



Helium Leak Testing of Dangerous Goods Packagings

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Abstract. The International Dangerous Goods Regulations prescribe the immersion under water method (“bubble test”) as standard method for the leakproofness test of dangerous goods packagings. But this test procedure acts as a test method for leak localisation, not for quantitative leakage rates measurement. Additionally, the sensitivity in detecting leaks of small diameters is restricted, depending on the test liquid and the test pressure. The bubble test is not suitable for a comparison with quantitative limit leakage rates based on realistic transport conditions. This is especially important when estimating the risk of the formation of an explosive atmosphere during the intercontinental carriage of dangerous goods packagings in freight containers.

To compare measured leakage rates with limit leakage rates, a quantitative leak testing procedure is required. Therefore a new approach for dangerous goods packagings is implemented: The pressure technique by accumulation using Helium as a tracer gas.

This work presents the test equipment necessary for the quantitative measurement of Helium leakage rates through closures of different kinds of dangerous goods packagings. The essential steps to achieve good repeatable results are: A controlled Helium filling process to reach a defined test pressure in the test sample, a sufficient homogenisation of the Helium-air-mixture inside the test sample and the ensuring of a constant pressure level of the test sample during the test. The Helium loss rate of the accumulation chamber has to be measured separately to receive a correction factor for the measured leakage rates. Different constructional measures are introduced to prevent a disturbing influence of the Helium leakage rate of the filling valves on the measurement results. Methods to estimate the disturbing effect of Helium permeation through permeable parts of the test sample are also presented. As a supporting method for the experimental investigations the Helium sniffer test can be applied.

This practical application-oriented advice can enable other users to establish a pressure technique by accumulation for their own technical field.

Introduction

The standard test procedure for the leakproofness test of dangerous goods packagings is the immersion in water, the so called “bubble test” (UN Model Regulations 6.1.5.4 [1]; technique C.1 [2]). The test pressure to be applied is 300 mbar for packaging group I (substances presenting high danger) and 200 mbar for packaging groups II and III (substances presenting medium and low danger). The test duration is 5 minutes. Criterium for passing the test is that no leakage should be detected by the rising of bubbles. This technique is suitable for leak localisation, but not for quantitative leakage rate measurement



[2]. Its sensitivity in detecting leak diameters is restricted: When using water and a test pressure of 200 mbar, only leaks of approximately 16 μm diameter can be detected [3]. Another disadvantage is that the results cannot be compared with limit leakage rates.

Therefore a new experimental approach for dangerous goods packaging was chosen: Quantitative leakage rate measurement, using the pressure technique by accumulation (technique B.3 [2]) with Helium as a tracer gas. The Helium leakage rates measured for different closure types of dangerous goods packaging were compared with Helium limit leakage rates calculated for a worst case transport scenario [4]. This approach allows a statement if the lower explosion limit in freight containers is reached under transport conditions.

This article describes the steps necessary for the application of a quantitative leakproofness test using the pressure technique by accumulation. As a practical example for each step, the measurement of the Helium leakage rates through the steel plug screw closure of a 6 l steel drum is illustrated (fig. 1).



Fig. 1. 6 l steel drum with steel plug screw closure.

Application of a quantitative leakproofness test

1.1 Calculation of Limit Leakage Rates

For the application of a quantitative test procedure, the user has to determine the maximum permissible leakage rate (leakage rate requirement). This is dependent on the specific technical situation. For dangerous goods packagings, the lower explosion limit may work as limit value. The worst case scenario is the intercontinental transport of dangerous goods packagings in freight containers. Helium leakage rates were calculated dependent on duration of transport, air change rate of the freight container, gauge pressure inside the packaging, transport temperature and individual substance-specific parameters [4]. The limit leakage rates (100 % Helium) for 300 mbar test pressure are between $2 \cdot 10^{-5}$ Pa m^3/s (substances of PG I, relatively high vapour pressures) and 0.01 Pa m^3/s (substances of PG III, relatively low vapour pressures).

1.2 Selection of a test procedure and a test gas

An overview of the essential criteria to for the selection of a suitable test procedure is provided by [2] [5] [6]. Following points should be considered:

- *Sensitivity of the test method and leakage rate range:* The minimum detectable leakage rate (sensitivity) and the measuring range are the most important parameters for choosing a test procedure [2] [5] [7].
- *Objective and extent of the investigation:* The objective can be a quantitative leakage rate measurement or a leak localisation. The extent can be the total area of the test object or a local area [2] [5].

- *Operation conditions and testing conditions:* Generally a test fluid other than the operation fluid is used [2]. Important parameters are also the test pressure conditions (pressure level and direction of flow) and the testing temperature [2] [5] [8].
- *Test object design:* The dimensions of the test object, openings for test gas supply and limit values concerning pressure and vacuum may have an influence [2].
- *Safety and environmental requirements:* Some test gases are toxic (e. g. Ammonia) or can cause damage for the upper atmosphere (Halogen-containing gases) [2].
- *Other aspects:* Automatability for series production, reliability and the cost (acquisition costs, operating costs) [9] [8].

[2] provides an overview about different test methods and techniques. For the present leak testing application, the measurement of leakage rates through the screw closure of a steel drum, the pressure technique by accumulation (technique B.3 [2]) using the tracer gas Helium was chosen. This procedure is suitable for test samples which must not be evacuated due to their weak structure [10].

1.3 Experimental Setup for the measurement of leakage rates

Figure 2 displays schematically the experimental setup for the measurement of the leakage rate through the screw closure of the 6 l steel drum, on the basis of a 3 l accumulation chamber (1). The green arrows indicate the direction of the gas flow.

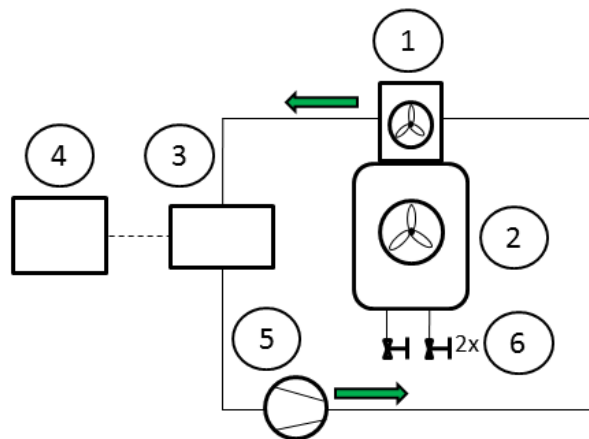


Fig. 2. Experimental setup for the pressure technique by accumulation.

Car tire valves (6) are used to fill the test sample (2) with Helium. After the Helium supply is finished, the accumulation chamber (1) is put over the closure area of the test sample. The accumulation chamber is fixed by its own weight (mass approx. 8560 g) on the flat surface of the test sample. A leakage through the closure of the test sample will provoke a continuous increase of Helium concentration inside the chamber. The pump (5) ensures a constant circulation flow of the Helium-air-mixture from the chamber to the T-Guard Helium leak detection sensor (Inficon) (3). The detector measures the Helium concentration of the Helium-air-mixture inside the chamber. The individual detection limit of this experimental setup is $1.18 \cdot 10^{-7}$ Pa m³/s [4].

1.4 Essential steps to receive good repeatable results

1.4.1 Controlled Helium Filling Process

Before starting the test, only air at actual ambient pressure is inside the test sample. It is necessary to receive a defined test pressure level inside the test sample (gauge pressure).

The aim is to add Helium into the test sample to reach the gauge pressure levels 100 mbar, 200 mbar, 300 mbar, 400 mbar and 500 mbar. Figure 3 schematically shows the Helium filling station.

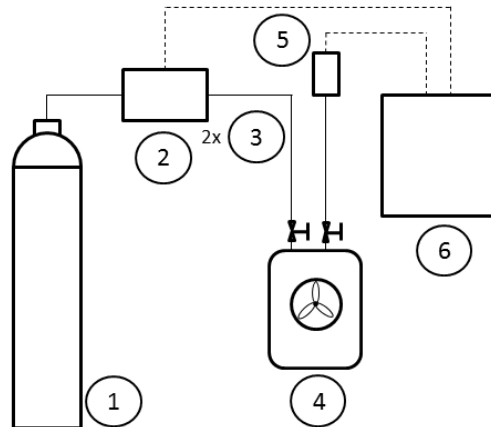


Fig. 3. Helium filling station.

The gauge pressure setpoint is entered into the program DASYSLab 10.0 (6) and the Helium filling is started. The Helium mass flow is regulated by the mass flow meter and controller “Mass-Stream” D-6300 (M+W Instruments) (2) to approximately 900 mg/min. The Helium supply from the Helium gas cylinder (1) into the test sample (4) is stopped automatically by DASYSLab when the pressure level is reached.

1.4.2 Homogenisation of Helium-air-mixture inside the test sample

It is necessary to generate a sufficient homogenisation of Helium-air-mixture inside the test sample to achieve a leakage gas flow of constant concentration. To obtain this homogenisation, a fan was installed inside the test sample (see fig. 2 and 3). The fan had a volume flow of 17.8 l/min. A test was carried out to check a sufficient homogenisation. A prepared test sample was filled with Helium. The nominal value of the Helium concentration inside the test sample was calculated from the inner free volume of the test sample and the added mass of Helium. As an indicator for sufficient homogenisation, local concentration differences of up to 10 % from the average concentration are tolerable [11]. In steps of 60 seconds intervals, gas samples were taken with a syringe from the prepared test sample. The Helium concentration of the samples was analysed by the Helium leak detector T-Guard. Figure 4 shows the results for 300 mbar test pressure.

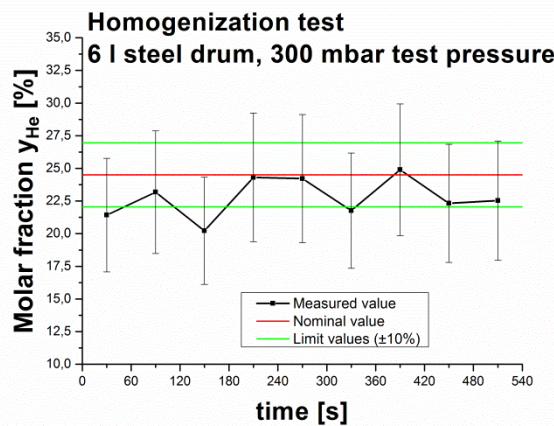


Fig. 4. Helium molar fraction inside test sample dependent on time [12].

For the current constellation (300 mbar gauge pressure in the 6 l steel drum), the nominal value of the Helium molar fraction is 24.5 %. It is apparent that a sufficient homogenisation is reached after 90 s.

1.4.3 Constant pressure level inside the test sample

For the pressure technique by accumulation it is important to reach an approximate constant pressure level inside the test sample during the testing time. Because the gauge pressure inside the test sample is the driving force for leakage flow, a decreasing gauge pressure would lead to a decreasing leakage rate during the test. The main influence factor is the filling valve for Helium filling. Pressure retention tests were carried out using two different valve types: Schott valves and car tire valves. The aim was to examine if the test samples were able to hold the applied test gauge pressure over a period of time (2 minutes). Figure 5 displays the results for the pressure retention test using Schott valves, figure 6 the results using car tire valves for the same packaging type (6 l steel drum).

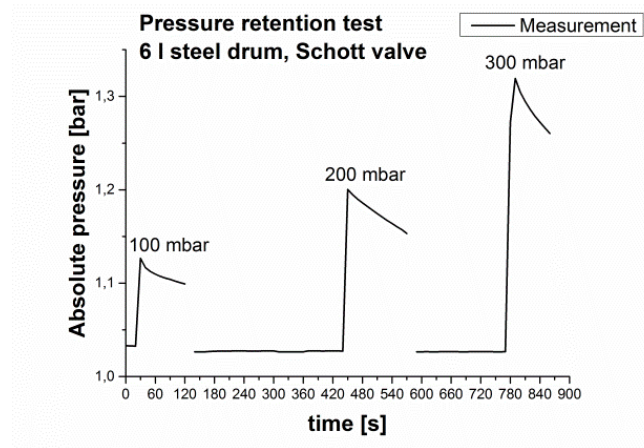


Fig. 5. Pressure test using Schott valves [12].

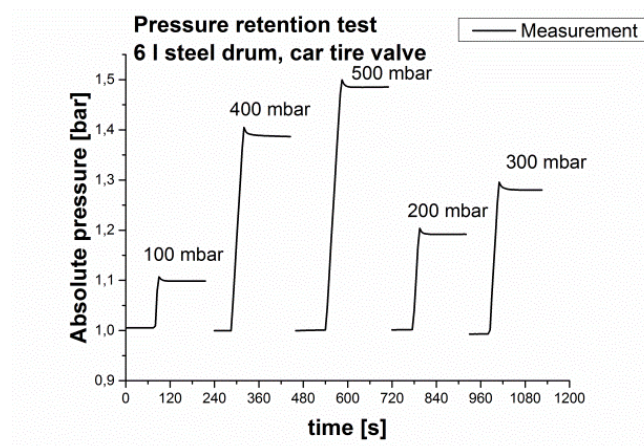


Fig. 6. Pressure test using car tire valves [12].

The test samples prepared with the Schott valves show a rapid pressure decrease. The maximum pressure decrease was 61 mbar. The reason was that the attachment of the Schott valves in the borehole of the packaging wall was not optimal. Using car tire valves with a flexible outer form, the maximum pressure decrease was only 15 mbar in 2 minutes. Therefore car tire valves were used for the Helium filling process for all test samples.

1.4.4 Helium loss rate of accumulation chamber

No accumulation chamber is absolute tight for the test gas Helium. Therefore the determination of the Helium loss rate is necessary. As a model, the determination of the air change rate according to [11] is used. It is defined as the quotient of the air flow inside a zone \dot{V}_L and the zone volume V_R :

$$n = \frac{\dot{V}_L}{V_R} \quad (1)$$

For the measurement of the air change rate, the concentration decay method can be applied. A tracer gas is injected into a room and the decay in concentration is measured, as function of time [11]. The resulting Helium loss rate n_{He} can be calculated based on [11]:

$$n_{He} = \frac{1}{t_1 - t_0} \cdot \ln \frac{c_{He,0}}{c_{He,1}} \quad (2)$$

Figure 7 shows an exemplary concentration decay test of the 3 l chamber.

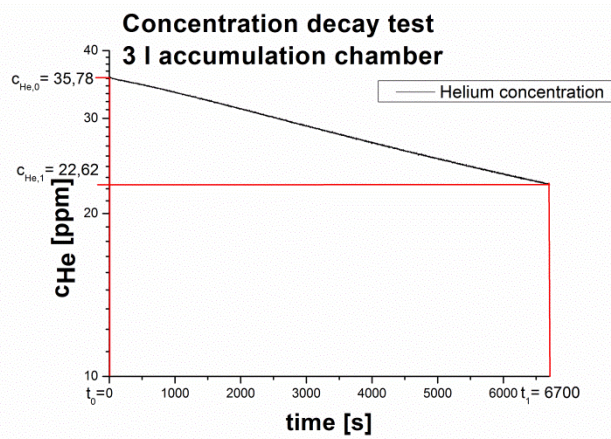


Fig. 7. Concentration decay test of the 3 l accumulation chamber [12].

The leakage rate for the accumulation method can be determined by [4]

$$q_{He} = V_{chamber} \cdot p \cdot \frac{\Delta c_{He}}{\Delta t} \cdot e^{n_{He} \cdot \Delta t} \quad (3)$$

In this expression, $V_{chamber}$ is the free volume of the accumulation chamber, p is the absolute pressure inside the chamber, Δc_{He} the concentration increase and Δt the test time. The average Helium loss rate of the 3 l chamber is 0.27 h^{-1} [12].

1.4.5 Methods to estimate the disturbing effect of Helium permeation

If there is a permeation of the tracer gas Helium through permeable parts of the test sample, this permeation leakage rate is a disturbance for the measurement of the leakage rate [13] [4]. Two different parameters can be calculated to estimate the effect of Helium permeation [4]. The induction time [14] (“time lag” [15]) is given by

$$t_{ind} = \frac{L^2}{6 \cdot D_{He}} \quad (4)$$

This expression is an approximation for the breakthrough time of Helium through the specific material of thickness L [16]. The steady-state gas permeation rate of Helium, expressed in the unit of a leakage rate, is [17]

$$q_{perm} = P \cdot \frac{A}{L} \cdot (p_1 - p_2) \quad (5)$$

The cross-sectional area A and the length of the permeation path L are dependent of the specific problem. The pressures p_1 and p_2 are the absolute partial pressures of the permeating substance Helium.

1.4.6 Helium sniffer for leak localisation

Additionally to the quantitative leak detection method, the sniffer technique (technique B.4 [2]) can be used. It is useful to localise undesired leaks in the experimental setup and to improve it. For the current investigation, the Helium sniffer leak detector Protec P3000(XL) (Inficon) was used.

1.4.7 Constructional measures to prevent disturbing influences of Helium leakage rate of filling valves

For the sniffer technique, it is important to reach a low Helium background concentration. A high Helium background concentration is an interfering influence. A critical source of undesired leakage rates are the filling valves. There are different possibilities to minimize them: Either a rigid casing can be used or flexible impermeable bags, for example made of Aluminium. Both can be fixed by Aluminium adhesive tape. Figure 8 shows a flexible impermeable bag in combination with a hood made of plexiglass.

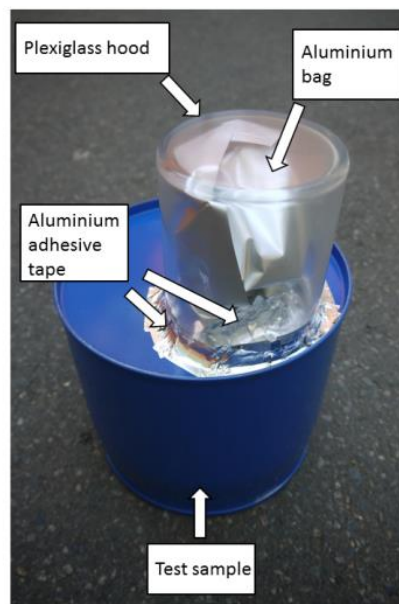


Fig. 8. Flexible impermeable bag in combination with a plexiglass hood for the reduction of the leakage rate of the filling valves [12].

2. Results

2.1 Leakage Rate Measurement of a test leak

Figure 9 displays the results for the measurement of the leakage rates of a capillary test leak for the gauge pressure levels 100 mbar, 200 mbar, 300 mbar, 400 mbar and 500 mbar. p_1 is the absolute pressure inside the test sample, p_2 is the atmospheric pressure.

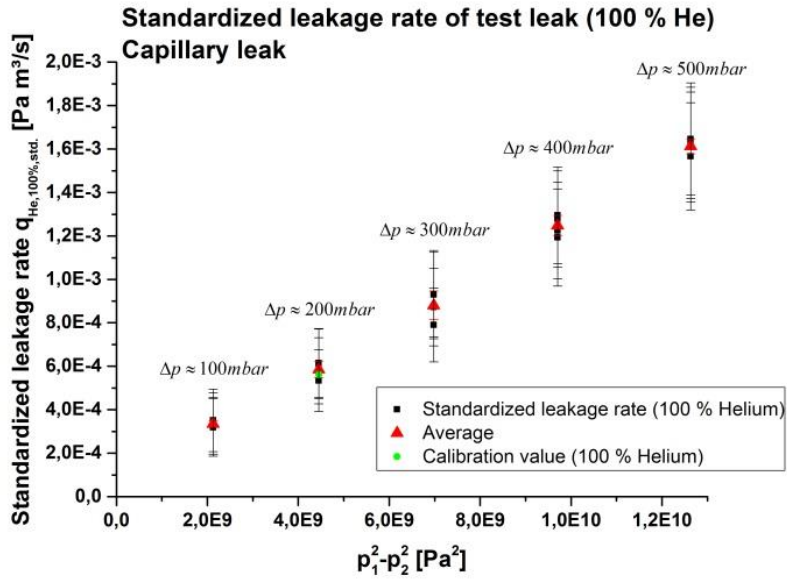


Fig. 9. Leakage rates of a capillary test leak [12].

The leakage rates show a linear increase as a function of the expression $p_1^2 - p_2^2$ which is typical for laminar flow under the assumption of a constant leak geometry. It is also obvious that the accordance with the calibration value is very good. [12]

2.2 Leakage Rate Measurement of closures

Figure 10 shows the exemplary results of the leakage rate measurement of the steel plug screw closure of the 6 l steel drum.

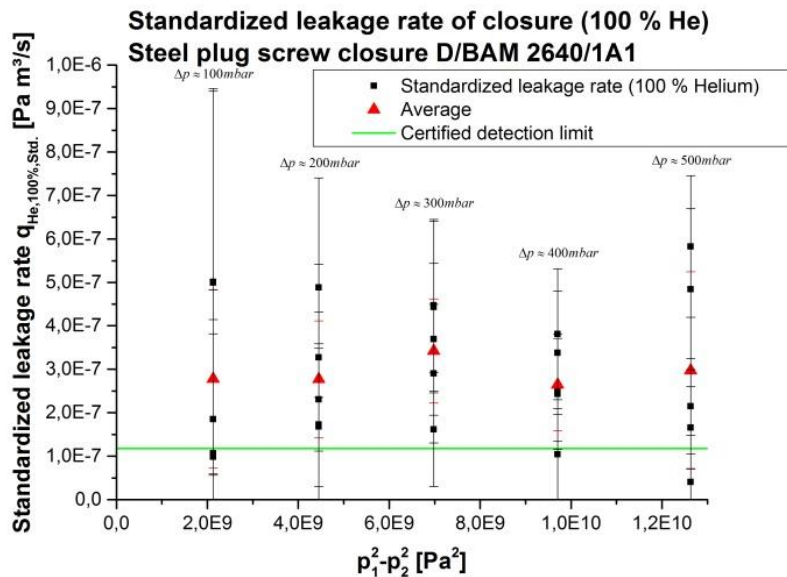


Fig. 10. Leakage rates (100 % Helium) for the steel plug screw closure of the 6 l steel drum.

The average values per pressure level are only slightly higher than the certified detection limit of the experimental setup. Compared to the calculated limit values for 300 mbar (see paragraph 1.1), the measured values are much smaller. Therefore, no explosive atmosphere can be created for this scenario under conditions of carriage.

3. Summary

Quantitative leakage rates through closures of dangerous goods packagings can be measured using the pressure technique by accumulation. This allows a clear assessment if there is the risk of the formation of an explosive atmosphere under conditions of carriage.

The successful application of a quantitative leakproofness test requires essential steps and preliminary tests.

4. Symbols

Symbol	Description	Unit
A	Area	m ²
c	Volume concentration	ppm
D_{He}	Diffusion coefficient of Helium	m ² /s
L	Length	m
n	Air change rate	s ⁻¹
n_{He}	Helium loss rate	s ⁻¹
P	Permeability coefficient	m ² /s
p	Absolute pressure	Pa
q_{He}	Helium leakage rate	Pa m ³ /s
$q_{He,100\%}$	Helium leakage rate (100 % Helium)	Pa m ³ /s
q_{perm}	Permeation leakage rate	Pa m ³ /s
t	Time	s
t_{ind}	Induction time ("time lag")	s
\dot{V}_L	Air flow	m ³ /s
$V_{chamber}$	Free chamber volume	m ³
V_R	Zone volume	m ³

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