The Use of Different Media in Leakage Tests

By Dr.-Ing. Manfred Schaaf and Stefan Hufnagel, Amtec North America, Inc.

Previously, in Europe, nitrogen was mainly used as the test medium in gasket testing. This medium was easy to handle, safe and cheap to buy. With increased requirements on sealing elements, however, a test medium with which these small leakage quantities can be reliably measured must be used. In laboratory testing, helium prevailed, by using a mass spectrometer these tightness requirements could be easily achieved. Other media, such as hydrogen and methane, were not used in standard tests because of their hazardous potential and detection limit.

Particularly in performance tests, a practice-oriented medium has always been favored in sealing tests in the USA. Methane is the first choice as the smallest hydrocarbon molecule. In ASME B16.20, a new performance test has been introduced, in which the tightness requirement of spiral wound gaskets in a leakage test with methane shall be checked.

Efforts are currently underway to establish hydrogen technologies as core elements of the energy transition to decarbonize production processes with the help of renewable energies. However, the use of hydrogen in industrial plants also places new demands on sealing technology. The tightness of the sealing elements when using hydrogen must be systematically examined for this purpose.

In this article, the difference in leakage testing between helium, methane, nitrogen, and hydrogen, in terms of use in the test laboratory, the measurement of leak rates and the detection limits between helium and methane, will be shown.

1. Conventional Standards for Gasket Leakage Measurements
The most common test procedures for leakage tests are the ones defined in DIN 3535, EN 13555, VDI 2440, ASTM F37, and ASTM ROTT. Since 2017, the performance test defined in ASME B16.20 is also very prominent.

In DIN 3535, the leak rate is measured at a defined stress level (32 MPa) and internal pressure (40 bar). The test medium used in DIN 3535 is nitrogen. There are several reasons for this specification; one is today´s specification of a high acceptable leak rate of 0.1 mg/m/s, and another is the date of the first issue of this standard (1986). At this time, all gasket materials were tested with nitrogen, and to achieve a good comparability of all the results, nitrogen is still used in this test procedure. In earlier days, the leak rate was measured with a gas burette or by the pressure drop method. Today, the differential pressure method is the most common measurement method used in this test procedure due to its higher accuracy.

The test methods in ASTM F37 are suitable for evaluating the sealing properties of gasket materials; a procedure for liquid leakage and gas leakage measurement is defined in this standard. For the liquid leakage measurement, Reference Fuel A is recommended, but nitrogen is the medium for the gas leakage measurement. The leak rate is measured in a very simple way by the change in the level of a sight-glass or of a water manometer. Within EN 13555, there are several test procedures defined. One of these is a set of leakage tests performed at several gasket stress levels, with one internal pressure which can vary from test to test. The stress is applied in loading and unloading sequences so that the sealing behavior of the gaskets in real applications can be simulated. Because of the goal to measure low leak rates, helium is used as the test medium, and the helium mass spectrometer was introduced into the sealing technology.

The standardization work item ASTM WK 10913, which has been known for decades as ROTT (Room Temperature Tightness test), is now approved as ASTM F2836-18. About 20 years ago, there was an attempt to harmonize ASTM and EN gasket testing procedures. As a result of this effort, the ROTT and the EN 13555 leakage measurements are similar relating to gasket stress levels, test medium and the required analyzer.

For the qualification of gasket materials as a high-grade sealing element in the first-time test of VDI 2440, the assembled bolted flange connection test fixture (DN40/PN40) is aged at the sealing element´s maximum service temperature. The test fixture´s assembly gasket stress is 30 MPa, related to the dimension of sheet gaskets. Following the aging step, a tightness test is carried out with helium at 1 bar internal pressure and the leak rate is determined by means of mass spectrometry.

The performance test method in ASME B16.20 uses methane as a test medium. For this procedure, a single gasket stress and test medium pressure are defined according to the class rating of the gasket sample. In all cases, the gasket is loaded to the defined stress, filled to the defined pressure, and the leakage is measured. Regardless of the stress/pressure combination, the maximum permissible leak rate is 0.0137 mg/(s•m) of methane with respect to the outside circumferential length of the gasket. Measurement of the methane leakage may be achieved with a flame ionization detector for hydrocarbons.

Standardization for the evaluation, testing and approval of gaskets for use with hydrogen is currently lacking. This places a significant burden on the gasket manufacturer to confirm that the products are suitable for the use in hydrogen services. There is no clear guideline or method that gives the manufacturer safety.

2. Flow Models

Table 1 summarizes the currently identified gas flow models. The density of the gas flow is described by the Knudsen-Number, Kn. At very small Knudsen numbers, laminar flow occurs, while at very high numbers, the flow is molecular. A detailed description can be found in EN 1591-1, Annex I.

Knudsen Number K_n	Type of Flow	Calculation Formula
$K_n \ll 1$	Laminar	$\dot{m} = \frac{M}{RT}\frac{nr^4\pi}{16\eta L}\left(p_i^2 - p_o^2\right)$
$1 \leq K_n \leq 100$	mixed	$\dot{m} = \left(\frac{nr^4\pi}{8\eta}\bar{p}\frac{M}{RT} + \frac{4}{3}\psi\sqrt{\frac{2\pi M}{RT}}nr^3\right)\frac{\Delta p}{L}$
$K_n \gg 100$	molecular	$\dot{m} = \frac{4nr^3}{3L}\sqrt{\frac{2\pi M}{RT}}\Delta p$

$K_n = l/d$	Mean free path length / capillary diameter	R	Universal gas constant
$\dot{m}$	Mass flow (Leak rate in gaskets)	T	Absolute temperature
ψ	Adzumi constant	M	Molar mass
η	Dynamic viscosity	n	Number of capillaries
$p_i - p_o$	External – , internal pressure	r	Radius of capillaries
$\bar{p}$	Mean pressure	L	Length of capillaries
Δp	Pressure difference		

TABLE 1: Type of flow depending on the Knudsen-Number, Kn

Table 2 provides the molar mass and dynamic viscosity of nitrogen, helium, methane and hydrogen, which are required to calculate the factors to convert a measured leak rate for a medium X to a leak rate of another medium Y.

	dynamic viscosity µPa · s	molar mass g
N_2	17,1	28
He	19,7	4
CH_4	10,8	16
H_2	8,6	2

TABLE 2: Molar Mass and Dynamic Viscosity of different test medias

Table 3 provides the calculated factors for the conversion of leak rates using the visco-laminar flow. Table 4 includes the leak rate factors using the molecular flow.

		Actual Test Gas			
		N_2	He	CH_4	H_2
Reference Gas	N_2	1,00	0,87	1,58	1,99
	He	1,15	1,00	1,82	2,29
	CH_4	0,63	0,55	1,00	1,26
	H_2	0,50	0,44	0,80	1,00

TABLE 3: Conversion factor using the laminar flow

		Actual Test Gas			
		N_2	He	CH_4	H_2
Reference Gas	N_2	1,00	0,38	0,76	0,27
	He	2,65	1,00	2,00	0,71
	CH_4	1,32	0,50	1,00	0,35
	H_2	3,74	1,41	2,83	1,00

TABLE 4: Conversion factor using the molecular flow

3. Leakage Measurement Methods

Depending on the expected amount of leakage and the test medium being used, several leakage measurement methods are available. The most common methods, described below, are:

- Differential Pressure,
- Helium Mass Spectrometer,
- Flame Ionization Detector, and
- Hydrogen Mass Spectrometer.

3.1 Differential Pressure Method

The schematic shown in Figure 1 shows the components used in the differential pressure method of leakage measurement. When using this method, two volumes are needed; the measuring volume (comprised of the volume between the filling valve 1 and the test sample) and the reference volume (comprised of a metallically sealed volume). The differential pressure meter is built between these two volumes.

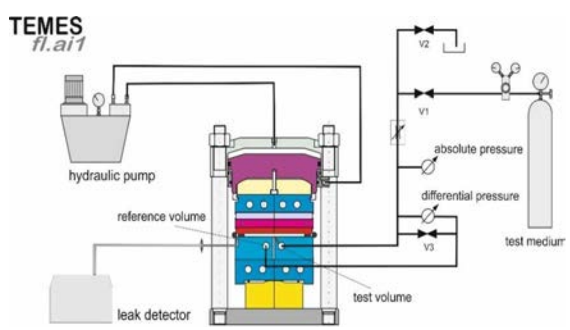

FIGURE 1: DIFFERENTIAL PRESSURE SYSTEM AND HELIUM MASS SPECTROMETER

The two volumes are separated at the start of measuring via the closing of valve 3. The differential pressure at this time/point equals 0 mbar. A leakage from the test sample generates a pressure loss of the measuring volume against the metallically sealed reference volume. The leak rate is calculated from the measured differential pressure with the gas equation, as the test volume, pressure, and temperature as well as the density of the gas and the measuring time are all known. The differential pressure method has a resolution range of 10 4 mg/m/s, referencing the average circumference of the gasket.

3.2 Helium Mass Spectrometry Method
The helium mass spectrometer works by creating a vacuum around the test specimen and measuring the helium that leaks from the sample into the vacuum, as seen in Figure 1 and in Figure 2. Therefore, the leak detector is connected via its inlet to the leakage plate.

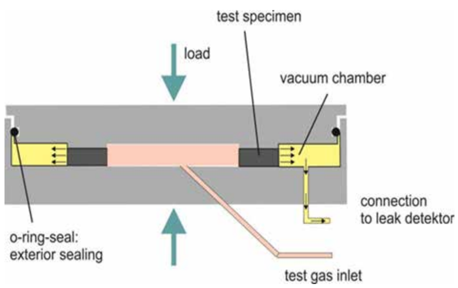

FIGURE 2: VACUUM CHAMBER FOR HELIUM LEAK DETCTOR

The outside area of the gasket is sealed from the outside environment by a rubber O-ring and evacuated by the turbo molecular pump in the leak detector. When helium leaks from the sample, it diffuses through the vacuum created by the turbo molecular pump and is measured by the leak detector to provide real-time leakage data. The measuring range of the leak detector is between 10-10 mbar*l/s and 10-1 mbar*l/s. The leak rate can be converted into a mass flow in mg/m/s using the gas equation, as the molar volume and the density of the gas are both known.

3.3 Flame Ionization Detector Method
With the flame ionization detector (FID), a direct measurement of hydrocarbon concentration in the flow of the flushing gas can be performed. Its connection to the test sample is similar to that of the helium leak detector and can be seen in Figure 3.

The FID provides accurate measurement of concentrations from a few parts per million (ppm) up to 100% by sampling air flow (flushing gas) from the perimeter of the test sample. This flushing gas through the test chamber must be provided by a bore hole within the lower leakage plate. This bore hole is placed at the side opposite of the plate connection to the inlet of the FID. The gasket's methane leakage (both face leakage and permeation through the material) is then introduced into the flushing gas and measured by the FID. The measuring range of the FID is between 0 ppm and 100,000 ppm. The concentration of methane in the flushing gas can be converted into a mass flow in mg/m/s, if the density of the gas and the flow rate of the flushing gas are known.

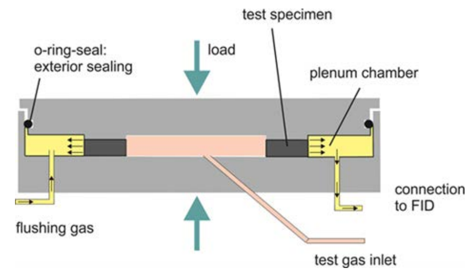

FIGURE 3: FLAME IONIZATION DETECTOR

3.4 Hydrogen Mass Spectrometer
The hydrogen mass spectrometer is simply adjusted from the helium mass spectrometer. For this method, a calibration gas is needed, for example, 50 ppm hydrogen. To measure with hydrogen, you must not only adjust the calibration curves, and media properties within the tool, but you must also make sure your test fixture is suitable for hydrogen with regards to having the proper pipes, fittings, and valves. It is also important for the hydrogen mass spectrometer to take into consideration the measurement time to reach a constant leak rate. As far as safety requirements go, some adaptations must be made when switching to measurement with hydrogen.

The measuring range of the leak detector for this method is between 10-7 mbar*l/s and 10 - 2 mbar*l/s. The leak rate can be converted into a mass flow in mg/m/s using the gas equation, as the molar volume and the density of the gas are both known.

4. Laboratory Requirements
Certain requirements for the environment of the laboratory must be met depending on the test media and/or the measurement method used. These requirements ensure a safe working environment and ensure the accuracy of the measurement method used. Nitrogen is a very popular test medium used in leakage tests across a variety of standards. With this medium, only the differential pressure method may be used. When using nitrogen, the oxygen concentration in the laboratory must be continuously monitored to ensure that it is at necessary levels for respiration of personnel. Nitrogen, from testing, may displace the oxygen in the laboratory if excess amounts have escaped during testing. Helium is another popular test medium and may be measured by either the differential pressure method, the mass spectrometry method, or a combination of the two. Similar to nitrogen, helium can displace oxygen in the laboratory if large amounts of helium escape. Therefore, an oxygen sensor should be deployed. Aside from displacing oxygen and the associated risks, a high concentration of helium in the lab can negatively affect the accuracy of the mass spectrometry measurement method. A high background concentration can make the measured leakage values of a test sample appear higher than they actually are. For these reasons, helium from leakage tests should be exhausted out of the lab to the outside environment. The use of methane and/or hydrogen as a test medium is gaining in popularity, and certain safety measures for its use must be taken. Sensors to measure the methane/hydrogen and oxygen mixture must be used to alert personnel if the oxygen levels are too low or if 20% of the lower explosion limit is reached. Furthermore, different federal, state or

local/municipal regulations may dictate how methane is able to be disposed of to the environment. For this reason, legal counsel must be consulted to ensure all applicable laws/regulations regarding the disposal of methane are adhered to.

5. Testing Results

In the following figures, the test results of an EN 13555 leakage measurement with different medias are shown.

In Figure 4, the test results obtained with different test media (methane, helium, and nitrogen), are summarized. The tests were carried out at 40 bar internal pressure for a spiral wound gasket with graphite filler. The leak rate measured with methane is, in most of the gasket stress levels, higher than the ones determined with helium and nitrogen. However, the difference between the leak rates measured with the three different media is less than an order of magnitude.

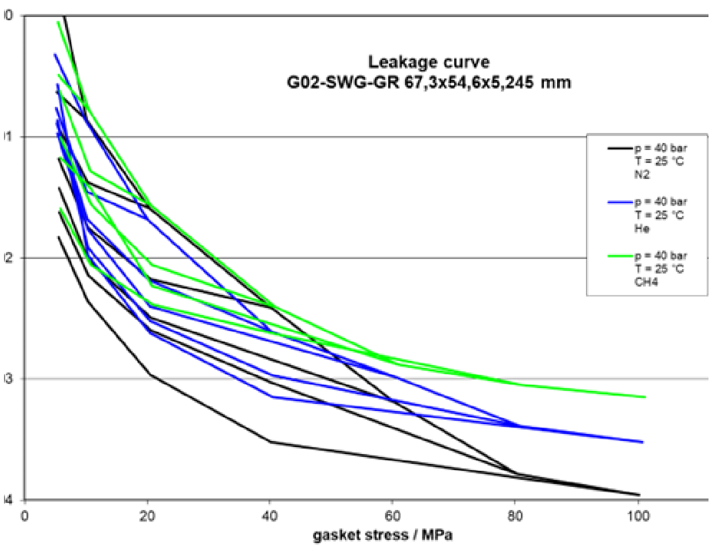

FIGURE 4: COMPARISON OF LEAKAGE TEST RESULTS OBTAINED WITH DIFFERENT TEST MEDIA (METHANE, HELIUM, NITROGEN)

Figure 5 compares the leak rates measured for helium and hydrogen at 40 bar internal pressure. At lower gasket stresses, hydrogen shows a higher leak rate than helium, and at higher gasket stresses, helium shows a higher leak rate.

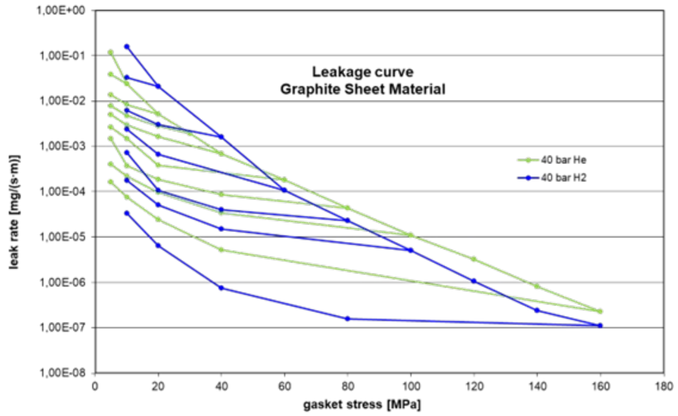

FIGURE 5: COMPARISON LEAKAGE TEST RESULTS OBTAINED WITH DIFFERENT TEST MEDIA (HELIUM, HYROGEN)

The conversion factor from He to H2 is 2.29 for laminar flow and 0.71 for molecular flow, see Tables 3 and 4. At low gasket stress, we see the laminar flow model, at high gasket stress, the molecular flow. This is proof that the theoretical conversion can't be used for the whole range of gasket stresses, and that the conversion factor is changing from low to high stresses, or throughout the course of a gasket test.

6. Conclusion

Changing sealing requirements in recent decades has led to new measuring methods in sealing technology. However, some of these measurement methods require the use of other test gases with new additional requirements.

Nitrogen continues to be the simplest test medium to use, but there is a limitation for low leak rates because no adequate detectors are available. Helium requires a tighter test setup because the measurement is sensitive to background concentrations. With helium, the lowest leak rates are detectable, but the helium mass spectrometer required to detect these low leak rates is the most expensive of the available analyzers.

Existing flow theories describe the leak rate through gasket materials only in a rough approximation. The effect of the gasket stress on the bolted flange connection, a critical parameter, is not directly considered in any of these flow models.