The Dangerous Goods Regulations currently do not include limit leakage rates or sensitivity requirements for industrial leak testing procedures that are equivalent to the bubble test, which is the prescribed test method for design type testing of dangerous goods packagings. During series production of such packagings, various methods are used, which often do not meet the requirements of the bubble test with regard to important criteria.

Sensitivity, flow direction, pressure level and automatability are particularly important factors when selecting a suitable industrial leak testing method.

The following methods are in principle both suitable and equally effective as the bubble test: pressure rise test (vacuum chamber), ultrasonic bubble leak detection and gas detection methods (pressure technique by accumulation and vacuum chamber technique).

To ensure a uniform test level during design type testing and production line leak testing and therefore a comparable safety level as required by the Dangerous Goods Regulations, it is necessary to include a more precise specification in these regulations. This requires, on the one hand, information about the sensitivity of the bubble test and, on the other hand, the inclusion of a list of suitable, equally effective industrial test methods with their specific boundary conditions.

**KEYWORDS**

bubble test, dangerous goods packagings, industrial leak testing, leakproofness test, series production

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1 | INTRODUCTION

According to the international United Nations (UN) Model Regulations—Recommendations on the Transport of Dangerous Goods, every packaging intended to contain liquids shall successfully undergo a suitable leakproofness test during its industrial series production before it is first used for carriage (UN 6.1.1.3). The requirement for the test method used is that it shows the capability of meeting the appropriate test level of the immersion test in water, the so-called ‘bubble test’, the standard leakproofness test method for the design type test of dangerous goods packagings (UN 6.1.5.4). The bubble test, when carried out as design type test, is a classic example of a laboratory test. However, it is impractical as an industrial test, except for production lines with very small manufacturing volumes. A basic criterion in selecting a suitable leak test procedure is its sensitivity.

The problem is that the UN Regulations do not clearly specify the sensitivity of the bubble test, but this would be the basis for choosing an industrial leak testing method. There is neither a specification of...
permissible limit leakage rates\(^4\) nor guidance about suitable industrial test methods, which must be at least equally effective. During production (piece-by-piece testing), various leak testing methods are used whose sensitivities differ considerably. As a result, an unequal safety level and distortions of competition are possible.\(^5\)

For the bubble test as defined in UN 6.1.5.4, the test sample is exposed to an internal air pressure (gauge) of 30 or 20 kPa, depending on the degree of danger, and is restrained under water for 5 min. The result is a simple fail/pass statement depending on whether bubbles rise or not (qualitative approach).\(^6,7\)

The minimum detectable leak diameter \(d_{\text{min}}\) of the bubble test under the test conditions stated in the UN Model Regulations is approximately 9.7 μm for 30-kPa gauge pressure and approximately 14.6 μm for 20-kPa gauge pressure.\(^7,8\) But this diameter is not a suitable measure for a quantitative comparison with other leak testing methods.\(^7\)

The first question in this work is what test level can be assigned to the bubble test as a specification for the sensitivity of an industrial leakproofness test.

The following approaches are compared below to assess the sensitivity \(q_{\text{min}}\) of the bubble test:

- Specification from leak testing standards \((q_{\text{min,stand}})\)
- Calculation of the minimum detectable leakage rate under the physical flow conditions of the bubble test \(q_{\text{min,UN,flow}}\) based on the test duration prescribed by the UN Model Regulations.
- Calculation of the minimum detectable leakage rate for a laminar viscous capillary flow \(q_{\text{min,UN, cap}}\) based on the minimum detectable leak diameter \(d_{\text{min}}\) of the bubble test under the test conditions prescribed by the UN Model Regulations.

The second main question is which industrial leak test method is equivalent in terms of sensitivity and other important parameters. An overview of common leak testing methods used in industrial series production is given. These procedures are compared and evaluated for their suitability for the series production of dangerous goods packagings.

The aim of this article is to establish a systematic way in choosing suitable and equally effective industrial leak testing methods for dangerous goods packagings. The results could serve as a basis for adding a list of such test methods into the UN Model Regulations.

2 | THEORY

2.1 | Leak test sensitivity

Absolute leakproofness does not exist.\(^9,10\) Instead, leakproofness is a relative term.\(^11,12\) One definition is the following: The test object is considered to be leakproof if the test method selected and its corresponding detection sensitivity cannot prove the passage of the test medium from one side to the other or to the outside.\(^13\)

The sensitivity of a leak testing procedure (detection limit) is the minimum detectable leakage rate \(q_{\text{min}}\); the test procedure is capable of detecting.\(^6,7,12\) The definition of the leakage rate is the pV-throughput of a specific fluid that flows through a leak under specified conditions.\(^5,14,15\) The lower the numerical value of \(q_{\text{min}}\), the higher the sensitivity. For a proper definition of a leakage rate, it is also important to state the prevailing test conditions.\(^12\)

One possibility is to specify the leakage rate with reference to the driving force: the pressure differential. The leakage rates given in ISO 12807 are standardised leakage rates (SLRs),\(^6\) normalised to the flow of dry air under the reference conditions of upstream pressure 101.3 kPa, downstream pressure 0 kPa, and a temperature of 298 K (25°C).

Another possibility is to express the leakage rate as released volumetric flow into the surrounding atmosphere at standard ambient pressure 101.325 kPa.\(^16\) The European standard DIN EN 1593 uses this type of indication to quantify the leakage rate of the bubble test on the basis of bubble diameter and bubble frequency.\(^17\) This type of leakage rate information only considers the amount of gas released, regardless of the exact level of upstream and downstream pressure.

2.2 | General criteria for selecting a suitable leak test method

When choosing a suitable leak test method for the respective application, various aspects must be taken into consideration in advance. These criteria can be divided into the following areas.\(^18–20\):

- Sensitivity of the test method: The permissible limit leakage rate of the test object determines the selection of the method.\(^14\) The sensitivity and the measuring range are the most important parameters for choosing a test procedure.\(^3\) The requirement for a test method is usually a sensitivity that is higher by a factor of 10 than the limit leakage rate to be detected.\(^21,22\)
- Objective and extent of the investigation: In general, two types of leak test methods can be distinguished: methods for leakage rate measurement (quantitative methods) and those for leak localisation (qualitative methods).\(^6,7,14,23\) Therefore, the objective can be a leakage rate measurement or a leak localisation. The range can be the total area of the test object or a single local area.\(^14,23\)
- Operation conditions and testing conditions: Test media are liquid or gaseous substances that must be specifically detectable after they have passed through a leak.\(^13\) Generally, a test fluid other than the operational fluid is used for the leak test to ensure better handling and higher sensitivity.\(^14\) Relevant parameters are also the test pressure conditions (upstream pressure, downstream pressure and direction of flow) and the testing temperature.\(^14,23\) For simplicity, the leak test is usually carried out at ambient temperature. The test pressure should be in the order of the operating pressure, and the flow direction should be the same as under operating conditions.\(^14,24\)
• **Test object design:** The dimensions of the test object, openings for test gas supply and limit values concerning pressure and vacuum also have an influence on choosing a test procedure. The test medium must be compatible with the object material.14

• **Safety and environmental requirements:** Many test methods involve the application of a pressure differential, either an over-pressure or a vacuum. This must not endanger the test personnel or the system. Some test gases are either toxic or harmful to the environment. Consequently, measures must be taken so that they cannot escape.14

If a leak test method is not to be used in the laboratory, but in industrial series production, there are further requirements. These are listed in the next section.

### 2.3 Special requirements for industrial leak test methods

Important requirements in the selection of a leak test method for series production are the ability for automation, the reliability of the method and the available economic opportunities (acquisition costs and operating costs).21,24,25 A central point is the compliance to a given production cycle.26 By deducting the handling time, the total test time, that is, the time available for the leak test, results from the production cycle time.27

Because of these additional requirements, sensitivity of one and the same leak test method is often lower under industrial conditions than under ideal laboratory conditions.24

### 2.4 Resulting requirements for a leak test method for industrial series production of dangerous goods packagings

From the requirements listed above, the following criteria are of particular importance for the industrial leak testing of dangerous goods packagings:

• **Sensitivity of the test method:** Limit leakage rates for dangerous goods packagings do not currently exist in the UN Model Regulations.4 The question is fundamental of how sensitive a method equivalent to the bubble test should be. Therefore, in Section 3, different approaches on how to assess the sensitivity of the bubble test, which is a requirement for an industrial process, are presented.

• **Objective and extent of the investigation:** Because the bubble test is performed by completely submerging the packaging, the industrial method must also allow a leak test on the total area of the packaging. However, the original closures may be removed (UN 6.1.1.3).1 The bubble test is primarily used as a method for leak localisation, even though a quantitative evaluation is possible by bubble counting or measuring of the escaped gas quantity.6,14,17,23 To select an equivalent method, a quantitative analysis of the bubble test is essential. Quantitative methods have an advantage over qualitative methods because they are more objective.

• **Operation conditions and testing conditions:** Depending on the degree of danger, either 20 or 30 kPa is specified as test over-pressure in the bubble test. Even with an alternative method, this pressure level should be selected, otherwise irreversible deformation of the test sample may result. The flow direction in the bubble test corresponds to the direction of flow, which is also present during the release of a dangerous substance under transport conditions, namely, from the inside to the outside. An equivalent industrial process should also ensure this flow direction.

• **Suitability for automated series production:** In this work, the automatability and the production cycle time are considered in particular. Because the focus is on the technical suitability of the individual procedures here, cost aspects are not considered.

Safety and environmental requirements are also not considered in the following. Of course, individual specifications of the industrial test method to be selected can only be made with reference to the specific application with its specific boundary conditions in the production line.

The question of how sensitive a method, equivalent to the bubble test, should be is fundamental. Therefore, in Section 3, different approaches on how to assess the sensitivity of the bubble test, which is a requirement for an industrial process, are presented.

### 3 REQUIRED SENSITIVITY QMIN OF THE INDUSTRIAL LEAK TEST METHOD

#### 3.1 Specification from leak testing standards (qmin, std)

Although the bubble test is classified as a method for leak localisation, information about its sensitivity is provided in various leak testing standards.6,14 ISO 12807 states the nominal sensitivity of gas bubble techniques under industrial conditions as 10⁻⁴ Pa m³/s (SLR).6 Higher sensitivities could be achieved using other test fluids than water that have lower surface tension.6,12 An equivalent value of 10⁻⁴ Pa m³/s for the sensitivity of the bubble test under industrial conditions is also mentioned in DIN EN 1779 for comparable flow conditions.6,23 The value qmin, std, SLR represents the sensitivity under the practical aspects of industrial leak testing.
Leakage rates can be converted from one condition to another assuming a constant leak geometry and a specific flow regime.\textsuperscript{6} For a given leak and one and the same type of gas, the relationship between the leakage rates at two different pressure levels is\textsuperscript{14}

\[ q_{\text{II}} = q_{1} \left( \frac{p_{1,\text{II}}^{2} - p_{2,\text{II}}^{2}}{p_{1,\text{I}}^{2} - p_{2,\text{I}}^{2}} \right) \]  \hspace{1cm} (1)

A conversion of the air leakage rate $q_{1} = 10^{-6}$ Pa m\textsuperscript{3}/s under SLR conditions (upstream pressure $p_{1,\text{I}} = 101.3$ kPa, downstream pressure $p_{2,\text{I}} = 0$ kPa) to a minimum leakage rate that results at the reference pressure level of the bubble test (upstream pressure $p_{1,\text{II}} = 121.3$ kPa, downstream pressure $p_{2,\text{II}} = 101.3$ kPa) under the assumption of laminar viscous flow leads to a value $q_{1}$ of approx. 5 $\times$ $10^{-5}$ Pa m\textsuperscript{3}/s ($q_{\text{min, UN}}$, ref). This information is also required for a subsequent comparison with suitable methods, because their sensitivity is often given on the basis of the flow of air at ambient pressure only.

### 3.2 Calculation of $q_{\text{min, UN}}$, flow

The leakage rate $q_{\text{flow}}$ under the physical flow conditions of the bubble test can be calculated by Equation 2.\textsuperscript{7,8,28}

\[ q_{\text{flow}} = \frac{p_{b}}{t} \frac{V_{b,\text{tot}}}{t} \]  \hspace{1cm} (2)

In this equation, $p_{b}$ is the effective absolute pressure inside the bubbles, and $V_{b,\text{tot}}$ is the total bubble volume and $t$ is the test time.

To evaluate the minimum detectable leakage rate $q_{\text{min, UN, flow}}$ under the test conditions of the UN Model Regulations, an extreme case is considered for the application of Equation 2. It is assumed that the first and only bubble exits the test sample exactly at the end of the test period of 5 min and is detected by the observer. The volume $V_{b,\text{tot}}$ therefore corresponds to the volume of this single bubble $V_{b}$. The smaller the diameter and thus the volume of this first single bubble, the better is—theoretically—the sensitivity of the bubble test. The detectable bubble size depends on various parameters, for example, the light conditions, the degree of contamination of the water and the attention of the test personnel.\textsuperscript{29} Under optimal circumstances, it is possible to detect a bubble of 1-mm diameter, which has a bubble volume $V_{b}$ of approximately 0.5 mm\textsuperscript{3}.\textsuperscript{8} This corresponds to the smallest specified bubble diameter for the example calculations in DIN EN 1593.\textsuperscript{17}

In addition, the following simplification is made: It is assumed that this bubble exits in the upper part of the test sample directly below the water surface. The hydrostatic head of fluid, as well as the surface tension restraint, can be considered insignificant.\textsuperscript{28,30} The bubble pressure $p_{b}$ is therefore equal to the atmospheric air pressure (101.3 kPa).

Under these extreme conditions, Equation 2 gives a value of $1.7 \times 10^{-7}$ Pa m\textsuperscript{3}/s for the minimum detectable leakage rate $q_{\text{min, UN, flow}}$. This value can be considered as theoretical maximum sensitivity of the UN bubble test when using Equation 2. Because of the assumed situation, however, this value is unrealistically low regarding the practical performance of the bubble test.

In this case, the leakage rate refers to the amount of air released from the component to the surrounding atmosphere.\textsuperscript{16}

This value for the sensitivity of the bubble test in the order of $10^{-7}$ Pa m\textsuperscript{3}/s is not feasible under industrial conditions from an economic and practical point of view.\textsuperscript{8,23} Therefore, the sensitivity value obtained in this way differs from the value of $q_{\text{min, UN}}$ obtained in Section 3.1 for industrial conditions in several orders of magnitude.

### 3.3 Calculation of $q_{\text{min, UN, cap}}$

The basic steps of this calculation are described in previous studies.\textsuperscript{7,8} The approach is to calculate the leakage rate of a reference gas that flows under defined conditions through a capillary. It is assumed that this capillary has a length $L$ corresponding to the wall thickness of the packaging type concerned and a diameter corresponding to the smallest detectable bubble diameter of the bubble test of the UN Model Regulations. This value for $d_{\text{min}}$, therefore represents the physical limit of the bubble test carried out under laboratory conditions. A laminar viscous flow regime is assumed. For this approach, representative packaging design types with their characteristic volumes (6, 60 and 216 L) and wall thicknesses $L$ (see Schlick-Hasper et al.)\textsuperscript{7} have to be taken into account. In this work, two different sensitivities of the bubble test are determined according to this method:

- **Sensitivity $q_{\text{min, UN, cap, He}}$** under normalised helium test conditions: 100% helium, temperature: 20°C, $\eta = 19.6$ $\mu$Pa s, upstream pressure $p_{1} = 131.3$ kPa (absolute), downstream pressure $p_{2} = 101.3$ kPa (absolute). The results are taken from Schlick-Hasper et al.\textsuperscript{7}
- **Sensitivity $q_{\text{min, UN, cap, air}}$** under the standard conditions of ISO 12807 (SLR of dry air at reference conditions, as given in Section 2.1). The dynamic viscosity of dry air under these conditions is 18.5 $\mu$Pa s.\textsuperscript{6}

Table 1 lists the results. In this work, only the results for packaging group I (30 kPa overpressure) are considered, as they lead to more severe results for the smallest detectable leakage rate. The sensitivity requirements for an equivalent method are thus stricter than for packagings for substances of packing groups II and III.

This calculation method shows that the bubble test theoretically leads to different sensitivities for packages of different sizes and materials and thus different wall thicknesses. The bubble test derived in this way has a higher sensitivity for packagings of higher wall thickness than for those of smaller wall thickness.\textsuperscript{7} For an industrial test method equivalent to the bubble test, this would mean formally that for different types of packagings, industrial leak tests with different sensitivities would have to be required.
The leakage rates obtained in this way are of the order of $10^{-4}$ to $10^{-5}$ Pa m$^3$/s and thus are between the values obtained in 3.1 and 3.2.

### 3.4 | Comparison of the different approaches for $q_{\text{min}}$

Table 2 presents the results of the three approaches.

The comparison shows that, depending on the boundary conditions, that is, the test medium and the flow conditions, the sensitivity of the bubble test varies by up to three orders of magnitude. Thus, the requirements for an industrial leak test method for dangerous goods packagings are fundamentally different, depending on the underlying approach. Therefore, if the UN Model Regulations require a test procedure equivalent to the bubble test, it should be clarified for which conditions this applies.

UN 6.1.1.3 specifies the level of the bubble test according to UN 6.1.5.4 as a reference for an industrial leak test. As mentioned in the introduction, the design type leakproofness test is a laboratory test. This means that the sensitivity of the bubble test under laboratory conditions must be chosen as basis for assessment.

Therefore, the values $q_{\text{min,SLR}}$ and $q_{\text{min,ref}}$ are not applicable, because the standards listed in Section 3.1 refer to the sensitivity of the bubble test under industrial conditions. The value for $q_{\text{min,flow}}$ results from the application of Equation 2 for an extreme case and is therefore unrealistically low (see Section 3.2).

As a consequence, the values obtained in Section 3.3 are the most realistic for assessing the sensitivity of the bubble test according to the UN Model Regulations. The SLRs $q_{\text{min,SLR}}$ are the most suitable as a reference. In the course of a conservative estimate, the smallest of these calculated values is chosen as the basis ($1.2\times10^{-5}$ Pa m$^3$/s, see Tables 1 and 2). It follows that the required sensitivity $q_{\text{min}}$ of an industrial test method must be in the order of $1.0\times10^{-5}$ Pa m$^3$/s (SLR). This value is used as the basis for the assessment of the industrial test methods in Section 5.

### 4 | RECOMMENDED METHODS FOR INDUSTRIAL LEAK TESTING OF DANGEROUS GOODS PACKAGINGS

The most commonly used leak testing methods are pressure decay test, pressure rise test, gas leak detection and bubble techniques.\(^5\)\(^,\)\(^10\)\(^,\)\(^26\)\(^,\)\(^31\) The pressure decay test used to be the classical and most common method for mass production, but leak testing with tracer gases has become increasingly important.\(^10\)

The UN Model Regulations do not provide information on suitable industrial leak test methods.\(^5\) Suitable leak test methods for industrial series production of dangerous goods packagings are recommended previously.\(^2\) Total immersion is not required for this purpose. Alternative methods that may be capable of meeting the requirements are\(^2\)

### TABLE 1 | Sensitivity of the bubble test $q_{\text{min,UN,cap,He}}$ (100% helium, temperature: 20°C, $\eta = 19.6$ µPa s, $p_1 = 131.3$ kPa, $p_2 = 101.3$ kPa, values taken from Schlick-Hasper et al.\(^7\)) and $q_{\text{min,UN,cap,air}}$ (100% dry air, temperature: 25°C, $\eta = 18.5$ µPa s, $p_1 = 131.3$ kPa, $p_2 = 0$ kPa)

<table>
<thead>
<tr>
<th>Sensitivity</th>
<th>Value (Pa m$^3$/s)</th>
<th>Packaging volume (L)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>$q_{\text{min,UN,cap,He}}$</td>
<td>$1.9\times10^{-4}$</td>
<td>6</td>
<td>Steel</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,He}}$</td>
<td>$1.9\times10^{-5}$</td>
<td>6</td>
<td>Plastics</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,He}}$</td>
<td>$6.4\times10^{-5}$</td>
<td>60</td>
<td>Steel</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,air}}$ (SLR)</td>
<td>$3.0\times10^{-5}$</td>
<td>6</td>
<td>Steel</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,air}}$ (SLR)</td>
<td>$9.9\times10^{-5}$</td>
<td>60</td>
<td>Plastics</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,air}}$ (SLR)</td>
<td>$2.0\times10^{-5}$</td>
<td>60</td>
<td>Steel</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,air}}$ (SLR)</td>
<td>$5.9\times10^{-5}$</td>
<td>216</td>
<td>Plastics</td>
</tr>
<tr>
<td>$q_{\text{min,UN,cap,air}}$ (SLR)</td>
<td>$1.2\times10^{-5}$</td>
<td>216</td>
<td>Plastics</td>
</tr>
</tbody>
</table>

Abbreviation: SLR, standardised leakage rate.

### TABLE 2 | Comparison of the different sensitivities $q_{\text{min}}$ leakage rate under specified flow conditions with upstream pressure $p_1$, downstream pressure $p_2$ and temperature $T$

<table>
<thead>
<tr>
<th>Sensitivity</th>
<th>Value (Pa m$^3$/s)</th>
<th>Flow conditions</th>
<th>Boundary conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>$q_{\text{min,SLR}}$</td>
<td>$10^{-4}$</td>
<td>Flow of dry air, $p_1 = 101.3$ kPa, $p_2 = 0$ kPa, $T = 298$ K (25°C) (SLR)</td>
<td>Industrial test</td>
</tr>
<tr>
<td>$q_{\text{min,ref}}$</td>
<td>$5\times10^{-5}$</td>
<td>Flow of dry air, $p_1 = 121.3$ kPa, $p_2 = 101.3$ kPa, $T = 298$ K (25°C)</td>
<td>Industrial test</td>
</tr>
<tr>
<td>$q_{\text{min,flow}}$</td>
<td>$1.7\times10^{-7}$</td>
<td>Flow of dry air, volumetric flow at atmospheric pressure 101.3 kPa</td>
<td>Laboratory test (extreme case)</td>
</tr>
<tr>
<td>$q_{\text{min,cap,He}}$</td>
<td>$7.6\times10^{-6}$</td>
<td>Flow of helium, $p_1 = 131.3$ kPa, $p_2 = 101.3$ kPa, $T = 298$ K (20°C)</td>
<td>Laboratory test (model of laminar viscous flow through capillary)</td>
</tr>
<tr>
<td>$q_{\text{min,cap,air}}$</td>
<td>$1.2\times10^{-5}$</td>
<td>Flow of dry air, $p_1 = 101.3$ kPa, $p_2 = 0$ kPa, $T = 298$ K (25°C) (SLR)</td>
<td>Laboratory test (model of laminar viscous flow through capillary)</td>
</tr>
</tbody>
</table>

Abbreviation: SLR, standardised leakage rate.
For these methods, it should be shown that they have the same level of sensitivity as the fully submerged leak test. Alternative methods should generally be applied to the entire packaging, as the fully submerged bubble test method, and not only to dedicated parts of the packaging, for example, the seams. Normally, when pressure decay, pressure rise or ultrasonic production leakproofness test methods are used, a pressure differential of at least 20 or 30 kPa between the inside of the packaging and the atmosphere should be applied. The test time should be adjusted so that the same sensitivity as in the bubble test is achieved. It may also be necessary to increase the test pressure difference to compensate for shorter test durations.2

In the following, the above-mentioned methods are examined with regard to the requirements described in Sections 2.4 and 3.4. All of these tests allow a leakproofness test of the entire surface of the packaging. When using a test closure, all these methods are nondestructive.

Because the field of leak testing is very strongly influenced by standards, these are initially mentioned before examples for typical industrial applications are presented.

The Vehicle Certification Agency2 describes in a later section how an industrial leak detection system shall be verified throughout the production period. This should be done by introducing a specially prepared test packaging (`leaker`) into the production line. This `leaker` should be a fault-free example of the packaging type being produced at this time, into which a hole of no greater than 0.4 mm in diameter has been precision drilled. This test packaging must be identified by the leak detector.

This in contradiction with the requirement of the UN Model Regulations that the industrial leakproofness test shall be equally effective. With the bubble test, one can detect much smaller leaks. Its minimum detectable diameter \(d_{\text{min}}\) is approximately 9.7 or 14.6 μm, as mentioned in Section 1. The specification of test leaks having diameters of 0.4 mm, that is, 400 μm, shows that in practice probably test methods are being used whose test level is not comparable with the bubble test. In this case, it is less than a test method equivalent to the bubble test, but rather a measure for quality assurance.

5.1. Pressure change detection

This quantitative method can be implemented in different ways: by measuring pressure decay, pressure rise or pressure rise in a vacuum chamber. In most cases, the test medium is air.14,23,32

5.1.1 Pressure decay test

Figure 1 presents the schematic diagram of the pressure decay test. The schematic diagrams in this section are based on the representation in ISO 12807.5 For the pressure decay test, the test object is subjected under a positive overpressure. This is done either by pressurisation or by placing it into a vacuum chamber. The pressure source is then isolated, and after a reasonable time for stabilisation, the readings of pressure and temperature are recorded at regular intervals. The pressure can be determined either as absolute pressure in the test object or as differential pressure between the test object and a leakproof reference vessel. This reference object must be designed so that it can assume the temperature of the test object.32

The sensitivity of the pressure decay test (technique D.1 in previous publications14,32) under industrial conditions is \(10^{-5}\) Pa m³/s (SLR), depending on object volume, test time and equipment.14 The standard ISO 12807⁶ specifies a range between \(10^{-2}\) and \(10^{-6}\) Pa m³/s (SLR) for the pressure drop test. The sensitivity is inversely proportional to the test object volume.6 Application examples16,27,33 relate only to small components with a maximum internal volume of 0.2 L, for example, plug-in systems or sensor systems. It can therefore be assumed that the sensitivities that can be realised for dangerous goods packagings tend to be in the less favourable range.

Problematic in the application of this method are thermally induced pressure changes. Small changes in ambient temperature can be buffered by using the differential pressure method using a reference tank of the same size. But if a component still comes hot from a previous production step and cools down, stronger temperature gradients are involved. This leads to a thermally induced pressure drop in the test sample. In this case, this amount should be estimated in advance in order to distinguish it from the pressure drop caused by leakage. Warm test pieces should ideally be cooled to room temperature through a cooling loop prior to testing. If this is not possible, a temperature compensation should be installed within the control program.16

5.1.2 Pressure rise test

When applying the pressure rise test, a lower pressure is generated inside the test object across the object boundary. For this purpose, the test object is either connected to a vacuum pump system...
(Figure 2) or placed in a pressurised chamber. After reaching the specified pressure difference, the test object is isolated, and the internal pressure is recorded at regular intervals.32

For the pressure rise test (technique D.2 in previous publications14,32), the same values for the sensitivities are given in previous studies6,14,32 as for the pressure decay test. In the practice of this process, the outgassing of water or other volatile components can be problematic. By this effect, an initial pressure increase can be generated that is above the pressure rise due to leakage.14,32

Because of the flow direction, which is directed from outside to inside in this method, this does not meet the requirements based on the bubble test.

5.1.3 Pressure rise test (vacuum chamber)

A third kind of pressure change method is the bell pressure change technique (technique D.3 in previous publications14,32).

The test object is enclosed by a rigid chamber (bell chamber), and a pressure difference is created between the test object and the chamber. If the test object is pressurised or the chamber is evacuated, any leakage across the boundary wall of the test object enclosed by the chamber will cause a pressure rise in the chamber (Figure 3). If the test object is evacuated or the chamber is pressurised, any leakage across the boundary wall of the test object enclosed by the chamber will effect a pressure decay in it.32

The sensitivity of the bell pressure change technique can theoretically reach values to \(10^{-6}\) Pa m\(^3\)/s, depending on free chamber volume, test time and equipment.14 Because of the requirements of the bubble test that the flow direction should be from the inside to the outside of the packaging, only the implementation is suitable in which the test object is pressurised or the chamber is evacuated (vacuum chamber).

In principle, the test pressure level specified for the bubble test (flow from 30 or 20 kPa gauge pressure level to atmospheric pressure level) can be applied for all these methods. All three methods can be automated.

Another test method that is formally classified as a pressure change method but is not mentioned previously2 is the flow measurement technique (technique D.4 in previous publications14,32). The extent of leakage is determined by measuring the flow rate of gas into or out of the test object. Its sensitivity is \(10^{-4}\) Pa m\(^3\)/s (SLR) under industrial conditions.14

Mass flowmeters can be used for the measurement. An ideal field of application is the testing of test objects with volumes up to a few litres. If the sensitivity in practice is an air leakage rate of 0.5 ml/min at ambient pressure (approximately \(8.4 \times 10^{-4}\) Pa m\(^3\)/s).16 Because the nominal sensitivity of this method is smaller than for the three methods just mentioned, it is no longer listed below.

Table 3 lists the individual aspects of the three methods.

Because the flow direction is exactly opposite to that of the bubble test in the pressure rise method, this method is in principle not considered as the bubble test equivalent method. When comparing the pressure decay method and the pressure rise method using an external vacuum chamber, the latter method is found to be more appropriate. On the one hand, it has a higher sensitivity (SLR); on the other hand, the use of a vacuum reduces the temperature sensitivity of the measurement.34

On the basis of the parameters considered, the third method is best suited for the industrial leak testing of dangerous goods packagings. If in practice a test pressure difference of more than 30 kPa is applied, the leakage rates determined in this way should be converted to the test pressure level of the bubble test using the correlations in ISO 12807.6

5.2 Ultrasonic leak detection

In the application of ultrasound for leak detection, there are two different variants: in the field of airborne ultrasonic leak testing and for the detection of air bubbles in the water bath in the bubble test.

5.2.1 Airborne ultrasonic leak detection

Airborne ultrasound detection is an inspection technique applied to locate leaks in pressurised systems. Gas leakage generates sound waves when the gas flow through the leaks is accompanied by turbulence. Airborne ultrasound can be detected at a distance from its source with directional scanning microphones or acoustic probes (Figure 4). For applicability, it is necessary that the leakage rate is sufficiently large (\(10^{-4}\) Pa m\(^3\)/s or higher for air at ambient pressure) and thus the flow regime is in the turbulent range.12
ASTM E1002 specifies a value of approximately $1.5 \times 10^{-2}$ Pa m$^3$/s for this leakage rate.\(^3\)^5

In a contribution about the current developments of the acoustic ultrasonic leak detection, the theoretical sensitivity is given as $10^{-3}$ Pa m$^3$/s (flow of leaking air at ambient pressure). In practical application, it is more likely to be $10^{-1}$ Pa m$^3$/s.\(^3\)^6

### 5.2.2 Ultrasonic gas bubble detection

In the case of industrially used bubble test, the manual underwater visual inspection often fails because it is not sufficiently reproducible and only rarely economical.\(^3\)^7 As mentioned above, the attention of the inspection personnel is not guaranteed over the entire test period and the visibility may be poor.\(^2\)^9 When integrating the bubble test into series production, automated ultrasonic gas bubble detection offers an alternative\(^3\)^7 (Figure 5).

Ultrasonic waves are scattered when they hit gas bubbles on their way through a liquid.\(^3\)^7 From an ultrasonic sensor, which can be operated both as a transmitter and as a receiver, a wave packet is emitted in the water basin. If there is a leaking part and thus air bubbles in the basin, the wave packet is scattered prematurely on them. The evaluation of the scatter signal is carried out via digital signal processors. Because of the propagation time of the sound, the system calculates the position of the bubbles and evaluates the component as leakproof or leaking on the basis of the specified limit leakage rate.\(^2\)^9

The advantages of the ultrasonic bubble detection in the bubble test are that even with turbid test liquid, the sensitivity of the system is maintained and that even under conditions of series production, a quantification is possible.\(^3\)^7

With ultrasound, air bubbles with a diameter of only 0.1 mm can be detected— in contrast to optical monitoring. The theoretical detection limit of this method is therefore $10^{-9}$ Pa m$^3$/s (volumetric flow at atmospheric pressure, 60 s measurement time). Sensitivities between $10^{-5}$ and $10^{-6}$ Pa m$^3$/s are realistic under real conditions of series production.\(^3\)^8

Examples for the application of this method are the testing of compressed air tanks with a volume of 10 to 60 L in 12 s cycle time with a limit leakage rate of $5.6 \times 10^{-4}$ Pa m$^3$/s\(^3\)^9 and the testing of small

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**TABLE 3** Individual aspects of the industrial leak test methods: Pressure decay test, pressure rise test and pressure rise test in vacuum chamber

<table>
<thead>
<tr>
<th>Leak test method</th>
<th>Objective</th>
<th>Flow direction of bubble test feasible</th>
<th>Pressure level of bubble test feasible</th>
<th>Sensitivity $q_{\text{min,SLR}}$ (Pa m$^3$/s) (SLR)</th>
<th>Sensitivity $q_{\text{min,ref}}$ (Pa m$^3$/s) (air at ambient pressure)</th>
<th>Ability for automation; limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure decay</td>
<td>Quant.</td>
<td>Yes</td>
<td>Yes</td>
<td>$10^{-5}$ ... $10^{-2}$ (ISO 12807)(^6)</td>
<td>-</td>
<td>Yes; hot test objects</td>
</tr>
<tr>
<td>Pressure rise</td>
<td>Quant.</td>
<td>No</td>
<td>Yes</td>
<td>$10^{-5}$ (DIN EN 1779)(^1); $10^{-6}$ ... $10^{-2}$ (ISO 12807)(^6)</td>
<td>-</td>
<td>Yes; outgassing</td>
</tr>
<tr>
<td>Pressure rise (vacuum chamber)</td>
<td>Quant.</td>
<td>Yes</td>
<td>Yes</td>
<td>$10^{-6}$ (DIN EN 1779)(^1)</td>
<td>-</td>
<td>Yes; –</td>
</tr>
</tbody>
</table>

Abbreviation: SLR, standardised leakage rate.
barrels (kegs) with a volume of 10 L to 50 L in 15 s cycle time with a limit leakage rate of $10^{-3}$ Pa m$^3$/s.\(^{38}\)

Table 4 lists the individual aspects of the two methods.

The airborne ultrasound detection does not have the necessary sensitivity $q_{\text{min,standard}}$ of $5 \times 10^{-5}$ Pa m$^3$/s (see Table 2). In addition, it is questionable whether at the relatively low overpressure level, as prescribed in the bubble test, a flow can be caused in a magnitude which lies in the turbulent-viscous flow region.

In terms of sensitivity, ultrasonic bubble leak detection would theoretically be appropriate. The suitability with regard to the cycle time of the respective production line would have to be checked.

5.3 | Gas leak detection

In the field of test gas method, the following two cases can basically be distinguished: gas flow into the test object and gas flow out of the test object.\(^{14,40}\) Because the test gas methods, which are based on a gas flow from the outside into the test object, contradict the requirement for the preferred direction of flow in the case of dangerous goods packagings mentioned in Section 2.4, this will not be discussed in the following.

In the field of test methods with a gas flow from the inside of the test object to the outside, there are generally three methods allowing an examination of the total area of the packaging.

5.3.1 | Pressure technique by accumulation

The test object is pressurised with tracer gas and is the placed into a test chamber. The tracer gas, usually helium or a halogen, flows out through leaks into the surrounding volume, causing a concentration increase in the chamber, whose free inner volume is homogenised with fans. After a certain accumulation period, this concentration increase is measured with a suitable leak detector. This method (technique B.3 in previous publications\(^{14,40}\)) can be used for objects that can be filled with a tracer gas at a pressure greater than atmospheric pressure. Its sensitivity is $10^{-7}$ Pa m$^3$/s (SLR) under industrial conditions, depending on accumulation period\(^{14}\) (Figure 6).

Because in this method the chamber does not have to be evacuated but is under atmospheric pressure, no mass spectrometer is required as leak detector. In the automated helium leak test, a system that uses a quartz membrane sensor without a vacuum chamber has instead been used for some time.\(^{41,42}\) In the test chamber, the test piece is charged with helium via its test gas connection. The quartz membrane sensor is capable of safely resolving increases in helium concentration of 0.025 ppm. Under laboratory conditions, leaks in the order of $10^{-7}$ Pa m$^3$/s (release of helium at ambient pressure) can be reliably detected.\(^{42}\) In production mode, with a free volume in the test chamber of 5 L, leakage rates of $10^{-5}$ Pa m$^3$/s (release of helium at ambient pressure) can be detected within a test time of 30 s. This method is also suitable for test samples made of plastic or warm test samples.\(^{43}\)

This method can also be used to measure the leakage rates of closures of different types of dangerous goods packagings under laboratory conditions.\(^{4,20}\)

The just mentioned sensitivities, expressed as helium leakage rates, can be equated with the air leakage rates assuming a laminar viscous flow regime, because the dynamic viscosities of these two gases are similar.\(^{44,45}\)

It is also possible to combine the immersion bubble test and the pressure technique by accumulation.\(^{46}\) The test sample is filled with helium and is immersed in a water bath, which is placed in an accumulation chamber connected to a helium mass spectrometer in sniffing operation. The advantage of this combined test procedure is that it does not require visual observation in the bubble test and that it leads to a quantitative result. Its sensitivity is $2 \times 10^{-6}$ Pa m$^3$/s, expressed as 100% helium leakage rate at ambient pressure for an accumulation time of 1.5 min.

Table 4

<table>
<thead>
<tr>
<th>Leak test method</th>
<th>Objective</th>
<th>Flow direction of bubble test</th>
<th>Pressure level of bubble test</th>
<th>Sensitivity $q_{\text{min,standard}}$ (Pa m$^3$/s) (SLR)</th>
<th>Sensitivity $q_{\text{min,standard}}$ (Pa m$^3$/s) (air at ambient pressure)</th>
<th>Ability for automation; limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Airborne ultrasonic leak detection</td>
<td>Loc.</td>
<td>Yes</td>
<td>Yes</td>
<td>$10^{-3} \ldots 10^{-1}$</td>
<td>$10^{-3} \ldots 10^{-1}$</td>
<td>No; for bubble test pressure level: No turbulent flow</td>
</tr>
<tr>
<td>Ultrasonic bubble leak detection</td>
<td>Quant./loc.</td>
<td>Yes</td>
<td>Yes</td>
<td>$10^{-6} \ldots 10^{-5}$</td>
<td>$10^{-6} \ldots 10^{-5}$</td>
<td>Yes; –</td>
</tr>
</tbody>
</table>

Abbreviation: SLR, standardised leakage rate.
5.3.2 | Pressurisation–evacuation (bombing test)

This method (technique B.5 in previous publications) is applicable to test objects that are sealed prior to leak testing and that are not equipped with a connection for filling with test gas. The object is first placed in a bombing chamber and is pressurised with tracer gas (Figure 7). The tracer gas flows through leaks from the outside into the test object. After this ‘bombing period’, the object is placed in a vacuum chamber connected to a mass spectrometric leak detector. These objects are generally of small dimensions (for example, semiconductor devices and hermetically enclosed relays). The magnitude of its sensitivity is between $10^{-9}$ and $10^{-6}$ Pa m$^3$/s (SLR) under industrial conditions. Test gas adsorbed on the surface represents a disturbing influence during the measurement. Therefore, test objects shall be flashed with tracer-gas free air or nitrogen before testing in the vacuum chamber.

ISO 12807 indicates a sensitivity between $10^{-9}$ and $10^{-4}$ Pa m$^3$/s (SLR) for this procedure.

The bombing test is used, for example, in the leak testing of cardiac pacemakers, components for mobile phones or encapsulated electronic modules for applications in the automotive or aerospace sector. For economic reasons, this method can only be used for components with a volume of approx. 10 cm$^3$, otherwise the times for pressure storage, flushing and test time will take too long.

An application of this method in dangerous goods packagings is therefore out of the question. It can also be assumed that most types of dangerous goods packagings are not suitable for withstanding an external high vacuum, as is required in the operation of a mass spectrometer.

5.3.3 | Vacuum chamber technique

The test object, filled with tracer gas, is placed into a test chamber, which is evacuated to a pressure lower than the internal pressure of the test object (Figure 8). Tracer gas flowing through leaks into the chamber is measured using a leak detector (technique B.6 in previous publications).

The maximum sensitivity of this method is indicated by $10^{-9}$ Pa m$^3$/s (SLR).

A typical industrial application of this procedure is the testing of 200-L steel drums. In this application, the test gas may be either pure air or an air–helium mixture. The drums are filled with the test gas under ambient pressure conditions, closed and placed in a large test chamber, which is evacuated to high vacuum to allow testing with a mass spectrometer. The production cycle is 360 parts per hour. The entire measuring process including evacuation and aeration is completed within a period of approximately 5 s. Because the test pressure difference is 100 kPa, the drums must be stabilised in the bottom area and lid area so as not to be irreversibly deformed. When using helium as the test gas, its content in the helium–air mixture is 1%. This means a consumption of about 2 L of helium per test.

The sensitivity of this application under production conditions, expressed as 100% helium leakage rate, is $<10^{-9}$ Pa m$^3$/s. Because of the use of a mass spectrometer and the necessary presence of a high vacuum, the pressure level does not match that of the bubble test. When testing other types than steel drums, it is to be expected that they will be irreversibly deformed by the test and thus destroyed.

Table 5 lists the individual aspects of the three methods.

Of these three gas detection methods, the pressure technique by accumulation formally has the best conditions for industrial leak testing of dangerous goods packagings. The vacuum chamber technique is also suitable, provided that it is possible to prevent the packaging from irreversible deformation.

5.4 | Soap solution applied to the entire packaging

This test corresponds to the soap bubble test in ISO 12807 or the bubble test with liquid application in DIN EN 1779. The test item is pressurised, and its surface is coated with a soap film. A leak is indicated by a soap bubble on the surface. Its nominal test sensitivity is $10^{-4}$ Pa m$^3$/s (SLR).
Depending on the surface tension of the soap solution used, it is even possible to detect leaks whose diameter lies below the minimum detectable leak diameter $d_{\text{min}}$ of the conventional bubble test.$^{50,51}$ Theoretically, therefore, the sensitivity, expressed as a quantitative leakage rate, would be higher than in the actual bubble test.

Table 6 lists the individual aspects of this method.

The sensitivity and the other parameters correspond to the liquid immersion bubble test. It is questionable whether in the practice of mass production, this method is suitable for automation and for production lines with high cycle times. In practice, it is mainly used for leak testing of pressure vessels, tanks or other large structures.$^{12}$ A problem is to ensure simultaneous coverage over the complete area.$^{6}$
5.5  Summary of suitable methods

Because of the individual consideration of the different methods in Sections 5.1 to 5.4 with regard to the criteria in Section 2.4, the following procedures are fundamentally excluded: the pressure rise test because of its opposite flow direction compared with the bubble test; the airborne ultrasonic leak detection, because this test procedure requires a turbulent flow regime; the bombing test, because this test method is limited to test specimen up to a volume of approx. 10 cm³.

It follows from Section 3 that the required sensitivity $q_{\text{min}}$, of an industrial leak test method is $1.0 \times 10^{-5}$ Pa m³/s (SLR). The pressure decay test can theoretically have sensitivity values in this range (Table 3). However, it is questionable whether this sensitivity can be realised for the practice of testing dangerous goods packagings, with regard to their comparatively large volumes, high cycle times during production and warm test specimens in the case of plastics packagings.

Table 7 shows a summary of the test methods that can realistically be applied in the practice of series production of dangerous goods packagings. In principle, these methods are suitable and equally effective as the bubble test. However, it should be noted that a final statement can only be made in the context of the specific production line with its individual conditions. This affects in particular the soap bubble test. This test has formally been included into Table 7 because it is theoretically equivalent to the immersion bubble test. However, it cannot be automated and is therefore only suitable for low quantities in production practice.

6  CONCLUSIONS

Because the International UN Model Regulations do not contain specific requirements for suitable industrial leak testing procedures, this work evaluates common leak testing methods for their suitability for series production of dangerous goods packagings.

First, the requirements for an equivalent leak test method are analysed, which must be equally effective as the standard test procedure (bubble test). The most relevant aspects are essentially the following: sensitivity, extent and objective of the investigation, testing conditions (pressure level and direction of flow) and ability for automation. Because the sensitivity of the bubble test can be determined in different ways, different approaches are presented and compared. The values of the sensitivity differ from each other by several orders of magnitude. The design type leakproofness test is mentioned in the UN Model Regulations as a reference for an industrial method. With this type of implementation, the bubble test is a laboratory test. In this way, it can be estimated that its sensitivity is in the order of $1.0 \times 10^{-5}$ Pa m³/s (SLR).

Subsequently, common test methods in industry are compared with these derived requirements. The comparison shows that some of these common leak testing methods are not suitable for industrial leak testing of dangerous goods packagings. This concerns the pressure decay test, the pressure rise test and the airborne ultrasonic leak detection. In these cases, either their sensitivity is insufficient or the flow direction is different than intended. The bombing test cannot be used on dangerous goods packagings because it is only suitable for smaller test items.

Other methods such as the pressure rise method (vacuum chamber), the ultrasonic bubble leak detection or the soap bubble test are basically able to fulfil the requirements. In the last two, however, the suitability for production lines with high cycle times must be checked.

In the field of gas detection methods, the pressure technique by accumulation and the vacuum chamber technique are most suitable.

Currently, leak testing methods are often used in industrial series production, which do not correspond to the requirements of the bubble test. These test methods are not equally effective procedures, as required by the UN Model Regulations. They are more likely to ensure a certain level of quality.

Therefore, with the aim of achieving a uniform level of safety, in the future, it is necessary to clarify the international UN Model Regulations. This concerns, on the one hand, information on the specific sensitivity of the bubble test and, on the other hand, a catalogue of suitable and equally effective industrial leak testing methods for dangerous goods packagings. The definition of limit leakage rates in the dangerous goods regulations can also be a first step.

SYMBOLS

- $d_{\text{min}}$: minimum detectable leak diameter (m)
- $L$: wall thickness (m)
- $p_0$: absolute pressure inside the bubble (Pa)
- $p_1$, $p_2$, $p_1$, $p_2$: absolute upstream gas pressure (in Condition I or II) (Pa)
- $q_{\text{flow}}$: absolute downstream gas pressure (in Condition I or II) (Pa)
- $q_{\text{flow}}$: leakage rate (pV-flowrate) of a fluid (Pa m³/s)
- $q_{\text{min}}$: sensitivity (minimum detectable leakage rate) (Pa m³/s)
- $q_{\text{min, std}}$: sensitivity, specification taken from leak testing standards (Pa m³/s)
- $q_{\text{min, std, ref}}$: sensitivity, value obtained by conversion of $q_{\text{min, std, SLR}}$ to reference pressure level of the bubble test (flow of dry air, $p_1 = 121.3$ kPa, $p_2 = 101.3$ kPa, $T = 298$ K (25°C) (Pa m³/s))
- $q_{\text{min, std, SLR}}$: sensitivity, specification taken from leak testing standards, under SLR conditions (Pa m³/s)
- $q_{\text{min, UN, cap}}$: sensitivity for a laminar viscous capillary flow, based on the minimum detectable leak diameter $d_{\text{min}}$ of the bubble test (Pa m³/s)
- $q_{\text{min, UN, cap, air}}$: expressed as flow of dry air, $p_1 = 101.3$ kPa, $p_2 = 0$ kPa, $T = 298$ K (25°C) (SLR) (Pa m³/s)
- $q_{\text{min, UN, cap, He}}$: expressed as flow of helium, $p_1 = 131.3$ kPa, $p_2 = 101.3$ kPa, $T = 298$ K (20°C) (Pa m³/s)
- $q_{\text{min, UN, flow}}$: sensitivity under the physical flow conditions of the UN bubble test (Pa m³/s)
leakage rate under the flow conditions I or II (Pa m³/s)
SLR standardised leakage rate, normalised to the flow of dry air under reference conditions ($p_1 = 101.3$ kPa, $p_2 = 0$ kPa, $T = 298 K (25°C)$) (ISO 12807)\(^6\) (Pa m³/s)
t test time (s)
$V_{b,tot}$ total bubble volume (m³)
$\eta$ dynamic viscosity (Pa s)

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