# Leak Detection in the **Food Industry**

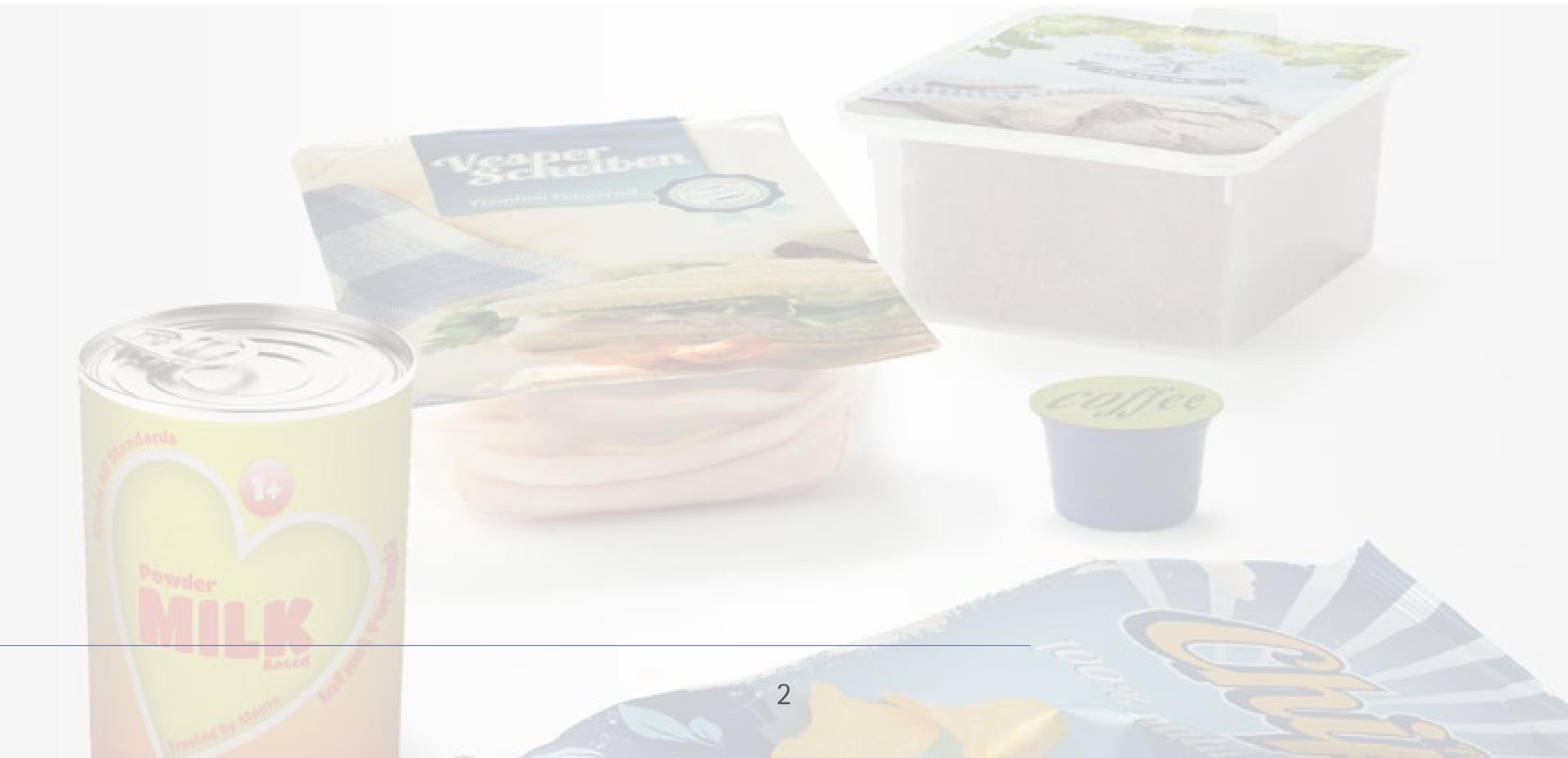
## A Guide

# Introduction

Requirements for food producers are continuously increasing. One reason for this increase is that today's consumers are better informed and more demanding. Another reason is stricter legal stipulations for manufacturers and distributors with the goal of optimum safety for the consumer. There are many factors manufacturers must eliminate to assure they deliver a safe product. Microbial growth, oxidation, moisture or pests can cause products to spoil well before their expiration date.

Proper packaging and the appropriate atmosphere are two of the most important criteria to make food durable. Specifically, composed protective gas atmospheres (Modified Packaging Atmospheres, MAP) contribute to the longevity of food stuffs – as long as the concentrations of the different gases do not change for the duration of a product's shelf life. This is achieved by sealed packages, which is why leak detection in the food industry is becoming increasingly important. The choices of testing methods are extensive and food manufacturers should carefully analyze their requirements before deciding on a process that is right for them.

This e-book is intended to provide you with an overview of the most important testing methods and to illustrate the challenges associated with leak detection for food packaging. We invite you to contact us to talk about your specific application needs.



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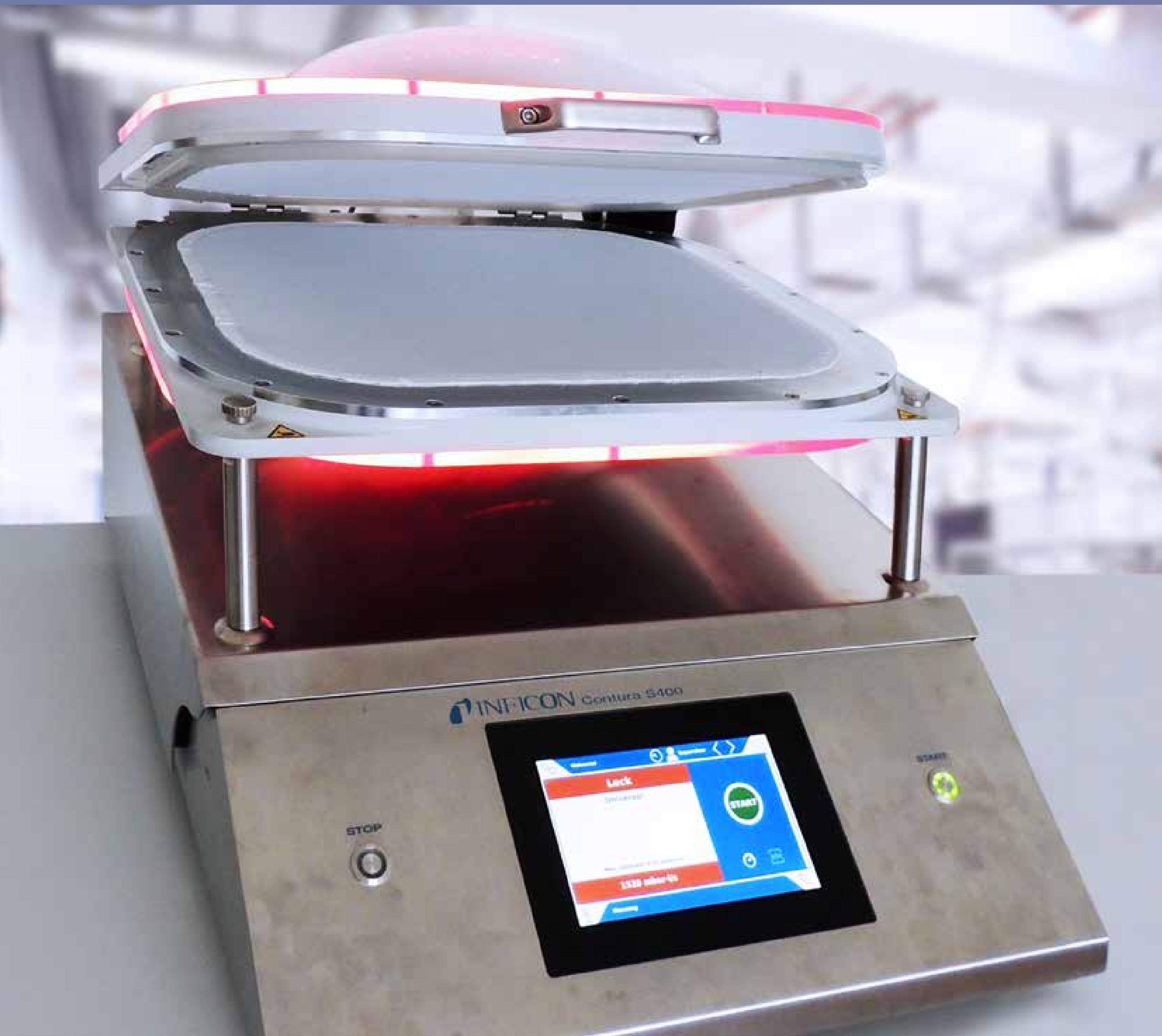
# Part 1
# Principles of Leak Detection in the Food Industry



# 1. Leak Rates and Types of Leaks

## 1.1 Leakage Types, Tightness and Measuring Units

A leak is a structure in the wall of an object through which gases or liquids can escape. This may be a simple hole, a permeable, porous region as well as capillaries, which are often difficult to identify. The leak rate indicates how much gas or liquid passes through one or more leaks at a given pressure difference within a defined time interval. For example: If in exactly one second one cubic centimeter of gas at an overpressure of one bar exits through a leak, the leak rate is 0.001 liters times 1000 mbar divided by 1 second, or in short: 1 mbar·l/s. One could also say that after exiting, the gas has a volume of 1 $cm^3$ at a pressure of 1 bar. Here is another way to calculate the unit: If the pressure inside a container with a volume of 1 liter changes by 1 millibar per second, the leak rate is 1 mbar·l/s. Leak rates measured in mbar·l/s are usually expressed in an exponential scientific notation: instead of 0.005 mbar l/s, it is written as $5x10^{-3}$ mbar·l/s.

In Europe, the measuring unit "mbar·l/s (or ccm/s)" has been widely accepted, but volumes and pressures may, of course, also be expressed in alternative units, resulting in a different measuring unit for the leak rate. The SI units are internationally recognized, resulting in the leak rate being expressed as Pa·$m^3$/s. In the US, measurements are often made in atm cc/s while, especially during the pressure drop test, the "standard cubic centimeter per minute" (sccm) is commonly used as the unit for the leak rate.

Following is a list for the conversion of units:

| | | |
|---|---|---|
| 1 atm cc/s | ≈ | 1 mbar·l/s |
| 1 Pa $m^3$/s | = | 10 mbar·l/s (SI unit) |
| 1 sccm | ≈ | 1/60 mbar·l/s |

## 1.2 Correlation Between Pore or Channel Size and Leak Rate

Understanding the relationship between a leak rate measured in mbar·l/s and the size of a leak of pores, capillaries or channels is worthwhile. In other words: What diameter must a circular hole or capillary actually have to cause a certain leak rate? Provided the diameter of the hole is significantly larger than the wall thickness of the foil or the packaging material, a hole with a diameter of 0.1 mm at a pressure difference of 1 bar results in a leak rate of about 1 mbar·l/s. In the case of a channel or a fine capillary, the leak rate changes to the effect that the gas exchange takes place at a much slower rate since the gas has to travel a longer distance. This effect is illustrated in Figure 1 and Figure 2. Accordingly, leak rates always change whenever the pressure difference between the interior of a test object and its surrounding atmosphere changes. In order to make the leak rate data comparable, they are normalized to a pressure difference of 1 bar (1000 mbar). If the pressure differential has a value other than 1, this value must be specified.

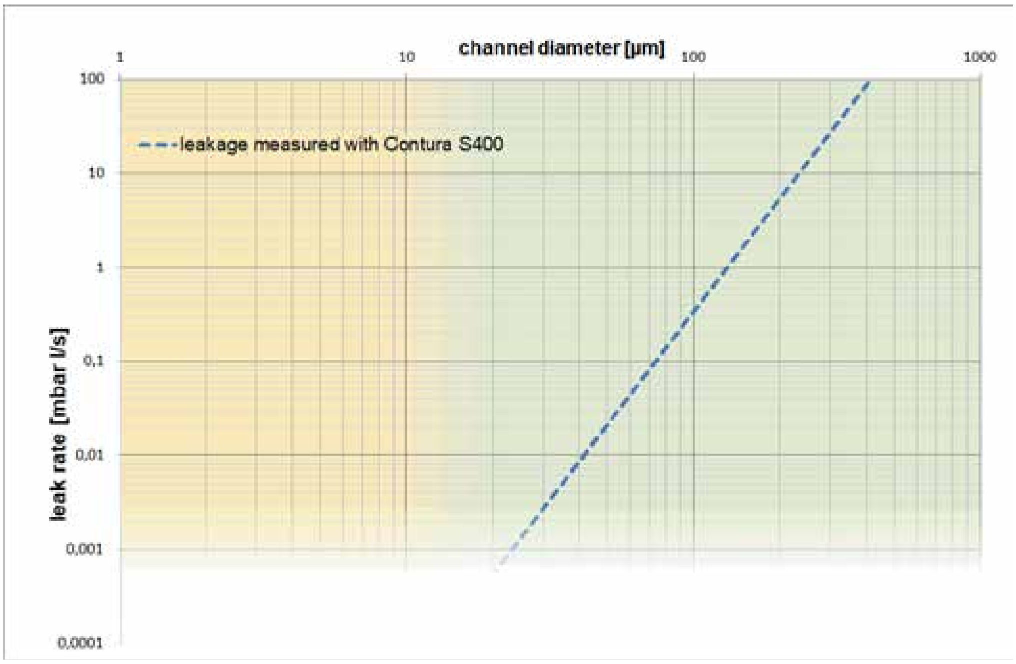



Figure 1: Correlation between pore diameter and measured leak rate at a foil thickness of 50 µm.

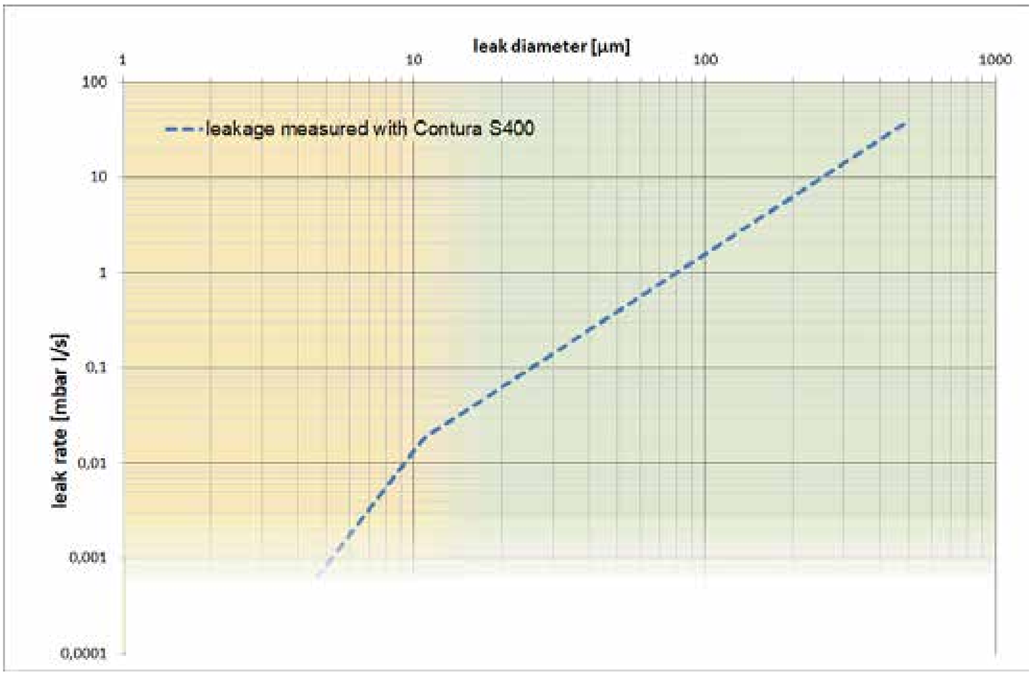



Figure 2: Correlation between channel diameter and measured leak rate for a channel length of 2 cm.

The precise leak rate still acceptable in any particular case, and at what point the packaging no longer passes the leakage test, is always dependent on the specific quality specifications in the production process.

Accordingly, the maximum permissible leak rate should always be taken into consideration when selecting the test method.

### 1.3 Potential Weak Spots of Packages

Often, food manufacturers are forced to calculate packaging materials and manufacturing processes, such as sealing, very closely to keep margins at a profitable level. For this reason, manufacturers have to resort, for example, to more economical foils which may develop larger pores and micro cracks or even tear as a result of the stress during the thermoforming. During the sealing process, undesired capillaries are easily formed by things such as residual product left close to the welding seam or plastic chips produced during the cutting of the packaging. Improperly matched adhesives and foil materials may also lead to poor quality seals. Especially small leaks like these are overlooked by many traditional testing methods, like the water bath, for example, allowing leaky packages to be placed on the market.

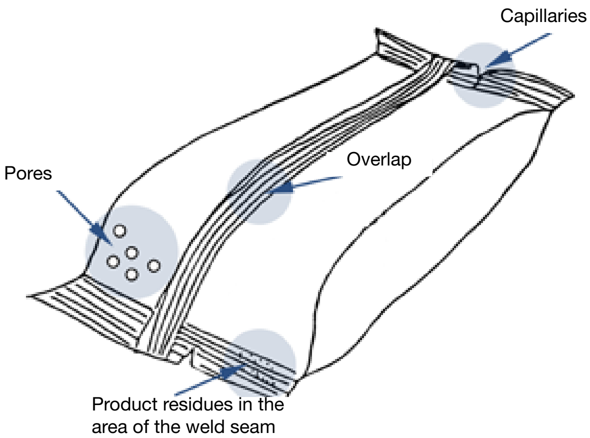



Figure 3: Graphical representation of the most common weak spots of packaging: Pore size, poorly sealed welds, and too narrow overlaps.

## 2. Common Leak Detection Methods in the Food Industry

The right leak detection method is determined by many factors: What type of packaging will be tested? Is a non-destructive test required? Does the packaging contain protective gas or sufficient head space? Which leak rate - and in this context also which shelf life date - are acceptable? Before deciding on a method, manufacturers should be aware of these requirements. Following is an overview of the most frequent test methods used in the food industry:

### 2.1 Water Bath

In the food industry, the water bath is in relative terms the easiest and most widely used testing method. The water bath method is also referred to as a bubble test or the "bicycle tube" method, whereby the test product is submerged in a water tank while the tester watches for emerging bubbles. In practice, due to the small difference in pressure and the surface tension of the water, often either none or only very large bubbles will emerge. Table

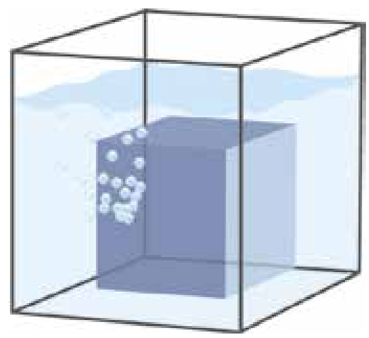


Figure 4: Schematic representation of the water bath method

1 shows the bubble rates of different leak rates under ideal conditions, which can almost never be achieved in real life. To increase the pressure difference, the air space above the water level must be evacuated to create negative pressure. This causes the pressure difference between the interior of the packaging and its environment to increase and allows the gas to pass through the leak more easily. However, the created low pressure must not be too low, otherwise the packaging will burst. The advantages of the water tank, namely its simple and intuitive handling, are invalidated by the bloating of the packaging.

| Leak rate (mbar·l/s) | Leak rate (sccm) | Bubble rate under ideal conditions |
|---|---|---|
| $10^{0}$ | 100 | 1000 x bubbles per second |
| $10^{-1}$ | 10 | 100 x bubbles per second |
| $10^{-2}$ | 1 | 10 x bubbles per second |
| $10^{-3}$ | 0.1 | 1 x bubbles per second |

Table 1: Correlation between different leak rates and the respective bubble rate in the water bath. The leak size determines the time escaping gas requires to form a bubble with a diameter of 3 mm. The test operator might not always recognize it as such.

On closer inspection, other pitfalls are revealed. One of the biggest problems, food manufacturers are subsequently unable to sell the tested products and are forced to discard them. Another limiting aspect is the tester him- or herself – the human factor. Whether bubbles are detected at all is always dependent on the individual tester and subject to his/her subjective assessment. Bubbles could, for example, get caught in the fold or overlap. Therefore, due to human error defective products may be put into circulation. Finally, the different types of leaks are of equal importance, especially leaks often found in the food industry that cannot be reliably identified in the water bath, due to their leak rate. Leaks caused by insufficient welding seams are often not large enough to be easily recognizable in the water bath. On the other hand, gross leaks cannot be detected either. When submerged in water, the packaging fills completely with water, leaving no air to escape during the testing process. In addition, there is the inevitable contamination process. If, for example, test material enters the water, the water will be contaminated. But even if this does not happen, the water in the test tank will become cloudy after four to eight weeks at the latest, and must be replaced. This usually generates additional costs. In order to promote the formation of bubbles, chemicals intended to reduce the surface tension of the water are often added to the water. The content of the tank must then be disposed of as hazardous waste, which in turn generates more costs.

## 2.2 Gas-based Testing Methods

Leak detection with tracer gas is based on a pressure difference being generated between the interior and the exterior of a test object, such that the tracer gas - in the food industry mostly $CO_2$ or helium - is able to flow out of the package through any existing leak, generating a measurably changed partial pressure in the chamber. Carbon dioxide at different concentrations is a component of most protective gases. Its use as a tracer gas in leak detection is recommended, since no other gas needs to be added. However, this process has severe limitations. A food manufacturer can only test products featuring a specifically required concentration of $CO_2$. Depending on the device being used, these minimum values range between 10 and 20 percent. For the testing of flexible packages, like bags of chips, with the $CO_2$ method, some restrictions apply. Since leak detection is carried out in a rigid chamber, there is the risk that the packaging will be damaged by the large pressure difference that is required, like 1000 mbar in the packaging and 30 mbar in the test chamber, for example. As a result, the bag will burst. If, on the other hand, the difference is too low - for example, 1000 mbar in the packaging versus 800 mbar in the test chamber - fine leaks (microleaks) will not be detected. As a rule, the detectable leak rate using a $CO_2$-based test ranges between $10^{-1}$ and $10^{-2}$ mbar l/s. As with the water bath described above, there is also the risk for very large leaks (gross leaks) that $CO_2$ will escape completely from the packaging while the test chamber is being evacuated. During the actual testing procedure, the pressure in the chamber will not change and the product will be incorrectly designated as properly sealed.

As with any $CO_2$-based leak detection, testers should closely monitor the $CO_2$ content in the environment. In normal air the $CO_2$ content is about 0.04 percent. Human breathing air, on the other hand, contains a concentration of up to four percent and may falsify test results if the tester is standing too close to the device. Prior tests as well may enrich the ambient air with carbon dioxide, affecting the reliability of measuring instruments. The measurement of the residual oxygen in the head space of a package must be strongly differentiated from the leak detection. This measurement checks the correct composition of the protective atmosphere (Modified Atmosphere Packaging, MAP).

## 2.3 Differential Pressure Method

Another option for leak testing food packages is the differential pressure method, where the device determines the pressure difference between the product to be tested and a reference object. For this purpose, the tester places the product in a test chamber which is subsequently evacuated. If gas escapes through a leak, the pressure in the test chamber changes and the difference can be detected in the form of a leak rate.

## 2.4 Pressure Increase Method in Vacuum Foil Chamber

The pressure increase method in the foil chamber is one of the most accurate test methods for the food industry. The tester places the packages between the elastic membranes in the foil chamber. The evacuation of the foil chamber creates a pressure drop between the test object and the foil chamber. Gas then flows through any existing leaks from the packaging into the chamber where it increases the pressure (Figures 5 and 6). In contrast to the above-described methods, there is no risk of bursting since the elastic membranes attach themselves completely to the test object and absorb any mechanical stress at the sealing seams.

The device calculates the respective leak rate based on the detectable pressure increase. The pressure increase method therefore does not require any additional tracer gas and is completely non-destructive. Of special interest to the food industry is the ability to detect leaks that are smaller than 10 µm. The device should furthermore allow short test cycles, reliable reproducibility of the results, and fast cycle times.

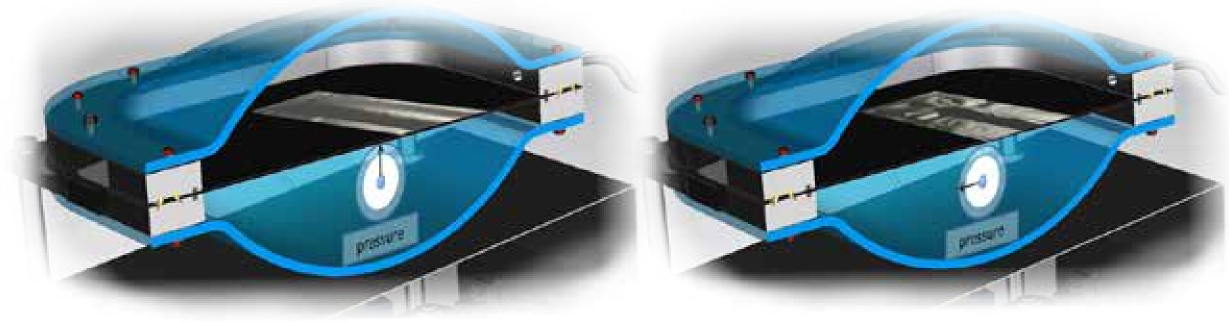



Figure 5: Schematic representation of the Contura S400 Leak Detector.

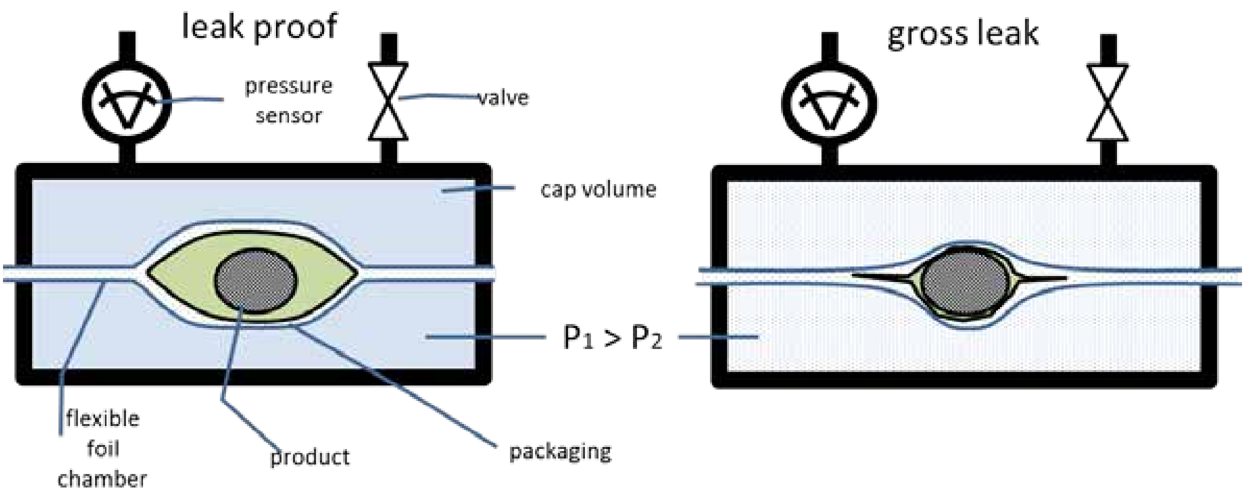



Figure 6: Operating principle of the pressure increase method in the foil chamber.

## 2.4.1 Gross Leak Detection

A leak detector for the pressure increase method should also be able to detect both gross and microleaks in small gas compartments. The detection of very large leaks - the "gross leaks" caused by gross damage to the packaging - is a problem for many leak detection methods. In case of a gross leak caused, for example, by torn packaging or a non-welded sealed seam, the testing device, through pumping action, completely evacuates the gas contained in the packaging. After this evacuation, there is no pressure difference. As a result, a test object with a gross leak is incorrectly identified as sealed. Packaging with a very small head space exhibits a similar problem. Using a patented process, the foil chamber principle can be enhanced to include gross leak detection. The test operator enters the volume of the packaging to be tested into the testing device. The device detects the test object's volume after the evacuation and is able to identify packages with gross leaks.

# Part 2
# Challenges of Leak Detection in the Food Industry

The food sector is subject to strict national and international guidelines. After all, unsafe (e.g., spoiled) foods represent potential health risks to consumers, which is why food producers must guarantee the safety of their products. The HACCP (Hazard Analysis and Critical Control Points) principle has established itself as the basis for laws that regulate the production and distribution of food items. The included standards are designed to enable food producers to identify and control potential hazards and, based on this information, to produce safe foods. The intention is to ensure that the products are safe for the consumer during manufacture, transport and storage, right up to the actual sale and consumption. In Germany, nearly all food items must additionally be labeled with a minimum shelf life ('best before') date or a consumption ('use before') date.  These dates indicate the period in which consumers can consume a food item when properly stored, without substantial loss of taste and quality, and without any health risk.

Packaging is an important factor in compliance with these guidelines. They not only provide stability, but also protect the food from the impact of light and microorganisms as well as from foreign odors, steam, carbon dioxide and undesired flavors, as shown in Fig. 7.

In order to also extend the shelf life of food items without the addition of artificial preservatives, Modified Atmosphere Packaging (MAP) has for several decades become one of the prevailing packaging methods. The principle of this method is a modification of the natural air atmosphere (78 percent nitrogen, 20.96 percent oxygen, 1 percent argon and 0.04 percent carbon dioxide) to extend the shelf life of products. The effects of different gases have already been used since the 1970s to inhibit the chemical, microbial and enzymatic spoilage processes. Since oxygen is the basis for damaging oxidation processes, it is usually displaced - often by a protective gas, which extends the shelf life date even further. For example, $CO_2$ - thanks to its bacteriostatic effect at a concentration of 15 to 20 percent - inhibits the growth of aerobic bacteria and mold.  However, the ability to ensure the necessary specific balance of the gas mixtures over the entire period of the minimum shelf life requires leak-proof packaging. The specific requirements for such packaging will be explained in the next chapter.

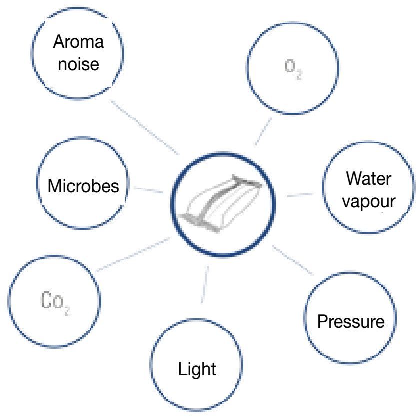



Figure 7: Sealed packages protect food from numerous undesirable external effects.

## 1. Dry Products

Dry products such as nuts, dried fruits, chips or other spicy baked goods products are usually compromised by two processes: oxidation by oxygen and ingress of moisture. Oxidation causes fats and oils to become rancid and products to spoil more quickly. Therefore, the packaging is most often flooded with pure nitrogen in order to replace the present oxygen up to a maximum concentration of two percent. Moisture is another factor affecting dry foods. When moisture permeates the packaging of chips or other snack foods, they lose their crispness and the overall quality of the product decreases. Fig. 8 shows how quickly water vapor is able to enter leaky food packages. Dried fruits are also at risk, since they are prone to mold if too much moisture penetrates the packaging. Thorough leak testing of the packaging is therefore essential - not only to ensure the quality of the product's taste, but also to assure the consumer's health is not at risk.

### Oxidation: The Impact of Oxygen on Food Products

There are two types of oxidation: auto-oxidation, a chain reaction independent of light, and photo-oxidation via sensitizers triggered by light. Auto-oxidation occurs primarily in unsaturated fatty acids and leads to the "rancidity" of fatty foods. In the second type of oxidation, incoming light activates trigger substances like riboflavin, which in turn induces oxygen to start oxidation.

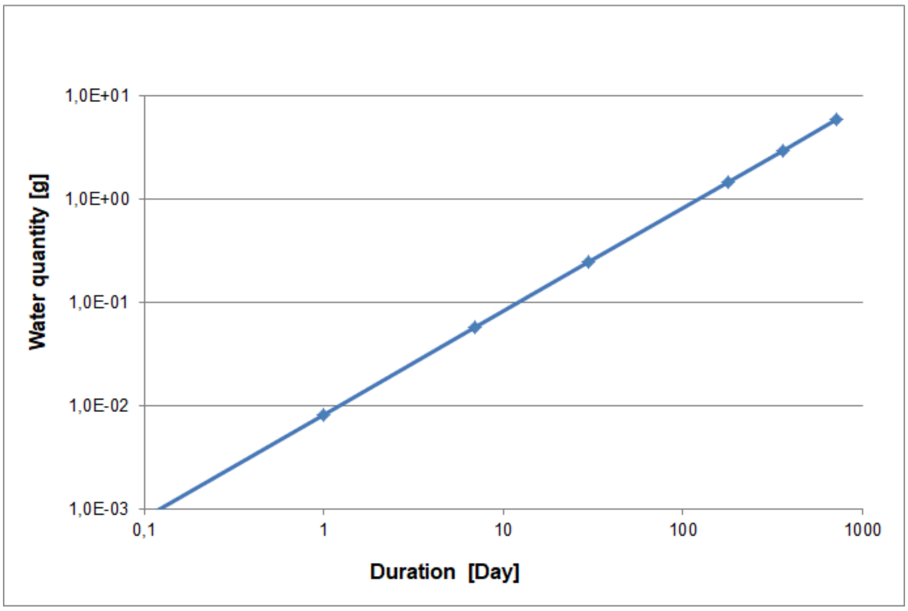



Figure 8: Water vapor entering food packaging in days at a leak rate of $5x10^{-3}$ mbar·l/s and 70 percent relative humidity.

## 1.1 Coffee

Since coffee is one of the most popular foods, the corresponding market is highly competitive. Due to their major impact on the flavor and taste experience, the correct packaging and storage of coffee beans and ground coffee represent a non-negligible competitive advantage for coffee distributors because coffee is particularly sensitive to external influences. After two weeks of storage at room temperature, roasted coffee shows a significant loss of freshness and flavor. Only a few minutes after its first contact with oxygen, ground coffee starts to oxidize and begins to lose a considerable amount of flavor. The entry of water vapor is a risk as well, which, however, can be mitigated by safe and tight packaging. To slow down the molding process as well as the oxidation in the coffee, a mixture of nitrogen and $CO_2$ is frequently injected into the packaging. A tight water barrier is also essential.

## 1.2 Potato Chips

Unsaturated fatty acids are particularly susceptible to oxidation with oxygen. The fatty acids degrade into reaction products that often smell and taste unappealingly, which in the case of potato chips is often concealed by strong seasonings. However, for health reasons such products, are no longer suitable for consumption, as the fat has turned rancid. Since unsaturated fatty acids are an important component of chips products, they must be packaged under a special oxygen-suppressing protective gas to prevent oxidation - thereby ensuring both the health of the consumers and their taste experience. The increase in oxygen concentration caused by various leak rates is shown in Fig. 9.

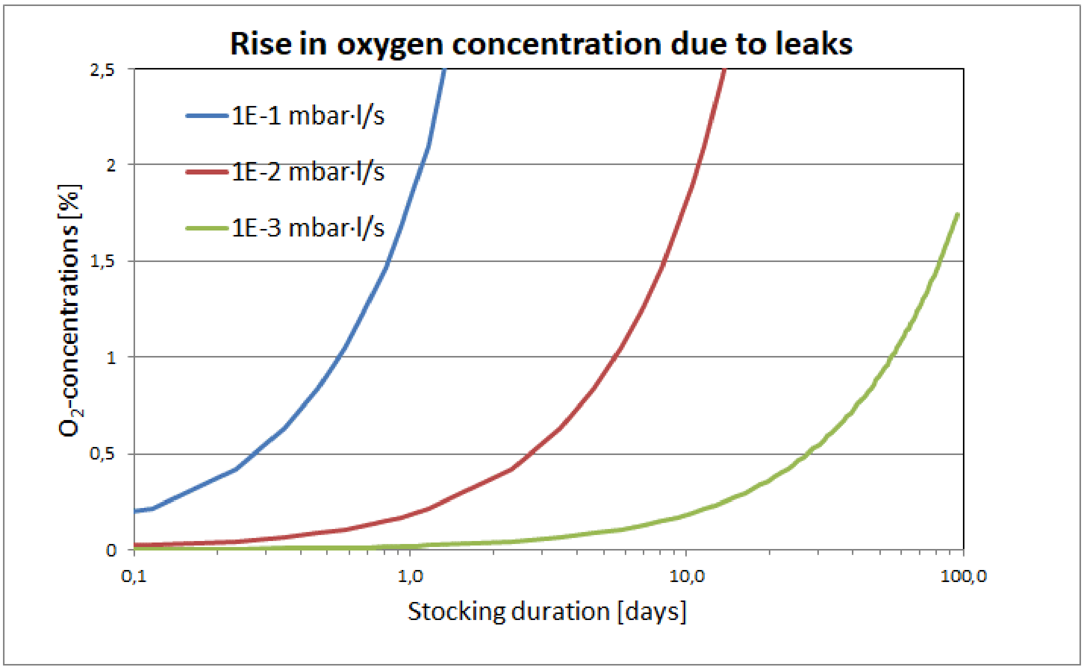



Figure 9: Starting point: Residual oxygen concentration of 0.5 percent in a package. Representation of the increase of oxygen as a function of the storage time and the level of the leak rate.

From the Field

INFICON has already worked with numerous food producers to optimize their packaging processes and ensure leak-proof packaging. For example, in this book we present some field-tested application scenarios for INFICON's Contura S400 Leak Detector.

A manufacturer of snack products notes irregularities during leak tests in their production. To ensure that all their packaging is leak-proof, the tester once again wants to check a conspicuous batch of products in tubular bags individually for leaks. For these tests, the tester needs a device which performs the test quickly, reliably and, above all, in a non-destructive manner. With the Contura S400, the tester can process such leak tests in a time-saving manner. The employee can place the device directly next to the production line and carry out the test there. Since the Contura S400 is designed to be especially user-friendly, this requires only minimal effort because the device does not require any calibration. The tester places the products, individually or several, between the two diaphragms. When the cover is closed, the test process starts automatically. The results of the test are then read by the tester in the form of the exact leak rate on the display, or by watching for the color and audio tone signal. Even in the noisy and distracting environment of production, he/she is able to carry out the test reliably. Last but not least, Contura S400 documents all test results, which the manufacturer can read out via a USB connection and subsequently archive in their quality management system.

## 2. Semi-Dry Products

Semi-dry products such as baked goods, frozen pizzas, fresh pasta or cheeses are mostly compromised by the rapid growth of microorganisms, as shown in Table 2.

| Pore Size | Microbial growth |
|---|---|
| 20 µm<br>15 µm | Growth in approx. in 5 days<br>Growth in approx. in 14 days |

Table 2: Correlation between pore sizes and microbial growth. Source: Vishwas Pethe, Alex Terentiev, Mike Dove: Integrity Testing of Flexible Containers. In: BioPharm International, 24 (2011), H. 11.

These microorganisms multiply best in an oxygen-containing atmosphere and spoil the product. A high oxygen content must therefore be avoided at all times, because among other things, it also leads to an accelerated vitamin breakdown. Meat - as well as cheese - however are oxygen-depleting foods and already deprive the atmosphere of a certain amount of oxygen. This should be considered in regard to the concentration of the protective gas. For bakery products, on the other hand, there is a risk of mold due to airborne spores; a reason for manufacturers to pay attention to a controlled atmosphere. This also always means a thorough leak test of the packaging. Moisture as well is problematic for semi-dry foods and should not be underestimated. A high level of moisture not only drives mold, but also makes baked goods simply unappetizing. For this reason, manufacturers often use a protective gas made of a combination of nitrogen and carbon dioxide. The carbon dioxide does not only act to displace the oxygen, but when it reacts with water, carbonic acid is formed in the packaging, which lowers the pH and has an antimicrobial effect. The water vapor permeability of a package is therefore a key factor in the packaging of baked goods. It specifies the amount of water vapor permeating one square meter of foil surface per day at a defined drop in atmospheric humidity and a constant temperature.

### From the Field

A producer of baked goods discovers repeatedly that his products packaged under protective gas contain too much oxygen and are spoiling quicker. While checking his packaging for leaks, he notes that they exhibit a high leak rate. In order to prevent costly and damaging complaints, the manufacturer decides to thoroughly examine his packaging plant. He is aware that leaky packages can be caused by a variety of reasons – the wrong foil, worn parts on the machine, or to product residues in the weld which can create capillaries. There is, however, also the chance that the settings on the machine need to be readjusted, for example the pressure, the temperature, or the overlapping of the weld seams. In order to determine the actual cause, the tester wants to check various parameters. If he uses a modern pressure increase tester with a foil chamber, like the Contura S400, the device will be able to provide the precise actual value of the measured leak rate after each adjustment quickly and reproducibly. The tester could, for example, test a new foil to see if the leak rate improves. Or he might exchange the welding jaws of his packaging machine to make sure they are not worn. Using the exclusion principle, he can quickly find the fault thanks to the incoming leak rate detection, rectify the error and subsequently achieve optimum tightness again. In order to avoid such complex tests in the future, the producer could also use the Contura S400 to establish a process of continuous improvement in their production.

## 3. Moist Products

Moist products such as fresh meat, ready-to-eat foods, extracted or fermented dairy products, fresh fish and poultry are particularly susceptible to the rapid growth of microorganisms. The main objective during production and storage is therefore the prevention of residual oxygen and the ingress of additional oxygen – usually through the use of a MAP protective gas atmosphere. Different gas mixtures are used to meet this objective. Hard cheese, which thanks to MAP can be stored for up to 10 weeks, for example, is packaged under an atmosphere consisting of 80 percent to 100 percent $CO_2$ and 0 to 20 percent $N_2$. The shelf life of yogurt of up to 25 days is guaranteed by a composition of zero to 30 percent $CO_2$ and 70 percent to 100 percent nitrogen. A combination of carbon dioxide and nitrogen avoids not only the formation of mold, but also oxidation.

### 3.1 Fresh Meat and Meat Products

Fresh meat products and sausages are highly sensitive to oxidation and infestation by microorganisms, placing special demands on the packaging. Since customers regard only meat with a light red surface as truly fresh and purchase it accordingly, proper protective gas combination is of utmost importance. A special case is the so-called "High-Ox Mix," a gas mixture consisting to a large extent (up to 80 percent) of oxygen. This causes the meat to retain its rosy color for a long time. At the same time, however, oxygen is known to make meat tough and the contained fat rancid, drawing criticism of its use.

## From the Field

Due to the highly perishable nature of his products, a meat producer attaches great importance to leak-proof packaging. He has furthermore determined that in the future, the market will move towards 100 percent audit testing. This means all units will be individually tested for leakage rather than individual samples only. That's why the meat producer is looking for a solution that can be easily integrated into production and which will transparently test all products. His production includes multiple lines for fillets, chops and ham, but he does not want to invest in multiple devices. Therefore, he opts for a modern pressure increase tester with a foil chamber - in our case, the Contura S400. Immediately, he is impressed with its easy operation. Furthermore, the testing device allows the documentation of the leak rates from various production lines. An employee scans the product via the bar code scanner, and the device reads out the stored settings. The employee then tests the product for leaks in the flexible foil chamber. He can easily read the detected leak rate on the display. The system automatically assigns the results to the different product lines. This data can then be read via the USB port and transferred to the quality management system.

# Conclusion

Sophisticated testing of food packaging requires a device that meets the highest standards, offers flexible use and delivers reliable results. The Contura S400, which INFICON has developed especially for flexible and MAP packaging, is based on the pressure increase method in a flexible foil chamber allowing it to detect gross as well as fine leaks. This leak detector operates without a tracer gas and completely non-destructively. This allows the Contura S400 to be easily integrated into any quality assurance process and enables continuous improvement of the manufacturing process.

If you would like to learn more about INFICON's innovative solution for leak detection in the food industry, please contact us. We look forward to helping you with your individual requirements.

Further information on the Contura S400 is available at:
http://www.inficonpackaging.com/de-de/verpackungde.aspx

or as a video:
https://www.youtube.com/watch?v=eKZkNEI0bSE

# Appendix

## Related Literature

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# About INFICON

INFICON GmbH in Cologne, Germany is one of the world's leading developers, producers and suppliers of instruments and devices for leak detection. The leak detectors are used in the production and quality control of demanding industrial processes and cover a wide range of applications. INFICON'S primary customers include manufacturers and service companies for air-conditioning and refrigeration equipment, the automotive and automotive supply industry, the semiconductor industry and manufacturers of leak detection systems. With its many years of experience in leak testing and leak detection, INFICON now also wants to support the food industry and has therefore developed the patented Contura S400 leak detector.


INFICON has more than 50 years of experience in leak detection technology. INFICON processes worldwide sales through production facilities in Cologne (Germany), Balzers (Liechtenstein), Linköping (Sweden), Syracuse (USA), and Shanghai (China), as well as sales offices in all major industrialized countries and an extended network of sales partners. In fiscal 2016, INFICON AG with its approx. 1000 employees, generated worldwide sales of approx. US$310 million. The registered shares of INFICON (IFCN) are traded at the SIX Swiss Exchange.

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