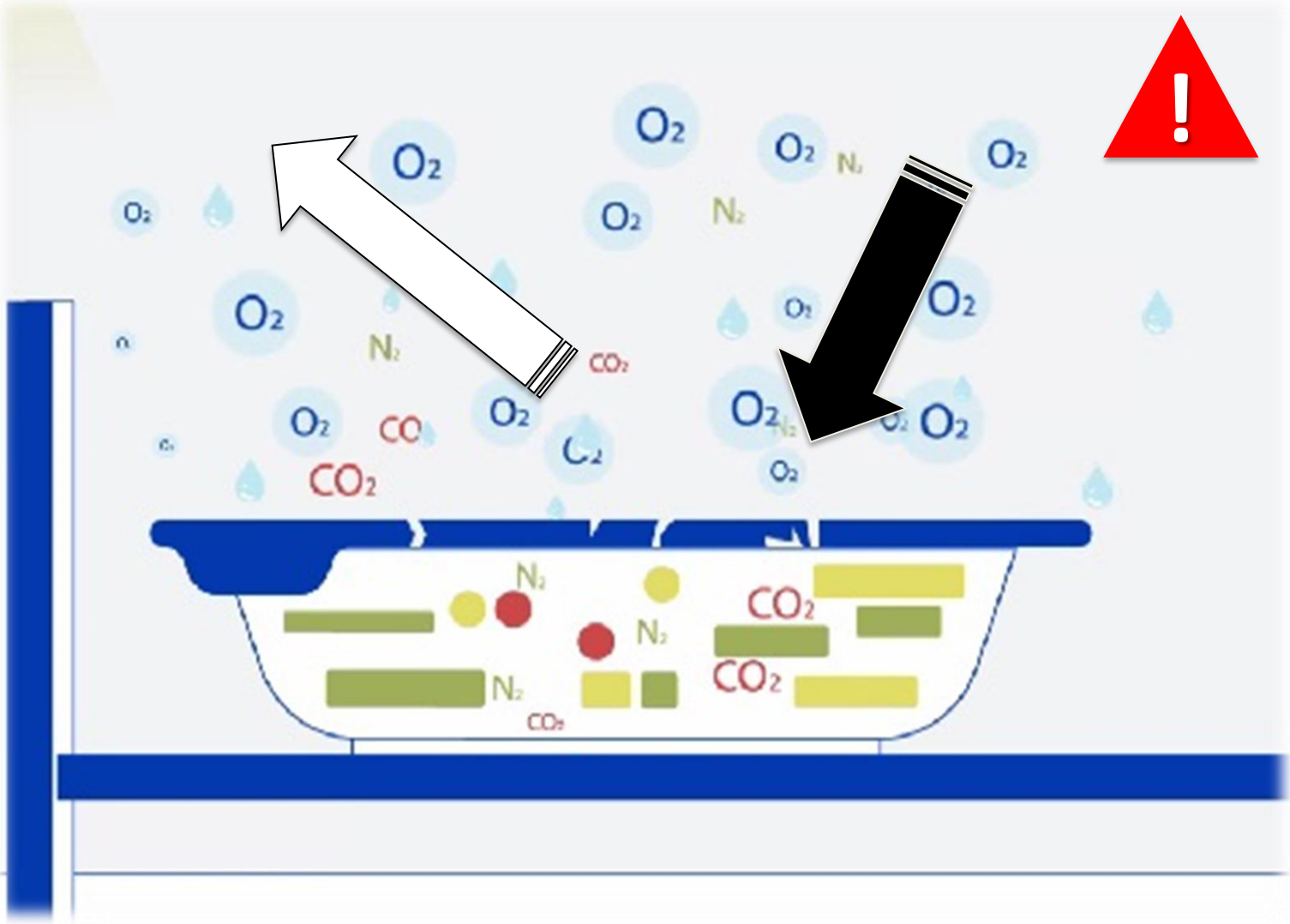
What is a leak?

"a hole in a container or covering, through which liquid or gas may accidentally pass."

This definition is fine but incomplete we should say:

"a hole in a container or covering through which liquid or gas may accidentally pass, **but also creates an exchange with the environment**"

Environmental exchange due to MICRO LEAKS



- Humidity
- Risks of bacterial contamination
- Oxygen balancing
- CO_2 or N_2 loss

Method / Detector	Tracer gas	Tested object under overpressure	Tested object under vacuum	Quantitative test	Localization	mbar l/s											
						10^{0}	10^{-1}	10^{-2}	10^{-3}	10^{-4}	10^{-5}	10^{-6}	10^{-7}	10^{-8}	10^{-9}	10^{-10}	10^{-11}
Description						Water dropping		Water tight		Bacteria tight		Virus tight		Gas tight		Technically tight	
Leak diameter						100 µm		30 µm		10 µm		3 µm		0.8 µm		0.1 µm	
Escape time of a bubble with 1 cc						10 s		> 15 min		> 1 day		> 100 days		> 30 years		> 1000 years	

ALL methods are sensitive enough for leak detection..
The difference is the time allowed to the test…

ESCAPE TIME FOR 1 cc	
100 µm	10 sec
30 µm	>15 minutes
10 µm	> 1 day
3 µm	>100 days
0,8 µm	>30 years
0,1 µm	>1000 years

Next: we must define our requirements

Which ones:

Technical variables

- Easy opening
- Storage
- Volume of generated waste
- Preservation after opening
- Weight

Environmental variables

- Visual
- Taste, flavours
- Colour, aspect
- Texture
- Traceability
- Health

PRESERVING THE CONTENT

(Best envelope , leak tightness)

REASSURING THE CONSUMER

(A firm commitment on shelf life: lower leak possible, conserving gas(MAP)

USING ACTIVE COMPONENT

(Active chemical components and materials, digital or electronic means, barrier films in case of Modified atmosphere)

How to do it ?

ONLY ONE METHOD: MASTERING THE FOLLOWING KEY POINTS

1. To have a metrological reference
2. To organize the control means by increasing sensitivity
3. To obtain records of leak rate sizing in regards of effective shelf life
4. To measure and know before judging
5. To identify influence variables affecting the performance
6. To optimize each single variable independently from others
7. To collect and to analyze
8. The production release limit
9. A satisfying product (safety + economy)

A BETTER COMMITMENT WITH ALL THE PLAYERS

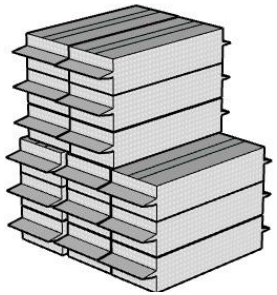
TO COMMIT TO EFFECTIVE SHELF LIFE

Reducing factors of defects:

- Leak
- Sealable
- Permeation
- Mechanical resistance…

TO SELECT AND MEASURE:

-A quality of sealing
-A reliable and repeatable process
-Define machinery specifications and settings (speed/quality)
-Select the appropriate material

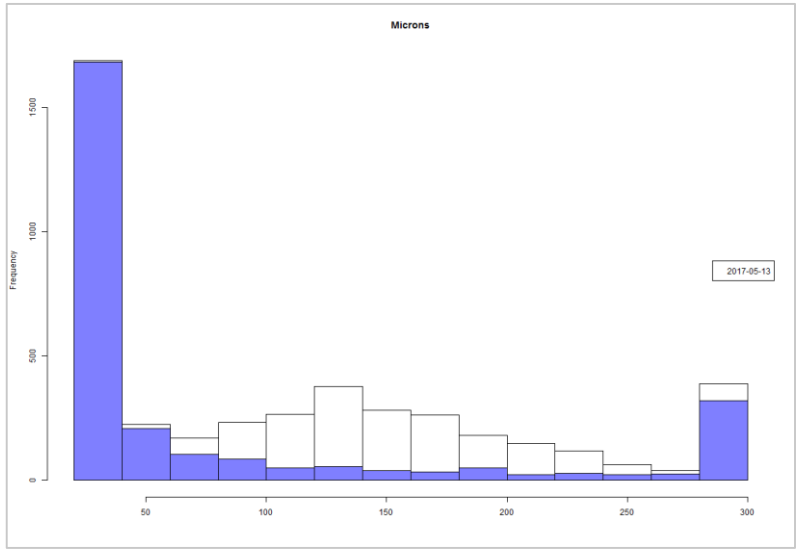


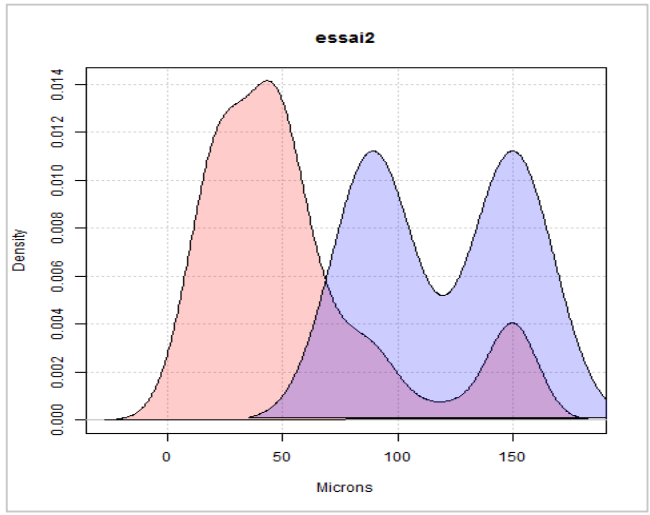
? ? ?

Next: How to we help to achieve these goals?

How do we help to **ACHIEVE** these GOALS?

Share Data analysis and metrological references with all players



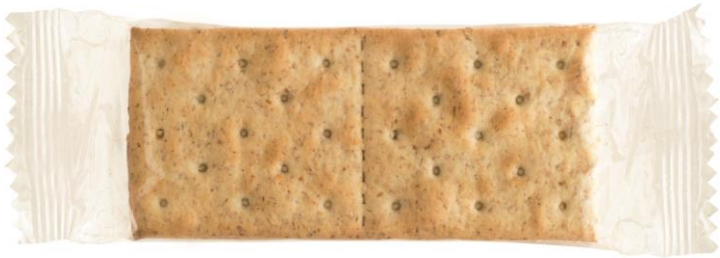


For example collect and analyse data for HUMIDITY or OXYGEN UPTAKE according to leak size

Achieving consistent crunchiness is a satisfied consumer

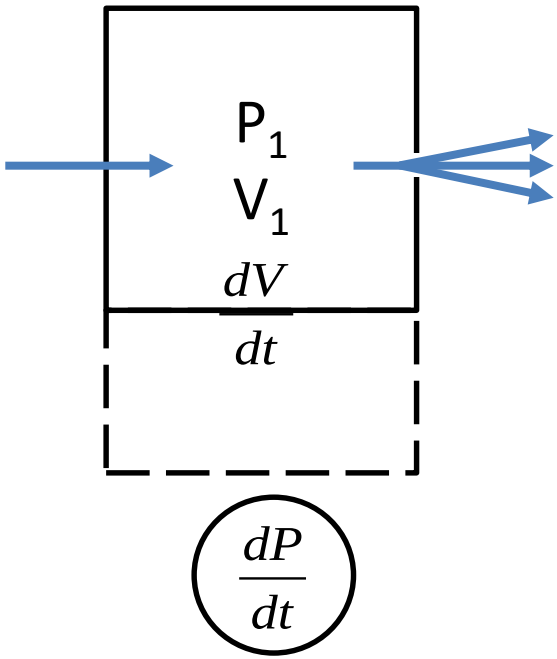
Once you have shared the data what is needed to get further commitments?

- A good understanding of the Standards
  - Selecting the best method



EXTRACT OF DIN 55508-1 STANDARD

The leak rate is measured by a physical loss of volume of fluid over time at a given differential pressure, and expressed in (**Pa.m³/sec**)
THIS IS NOT THE PERMEATION RATE



$\frac{dm}{dt}$

LEAK RATE in **Pa.m³/sec**

Homogeneous with energy

$P_2 = P_{atm}$

V_∞

$$Q_{PV} = \frac{d(P.V)}{dt} = V.\frac{dP}{dt} + P.\frac{dV}{dt}$$

Variation of pressure at constant volume

Flow at constant pressure

More variables : pressure –Volume-Time

Next :an other standard

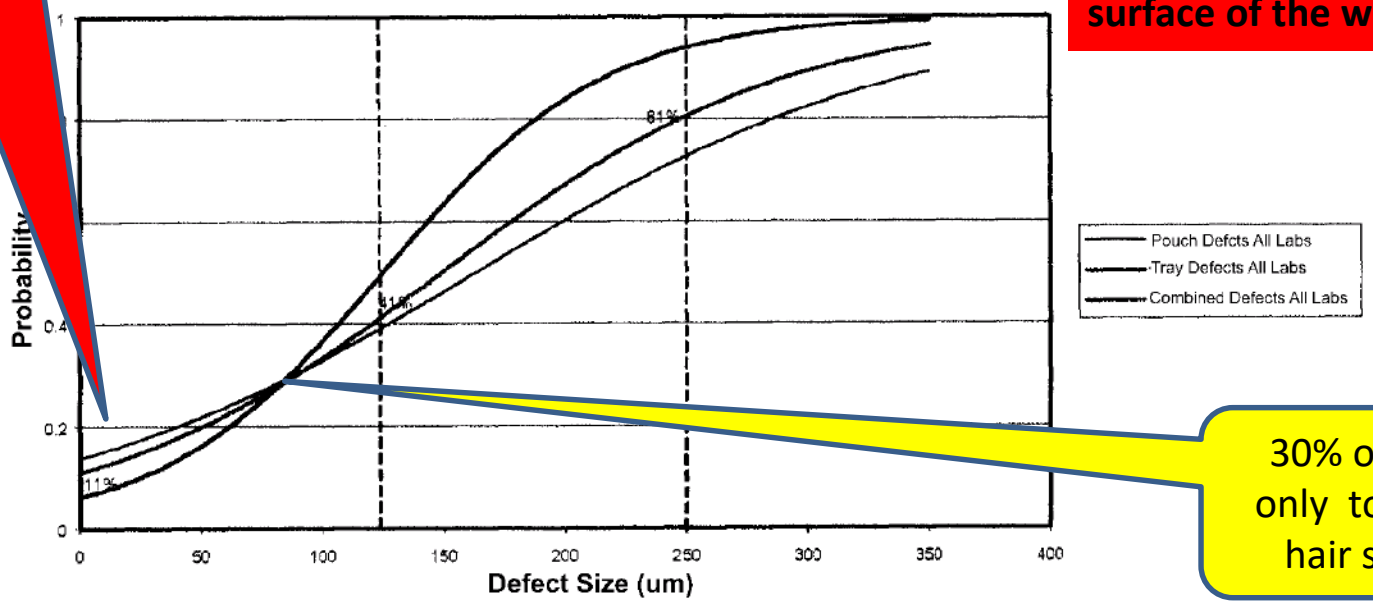
11% of chance to see bubbles which are not leaks because or air trapped

Water bath/ basin

This standard shows the probability to detect a leak rate in a water bath.
The sensitivity depends on the rigidity of the pack.
The water bath shows the major leaks and hides micro-leaks existing at the surface of the whole package.

F 2096 – 02$^{\epsilon 1}$

Probability of Rejection
Combined Laboratory Results



30% of chance only to detect a hair size leak

FIG. 2 Probability of Rejection-Combined Laboratory Results

F 2096 standard

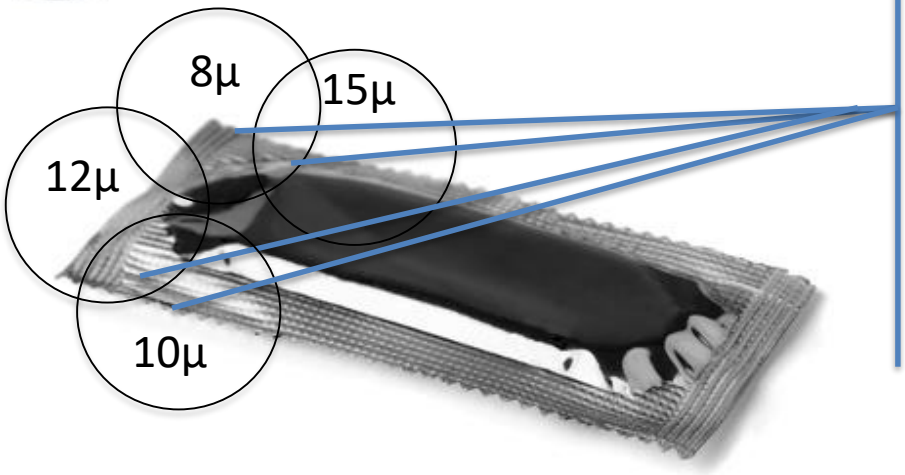
Leak test by immersion

DRAWBACKS:

> If there is only one perforation, the water does not enter the packaging, unless it comes to the surface of the water, which requires manipulation

> It can't quantify leaks objectively, and it requires time for both manipulation and subjective observation by the operator.

Based on the standard F2096, let's immerge a packaging with a number of micro leaks due to a poor sealing jaw or any temperature issue happening at the wrong time during production:

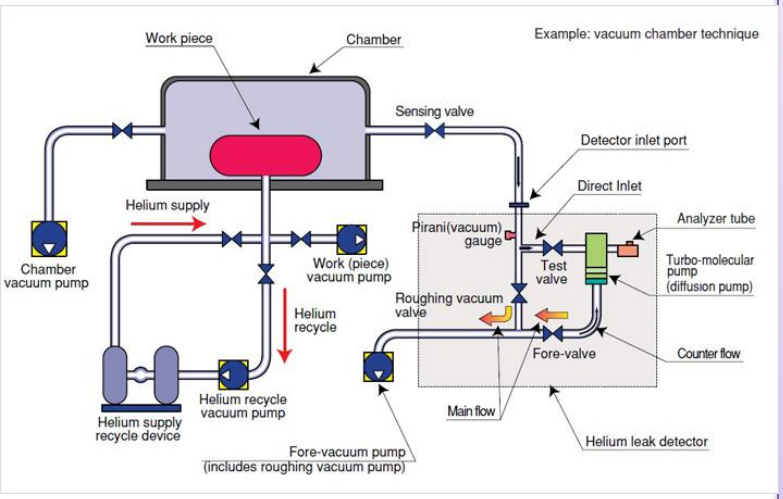


If we **follow what the standard says** none of those micro leak will be detectable under water individually but added to each other it will provide an eventual large surface of exchange with the environment. More than enough to let the gas escape, humidity to penetrate the packaging or even worst.. Bacteria, virus …
but it definitely shortens the shelf life of the product.

In addition, water is, in many cases, banned from production workshops for hygiene and safety hence water bath is not a 100% "non destructive method" , risks are too high and it is not responsible to reconditioned a product after being under water.

- Virus size: 0.1µm
- Bacteria: 1µm
- Blood cell: 10µm
- Human hair: from 40 to 80µm

Tracer gas systems



Helium test:

- Not simple
- Expensive to run
- Expensive to maintain

Tracer gas systems such as He or CO_2 are quite costly **and not so effective if we consider all variables that might alter the result of the test:**

1- this test **is dependant of the "pollution"** of the testing environment especially if a large leak had occurred .

2- This test is **100% dependant of operator's skill.**

3- this test is **dependant of the vacuum level in the chamber and the speed the chamber is emptied**

4- This test is **dependant of the product volume and the dead volume of the chamber**

To increase sensitivity of this method it is necessary to increase vacuum level and/or increase test time , those two factors could create fatigue on seals and introduce extra micro leaks independently from the manufacturing process.

It is compulsory to master all variables to define a repeatable leak rate.

This method consists in inflating the package at a pre-defined pressure, then stopping the air flow and measuring the pressure variation over time.

The problem with soft packaging is .. They are soft .. Hence they grow or shrink over pressure variations …

-Directly dependant on packaging internal dead **volume with the product inside** ….
We can recall the law of perfect gases at constant temperature[PV=Cst]
Pressure x Volume = Mass x Constant xT
- Directly dependant on temperature variations while testing

$$LEAK_{(cc/mn)} = 60 \times 10^{-5} \times V_{(cc)} \times dP/dt_{(pa/sec)} \text{ at } T° = cst$$

QUIZ: Using the above formula, calculate the dP/dt with a volume of 100 cc resulting from a a leakflow of 1 mL/mn at T°= 20degC.
Then compare the result using when the Volume shrinkage is 10 % …(90 cc) **..and write down the value you will set as a level to discriminate a good from a fail product ?**

The different means of checking for leaks their pros and cons:

The direct measurement of loss of material (ABISS® Open System):

This method combines the best of all methods WITHOUT THEIR INCONVENIENT , a great methodology, easiness of set up , data recording and a very high sensitivity coupled with only mastered variable.

EXOS : Micro leak measurement system ABISS®

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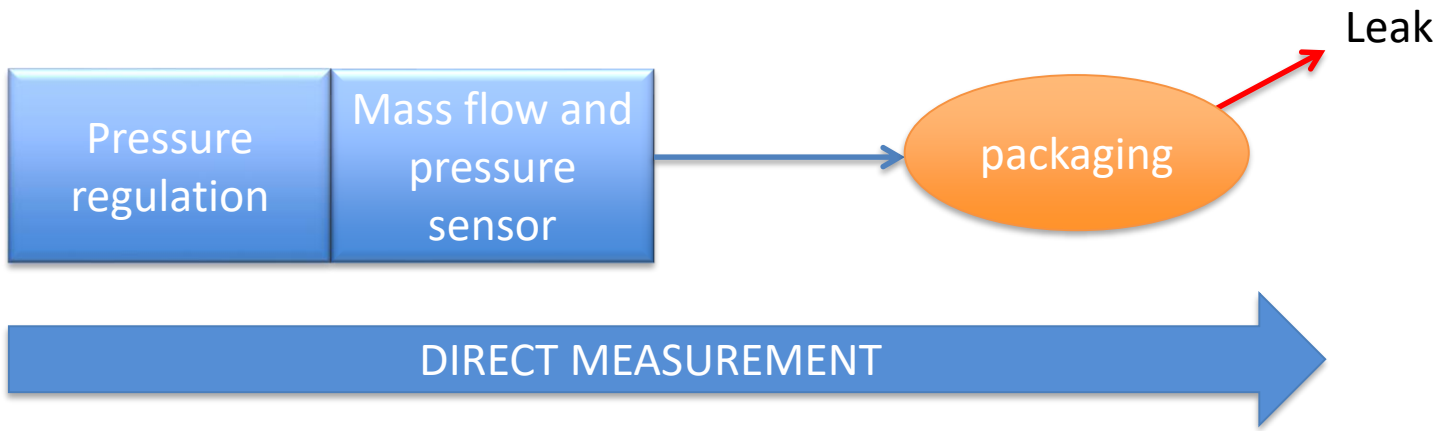
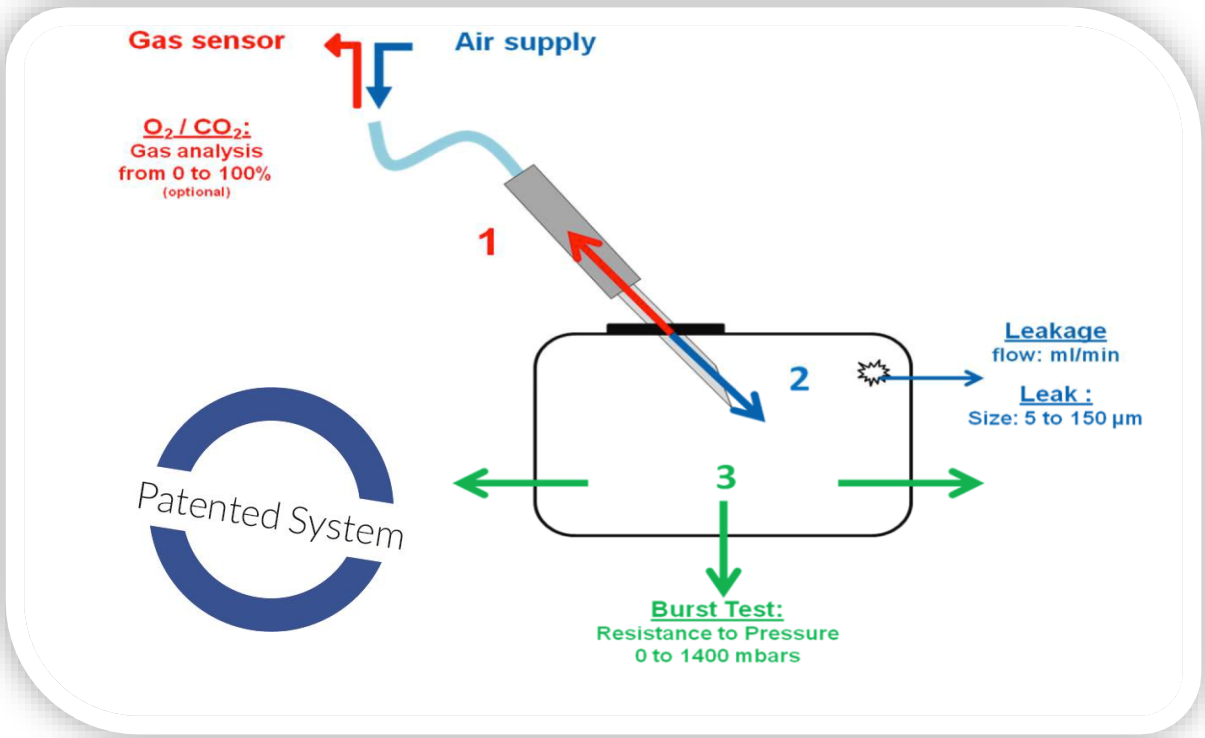


ABISS EXOS : FAST, SIMPLE, EFFICIENT, Independent from unknown variables

Our EXOS and OXYLOS : 1 instrument, 1 sample , 3 tests:

- Gas analysis (O_2 CO_2)
- Micro leak test
- Burst or creep test



abiss

Gas analysis

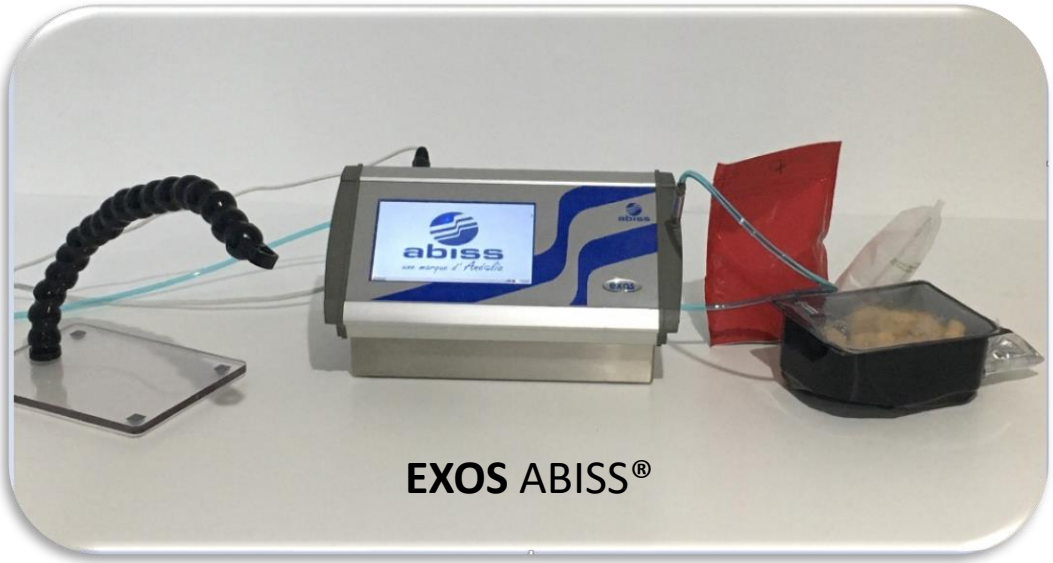
Patented System

MADE IN FRANCE

Patented technology:

Measuring and determining the leak rate in combination with gas analysis to evaluate the gas exchange rates.

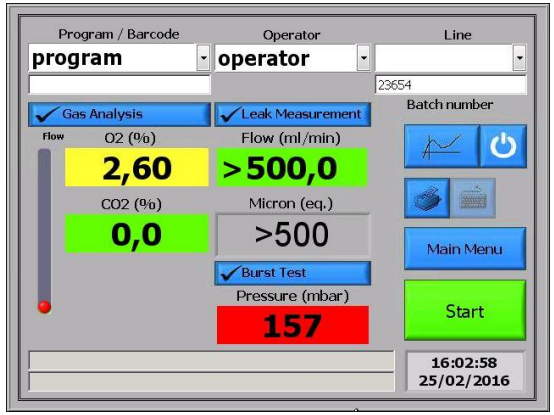
GAS + MICRO LEAK + BURST TEST
in 1 sample



EXOS ABISS®

OXYLOS Hygienic Design ABISS®

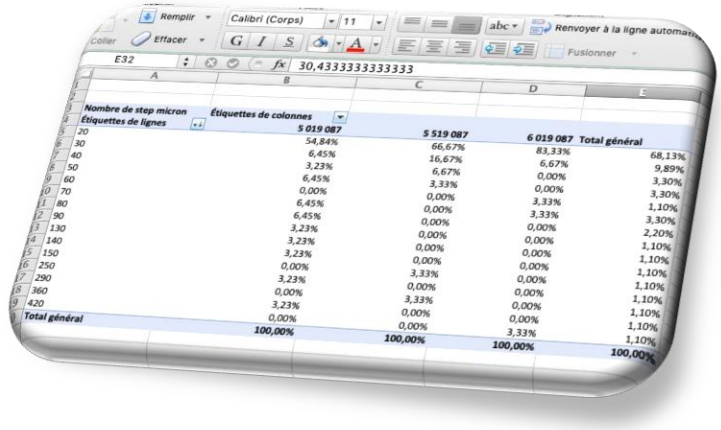
EXOS ABISS®: An easy way to solve problems



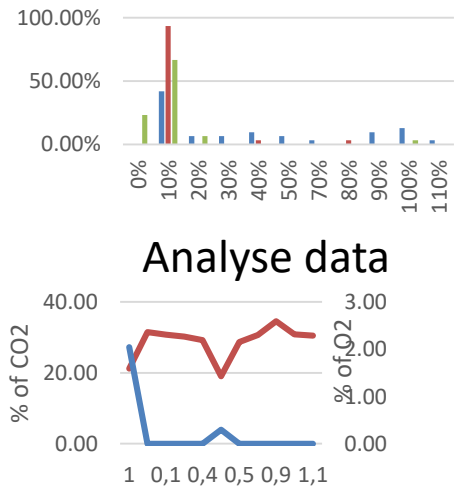
Viewing data
[O_2,CO_2 Leak and burst]

Observing phenomenon
[measuring curves]

Collecting data



Analyse data



Find the solutions

« A GOOD METHODOLOGY coupled with
Data collection and analysis will inevitably
Guide you to THE SOLUTION of your issues
To make **consumers safer** and happier
together with saving time
And MONEY to the manufacturer »

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Gas analysis and micro leak measurement

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