

A PASSION FOR PERFECTION

PFEIFFER  VACUUM

LEAK DETECTION

COMPENDIUM

Masthead

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Note

Leak Detection Compendium

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1 Leak Detection – Introduction

1.1 What Is A Leak? – Definition

A leak is defined as a structure in the wall of an object which is capable of passing gases or liquids from one side of the wall to the other. This structure can be a hole, porosity, permeable element or any other structure. Every technical object has such imperfections. For that reason we cannot expect that media transport through the wall is zero. Hence “tightness” or “leak-tight” are expressions which cannot be used absolutely – there is no such thing as an “absolutely tight” object. Leak tightness always refers to requirements of a certain machine, a specific system, or a respective product. Criteria for leak-tightness have to be defined and quantified on an individual basis for the respective needs.

Media transport through the wall can be described with a “leakage rate”. The commonly used expression “leak rate” can be understood as the frequency of occurrence of a leak. For that reason “leakage rate” is the correct technical expression which we are going to use in this booklet. It is defined as the throughput of a specific fluid which passes through a leak under specific conditions. Throughput can be expressed e.g. as mass loss per time or gas flow (amount of gas, described as product of pressure times volume per time).

Driving effects of media transfer can be pressure gradient or concentration difference across the wall.

So as described in the first paragraph we do not necessarily need a hole for media transport though a wall or a solid part. Gases can also be transported through a food packaging foil or the elastomer seal of a vacuum system. This transport mechanism is called “permeation”. Basically, permeation is composed of three steps:

1. Adsorption of a medium on the surface of the solid.
2. Diffusion through the bulk of the solid. Diffusion is a natural mixing process which lasts until there is no concentration gradient left. An example is mixing of a drop of ink in water. If you wait long enough there will be homogeneous blue color throughout the glass.
3. Desorption of the medium from the other surface of the solid.

Please see Figure 1-1 for a summary of interactions between the wall of a vacuum chamber and the respective gases.

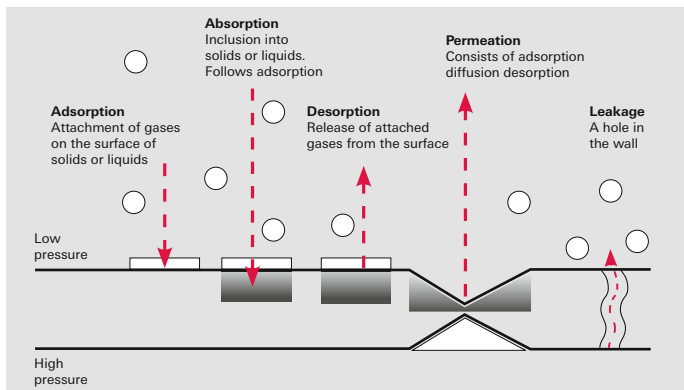


Figure 1-1: Interactions between the wall of a vacuum chamber and the respective gases

In case that the wall of a test object really has a small hole and there is a pressure difference from one side of the wall to the other we can observe media flow from the high-pressure side to the low-pressure side. Both liquids and gases can then flow through this hole.

The impacts of a leak can be very diverse. A leaky water tap in a private household is just nerve-racking with little consequences for function, environment, or waste of energy. However, if it is discovered that a water supplier in a big city loses 26 % of drinking water in his pipe network (which is a real case) then this is a massive waste of resources and money which has to be addressed. Discovering water droplets in your water-tight wrist watch is just awkward and means that you need to read the time through water bubbles in the future. Penetration of water into the igniter or gas generator of a car's airbag can lead to malfunctions and cost lives.

Massive leak impacts also can be release of corrosive or toxic substances. One of the most severe incidents after failure of a seal was the Space Shuttle *Challenger* disaster in January 1986.

Leaks can result in malfunctions or reduced lifetime of many technical products. Examples are:

- Refrigeration and air conditioning products in domestic and automotive applications
- Light-alloy rims in wheels
- Fuel and oil tanks
- Process equipment in chemical and pharmaceutical industry
- Condensers and steam lines in power plants

Leak detection of machines and production equipment often is the indispensable prerequisite for product quality.

1.2 How Large Is A Leak?

In order to get a feeling for diameters of leaks one can calculate the diameter of a leak assuming that it is an ideal tube with smooth walls.








| |  |  |  |  |  |  |  |
|--|---|---|---|---|---|---|---|
| Description | Technically tight | Gas tight | Virus tight | Bacteria tight | Water tight | Water dripping | Water running |
| Leak diameter | 0.1 μm | 0.8 μm | 3 μm | 10 μm | 30 μm | 100 μm | 1 mm |
| Leakage rate | 10^{12} Pa $\text{m}^3 \text{s}^{-1}$ | 10^{10} Pa $\text{m}^3 \text{s}^{-1}$ | 10^8 Pa $\text{m}^3 \text{s}^{-1}$ | 10^6 Pa $\text{m}^3 \text{s}^{-1}$ | 10^4 Pa $\text{m}^3 \text{s}^{-1}$ | 10^2 Pa $\text{m}^3 \text{s}^{-1}$ | 1 Pa $\text{m}^3 \text{s}^{-1}$ |
| Escape time of a bubble with 1 cc | > 1000 years | > 30 years | > 100 days | > 1 day | > 15 min | 10 s | 0.1 s |
| Leak location by sniffing | | | | | | | |
| Leak quantification under atmospheric conditions | | | | | | | |
| Local and integral vacuum test | | | | | | | |
| Test of sealed objects | | | | | | | |
| Test methods | | | | | Visual | | |
| | | | | | Ultrasonic | | |
| | | | | | Pressure decay | | |
| | | | | | Bubble test | | |
| | | Tracer gas at ambient pressure | | | | | |
| | Tracer gas in vacuum | | | | | | |

Figure 1-2: Magnitudes of leak size and leakage rate

From Figure 1-2 we can learn that the dimensions of most technical leaks are smaller than the optical resolution of the eye. So it is useless to try optical inspection once you have identified a leaking part by a tracer gas method.

On the other hand we can see that leak dimensions are at least 1,000 times bigger than the diameter of a tracer gas atom or molecule (which is in the 10^{-10} m range). This means any tracer gas can pass through a leak channel. We are going to see in the next chapter what the differences are with respect to different tracer gases.

2 Basic Leak Detection Theory

In the last chapter we have introduced “leakage rate” as throughput of a specific fluid which passes through a leak under specific conditions. Several questions arise from this definition:

- How is gas transported through the leak?
- What is the geometry of the leak?
- Which influence do “conditions” have?

The geometry of the hole is unknown in most cases. Test personnel does not know whether a leak channel is a circular hole with smooth walls or whether they have to deal with a crack, an open joint, or a gap. Calculation can only be made for ideal geometries. Since we do not know the real leak geometry we have to make assump-

tions. Calculations for classical flow regimes provide an upper limit for media transport through the hole.

Tracer gas throughput can occur in various different flow regimes:

- Laminar flow
- Transition or Knudsen flow
- Molecular flow

In laminar flow we have high pressure and high gas density with strong interaction between individual atoms or molecules of the tracer gas. So we have laminar transport through a leak. As a very basic role of the thumb this flow regime is dominant at leak rates higher than $10^5 \text{ Pa m}^3 \text{ s}^{-1}$.

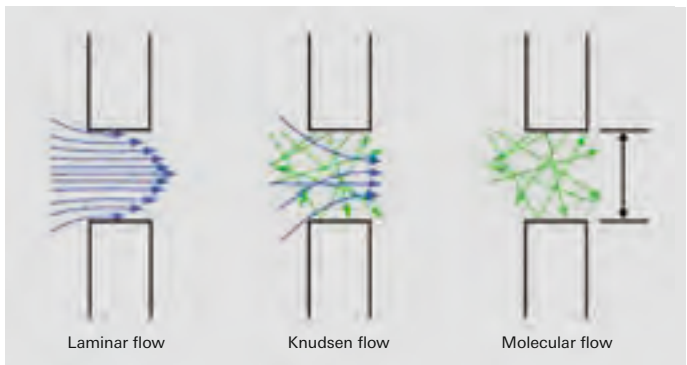


Figure 2-1: Flow regimes

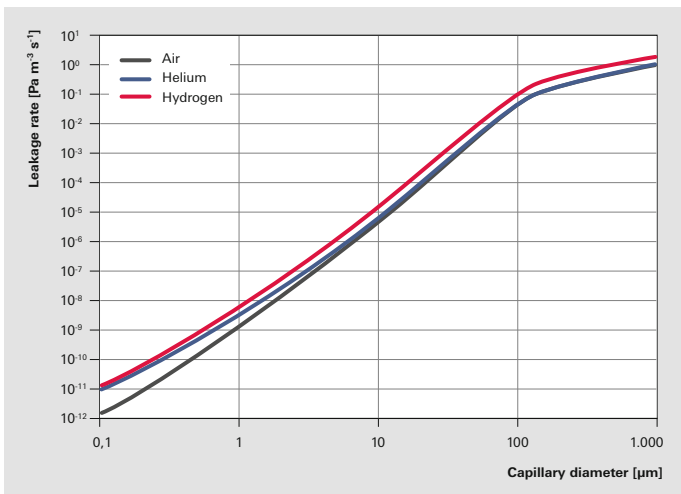


Figure 2-2: Transport of different media through a leak
(calculated for a temperature of 20 °C and a wall thickness of 1 cm)

In molecular flow we have low pressures and a low gas density. There are almost no interactions between individual atoms or molecules of the tracer gas. The molecular flow regime is dominant at leak rates lower than $10^{-8} \text{ Pa m}^3 \text{ s}^{-1}$.

The transition between laminar-viscous flow and molecular flow is not instantaneous. The transition regime is called Knudsen flow. If you are not certain, which flow regime you are in please calculate both alternatives and choose the worst case.

In Figure 2-2 you can see that the blue curve for helium correlates with the grey curve for air from very large leakage rates to roughly $10^{-5} \text{ Pa m}^3 \text{ s}^{-1}$. Then the helium curve

moves away and overlaps with the green curve for hydrogen at leak rates lower than $10^{-8} \text{ Pa m}^3 \text{ s}^{-1}$. This is a practical consequence of the aforementioned flow regimes. We shall show later how this can be described quantitatively (see 2.3.7 Leak Conversion Between Gases in Laminar Flow and the following chapters).

2.1 Mathematical Basics

Leak detection has to deal with a broad dynamic range of leakage rates. Data mostly are displayed as exponential numbers. Please see Table 2-1 for a compilation of relevant displays and prefixes.

| billion | | | million | hundred thousand | ten thousand | thousand | hundred | tens | single | tenth | | hundredth | thousandth | ten-thousandth | hundred-thousandths | millionth | | billionth | prefix | exponential | |
|---------|---|---|---------|------------------|--------------|----------|---------|------|--------|-------|---|-----------|------------|----------------|---------------------|-----------|---|-----------|--------|-------------------|-------------------|
| 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | | | giga | $1 \cdot 10^9$ | |
| | | | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | | | mega | $1 \cdot 10^6$ | |
| | | | | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | | | | | | $1 \cdot 10^5$ | |
| | | | | | 1 | 0 | 0 | 0 | 0 | | | | | | | | | | | $1 \cdot 10^4$ | |
| | | | | | | 1 | 0 | 0 | 0 | | | | | | | | | | kilo | $1 \cdot 10^3$ | |
| | | | | | | | 1 | 0 | 0 | | | | | | | | | | hecto | $1 \cdot 10^2$ | |
| | | | | | | | | 1 | 0 | | | | | | | | | | deca | $1 \cdot 10^1$ | |
| | | | | | | | | | 1 | | | | | | | | | | | $1 \cdot 10^0$ | |
| | | | | | | | | | 0, | 1 | | | | | | | | | deci | $1 \cdot 10^{-1}$ | |
| | | | | | | | | | 0, | 0 | 1 | | | | | | | | centi | $1 \cdot 10^{-2}$ | |
| | | | | | | | | | 0, | 0 | 0 | 1 | | | | | | | milli | $1 \cdot 10^{-3}$ | |
| | | | | | | | | | 0, | 0 | 0 | 0 | 1 | | | | | | | $1 \cdot 10^{-4}$ | |
| | | | | | | | | | 0, | 0 | 0 | 0 | 0 | 1 | | | | | | $1 \cdot 10^{-5}$ | |
| | | | | | | | | | 0, | 0 | 0 | 0 | 0 | 0 | 1 | | | | micro | $1 \cdot 10^{-6}$ | |
| | | | | | | | | | 0, | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | nano | $1 \cdot 10^{-9}$ |

Table 2-1: Numbers and prefixes

2.2 Units

We have already used it several times without definition: the SI leakage rate unit is "Pa m³ s⁻¹". If we analyze the unit we can find the following physical properties:

- Pressure [Pa]
- Volume [m³]
- Time [s]
- Amount of gas [Pa m³]
- Pumping speed [m³ s⁻¹]
- Gas throughput [Pa m³ s⁻¹]

So basically a leakage rate can be understood as a gas flow or a certain amount of gas which flows through a leak channel in a certain time. In order to describe the quantity of gas we need the product of pressure and volume since gases are compressible or can be expanded. This is the big difference to liquid media where volume alone is sufficient to describe a quantity.

All other leakage rate units are following the same concept. If we find a unit which is constituted e.g. of volume and time only, there is always a reference to "standard conditions". Those contain a pressure reference. According to this, we can convert any leakage rate units by converting the individual physical properties. Please see chapter 11 for a leakage rate conversion table.

2.3 Formulas for Leak Detection Calculations

2.3.1 Calibration

If a leak detector is operated in parallel to a roughing pump or a roughing group (e.g. a roots pumping group) one should always measure signal response time and partial flow with a calibrated leak. The correct location of the calibrated leak is shown in Figure 2-3. Comparison of the leakage rate value

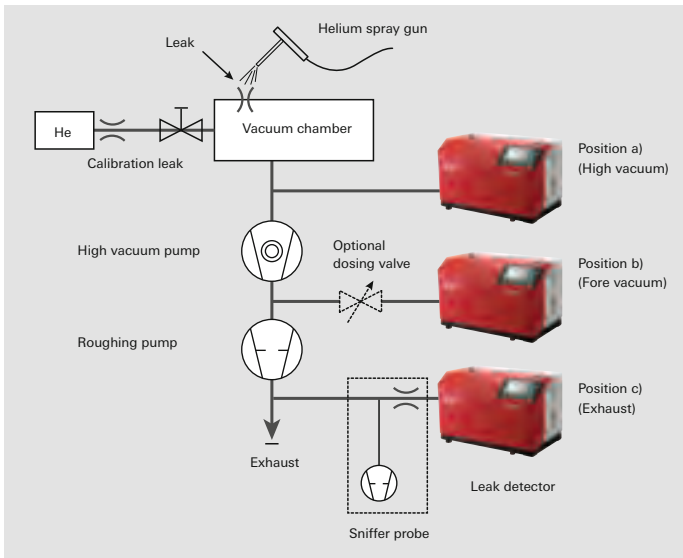


Figure 2-3: Possible connections of a leak detector on a vacuum system

directly measured on the leak detector and in the shown position gives precise information about the split of the tracer gas flow. Tracer gas flow is divided into two streams: one is transported to the leak detector and the other one is pumped away via the pumping system.

If over pressure is avoided in the line between calibration leak and valve a precise determination of the response time is also possible.

2.3.2 Pump Down Time

The pump down time in laminar-viscous flow regime is given in Formula 2-1.

$$t = \frac{V}{S_{eff}} \ln \frac{p_1}{p_2}$$

Formula 2-1: Pump down time

- t Pump down time
- V Volume
- S_{eff} Effective pumping speed
- p_1 Start pressure
- p_2 End pressure

The effective pumping speed is linear in the denominator. So a roughing pump in parallel operation to the leak detector as shown

in Figure 2-3 accelerates the pump down. Since particle transport and water vapor desorption are processes which occur at high pressures, a parallel pump also protects the leak detector, enhances its life time and prolongs the maintenance interval. Furthermore, maintenance on a vacuum pump normally is cheaper than maintenance on a leak detector.

2.3.3 Effective Pumping Speed

In a real pumping system the specified pumping speed of the pumps used is reduced due to flow resistances, the so-called conductance of installed components. The effective pumping speed is given in Formula 2-2.

$$\frac{1}{S_{eff}} = \frac{1}{S_{Nenn}} + \frac{1}{C}$$

Formula 2-2: Effective pumping speed

| | | |
|-----------|-------------------------|-------|
| S_{eff} | Effective pumping speed | [l/s] |
| S | Nominal pumping speed | [l/s] |
| C | Conductance | [l/s] |

Example: A turbopump with a pumping speed of 67 l/s is connected to a DN 63 flange on a vacuum chamber. It is supposed to act as a booster pump in leak detection (see 2.3.5 Signal Response Time). We want to isolate the pump with a valve. We can either choose an angle valve with a conductance of 160 l/s or a gate valve (which is more expensive) with a conductance of 550 l/s. Which valve should we choose?

With the angle valve we achieve an effective pumping speed of 47 l/s, meaning we are losing 30% of pumping performance. With the gate valve we can reach a nominal pumping speed of 60 l/s and we are losing 11% only. As a result, the more expensive gate valve is well worth its money.

2.3.4 Conductance in Series

In most cases more than one component is built in series between test object or chamber and the pumping system. As shown in Formula 2-3 individual conductance behaves like resistors in series. So the part with the smallest conductance determines the performance of the complete system.

$$\frac{1}{C_{Ges}} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_3} + \dots + \frac{1}{C_n}$$

Formula 2-3: Conductance in series

Example: You have a high-performance leak detector with a pumping speed at the inlet of 7 l/s. You connect it to a part you have manufactured and which is lying on a workspace. The part has a DN 16 flange connection with a DN 16 isolation valve. You connect it with a 2 m long tube DN 16 with you leak detector. The remaining conductance in molecular flow is:

$$\frac{1}{C_{Ges}} = \frac{1}{C_{valve}} + \frac{1}{C_{line}} + \frac{1}{5 \text{ l/s}} + \frac{1}{0.25 \text{ l/s}}$$

$$C_{ges} = 0.24 \text{ l/s}$$

According to Formula 2-2 the effective pumping speed of the leak detector is reduced from 7 l/s to 0.23 l/s and we maintain 3% of the original pumping speed with consequences for the response which are shown in paragraph 2.3.5 Signal Response Time.

There is nothing we can do about the valve. But we can connect a reducer directly after the valve to expand the diameter to DN 40. Also, we can try to place our leak detector closer to the part and save 1 m line length. With this set-up we can retrieve an effective pumping speed of 2.1 l/s and measure ten times faster than with the previous set-up.

Therefore, you should select a connection which is as short and thick as possible. If you need to compromise between length

and diameter of the connection, then chose the bigger diameter. Conductance depends linearly on the length but with power to 3 on diameter in molecular flow.

2.3.5 Signal Response Time

If a container is connected to a leak detector and pumped, there will be a time delay between tracer gas spraying and signal response. There are build-up time (or time constant), dead time, and a ramp-up time.

The build-up time or time constant is a time which elapses until a volume is filled with tracer gas to a certain equilibrium level. It occurs in every flow regime. Per definition the time constant of a vacuum system is the time when 63% of the equilibrium value is reached (see Formula 2-4).

$$\tau_{63\%} = \frac{V}{S_{\text{eff}}}$$

Formula 2-4: Signal response time

$\tau_{63\%}$ Time constant
 S_{eff} Effective pumping speed
 V Tested volume

Please note that the effective pumping speed is in the denominator. This explains the importance of the examples from the last paragraph. So if we assume a volume of 30 l and take the calculated effective pumping speed with the two different gas lines, we end up with the values in Table 2-2. So just by the selection of a line we can influence the time consumption of a measurement drastically.

| Component | Pumping speed/ conductance (DN 16) | Pumping speed/ conductance (DN 40) |
|-------------------------|--|--|
| Helium leak detector | 7 l/s | 7 l/s |
| Isolation valve DN 16 | 5 l/s | 5 l/s |
| Gas line DN 16, 2 m | 0.25 l/s | |
| Gas line DN 40, 1 m | | 7.7 l/s |
| Effective pumping speed | 0.23 l/s | 2.1 l/s |
| Signal response time | 131 s | 14 s |

Table 2-2: Example signal response time

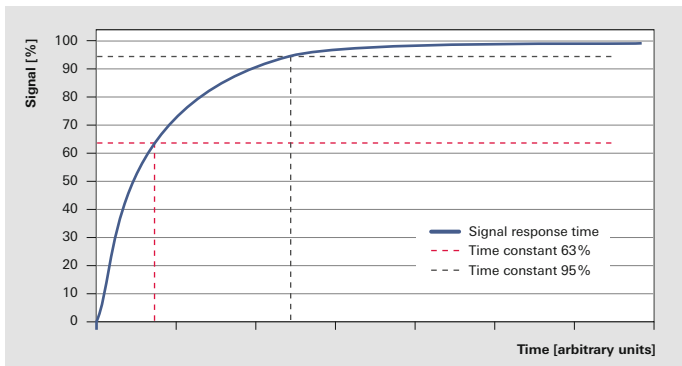


Figure 2-4: Signal response time

The exponential rise of tracer gas concentration can be modeled and the respective tracer gas concentration versus time can be calculated according to Formula 2-5.

$$q_{He}(t) = q_{He,end} \cdot [1 - e(-\frac{S_{eff} \cdot t_s}{V})]$$

Formula 2-5: Tracer gas partial pressure versus time

$q_{He,end}$ Stabilized leakage rate
 t_s Signal rise time

Theoretically there is only exponential convergence to the final value. However, after a time of 3 x the time constant $\tau_{63\%}$ a value of 95% is already reached.

Thus, a high vacuum pump in series operation to the leak detector accelerates the measurement due to its much higher effective pumping speed for the tracer gas helium.

Example: If a volume of 100 l is tested, the signal response time $\tau_{63\%}$ is 100 s if a leak

detector with an effective pumping speed of 1 l/s is used. This time constant can be reduced to one second if the leak detector is operated as the "backing pump" of a 100 l/s turbopump directly attached on the test object.

Working with turbopumps in series is a very effective tool to accelerate cycle time in industrial production or to test very large objects in high vacuum.

A dead time occurs only in laminar-viscous flow regime. As a first-order approximation it depends on the gas transfer length and the gas flow velocity of the tracer gas (see Formula 2-6).

$$t_0 = \frac{l}{v}$$

Formula 2-6: Dead time

t_0 Dead time
 v Gas flow velocity
 l Gas transfer length

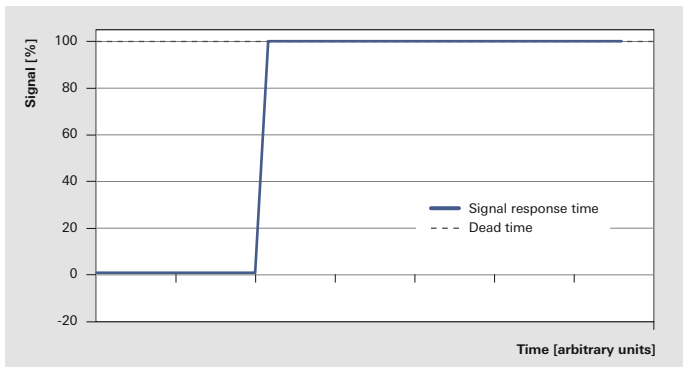


Figure 2-5: Dead time

Signal response is comparatively fast and with a steep slope, so signals can be identified easily even over a large background. Since a dead time occurs only in laminar-viscous flow regime, one can use it technically in order to increase test pressure. A transition from molecular flow to laminar flow is achieved in order to accelerate the test time. This is particularly helpful with conductance-limited parts like long tubes with a small diameter.

An induction time occurs if tracer gas needs to diffuse over a certain length without active gas transport to the leak detector. This can happen if a leak detector is connected to the tested object via a long tube at high pressure with a dosing valve just on top of the leak detector. In this case diffusion through the long tube is a time-consuming step and should be avoided, if possible. On the other hand there may be samples with several seals in series connected with small dead volumes each. Samples of that type are difficult to test and need long cycle times. For a part that needs to be tested in serial production this consideration should be made in the design phase already.

$$t_i = \frac{L^2}{2 \cdot D}$$

Formula 2-7: Induction time due to diffusion

- t_i Induction time
- L Permeation length
- D Diffusion coefficient

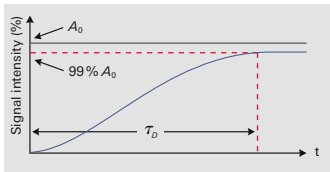


Figure 2-6: Induction time

The resulting response time profile is shown in Figure 2-6. In the initial test phase the signal rise is much slower than with a normal time constant. It is also important to notice that dependence on diffusion length is quadratic. Therefore, it is important to keep diffusion length short.

2.3.6 Permeation

Permeation is the transport process if gas does not penetrate through holes in a so-called “capillary leak” but penetrates through materials. Materials that are penetrable by gases can be elastomer seals, glues, quartz,... Permeation consists of adsorption on one side of the material, diffusion through the wall and desorption from the other side of the material. So there are subsequent interactions between gas and material which are strongly depending on the materials and gases used.

The leakage rate is proportional with the permeation area and pressure gradient. It decreases linearly with the permeation distance. The dependence on materials is expressed with a permeation coefficient.

$$q_{Gas}^{permeation} = P \cdot \frac{A}{L} \cdot (\rho_1 - \rho_2)$$

Formula 2-8: Permeation

- P Permeation coefficient
- A Cross-section area
- L Permeation length
- ρ Pressure

Example: If we a rebuilding a vacuum system with many flanges it is interesting to get an idea about the gas load by permeation through an o-ring. Let us take the chamber seal of a rectangular door with a length of 500 mm on each side. The diameter of the o-ring is 5 mm. This means we have a cross-section area of $A = 4 \cdot 500\text{mm} \cdot 5\text{mm} = 10,000\text{mm}^2 = 1 \cdot 10^{-2}\text{m}^2$

The permeation length is 5 mm and the pressure gradient is from atmospheric to vacuum.

The permeation coefficient can be found in several vacuum text books. For an FKM seal we can assume a value of $4 \cdot 10^{-3}$ mbar l/s mm/(m² bar). In the unit we have the dimensions of a gas flow in mbar l/s, a permeation length in mm, a permeation area in m² and the pressure gradient in bar. Many permeation coefficients have been published in pre-SI unit times. So we are going to use these units in our estimate.

$$P = 4.0 \cdot 10^{-3} \frac{\text{mbar} \cdot \text{l} \cdot \text{mm}}{\text{s} \cdot \text{m}^2 \cdot \text{bar}}$$

$$q_{\text{Gas}}^{\text{permeation}} = 4.0 \cdot 10^{-3} \frac{\text{mbar} \cdot \text{l} \cdot \text{mm}}{\text{s} \cdot \text{m}^2 \cdot \text{bar}} \cdot \frac{1 \cdot 10^{-2} \text{ m}^2}{5 \text{ mm}}$$

$$\frac{1 \cdot 10^{-2} \text{ m}^2}{5 \text{ mm}} \cdot (1 \text{ bar} - 0 \text{ bar}) = 8.0 \cdot 10^{-6} \frac{\text{mbar} \cdot \text{l}}{\text{s}}$$

$$q_{\text{Gas}}^{\text{permeation}} = 8.0 \cdot 10^{-7} \text{ Pa m}^3 \text{ s}^{-1}$$

Figure 2-7 shows that permeation coefficients (at 20 °C) through metallic walls are very small. With the exception of special hydrogen storage materials permeation, through metals does not affect the perfor-

mance of a vacuum system. The permeation gas load in a vacuum system is dominated by permeation through elastomer seals.

Gas permeation is an interesting parameter in the packaging industry. In many cases measurements are made with real material/gas pairs. However, it has been shown that helium can act as a fast modeling gas for fast measurements.

2.3.7 Leak Conversion Between Gases in Laminar Flow Regime

We can convert leak rates measured in a capillary leak from one tracer gas to another. In laminar flow regime the leakage rate of two gases are reversely proportional to their dynamic viscosities.

$$\frac{q_{\text{Gas1}}}{q_{\text{Gas2}}} = \frac{\eta_{\text{Gas2}}}{\eta_{\text{Gas1}}}$$

Formula 2-9: Leak conversion between gases in laminar flow regime

Example: What is the difference between a helium leakage rate and an air leakage rate in laminar flow regime?

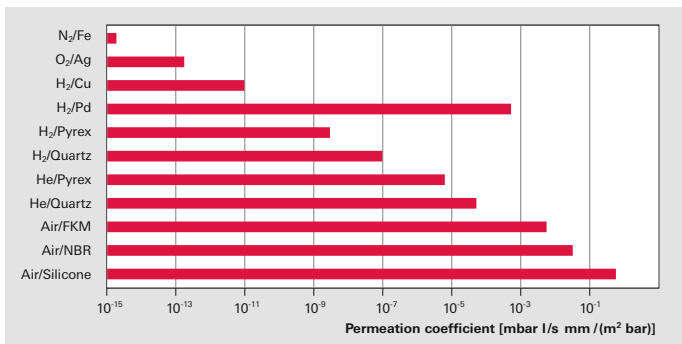


Figure 2-7: Permeation coefficients

| Medium | Name | Formula | Dynamic viscosity [10 ⁻⁶ Pa s'] | To convert from gas to helium multiply gas leakage rate with | To convert from helium to gas multiply He leakage rate with |
|-----------------|---------------------------|--|--|--|---|
| H ₂ | Hydrogen | H ₂ | 8.80 | 0.45 | 2.23 |
| He | Helium | He | 19.60 | 1.00 | 1.00 |
| NH ₃ | Ammonia | NH ₃ | 9.91 | 0.51 | 1.98 |
| N ₂ | Nitrogen | N ₂ | 17.48 | 0.89 | 1.12 |
| - | Air | N ₂ +O ₂ +Ar+CO ₂ | 18.19 | 0.93 | 1.08 |
| O ₂ | Oxygen | O ₂ | 20.24 | 1.03 | 0.97 |
| Ar | Argon | Ar | 22.11 | 1.13 | 0.89 |
| CO ₂ | Carbon dioxide | CO ₂ | 14.63 | 0.75 | 1.34 |
| SF ₆ | Sulfur hexafluoride | SF ₆ | 15.00 | 0.77 | 1.31 |
| R50 | Methane | CH ₄ | 10.87 | 0.55 | 1.80 |
| R134a | 1,1,1,2-Tetrafluoroethane | CH ₂ F-CF ₃ | 11.61 | 0.59 | 1.69 |

Table 2-3: Leakage rate conversion factors in laminar flow regime

$$q_{air} = q_{Helium} \cdot \frac{\eta_{Helium}}{\eta_{air}} = q_{Helium} \cdot \frac{19.6 \mu Pa s}{18.2 \mu Pa s} = 1.08 \cdot q_{Helium}$$

Table 2-3 summarizes several conversion factors between frequently used gases. Please observe that this conversion factors cover gas nature only but not variations in temperature or pressures. We are going to cover these aspects in the following chapters.

2.3.8 Leak Conversion Between Gases in Molecular Flow Regime

In molecular flow regime the leakage rate of two gases are reversely proportional to their molar masses.

$$\frac{q_{Gas1}}{q_{Gas2}} = \sqrt{\frac{M_2}{M_1}}$$

Formula 2-10: Leak conversion between gases in molecular flow regime

M molar mass

Example: What is the difference between a helium leakage rate and an air leakage rate in molecular flow regime?

$$q_{air} = q_{Helium} \cdot \sqrt{\frac{M_{Helium}}{M_{air}}} = q_{Helium} \cdot \sqrt{\frac{4}{29}} = 0.37 \cdot q_{Helium}$$

Table 2-4 summarizes several conversion factors between frequently used gases. Please observe that this conversion factors cover gas nature only but not variations in temperature or pressures. We are going to cover these aspects in the following chapters.

2.3.9 Leak Conversion Between a Liquid and a Gas

If we observe an amount of water (which can be described as a volume) which escapes from a leak per time, we can measure a water leakage rate. Observing pressure conditions and media viscosities we can calculate a tracer gas leakage rate.

$$q_{Gas} = \frac{\eta_{liquid}}{\eta_{gas}} \cdot q_{liquid} \cdot \frac{p_1 + p_2}{2}$$

Formula 2-11: Tracer gas leakage rate and liquid leakage rate

Example: A leaky water tap produces a bubble with a diameter of 4 mm every second. Pressure in the water supply line is 0.5 MPa. How big is the leakage rate with respect to the tracer gas helium?

| Medium | Name | Formula | Molecular mass [u] | To convert from gas to helium multiply gas leakage rate with | To convert from helium to gas multiply He leakage rate with |
|-----------------|---------------------------|--|--------------------|--|---|
| H ₂ | Hydrogen | H ₂ | 2.02 | 0.71 | 1.41 |
| He | Helium | He | 4.00 | 1.00 | 1.00 |
| NH ₃ | Ammonia | NH ₃ | 17.03 | 2.06 | 0.48 |
| N ₂ | Nitrogen | N ₂ | 28.01 | 2.65 | 0.38 |
| – | Air | N ₂ +O ₂ +Ar+CO ₂ | 28.96 | 2.69 | 0.37 |
| O ₂ | Oxygen | O ₂ | 32.00 | 2.83 | 0.35 |
| Ar | Argon | Ar | 39.95 | 3.16 | 0.32 |
| CO ₂ | Carbon dioxide | CO ₂ | 44.01 | 3.32 | 0.30 |
| SF ₆ | Sulfur hexafluoride | SF ₆ | 146.05 | 6.04 | 0.17 |
| R50 | Methane | CH ₄ | 16.04 | 2.00 | 0.50 |
| R134a | 1,1,1,2-Tetrafluoroethane | CH ₂ F-CF ₃ | 102.03 | 5.05 | 0.20 |

Table 2-4: Leakage rate conversion factors in molecular flow regime

Volume of the water drop:

$$V = \frac{4}{3} \cdot r^3 \cdot \pi = \frac{4}{3} \cdot 2^3 \cdot \pi = 33.51 \text{ mm}^3 = 3.35 \cdot 10^{-2} \text{ cm}^3$$

Water flow:

$$q_{\text{water}} = \frac{V}{t} = \frac{3.35 \cdot 10^{-2} \text{ cm}^3}{1 \text{ s}} = 3.35 \cdot 10^{-2} \text{ cm}^3 \text{ s}^{-1} = 3.35 \cdot 10^{-8} \text{ m}^3 \text{ s}^{-1}$$

For tracer gas helium and water we can write Formula 2-11 in the form:

$$q_{\text{Helium}} = \frac{\eta_{\text{water}}}{\eta_{\text{Helium}}} \cdot q_{\text{water}} \cdot \frac{\rho_1 + \rho_2}{2}$$

With the values above we achieve:

$$q_{\text{Helium}} = \frac{1,002 \cdot 10^{-6} \text{ Pa s}^{-1}}{19.6 \cdot 10^{-6} \text{ Pa s}^{-1}} = 3.35 \cdot 10^{-2} \text{ m}^3 \text{ s}^{-1}$$

$$\frac{0.5 \cdot 10^6 \text{ Pa} + 0.1 \cdot 10^6 \text{ Pa}}{2} = 0.60 \text{ Pa m}^3 \text{ s}^{-1}$$

2.3.10 Leaks Blocked by Liquids

A leak can be tight with respect to liquids in case of a small leak diameter and high surface tension of the respective fluid. Formula 2-12 shows the maximum diameter of a leak which is just tight for the respective medium. Through any larger leak the medium will escape.

$$d_{\text{max}} = \frac{4 \cdot \sigma \cdot \cos \phi}{\Delta p}$$

Formula 2-12: Leaks blocked by liquids

- σ Surface tension
- $\cos \phi$ Contact angle
- Δp Pressure gradient

Table 2-5 shows selected surface tensions of some media.

| Liquid | Formula | Surface tension at 298 K (σ in N m ⁻¹) |
|---------------------|--|--|
| Water | H ₂ O | 72.3 · 10 ⁻³ |
| AdBlue® | (CO(NH ₂) ₂); 35 % in H ₂ O | 1.4 · 10 ⁻³ |
| Benzene | C ₆ H ₆ | 28.9 · 10 ⁻³ |
| Ethanol | C ₂ H ₅ OH | 22.5 · 10 ⁻³ |
| R134a (refrigerant) | CF ₃ -CH ₂ F | 8.1 · 10 ⁻³ |

Table 2-5: Selected surface tensions

From the table we can clearly see that refrigerants have the lowest surface tension and will escape through leaks which still are blocked by water. Although AdBlue® (used in

combustion processes for selective catalytic reduction of NO_x) is just an aqueous solution of urea, its surface tension is more than 50 times lower than the one of water itself. Consequently different leakage criteria have to be applied.

Great care has to be taken when using the aforementioned values. Detergents may reduce surface tension drastically. Please observe that surface tension values are measured for defined surfaces. Surface tension of water on a steel plate is completely different from water surface tension on a hydrophobic plastic surface. So leakage rate specifications of "water tightness" will vary over a wide range and be strongly depending on materials and water additives.

Often we do not know the contact angle. With a conservative approach we can define $\cos \phi = 1$. This is the worst-case scenario, since it uses the upper limit of cosinus for our calculations.

Example: If we assume a pressure difference of 0.3 MPa we end up with a diameter of a little bit less than $1 \mu\text{m}$ for water. For AdBlue® the calculation yields less than $0.2 \mu\text{m}$. So AdBlue® will creep through holes which are blocked by water.

Now the question is whether we can still detect this leak with a tracer gas method. Let us consider the gas transport through a pipe according to the Hagen-Poiseuille Formula:

$$q_{pV, \text{la min ar}} = \frac{\pi}{8} \left(\frac{d}{2} \right)^4 \frac{\bar{p}}{\eta \cdot L} \cdot (\rho_1 - \rho_2) \text{ with } \bar{p} = \frac{(\rho_1 + \rho_2)}{2}$$

Formula 2-13: Hagen-Poiseuille Formula

If we insert the maximum diameter from Formula 2-12 into this general form of the Formula and insert the condition for \bar{p} we achieve:

$$q_{pV, \text{la min ar}} = \frac{\pi}{8} \left(\frac{d_{\text{max}}}{2} \right)^4 \frac{(\rho_1^2 - \rho_2^2)}{\eta \cdot L \cdot 2}$$

If we calculate with the aforementioned pressure gradient of 0.3 MPa, the dynamic viscosity for helium and if we assume a wall thickness of 1 mm, we end up at a leakage rate of $1.6 \cdot 10^{-7} \text{ Pa m}^3 \text{ s}^{-1}$. According to this, we could measure this leak with a standard helium leak detector without any problem. However, if we try to do so after a water pressure burst test, we will not be able to detect the leak since it is masked by water which is still in the leak channel.

2.3.11 Mass Loss Rates and Volume Leakage Rates in Laminar Flow Regime

Conversion of mass loss rates in pV tracer gas leakage rates is calculated in two steps. First the mass loss rate of the respective medium is converted into a pV leakage rate of the medium. In the second step, medium conversion is calculated according to the formulas valid for the respective gas flow regime. So we can either use formulas for laminar-viscous flow or molecular flow. In case that we do not know which flow regime we are in, we need to calculate both and assume the worse case.

Example: An air conditioning system must not lose more than 1 g refrigerant R134a per year in order to maintain functionality – what is the helium leakage rate at a temperature of 25 °C?

First step: Conversion of refrigerant mass loss in a refrigerant pV gas flow

$$q_{pV} = q_M \cdot \frac{R \cdot T}{M}$$

Formula 2-14: Conversion of mass loss in pV gas flow

| | |
|----------|-------------------|
| q_{pV} | Leakage rate |
| q_M | Mass loss rate |
| R | Gas constant |
| T | Temperature (abs) |
| M | Molar mass |

$$q_{pV, R134a} = \frac{1 \text{ g/a}}{365 \cdot 24 \cdot 3,600 \text{ s/a}} \frac{83.14 \text{ mbar} \cdot \text{l} \cdot \text{mol}^{-1} \cdot \text{K}^{-1} \cdot 298 \text{ K}}{102 \text{ g} \cdot \text{mol}^{-1}} = 7.7 \cdot 10^{-6} \text{ mbar l/s}$$

$$q_{pV, R134a} = 7.7 \cdot 10^{-7} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

Second step: Conversion of refrigerant pV gas flow into a helium pV gas flow (i. e. leakage rate).

$$q_{pV, \text{Helium}} = q_{pV, R134a} \cdot \frac{\eta_{R134a}}{\eta_{\text{Helium}}} = 7.7 \cdot 10^{-7} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1} \cdot \frac{11.6 \mu\text{Pas}}{19.1 \mu\text{Pas}} = 4.7 \cdot 10^{-7} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

2.3.12 Mass and Volume Leakage Rates in Molecular Flow Regime

In the example above we have assumed laminar flow regime. So what happens in molecular flow regime?

Example: An air conditioning system loses 0.01 g refrigerant R134a per year – what is the helium leakage rate under identical pressure conditions at a temperature of 25 °C?

$$q_{pV, \text{Helium}} = q_{pV, R134a} \cdot \sqrt{\frac{M_{R134a}}{M_{\text{Helium}}}} = q_{M, R134a} \cdot \frac{R \cdot T}{M_{R134a}} \cdot \sqrt{\frac{M_{R134a}}{M_{\text{Helium}}}}$$

$$q_{pV, \text{Helium}} = \frac{0.01 \text{ g/a}}{3.15 \cdot 10^7 \text{ s/a}} \frac{83.14 \text{ mbar} \cdot \text{l} \cdot \text{mol}^{-1} \cdot 298 \text{ K}}{102 \text{ g} \cdot \text{mol}^{-1} \cdot \text{K}} \cdot \sqrt{\frac{102}{4}} = 3.9 \cdot 10^{-7} \text{ mbar l/s}$$

$$q_{pV, \text{Helium}} = 3.9 \cdot 10^{-8} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

2.3.13 Standardized Leakage Rate – Pressure Dependence

A standardized leakage rate is the equivalent quantity of air which would flow through a measured leak over time under the following conditions:

- inlet pressure 1013.25 hPa
- exit pressure “0” hPa
- room temperature.

There is no pressure “0” Pa. This figure shall just indicate that the exit pressure is negligibly small against the inlet pressure. “Room temperature” also needs to be defined. There are standard definitions in the range between 20 °C and 25 °C. This difference

is comparatively small. However, there are also standard definitions which are defined at 0 °C (273 K). Before this leads to irritations all reference parameters should be agreed between supplier and customer.

One can calculate from operating conditions to a standardized leakage rate. Again, we have to distinguish between viscous (laminar) and molecular flow. For laminar and molecular flow the following equations are valid:

$$\frac{q_{\text{Gas, Operation}}}{q_{\text{Gas, Standard}}} = \frac{(p_{\text{Inlet, Operation}}^2 - p_{\text{Exit, Operation}}^2)}{(p_{\text{Inlet, Standard}}^2 - p_{\text{Exit, Standard}}^2)}$$

Formula 2-15: Standardized leakage rate – laminar flow regime

$$\frac{q_{\text{Gas, Operation}}}{q_{\text{Gas, Standard}}} = \frac{(p_{\text{Inlet, Operation}} - p_{\text{Exit, Operation}})}{(p_{\text{Inlet, Standard}} - p_{\text{Exit, Standard}})}$$

Formula 2-16: Standardized leakage rate – molecular flow regime

Example: A large container is pressurized to 30 bar (relative) in operation. The specified leakage rate under operating conditions is $10^{-4} \text{ Pa m}^3 \text{ s}^{-1}$. For safety reasons we do not want to perform the test at operational pressure but from atmospheric pressure against an evacuated chamber. These are the standard conditions as defined above.

Conversion of Formula 2-15 yields:

$$q_{\text{Gas, Standard}} = q_{\text{Gas, Operation}} \cdot \frac{(p_{\text{Inlet, Operation}}^2 - p_{\text{Exit, Operation}}^2)}{(p_{\text{Inlet, Standard}}^2 - p_{\text{Exit, Standard}}^2)}$$

Test conditions are from ambient pressure (1 bar) against vacuum (“0” bar). In operation relative pressure is 30 bar, which is an absolute pressure of 31 bar. The container is leaking against atmospheric pressure (1 bar), which results in the following numbers:

$$q_{\text{Gas, Standard}} = 10^{-4} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1} \cdot \frac{(1^2 - 0^2)}{(31^2 - 1^2)} = 1 \cdot 10^{-7} \text{ Pa} \cdot \text{m}^3 \cdot \text{s}^{-1}$$

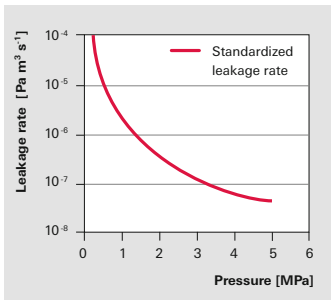


Figure 2-8: Dependency of standardized leakage rate of operational pressure in laminar flow regime

So the need for safety during a test means that we need to detect a signal which is 1,000 times smaller than the real leakage rate in operation. This concept of higher or lower test pressures often is used to optimize

- safety
- tracer gas consumption
- cycle time.

On the other way around you can e.g. increase test pressure and/or use gas mixtures to achieve short test times and low tracer gas consumption.

Figure 2-8 shows the dependency of the standardized leakage rate (assumed a measured leakage rate of $1.0 \cdot 10^{-4} \text{ Pa m}^3 \text{ s}^{-1}$) of operational pressure in laminar flow regime.

2.3.14 Temperature Dependence of Gas Transport through a Leak

Temperature dependence of gas transport through a leak can easily be derived from the ideal gas law. We achieve:

$$\frac{q_{\text{Gas, Operation}}}{T_{\text{Operation}}} = \frac{q_{\text{Standard}}}{T_{\text{Standard}}}$$

Formula 2-17: Temperature dependence of gas transport through a leak in laminar flow regime I

Conversion of Formula 2-15 yields:

$$q_{\text{Gas, Operation}} = q_{\text{Gas, Standard}} \cdot \frac{T_{\text{Operation}}}{T_{\text{Standard}}}$$

$$q_{\text{Gas, Standard}} = q_{\text{Gas, Operation}} \cdot \frac{T_{\text{Standard}}}{T_{\text{Operation}}}$$

Formula 2-18: Temperature dependence of gas transport through a leak in laminar flow regime II

Example: The leakage rate of a tube in an automotive application is defined at $1.0 \cdot 10^{-6} \text{ Pa m}^3 \text{ s}^{-1}$ for a maximum temperature of $120 \text{ }^\circ\text{C}$. The test is performed at ambient temperature of $20 \text{ }^\circ\text{C}$.

$$q_{\text{Gas, Standard}} = 1.0 \cdot 10^{-6} \text{ Pa m}^3 \text{ s}^{-1} \cdot \frac{293 \text{ K}}{393 \text{ K}}$$

$$= 7.5 \cdot 10^{-7} \text{ Pa m}^3 \text{ s}^{-1}$$

Please note that we need to insert absolute temperatures in [K] into the Formula. This results in comparatively small variations as a result of temperature changes. Formula 2-17 is valid for laminar-viscous flow regime. In molecular flow regime Formula 2-19 is valid.

$$\frac{q_{\text{Gas, Operation}}}{q_{\text{Gas, Standard}}} = \sqrt{\frac{T_{\text{Operation}}}{T_{\text{Standard}}}}$$

Formula 2-19: Temperature dependence of gas transport through a leak in molecular flow regime

2.3.15 Accumulation Testing

In chapter 6 we describe an integral test which is performed with a sniffer probe at atmospheric pressure, the so-called “accumulation test”. In case of a leak, the concentration in an envelope around the part to be tested can be described as follows:

$$q_{pV} = \frac{\Delta c \cdot 10^{-6} \cdot V_{\text{chamber}} \cdot p_{\text{chamber}}}{t}$$

Formula 2-20: Accumulation testing

| | | |
|----------------------|---|-------------------|
| Δp | Concentration rise of tracer gas in chamber | [ppm] |
| V_{chamber} | Net chamber volume | [m ³] |
| p | Chamber pressure | [Pa] |
| t | Time | [s] |

This concentration rise occurs on the natural background of the tracer gas used. With helium we have to deal with the natural background of 5 ppm in air. So we need to decide which concentration rise we define as unambiguous over the background signal.

Example 1: For a part to be tested the leakage rate of $1 \cdot 10^{-6} \text{ Pa m}^3 \text{ s}^{-1}$ was defined. Minimum time shall be determined which still allows process-safe measurement of a 5 ppm concentration rise. The test pressure is 4 bar (rel). The chamber pressure is atmospheric (100,000 Pa). If we assume a small part like a fuel filter, the net volume in the envelope, i.e. the volume of the empty chamber minus the volume of the part to be tested, is basically determined by the fan one needs for gas recirculation: let us assume a net volume of 100 cc (= 10^{-4} m^3).

$$t = \frac{\Delta c \cdot 10^{-6} \cdot V_{\text{chamber}} \cdot p_{\text{chamber}}}{q_{pV}}$$

$$= \frac{5 \cdot 10^{-6} \cdot 10^{-4} \cdot 1 \cdot 10^5 \text{ m}^3 \text{ Pa s}}{1 \cdot 10^{-6} \text{ Pa m}^3}$$

$$t = 5 \cdot 10^{-6-4+5+6} \text{ s} = 5.0 \cdot 10^1 = 50 \text{ s}$$

Hence, in this example we have to put atmospheric pressure of roughly 100,000 Pa in the numerator. The internal pressure of the tested object does only play a role as a driving force for gas to penetrate the leak from the interior of the tested object.

A typical graph of such a measurement is a linear curve starting from the tracer gas background level (see Figure 2-9).

Example 2: Let us assume that a manufacturer of tubular heat exchangers occasionally has to check integrity of tubes versus the shell. So in the manufacturing process the tubes are pressurized. The shell is not yet installed to maintain the opportunity of repair once a leak is discovered. Let us assume the pre-assembly is on trestles and the free volume is 5 m³. The part is covered with a plastic foil which is taped to the ground. A fan under the foil allows for homogeneous gas concentration. With an allowed leakage rate of $1 \cdot 10^{-4} \text{ Pa m}^3 \text{ s}^{-1}$ we get the following result:

$$t = \frac{\Delta c \cdot 10^{-6} \cdot V_{\text{chamber}} \cdot p_{\text{chamber}}}{q_{pV}}$$

$$= \frac{5 \cdot 10^{-6} \cdot 5 \cdot 1 \cdot 10^5 \text{ m}^3 \text{ Pa s}}{1 \cdot 10^{-4} \text{ Pa m}^3}$$

$$t = 2.5 \cdot 10^{1-6+5+4} \text{ s} = 2.5 \cdot 10^4 = 25,000 \text{ s} \approx 7 \text{ h}$$

In this case an accumulation test does not seem appropriate due to

- long time scale,
- danger of tracer gas loss by permeation through the foil,
- technical and handling limitations regarding foil and tape.

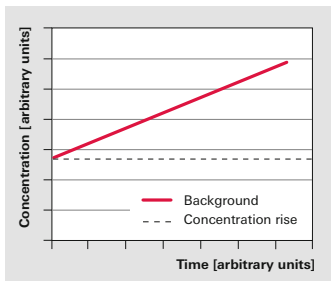


Figure 2-9: Accumulation test

3 Tracer Gases

3.1 Introduction

The simplest gas for leak detection is air. Compressed air is used for pressurizing samples and measuring pressure decay with a simple total pressure gauge. Air is penetrating a vacuum chamber and can be detected as a pressure rise. In processes at pressures below ambient with gas flow one can easily detect air leaks by mass spectrometry. We are going to explain this detector principle in chapter 4.

However, the examples above do not allow for leak location. The minimum detectable leakage rate is limited with pressure decay and pressure rise. In order to improve the sensitivity of leak detection we need to improve the selectivity for leak location and sensitivity for leakage rate measurements lower than achievable with e.g. pressure de-

cay. Possibly we also need a faster method than pressure decay with high throughput of parts to be tested. All these requirements lead the way to special tracer gases.

The requirements a tracer gas has to fulfill are:

- Low ambient concentration for good sensitivity in air
- High sensitivity detector technology
- Highly selective sensor technology
- Environmentally friendly
- Affordable
- Inert and nontoxic

These requirements are perfectly met with the dominant tracer gas helium. Over the past few years a mixture of 5% hydrogen and 95% nitrogen (also known as forming gas 95/5) has also reached some industrial acceptance. Table 3-1 indicates the pros and cons for each tracer gas.

| Requirement | Helium | Hydrogen |
|--------------------------------------|---|--|
| Low ambient concentration | 5 ppm in air | 0.5 ppm in air |
| High sensitivity detector technology | Mass spectrometry, $Q < 5 \cdot 10^{-13} \text{ Pa m}^3 \text{ s}^{-1}$ | Mass spectrometry, higher background than with helium transistor detector technology for sniffing only |
| Highly selective sensor technology | Mass spectrometry, no cross sensitivity on mass 4 u | Mass spectrometry, background on mass 2 u transistor detector sensitive to ambient hydrogen sources |
| Environmentally friendly | Noble gas, naturally in air, no green house potential | Naturally in air, no green house potential |
| Affordable | Medium gas cost | Low gas cost |
| Safe | Inert, non flammable, non explosive | Only in gas mixtures below lower explosion limit |

Table 3-1: Requirements of helium and hydrogen

Other tracer gases like refrigerants, SF₆, ammonia (NH₃), and several others are available. However, helium and to some minor extent forming gas 95/5 are the dominant tracer gases as the other gases have serious limitations.

3.2 Helium

Helium is the second most common element in the universe, representing about 23 % of the total matter. 76 % is hydrogen. All other elements represent an insignificantly small fraction of the total. Helium is a 100 % green gas and has absolutely no environmental impact on the atmosphere. Helium was discovered by spectroscopy in a solar eclipse on August 18, 1868. The discovery in the sun's chromosphere gave the new element its name: "helios" in Greek means "sun". While helium is very common in the universe most of it is in the stars: on earth it is actually not abundant. Since it is so light all the helium present during the formation of earth escaped to space.

Helium is created deep in the earth from the radioactive decay of uranium and thorium. On earth helium was discovered in 1881 by spectroscopy of Mount Vesuvio in Italy – the volcanic gases emanated by the mountain showed the same lines in the spectrum as already known from the sun. Helium concentration in the atmosphere is 5 times higher than the concentration of Krypton and 60 times higher than Xenon. The heavier noble gases are isolated from air rectification. In contrary, helium is "mined" from natural gas and oil wells and stored. Natural gas wells with a sufficient helium concentration for economic helium isolation are located in the USA, Russia, Algeria, Quatar and Poland. The annual world wide production is roughly 2.8 · 10⁷ m³ or 4,500 tons. Helium is constantly seeping up from the ground all around us, but it is so light that almost all of

it escapes into space fairly rapidly. There is a constant flow of helium from space and the sun to earth. This gives a dynamic equilibrium and is the reason for the world wide constant concentration of 5 ppm helium in ambient air. Helium is a very light colorless element and it is one of the six noble gases which means it does not react with anything for all practical intents and purposes.

Helium is used for the following applications:

- Protective gas for welding and cutting
- Filling (resonator) gas in lasers
- Carrier gas in gas chromatography
- Diving gas mixtures
- Cooling, e.g. during production of fiber optics
- Balloon filling gas
- Superconducting magnets (in liquid form)
- Low temperature physics (liquid)
- Nuclear magnetic resonance (liquid)
- Tracer gas in leak detection (12 % of annual Helium consumption)

Helium is commercialized in many different purity levels from ballooning gas with roughly 90 % of helium up to a purity of 6.0, which is 99.9999 % of helium. So which purity levels should one use for leak detection? Highest purity is rarely used in leak detection. This only makes sense e.g. for nuclear applications in order to avoid unwanted fission products or in UHP (Ultra High Purity) grade gas supply lines which are pre-treated by purging with ultra-pure argon and which are also particle-tested. Do not forget the influences of tubing and pressure reducer in these applications!

For standard industrial applications purity in the range of 97 % to 99 % is more than adequate. There is absolutely no risk of accuracy loss or contamination for the leak detector by using standard purity level of helium gas.

3.3 Hydrogen

Hydrogen (H₂) is the lightest element. It is a colorless, odorless, tasteless, flammable gas found at concentrations of about 0.5 ppm in the ambient air. Hydrogen is produced by several methods, including steam/methane reforming, dissociation of ammonia and recovery from by-product streams from chemical manufacturing and petroleum reforming. Hydrogen can be stored and transported either as a gas or a cryogenic liquid. Hydrogen is flammable in the concentration range 4 % to 77 % in air or oxygen and can detonate in the range 18 % to 60 % in air or oxygen. If hydrogen (H₂) and oxygen (O₂) are mixed and heated, they react and

create water vapor (H₂O). During this process more heat is generated which may (if the concentration is high enough) ignite the surrounding gas. If this process propagates, the gas explodes. At low concentration of hydrogen (< 4 % in air) the generated heat is not enough to ignite the surrounding gas. At concentrations in the range 4–12 % the combustion may spread only if actions are taken to prevent the generated heat from dissipating. It is a common misconception that hydrogen will explode as soon as the concentration exceeds 4 %. It may explode only if conditions are favorable for spontaneous propagation of combustion. When concentration exceeds 18 %, the combustion can spread by itself quickly in the gas. If it

| | Helium | Hydrogen | Forming gas 95/5 | Air |
|--|--|--|--|---|
| Natural concentration in air | 5 ppm | > 0.5 ppm | – | – |
| Molecular mass | 4 g mol ⁻¹ | 2 g mol ⁻¹ | 29 g mol ⁻¹ | 29 g mol ⁻¹ |
| Dynamic viscosity at 20 °C | 19.1 µPa s | 8.9 µPa s | 17.5 µPa s | 18.0 µPa s |
| Diffusion coefficient in air | 7.1 · 10 ⁻⁵ m ² s ⁻¹ | 7.2 · 10 ⁻⁵ m ² s ⁻¹ | – | 2.2 · 10 ⁻⁵ m ² s ⁻¹ (nitrogen) |
| Permeation coefficient in Silicone | 25,000 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ (1) | 50,000 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ | – | 12,000 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ (nitrogen) |
| Permeation coefficient in NBR | 900 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ | 1,200 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ | – | 90 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ (nitrogen) |
| Permeation coefficient in polyamides | 50 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ | 144 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ | – | 2 · 10 ⁻¹⁰ mbar l s ⁻¹ cm cm ² bar ⁻¹ (nitrogen) |
| Detector principles (2) | Mass spectrometry Quartz window sensor | Mass spectrometry transistor (MOSFET) | Mass spectrometry transistor (MOSFET) | Mass spectrometry Total pressure gauges |
| Availability of calibrated leaks | + | + | + | Not applicable |
| Lower explosion limit (concentration in air) | Not applicable | 4 % | Not applicable | Not applicable |
| Upper explosion limit (concentration in air) | Not applicable | 77 % | Not applicable | Not applicable |

Table 3-2: Selected physical properties of tracer gases

- (1) The unit describes a gas flow in mbar l s⁻¹ over a wall thickness in cm on an area in cm² and with a differential pressure in bar.
- (2) Please see chapter 4 for details of detector principles

propagates faster than the speed of sound then we have a detonation, a bang.

At hydrogen concentrations higher than 75%, there is not enough oxygen left for the gas to ignite. This is the upper explosion limit. If you use the recommended tracer gas of 5% H₂ / 95% N₂ and mix it with air there will either be too little hydrogen or too little oxygen to constitute a combustible gas mixture. Hence this gas mixture is classified as non-flammable.

3.4 Physical properties of tracer gases

Many applications in leak detection require calculations to optimize the test recipe for minimum tracer gas consumption, measurement of the required threshold in a certain time, and economics reasons. Please find in Table 3-2 some physical properties of tracer gases.

4 Sensor Technologies

Pfeiffer Vacuum uses three main detector technologies for leak detection in own products:

- Magnetic mass spectrometer
- Quadrupole mass spectrometer
- Quartz window sensor

4.1 Mass Spectrometric Analyzers

Mass spectrometry is one of the most commonly used analytical detector principles. Mass spectrometers analyze the constitution of chemical substances by partial pressure measurement in vacuum.

Partial pressure is the pressure that would be exerted by a gas or vapor if it alone was

present in an enclosure. Let us take air as an example: Air is a mixture of many gases. The most abundant one is nitrogen with a concentration of 78.1%. So if one would remove all other gases from air at a pressure of 1,000 hPa, the remaining nitrogen would exert a pressure of 781 hPa. By the way, this is how nitrogen was discovered in the late 18th century: by removal of oxygen by combustion and removal of gaseous reaction products. The remaining gas was mainly nitrogen.

Let us take another example: Helium as a commonly used tracer gas in leak detection has a concentration of 5 ppm ("parts per million") in air which results in a partial pressure of $5 \cdot 10^{-3}$ hPa.

Mass spectrometers are used for a large variety of analysis and process control.

Research and Development

- Catalytic Research
- Pharmaceutical development
- New materials

Process Control

- Metallurgy
- Chemical synthesis
- Semiconductor production
- Surface analysis

Doping control and environmental monitoring

- Aerosols
- Combustion gases
- Doping control
- Forensic analytics
- Isotop analysis for age determination and origin determination

Product analysis

- Chemical industry
- High purity gases
- Pharmacy
- Food monitoring
- Leak detection

In this booklet we are going to focus on leak detection applications.

A mass spectrometer analyses gaseous components. Solid or liquid media also can be analyzed if they are evaporated in an inlet system which vaporizes the respective media. Evacuation reduces total pressure and gas density to molecular flow regime. Then the gaseous mixture is ionized by electron bombardment. It is necessary to ionize the gas since mass separation is performed in an electrical or a magnetic field. We can deflect ionized, i.e. charged particles in an external field, but not neutral ones. In a following step the produced ions are separated in a mass filter by their mass-to-charge ratio.

Mass and Charge

- Total pressure is the sum of all partial pressures of a given gas mixture.
- In order to determine the partial pressure of a certain gas component it has to be isolated from the mixture.
- Prior to detection, separation of the gas mixture and isolation of the respective component is required.
- Isolation is made according to the mass-to-charge ratio of the respective component.

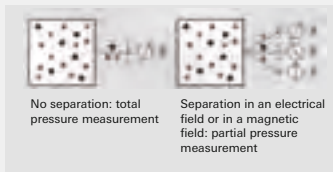


Figure 4-1: Total pressure measurement and partial pressure measurement

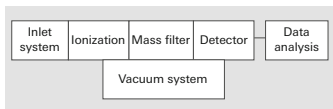


Figure 4-2: Components of a mass spectrometer

Figure 4-2 shows typical components of a mass spectrometer.

- The inlet system allows for introduction of the media to be analyzed. This can occur by a capillary or a dosing valve into a vacuum chamber.
- A vacuum system reduces the pressure of the introduced media to working pressure of the analyzer system and maintains it.

Analysis is made in a detection system under vacuum. We need vacuum for the following reasons:

- A heated wire, the so-called filament which is used for ionization would burn after a short period of operation at ambient air. This is due to the oxygen in air. So we need to remove the oxygen by pump down.
- At ambient pressure we have a particle density of more than $2.7 \cdot 10^{19}$ (27,000,000,000,000,000,000) particles per cm^3 (cubic centimeter). Due to this high density a particle would collide with another particle after a distance of only $6.8 \cdot 10^{-9}$ m (6.8 / 10,000,000 m). Under these conditions the medium that we want to analyze would never reach the detector. So we need to reduce the particle density (in other words the pressure) to a level which allows for a traveling distance of the particle (the so-called mean free path length) which is larger than the dimensions of the analytical device. This is achieved at a pressure level of roughly $1 \cdot 10^{-3}$ hPa.

Please see our Vacuum Know How Book and references therein for further details regarding vacuum physics.



The detector consists of the following components:

- An ion source which converts neutral gas atoms and molecules to charged (ionized) particles
- A mass filter which separates the charged particles according to their mass-to-charge ratio.
- A Faraday cup or a secondary electron multiplier which measures the ion current after separation by the mass filter. The measured current is an indication for the partial pressure of the respective gas component.
- A data analysis system processes ion currents which are measured by the detector and displays them in a user-friendly form. In a leak detector ion currents can be quantified after calibration and are displayed as leak rate.

There is a large variety of mass spectrometers. Main differences are in the mass filter. Frequently used technologies are shown in Table 4-1.

In this booklet we are going to describe magnetic sector field and quadrupole mass spectrometers only since they are the most frequently used analyzers in commercially available tracer gas leak detectors.

| Name | Magnetic sector field mass spectrometer | Time-of-Flight mass spectrometer | Ion trap mass spectrometer | Quadrupole mass spectrometer |
|-------------------|---|---|---|--|
| Working principle | Mass separation by different deflection radius of a charged particles with different masses in a magnetic field | Particles with the same energy but different masses have different velocities. Detectors with high time resolution allow for mass separation due to different flight times. | Ion trajectories are determined by an electrical field with high frequencies. | Resonance of moving ions in an electrical field with high frequency. |

Table 4-1: Mass spectrometer technologies

4.1.1 Magnetic Mass Spectrometer

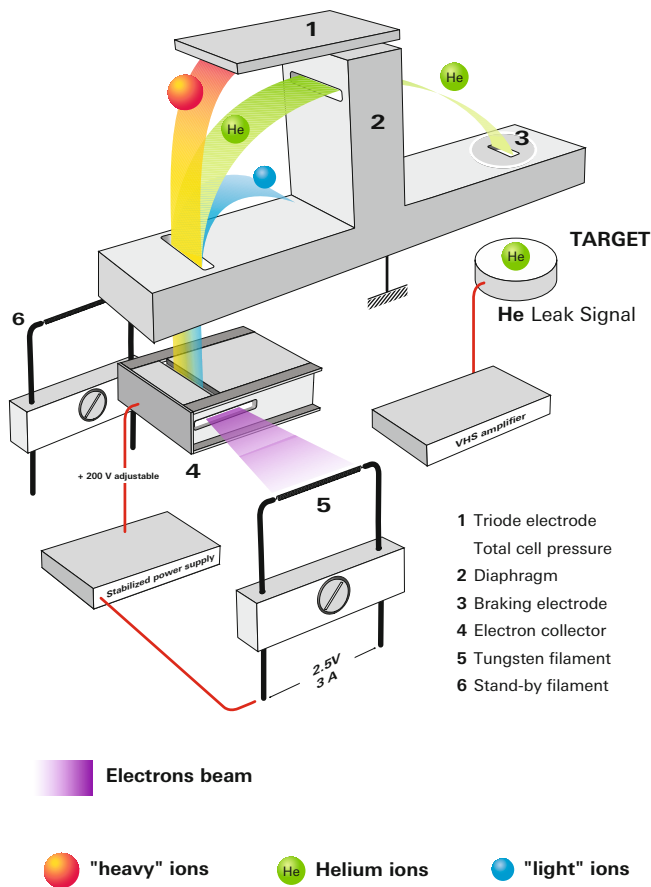


Figure 4-3: Working principle of a magnetic mass spectrometer

Magnetic sector field mass spectrometers are the dominant analyzers in helium leak detectors. The reason why they are dominating this application is their simple and rugged design with less sensitivity to contamination compared to other mass spectrometric analyzers. By default, the analyzer is set to mass 4 u for the most abundant helium isotope. Alternatively, the analyzer can be set to mass 3 u (e.g. the helium isotope with mass 3 u) or mass 2 (e.g. the hydrogen molecule with mass 2 u). Mass range limitation allows for construction of small and compact yet very powerful mass spectrometers with low detection limits.

Figure 4-3 shows the working principle of a magnetic mass spectrometer.

In a first step neutral gas atoms or molecules are ionized by electron bombardment in an ionizer cell (see Figure 4-4). We need ionized, i.e. charged particles in order to accelerate and deflect them in an electrical or magnetic field. This is not possible with neutral gas particles.

The electrons are generated by a heated wire, the so-called filament.

Molecule M in the ionization chamber (see Figure 4-3, Pos. 4) of the analyzer cell it is hit by an electron with high energy. This leads to emission of another electron from the shell of the molecule and to fission of the neutral molecule M to a positively charged ion M^+ and a negatively charged electron e^- :



The positive ion M^+ is then accelerated and injected into a zone subjected to a magnetic field. This magnetic field deflects the ions in different curves according to the mass of the respective ion. More precisely, the ions are deflected according to the mass-to-charge ratio since we can also have multiple ionized gases. Thus the injected ion beam which contains a mixture of various ions with different masses is divided into several beams. Each of the individual beams contains ions with one single mass only. For example, $^4\text{He}^+$ ions (mass-to-charge ratio $m/e = 4$) are

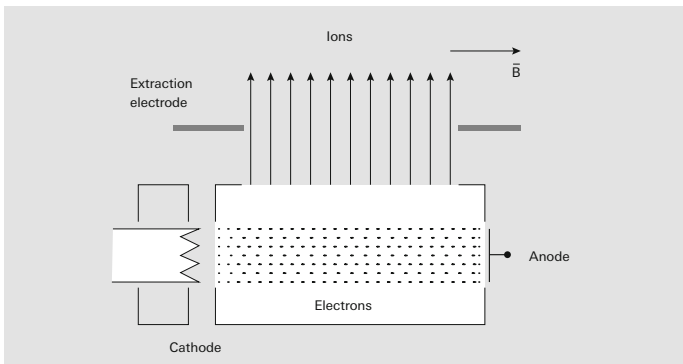


Figure 4-4: Ionization process in an ion source

separated from lighter ions (e.g. hydrogen species like H_2^+ , $m/e = 2$ or H^+ , $m/e = 1$) or heavier ions (e.g. nitrogen N_2^+ , $m/e = 28$ or oxygen O_2^+ , $m/e = 32$).

The triode electrode (see Figure 4-3, Pos. 1) collects the ions with a mass higher than the tracer gas mass. This ion current is calculated to give an indication about the total pressure in the analyzer cell.

The previously mentioned magnetic field is produced by an external permanent magnet and is constant. The accelerating field is adjusted so that the tracer gas ion is guided on a pre-determined path through an orifice (2) and arrives on the detector. This is the TARGET in Figure 4-3.

The detector converts the ion current of the respective tracer gas to an electrical current which is then processed in the leak detector electronics and displayed as the leak rate signal. The detector needs to provide high sensitivity to allow for minimum detectable leakage rates. Simple Faraday cups which have been used previously in leak detectors

do not meet these requirements any longer. In modern devices micro channel plates are used (see Figure 4-5).

MCPs allow for very compact geometry which is going along with high amplification and low noise. They consist of millions of small glass capillaries with a diameter of $8\ \mu\text{m}$. The capillaries have a conductive coating and are slightly tilted to the surface. Each capillary works as an independent secondary electron multiplier. If a tracer gas ion hits the surface of a capillary it generates a cascade of secondary electrons. The potential across the plate accelerates the electrons to the detector.

In order to separate the helium ions from "noise" caused by other ions or electrons, an electrode located in front of the target eliminates them. This electrode (Figure 4-3, Pos. 3) is called the "braking electrode".

The current of ions of the tracer gas is proportional to the partial pressure. By comparing the measured ion current to an ion current produced by reference (which is a

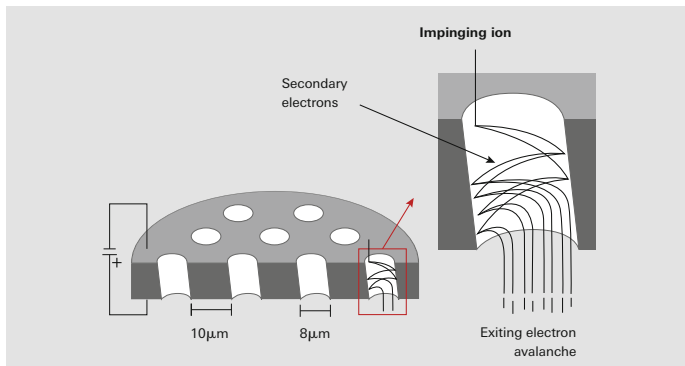


Figure 4-5: Micro Channel Plate

calibrated leak) we can find the flow rate of the leak that has been detected.

There is a spare filament (Figure 4-3, Pos. 6) in the analyzer cell. This is used as a back-up filament in case that the first filament breaks. It warrants continued operation of the leak detector during a measurement campaign or in serial leak detection which is integrated in a production process. However, once the first filament breaks the operator should prepare maintenance.

We can describe ion deflection in a mass spectrometer also in a more quantitative way:

We have generated a positive ion M^+ with the charge q . In the next step the positive ion M^+ is accelerated in an electrical field by a voltage U . Due to acceleration the kinetic energy of the particle E_{kin} increases.

$$E_{kin} = q \cdot U = \frac{m \cdot v^2}{2}$$

Formula 4-1: Kinetic Energy

E_{kin} Kinetic energy
 q Charge
 U Voltage (potential)
 m Mass
 v Velocity

Conversion of Formula 4-1 yields the velocity of the particles during their flight through the magnetic sector field.

$$v = \sqrt{\frac{2q \cdot U}{m}}$$

Formula 4-2: Particle velocity

If charge q is equal, then the velocity and the flight time for passing a defined distance are depending on the mass. The latter is applied in time-of-flight mass spectrometers.

In a sector field mass spectrometer the ions are guided on a circular trajectory by the homogeneous magnetic field. This is caused by the Lorentz force (named after the Dutch mathematician and physicist Hendrik A. Lorentz, 1853–1928) which acts upon the moving ions perpendicular to the plane which is defined by velocity and magnetic field.

$$F = q \cdot v \cdot B$$

Formula 4-3: Lorentz force

F Lorentz force
 B Magnetic field

On the circular trajectory with radius r the Lorentz force equals the centripetal force.

$$q \cdot v \cdot B = m \cdot v^2 / r$$

Formula 4-4: Equilibrium between Lorentz force and centripetal force

Conversion of Formula 4-4 yields the radius of the trajectory of particles during their flight through the magnetic sector field.

$$r = \frac{m \cdot v}{q \cdot B}$$

Formula 4-5: Radius

Combination of Formula 4-2 and Formula 4-5 replaces velocity v and leads us to

$$r = \sqrt{\frac{2mU}{qB^2}}$$

Formula 4-6: Radius as a function of mass, voltage, charge and magnetic field

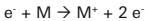
In magnetic sector field mass spectrometers the magnetic field B is fixed and defined by the external magnet. Please see Figure 4-3 for the direction of the magnetic field. The mass spectrometers in leak detectors are

designed and tuned for optimum trajectories of the tracer gas ^4He . All other masses cannot pass through the orifices and are neutralized after collision with the walls of the analyzer cell.

As can be seen in Formula 4-6 the radius r can be influenced by the acceleration voltage U . In commercially available leak detectors the available mass range is limited to masses 2 (hydrogen H_2^+), 3 (helium isotope $^3\text{He}^+$, possibly tritium ^3H), and 4 (helium $^4\text{He}^+$).

4.1.2 Quadrupole Mass Spectrometer

The first process that a neutral gas atom or molecule undergoes in a quadrupole mass spectrometer is exactly the same as in a magnetic mass spectrometer: ionization by electron bombardment in an ionizer cell (see Figure 4-4).



The positive ion M^+ is then accelerated and injected into a zone subjected to an electric

field. This electrical field is oscillating and it is applied to four cylindrical rods which are set parallel to each other (see Figure 4-6). The rods are forming the mass filter. Ideally, the rods have hyperbolic profile. However, since hyperbolic geometries are difficult to manufacture the ideal shape is approximated with cylindrical rods. The cylindrical rods have a specific ration of rod diameter to spaces between the rods.

The opposing rod pairs are connected electrically. A radio frequency voltage is applied between the respective two pairs of rods. The RF voltage is superimposed by a direct current voltage. One pair of rods is positively charged, the other one negatively. We can understand the motion of the ions generated in the ion source in a simplified form by looking at

- a) electrostatic attraction – same charges repel, opposite charges attract each other
- b) mass inertia of big and small ions

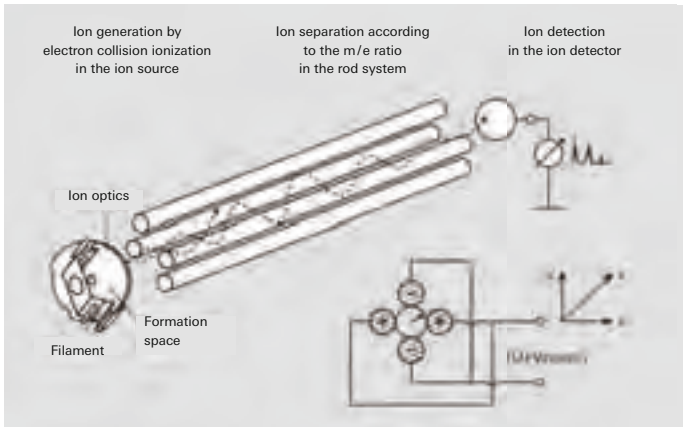


Figure 4-6: Working principle of a quadrupole mass spectrometer

So if we would inject two positively charged ions with different masses to the positive rod pair of the mass filter, they would just be repelled by the two positively charged rods and pass through the filter without any separation effect. If we add an RF alternating current the ions will see a temporarily negatively charged rod. This happens once the sinus curve of the RF alternating current turns negative and the negative charge is bigger than the positive DC component. Mass inertia of heavy ions is too big to enable the ion to crash into the temporarily negatively charged rod with its attracting electrostatic force. Once the heavy ion has started moving to the direction of the attracting negative charge the sinus curve is in the positive section again and the ion is repelled again. So a heavy ion will not crash into the rods on its way through the positive rod pair of the mass filter. In contrary a light positive ion can follow the fast charge changes in the RF field due to its lower inertia. So after a few cycles the light ion hits the rods, gets neutralized and pumped out of the quadrupole. In this sense the positive pair of the two rods is a high pass mass filter.

Let us take the same approach on the negative pair of rods: with only a DC charge, the positive ions would be attracted and not a single ion would pass through the mass filter. If we now apply the RF voltage there is no way for the heavy ion to escape for a long time from the negative attraction of the rods due to its inertia. After a few cycles it will crash into the rods, get neutralized and pumped away. In the contrary a light ion will be able to follow the RF cycles and pass through the mass filter. The negative pair of the two rods is a low pass mass filter.

Now we can combine what we have explained in two dimensions in three dimensions. If we mount the four rods as shown in Figure 4-6 we have the high pass mass filter in x/z-plane and the low pass mass filter in

y/z-plane. The ions will not follow a sinus-like trajectory but one like a corkscrew.

So by tuning DC and RF frequencies we can decide how wide the window of masses is we allow passing through the mass filter. We can sweep a band-pass with a width we can influence over a mass range. And we can either scan a mass range in order to get an idea of what is inside our vacuum chamber or we can keep the voltages stable and follow time-resolved processes. In helium leak detection we do the latter with the mass filter set to parameters which allow our tracer gas ions to pass through.

Let us describe this more in detail with some formulas:

The voltage between the two pairs of rods consists of a DC component U and an RF component with amplitude V and frequency $f = \omega/2\pi$.

$$U_{quad} = U + V \cdot \cos\omega t$$

Formula 4-7: Quadrupole Voltages

- U DC voltage
- V RF amplitude
- f Frequency
- ω Angular frequency

In order to solve the Formulas of ion motion through the quadrupole field we introduce two dimensionless variables, the so-called stability parameters a and q . These summarize the variable parameters of the quadrupole field.

$$a = \frac{8 \cdot Q \cdot U}{m \cdot r_0^2 \cdot \omega^2}$$

Formula 4-8: Stability Parameter a

- Q Ion charge
- m Ion mass
- r_0 Field radius

$$q = \frac{4 \cdot Q \cdot V}{m \cdot r_0^2 \cdot \omega^2}$$

Formula 4-9: Stability Parameter q

So we have reduced a six-dimensional problem to a two-dimensional one. This simplification yields the Mathieu differential Formulas of motions. The solutions of these Formulas define the range of stable ion trajectories. Stable ion trajectories are only in the section below the red triangle defined by a and q with amplitudes $r_{max} < r_0$ (see Figure 4-7).

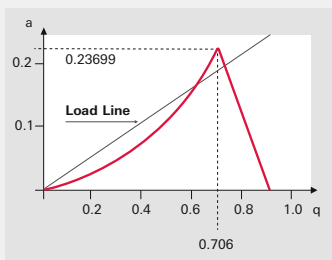


Figure 4-7: Stability diagram of a quadrupole

If you divide Formula 4-8 by Formula 4-9 you achieve

$$\frac{a}{q} = \frac{2 \cdot U}{V}$$

Formula 4-10: Stability Parameter a

This is the slope of the mass scan line of the quadrupole mass filter. By adjusting the slope of the mass scan line one can vary the width of the bandpass. In other words mass resolution can be tailored according to the respective application.

If mass resolution is maximized the mass scan line passes the peak of the triangle with the values

$$a_{peak} = 0.237 \text{ und } q_{peak} = 0.706.$$

The mass filter is only transparent for voltage ratios

$$\frac{U}{V} = \frac{a_{peak}}{2 \cdot q_{peak}} < 0.1678$$

Below these values the mass scan line overlaps with the stability range. All ions which are included in the small triangle which is defined by a , q and the mass scan line can pass through the mass filter.

Let us introduce the ratio $\frac{m_U}{e}$ with

$$m_U \text{ atomic mass unit } 1.6605 \cdot 10^{-27} \text{ kg}$$

$$e \text{ elementary charge } 1.6602 \cdot 10^{-19} \text{ A} \cdot \text{s}$$

$$\frac{m_U}{e} 1.0365 \cdot 10^{-8} \text{ kg A}^{-1} \text{ s}^{-1}$$

If we now multiply $\frac{m_U}{e}$ with the dimension-

less mass number M of the respective ion we end up with the following parameters for the voltages U_{peak} and V_{peak} at the tip of the stability diagram:

$$U_{peak} = k_u \cdot M \cdot r_0^2 \cdot f^2$$

Formula 4-11: Stability Condition for U

$$k_u 1.2122 \cdot 10^{-8} \text{ kg A}^{-1} \text{ s}^{-1}$$

$$V_{peak} = k_v \cdot M \cdot r_0^2 \cdot f^2$$

Formula 4-12: Stability Condition for V

$$k_v 7.2226 \cdot 10^{-8} \text{ kg A}^{-1} \text{ s}^{-1} \text{ kV}$$

The stability conditions are showing that a fixed frequency yields a direct proportionality between voltages and mass in a quadrupole mass filter. So with linear variation of voltage amplitudes you get a linear mass scale.

If the DC voltage is switched off ($U = 0$), all trajectories are stable with $q < 0.905$. According to Formula 4-9 these are all masses with

$$M > \frac{k_H \cdot V}{r_0^2 \cdot f^2}$$

Formula 4-13: High Pass Condition

$$K_H \text{ constant} \quad 1.0801 \cdot 10^7 \text{ A s kg}^{-1}$$

In this operational mode the quadrupole is a high pass filter. If we start from low masses and increase RF amplitude V , the trajectories of ever heavier ion species become unstable and are filtered. In this mode one can achieve an integral spectrum which yields a total pressure measurement.

Entrance conditions are decisive for ion transmission through the quadrupole. In an ideal case the ions are injected in the center of the quadrupole and move in parallel to the axis of the rods.

These conditions can be met easier with

- long distance between the rods (which also influences the rod diameter) and
- long rods

If rods become longer and thicker, then requirements regarding production tolerances are easier to meet. In our Know How Book we describe the advantages of Pfeiffer Vacuum's ion sources which result in high transparency and high sensitivity.

In practical operation band-pass tuning is made by variation of the voltage ratio $\frac{U}{V}$ to

keep line width ΔM constant. This means an increase of resolution $\frac{M}{\Delta M}$

proportional with increasing mass. Due to the proportionality of V and M as shown in Formula 4-12 the mass scale of a quadrupole mass filter is linear, in contrary to a magnetic sector field mass spectrometer.

One of the determining parameters for a quadrupole is RF power.

$$N_{RF} \approx \frac{C}{Q} \cdot M^2 \cdot f^5 \cdot r_0^2$$

Formula 4-14: RF power

- C Capacity of the system
- Q Circuit quality

The required RF power increases with high powers of f and r_0 . Increasing the field radius r_0 lowers relative mechanical tolerances and improves the behavior of the mass filter. Generally, an increase of both field radius and frequency leads to improvements. However, going to higher frequencies is limited according to Formula 4-14. Prolongation of the rods allows for lower frequencies again. Serial production of rods limits the length often to 300 mm.

The required mass range and the demanded resolution determine mass filter dimensions and operating frequency. In real quadrupoles rods with diameters of 6, 8 and 16 mm are used with adapted electronics.

Let us consider the relation between resolution and mechanical precision. If we assume a quadrupole mass filter which is working close to the tip of the stability diagram (see Figure 4-7). We can determine the DC voltage and RF amplitude according to Formula 4-11 and Formula 4-12.

$$U_{peak} = 1.2122 \cdot 10^{-8} \text{ kg A}^{-1} \text{ s}^{-1} \cdot M \cdot r_0^2 \cdot f^2$$

$$V_{peak} = 7.2226 \cdot 10^{-8} \text{ kg A}^{-1} \text{ s}^{-1} \cdot M \cdot r_0^2 \cdot f^2$$

If we assume that both voltages and the frequency can be tuned with infinite precision we achieve:

$$M = c_k \cdot \frac{1}{r_0^2}$$

c_k constant

After differentiation, division by M and definition of the absolute value we end up with the dispersion of the mass filter which is caused by the field radius r_0 .

$$\frac{\Delta M}{M} = \frac{2 \cdot r_0}{r_0}$$

Formula 4-15: Dispersion

Let us assume that field radius r_0 varies over the length of the mass filter by $\delta r_0 = 0.03$ mm. Now we consider the result of this variation on two mass filters with different dimensions. For optimum transmission the resolution of the mass filter (we define

$\frac{\Delta M}{M} = 0.01$) must be higher than dispersion

caused by r_0 . For a field radius of 3 mm the result is

$$\frac{\Delta M}{M} = \frac{2 \cdot r_0}{r_0} = \frac{2 \cdot 0.03 \text{ mm}}{3 \text{ mm}} = 0.02$$

So dispersion is bigger than the required resolution.

If we take a mass filter with a field radius of 12 mm the result is

$$\frac{\Delta M}{M} = \frac{2 \cdot r_0}{r_0} = \frac{2 \cdot 0.03 \text{ mm}}{3 \text{ mm}} = 0.005$$

So the required resolution can be achieved with the higher field radius, i.e. the bigger rod system. The paragraph above also means that transmission of the smaller quadrupole is close to zero with the required resolution whereas virtually all ions can pass the larger quadrupole.

The consideration above is a simplification which neglects several effects. However, what we can learn from this paragraph is that

- tolerance of field radius must be much lower than 1% over the whole length of the mass filter rods

- field radius deviations result in transmission loss
- the bigger the quadrupole, the lower the influences of absolute mechanical tolerances
- the higher resolution requirements in a high mass range are, the lower the relative precision of a mass filter must be.

4.2 Quartz Window Sensor

The mass spectrometric detectors described in the previous chapters are detecting the tracer gas by ionization of a gas mixture with following separation of ions in an electrical or a magnetic field. The quartz window sensor described in this chapter performs separation by different permeation properties of gases. Helium and hydrogen can permeate through a thin quartz diaphragm – other gases cannot. This effect is already applied since a long time in commercially available calibrated leaks. Helium from a reservoir with defined pressure passes through a quartz element and forms a small and very precise flow which then can be used for calibration of a leak detector.

A quartz diaphragm can be used to isolate helium from a gas mixture. If helium penetrates from the mixture into vacuum, the total pressure in vacuum is purely determined by the partial pressure of helium. So a quantitative measurement of helium can be performed with a simple total pressure gauge.

Serial production of suitable quartz membranes became possible with the availability of modern micromechanical technologies. The surface of a silicon wafer is oxidized in a controlled process. This produces a homogeneous quartz layer with a thickness of 7 μm (see Figure 4-8). In a micromechanical dry etching process several thousand holes with a diameter of 200 μm are etched from the other side of the wafer until the quartz

layer is reached. Thus all gases can reach the quartz layer and helium can selectively pass through the membrane. The measurement is the faster the thinner the diaphragm and the higher the temperature is. So the diaphragm is heated by a sputtered Platinum heater to temperatures of several hundred degrees C.

The diaphragm is then bonded to a glass housing with electrical feed-throughs for the total pressure gauge. The cell is evacuated through a glass tube to Extremely High

Vacuum (XHV) with pressures lower than 10^{-11} hPa. Then the tube is molten off and the cell is sealed. Pressure measurement is performed with a well-known ionization gauge (Penning type). Please see our Know How Book for further details of the Penning gauge and total pressure measurement.

Quartz window technology does not achieve the same sensitivity compared to mass spectrometric devices. Please see chapter 5 Working Principle for Test Equipment for a comparison of devices.

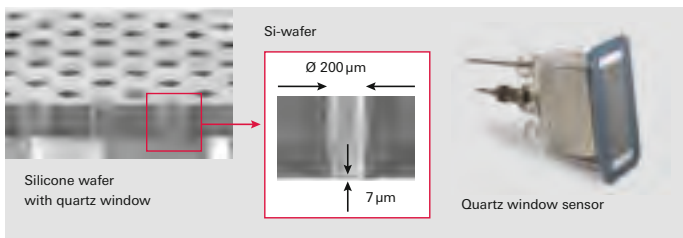


Figure 4-8: Quartz window

4.3 Comparison of Pfeiffer Vacuum's Detector Technologies

| | Magnetic sector field mass spectrometer | Quadrupole mass spectrometer | Quartz window sensor |
|---|---|--|--|
| Working principle | Mass separation of ions in a magnetic field | Mass separation of ions in an electrical field | Separation of helium by selective permeation through a quartz membrane, detection with total pressure gauge |
| Tracer gases | ^4He , ^3He , H_2 | All species which are gaseous or can be vaporized | He , H_2 |
| Leak localization Vacuum / Overpressure | + / + | + / o | + / - |
| Integral leak test Vacuum / Accumulation / Carrier gas | + / + / + | + / o / - | + / - / - |
| Virtual leak, outgassing | - | + | - |
| Quantitative leak test | + | + | + |
| Calibration | + | o | + |
| Permeation and desorption measurement | Helium and hydrogen only | Multi-gas, information about species, if unknown | - |
| Sensitivity range [Pa m ³ s ⁻¹] | Tracer gas helium: 10 ⁻¹³ to 10 ⁻³ | Strongly depending on tracer gas, for helium <10 ⁻¹¹ | Tracer gas helium: 10 ⁻⁸ to 10 ⁻³ |
| Response time [s] | < 1 | < 1 | < 1 |
| Selectivity | Tracer gas helium: Very high Tracer gas hydrogen Medium | Strongly depending on tracer gas | Tracer gas helium: Very high |
| Characteristics | <ul style="list-style-type: none"> + Can be used for leak location and quantification + Mature and field-proven technology + With tracer gas helium very good selectivity + Excellent sensitivity and lowest detection limit + Very wide dynamic range + Fast signal response | <ul style="list-style-type: none"> + Can operate with virtually every gaseous substance as tracer gas and unrivalled flexibility regarding adaptation to respective application + Identification of unknown gas components, e.g. in permeation or diffusion experiments + High time resolution + Can be used as measurement device but also for process regulation + Continuous monitoring of gas composition and tightness + Detection of virtual leaks | <ul style="list-style-type: none"> + Can be used for leak location and quantification + Low maintenance + Broader pressure range compared to mass spectrometer-based analyzers + Light weight allows for optimum use for traveling service staff |
| Limitations | <ul style="list-style-type: none"> - Limited mobility due to pumping technology and weight - Limited range of tracer gases | <ul style="list-style-type: none"> - Stand-alone analyzers need high vacuum systems, atmospheric systems need special sample inlet systems - In most cases stationary use only - Difficult calibration - Needs highest operator education level of all NDT methods | <ul style="list-style-type: none"> - Sorption pump in the sensor can be saturated after long-term use (several years) and needs to be exchanged - Limited sensitivity and higher detection limit compared to mass spectrometer-based analyzers - Detection limit depending on sample volume |

Table 4-2: Comparison of Pfeiffer Vacuum's Detector Technologies

4.4 Other Detector Technologies

There are numerous detector technologies used in non-destructive testing (NDT). The simplest detectors are our built-in sensors eyes and ears. They are used in classical vis-

ual inspection technologies like bubble test or sonic tests. On the other hand the most sensitive methods are mass-spectrometer-based. An overview of sensor technologies in NDT is summarized in Table 4-3.

| Method / Detector | Tracer Gas | Tested object under Overpressure | Tested object under Vacuum | Quantitative test | Localization | Leakage rate [Pa m ³ s ⁻¹] | | | | | | | | | | | | | | | |
|---|--|----------------------------------|----------------------------|-------------------|-----------------|---|------------------|------------------|------------------|------------------|-------------------|-------------------|--|--|--|--|--|--|--|--|--|
| | | | | | | 10 ⁻¹ | 10 ⁻³ | 10 ⁻⁵ | 10 ⁻⁷ | 10 ⁻⁹ | 10 ⁻¹¹ | 10 ⁻¹³ | | | | | | | | | |
| Foaming Agent | any | + | - | - | + | | | | | | | | | | | | | | | | |
| Bubble Test | any | + | - | - ¹⁾ | + | | | | | | | | | | | | | | | | |
| Sonic or Ultrasonic Sensor | any | + | - | - | + | | | | | | | | | | | | | | | | |
| Ultrasonic Bubble Detection | any | + | - | + | + | | | | | | | | | | | | | | | | |
| Pressure Rise | any | - | + | + | - | | | | | | | | | | | | | | | | |
| Pressure Decay | any | + | - | + | - | | | | | | | | | | | | | | | | |
| Flow Controller | any | + | - | + | - | | | | | | | | | | | | | | | | |
| Photo-acoustic detection after laser absorption | Tracer gases like SF ₆ , CO ₂ , N ₂ O | + | - | + | + | | | | | | | | | | | | | | | | |
| Refrigerant Sniffer based on Infrared Sensor | Refrigerant | + | - | - | + | | | | | | | | | | | | | | | | |
| Low-end Refrigerant Sniffer based on Infrared Sensor, Semiconductor Sensor, Rice Diode, ... | Refrigerant | + | - | - | + | | | | | | | | | | | | | | | | |
| Hydrogen Field Effect Transistor | Tracer gas containing hydrogen | + | - | + ²⁾ | + | | | | | | | | | | | | | | | | |
| Quartz Window Sensor | Tracer gas helium | - ³⁾ | + | + | + ⁴⁾ | | | | | | | | | | | | | | | | |
| Magnetic sector field mass spectrometer, sniffing | Tracer gas ⁴ He, ³ He, H ₂ | + | - | + ²⁾ | + | | | | | | | | | | | | | | | | |
| Magnetic sector field mass spectrometer, vacuum | Tracer gas ⁴ He, ³ He, H ₂ | - | + | + | + | | | | | | | | | | | | | | | | |
| Quadrupole mass spectrometer | any | + | + | + | + | | | | | | | | | | | | | | | | |

Table 4-3: Sensor technologies used in non-destructive testing (NDT) and detection limits

¹⁾ Possible with bubble collection and volumetric analysis

²⁾ Accumulation test method only, see chapter 6 Leak Detection Methods

³⁾ Not available from Pfeiffer Vacuum

⁴⁾ Spraying method only, see chapter 6 Leak Detection Methods

The lowest detection limit does not necessarily offer the best solution for every leak detection task. Considerations have to be made regarding economical, environmental and legal aspects. Industrial leak detection has to follow the demands given by production. If for example, a part is thermally treated and needs to be leak-checked in the next

step. The thermal behavior of the enclosed air may exclude pressure decay as a suitable test method. Another example is wetting of the tested part in a classical bubble test. If the part cannot be wetted or the following drying step cannot be performed, then the decision making process often leads to tracer gas leak detection methods.

5 Working Principle of Test Equipment

5.1 Sector Field Mass Spectrometer Helium Leak Detector

The operating principle of a sector field mass spectrometer has been explained in chapter 4. This mass spectrometer is integrated in a helium leak detector (see Figure 5-1) with automatic valve and pump control.

When the unit is not in test mode (stand-by or roughing mode), the leakage rate displayed corresponds to the internal helium background of the unit.

The mass spectrometer (8) for masses 2, 3 and 4 u (corresponding to tracer gases hydrogen, ³helium and ⁴helium) is mounted at the high vacuum flange of a turbopump (7). A roughing pump (5) is backing the turbopump which can be isolated with the exhaust valve (6). After cycle start the tested part is evacuated via the roughing valve (3). The exhaust valve (6) is closed. When the

inlet pressure measured on the gauge (2) reaches the cross over pressure, the exhaust valve (6) is opened. Now the tracer gas admitted through the tested object can reach the backing line between the turbopump (7) and backing pump (5). Due to the limited compression ratio of the turbopump (= the pressure ratio between exhaust pressure and inlet pressure of the turbopump which is depending on the admitted gas) the light tracer gas can diffuse backwards through the turbopump (7) to the analyzer cell (8) and be detected. This operating mode is called "counter flow" or "gross leak" mode. In this pressure regime the leak detector does not reach its full sensitivity due to the small proportion of gas flow which reaches the detector. In order to have higher sensitivity measurements one needs to pump down further. At a lower pressure the roughing valve (3) closes and the test valve (4) is opened. This operating mode is called "direct flow" or "fine leak" mode, it is the highest sensitiv-

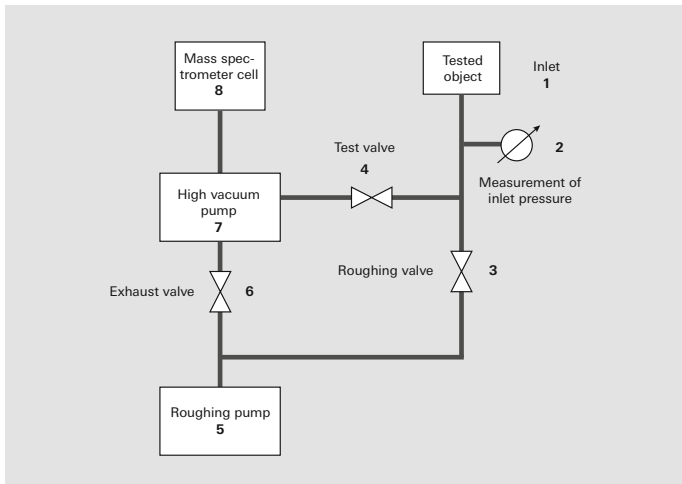


Figure 5-1: General vacuum circuit of a helium leak detector

ity test mode. A calibrated leak is in general present in the layout of such leak detectors to allow the auto-calibration of the system.

Counter flow mode is used e.g. when a big leak needs to be located. Other applications are protection of the analyzer cell against air inrush in case that the tested part might implode. Direct flow mode is mainly used for quantitative detection of small leaks.



Figure 5-2: ASM 340

| | Counter flow | Direct flow |
|---------------------------|--------------|-------------|
| Inlet pressure | High | Low |
| Pump down time | Low | High |
| Minimum detectable signal | Medium | Small |
| Air inrush stability | High | Medium |

Table 5-1: Counter flow and direct flow principle

5.2 Quadrupole Mass Spectrometer Helium Leak Detector

Quadrupole mass spectrometers are often used as partial pressure probe or residual gas analyzers (RGA) in high vacuum systems. Selection of the ion source, mass filter and detector provides for adaptation to mainly analytical applications. Leak detection by

analysis of an air fingerprint in the mass spectrum or by following a time-resolved helium signal is rather a by-product of the main application.



Figure 5-3: Gas analysis system OmniStar

Many processes which are monitored with quadrupole mass spectrometers are run at process pressures above the maximum operational pressure of a quadrupole of 10^{-4} hPa. An important component of a QMS-based gas analysis system like Pfeiffer Vacuum's OmniStar (see Figure 5-3) is a gas inlet system, which is tailored to the respective process pressure. Gas mixtures have to be transferred from process pressure to analysis without any change of the composition. Depending on the inlet pressure the following principles can be used:

- At inlet pressure > 10 hPa a capillary is used for pressure reduction. In case of condensable process gases the capillary can be heated. In the capillary we have laminar flow conditions. A dosing valve provides optimum pressure conditions in the ionizer of the mass spectrometer. As an option, the capillary can be equipped with an extra pump. This ensures fast response time and high pressure in front of the dosing valve going along with high sensitivity.

- At inlet pressure < 10 hPa pressure reduction is made via an orifice or skimmer with a differentially pumped mass spectrometer. An intermediate chamber between inlet and mass spectrometer housing is pumped with a turbopump.
- At pressures below $< 10^{-4}$ hPa the mass spectrometer can be used without an additional inlet system.

QMS-based gas analysis systems can e.g. be used for permeation measurements. Analysis can be performed either in a vacuum system through e.g. a foil or at ambient pressure with a capillary inlet system – like a sniffer probe. Quadrupole mass spectrometers offer maximum flexibility in selection of the permeating gas. Classical helium leak detectors can be used for permeation measurements with helium as a fast permeation gas. This may be used in case that faster process times than with heavier gases need to be achieved.

5.3 Quartz Window Helium Leak Detector

Mass spectrometers are separating a gas mixture after ionization with following separation in a magnetic or electrical field. Quartz window leak detectors are making use of different permeation properties of tracer gases.

Quartz window leak detectors do not have an integrated pumping system. In Figure 5-4 you see how they are connected to the vacuum line of the vacuum system which is used for chamber evacuation via an external valve (9).

An external calibration leak (7) provides for quantification and response time measurement. For fast signal recovery the sensor can be purged via the flow meter (5) and connected to an external pump via the valve (3). Pfeiffer Vacuum's MiniTest 300 is mainly used at inlet pressures up to 200 hPa in

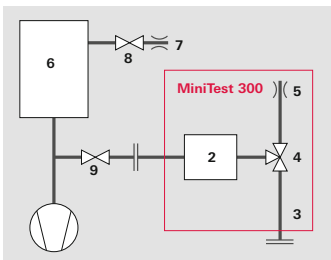


Figure 5-4: Quartz window leak detector on a vacuum system

dirty applications compared to a mass spectrometer-based leak detector. Due to the high maximum inlet pressure it can be used e.g. in steam lines without any protection. Dominant field of application is detection of large leaks (still smaller than the detection limit of any pressure decay measurement) for localization and repair.



Figure 5-5: MiniTest 300 with wireless remote control

5.4 Comparison of Detectors

In Table 5-2 we have summarized some parameters of leak detection equipment.

| | Quartz window leak detectors | Magnetic field mass spectrometer leak detector | Quadrupol mass spectrometer |
|-----------------|---|--|--|
| Localization | Yes | Yes | Yes |
| Quantification | Yes, external calibrated leak | Yes, internal and external calibrated leak | Yes, via pre-defined calibration gas mixture |
| Virtual leak | No | No | Yes |
| Permeation | No | No difference between permeation and desorption. | Yes, with any gas |
| Desorption | No | Can be used for permeation measurements with helium as a fast alternative for heavier process gases. | Yes, with any gas |
| Test pressure | Very high | High | Low, can also be used in gas analysis systems from ambient |
| Detection limit | $5 \cdot 10^{-9} \text{ Pa m}^3 \text{ s}^{-1}$ | $5 \cdot 10^{-13} \text{ Pa m}^3 \text{ s}^{-1}$ | Ion current depending on analyzer and detector |
| General | Qualitative and possibly quantitative measurement, best robustness. | Qualitative and quantitative measurement, best flexibility regarding test methods. | Rather a qualitative or semi-quantitative instrument. Difficult quantification. Best versatility regarding gas selection. Requires know-how. Delivers most information. Needs highest level of operator knowledge. |

Table 5-2: Comparison of detectors

6 Leak Detection Methods

Prior to starting a leak detection measurement the operator should think about the sample itself. Example: Due to its high inlet pressure and water vapor tolerance a steam line in a power station may be checked with a MiniTest 300 leak detector without any problem. However, a mass spectrometer leak detector should be protected by a condensate trap between sample and detector. In this example we are looking for very big leaks. In an industrial test for lower leak rates moisture can block leak channels. This is not due to the very small size of water molecules but inter-molecular forces which result in surface tension of water. In other words the sample needs to be dry. In most cases it is absolutely useless to perform an integrity test (a water pressure burst test) prior to tracer gas leak detection – the water in the leak channels will mask every leak.

Not only moisture or process residuals have to be removed prior to a leak test. This applies also for dust and particles. A human hair in a seal may cause leaks in the order of $10^{-3} \text{ Pa m}^3 \text{ s}^{-1}$. So cleanliness is the prerequisite for proper leak detection.

Regarding the forces which act on the sample one should always try to copy real life operation of the part to be tested. This means that the pressure direction during the test should be the same as in operation.

The same applies for sample temperatures. One of the big advantages of tracer gas

leak detection over pressure decay methods is low sensitivity regarding temperature effects. However, the tracer gas cannot influence temperature-triggered structural changes in the test object which may lead to opening or closing of a leak channel. Examples are cold leaks in low-temperature physics which may open due to contraction of the material.

6.1 Getting Started

Before starting with a leak test the operator should make sure that the detection equipment works properly. It is also recommended to auto-calibration of the leak detector and calibration of the complete set-up. This means that one should check response time and partial flow when working with pumps in parallel to the leak detector.

One should start the test procedure with an integral test. In case that no leaks are found the test is finished and there is no need to look further. Only in case that a part is found to be leaky in an integral test one should start with pinpointing. The operator should check the complete part and not finish work after identification of the first leak – one might need to repeat the test and lose time.

It is good working practice to mark the tested parts with stickers (e.g. green and red for non-leaking and leaking sections) and to temporarily seal leaking sections. This prevents random tracer gas entry through a

leak which was already identified and facilitates following tests. Sealing can be made with any material which shows low tracer gas permeation or memory effect and which can be removed easily without residual. Potential materials are a tape (possibly metalized), foil, automobile body glue or putty. Check suitability of your selected material before using it in real test campaigns.

After identification of the leak and repair, the leak test has to be repeated in order to prove that the repair action was successful. Now that the operator has proven that the part meets the tightness requirements he may fill the test protocol.

In vacuum technology the initial integral test often is made with a total pressure gauge which is installed on the chamber anyway. An example from semiconductor industry is the standard pressure rise leak test which is performed after any maintenance on the respective vacuum tool. All valves from the gas supply and to the pumping system are closed and the pressure rise is monitored over time. The time-resolved pressure rise is shown in Figure 6-1. There is always some gas adsorbed on the chamber walls. So at least the initial phase of a pressure rise leak test is dominated by surface desorption. Since the amount of gas adsorbed on the

chamber wall (in the initial phase we are talking about roughly 10^{19} atoms or molecules per square meter) will get less and less over time a first test may fail and a second test may already yield a positive result.

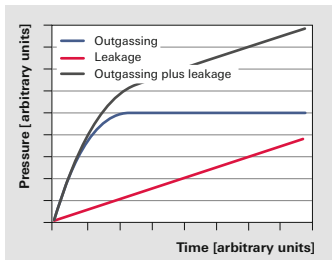


Figure 6-1: Time-dependent pressure rise

Since waiting time is long for an unambiguous detection of the curves displayed above an integral leak check with a tracer gas may be faster.

If we have a mass spectrometer installed in a vacuum system one can identify an air leak by analysis of the mass spectrum. The dominant gases in air, namely nitrogen, oxygen and argon will display signals on masses 28, 32, and 40 u. The ratio of these signals is a clear indication for an air leak.

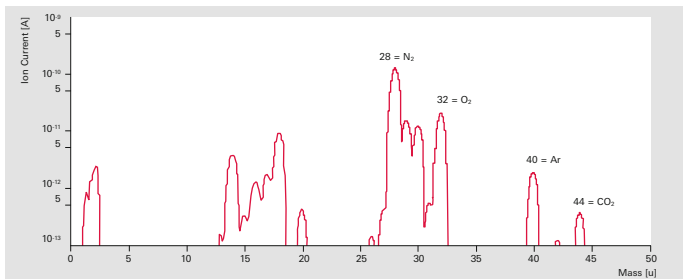


Figure 6-2: Mass spectrum air leak

From the sensitivity of the respective mass spectrometer for nitrogen (the sensitivity of the mass spectrometer is specified in the technical data) one can calculate the nitrogen partial pressure. This is possible only in vacuum systems at low pressures in order to identify small leaks with rates $< 10^{-5} \text{ Pa m}^3 \text{ s}^{-1}$.

$$p_{(N_2)} = \frac{i_{(N_2^+)}}{Se_{(N_2)}}$$

| | |
|---------------|--|
| $p_{(N_2)}$ | Nitrogen partial pressure |
| $i_{(N_2^+)}$ | Nitrogen ion current |
| $Se_{(N_2)}$ | Mass spectrometer sensitivity for nitrogen |

In a second step the nitrogen gas flow can be derived from nitrogen partial pressure and pumping speed:

$$Q_{(N_2)} = p_{(N_2)} \cdot S_{(N_2)}$$

| | |
|-------------|------------------------|
| $Q_{(N_2)}$ | Nitrogen leakage rate |
| $S_{(N_2)}$ | Nitrogen pumping speed |

This gives at least an indication for the order of magnitude of the leak which has to be found then with tracer gas spraying.

We have shown procedures for integral leak detection by means of total pressure and partial pressure gauges. This is possible with tracer gases also.

6.2 Integral Test of Enclosed Parts under Vacuum

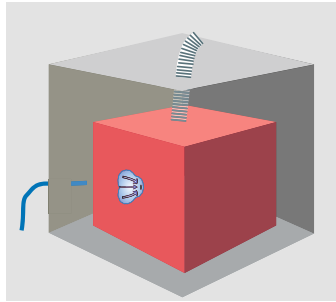


Figure 6-3: Test of an enclosed object under vacuum

In a test chamber, the part is connected to a leak detector and evacuated. Regarding the question of time consumption, response time and other theoretical background please see chapter 2. The chamber is filled with tracer gas. This allows for defined test conditions with precise parameters for tracer gas concentration and test pressure. Should a leak be present, the tracer gas will penetrate the part and be measured by the leak detector. The chamber can be very diverse. It can be a rigid metallic chamber which allows pre-evacuation. Refill from the evacuated chamber is a very fast process which ensures that tracer gas is distributed to any location around the chamber no matter how complicated the geometry may be. A more simple set-up of the test chamber can be a plastic foil which is taped around the part. Still tracer gas concentration can be controlled and test pressure is 1,000 hPa.

With a foil the pre-evacuation “pump” can be as simple as a vacuum cleaner. We do not need to go for extremely low pressure, we just need to make sure that tracer gas dis-

tribution process is not dominated by slow diffusion (velocity less than 1 cm per second) but by fast convection during the refill.

It is also possible to tape only sections of the part to be tested. This can be the flange of a large heat exchanger or a simple weld. In the first case one would still rather use a plastic foil which is taped on the heat exchanger. In the second case one might just tape the welded seam.

Let us imagine that the part to be tested has a welded seam and is evacuated by the leak detector. In order to have a fast check whether the seam is leaky one might cover the welded seam with a non sticking foil like an FKM tape. Then this FKM tape is covered with a sticky tape and tracer gas is injected under the FKM tape. The pressure differential between spray probe and atmospheric pressure leads to a fast gas distribution underneath the FKM tape and a fast integral test result.

The integral test of an evacuated part allows quantitative tests with very high sensitivity. For serial production a high level of automation is possible with high throughput and high repeatability.

Once we have determined that a part is leaking, we need to identify the location of a leak. If the part is still connected to the leak detector we can just remove the tracer gas used for quantitative leak detection by evacuation of the chamber or removal of the tape or foil and we do not need any further sample pre-treatment.

Main advantages:

- High level of automation possible
- Very high sensitivity
- High throughput
- High repeatability
- Can be tracked back to international regulations

6.3 Spraying Test

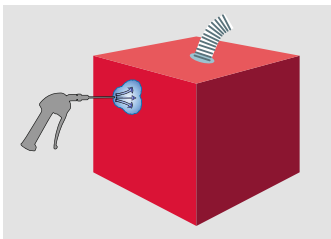


Figure 6-4: Spray test of an evacuated object

The leak detector evacuates the air inside the test part. After that, the tracer gas is sprayed on the external surface of the part. The detector measures the gas flow through the leak channel in the wall of the test part. The leak can be localized. Often this method is regarded as a quantitative measurement. However, since the operator cannot specify the quantity of tracer gas which is present on the entrance of the leak channel there is no way of quantifying the result.

When an operator needs to test a very large sample like a large cryogenic vessel or a large heat exchanger he does not have to take care of handling the connection between tracer gas bottle and spray probe if he connects a tracer gas-filled bladder directly to the connection of his spray gun.

For testing large devices it is also very helpful to have a wireless remote control like Pfeiffer Vacuum's RC 500 WL. (Figure 6-5).



Figure 6-5: Wireless remote control RC 500 WL

It is important to work with as little tracer gas flow as possible. Otherwise the test environment is flooded with an excess of tracer gas which will penetrate into unidentified leaks and increase the background signal. The first step to avoid this is using a good pressure reducer on the tracer gas bottle. "Good" means a two-stage pressure reducer with flow control. The low-pressure side should be set to a pressure slightly above ambient pressure. The flexible gas line between gas bottle and spray gun should be short in order to avoid accumulation. Triggering the spray gun should not result in a sudden release of a large amount of tracer gas which may flood the complete object to be tested. The spray gun should be a helium spray gun – not one for compressed air. The latter ones are too leaky and flow regulation is hardly possible. Tracer gas flow should produce roughly one small bubble in a glass of water or a very light gas flow that you can just detect with your tongue. When starting leak detection, the potentially accumulated tracer gas in the hose should be released by triggering the spray gun behind your back or anywhere but the direction of the test object.

The spraying test is one of the most commonly used tracer gas tests. It is easy to perform and allows for leak localization with very high sensitivity.

The spatial resolution of a spray test strongly depends on the spray gun used. With Pfeiffer Vacuum's spray guns one can even insert needles into the spray tube in order to minimize flow and allow for precise dosing in the targeted area.

Imagine you need to check a feed-through with many pins. You can provide for a constant tracer gas flow from the spray probe. The leak detector will show a certain leakage rate. Then you wet individual pins with alcohol (isopropanol) from a small spray bottle.

If you see a signal decrease you have identified the leaking pin.

Main advantages:

- Ability to locate the leak
- Very high sensitivity
- Easy to perform
- Local or global test
- Detector cost only, no tooling

Up to now we have shown leak detection methods with evacuated test objects. However, many objects are operated under pressures higher than ambient. Since one should always use the same pressure gradient in testing than in real life operation of the respective part, we also need methods for testing of pressurized objects. The simplest one is the

6.4 Sniffing Test

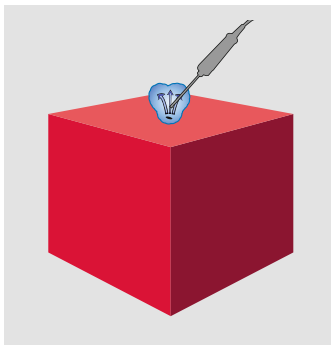


Figure 6-6: Sniffing test of a pressurized object

The test part is pressurized with tracer gas or a gas mixture containing the tracer gas. After that, the sniffer probe is moved around the part. If a leak is present, the leak detector will detect the escaping tracer gas, allowing leak localization.

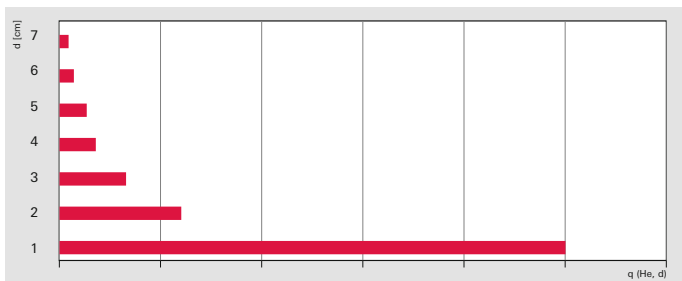


Figure 6-7: Displayed signal as a function of distance from leak

Throughput and hose length of the sniffer probe have an influence on the signal response time and displayed intensity. Figure 6-7 shows the quadratic signal decrease with distance from the leak for a sniffer probe with small throughput. This is ideal for high spatial resolution of leak localization. For fast measurements one should use a sniffer probe with high gas throughput.

A sniffer probe should always be located as closely to the leak as possible. Sniffing velocity should be in the range of 1 cm per second. Since there is a time delay due to gas transport through the sniffer line (depending on the length of the line) the operator will have passed the leak once the maximum signal is displayed. For that reason one should reverse test direction after leak detection and move the probe ever slower back and forth around the suspected leak. This together with slowly going left and right from the leak enables optimum leak localization.

Sniffing leak detection on a rack or gantry is optimum with light-weight leak detectors which still can be transported to the respective location. Often, this has to be made with a floor-standing leak detector whilst the operator is working on the gantry. In this case the hose length can reach a distance of dozens of meters. Pfeiffer Vacuum delivers

long hoses or hose extensions for this type of application. However, single response time is prolonged.

One can also cover an area to be leak-checked with plastic foil (minimize volume!) and wait until the tracer gas emanated from a single or several leaks has lead to an enhanced concentration under the foil. After a certain time the operator pierces the foil and can check whether or not he can detect a concentration rise. With this method one can test e.g. tube plates of heat exchangers with dozens or hundreds of tube joints. Checking this area completely or in sections under a foil can save time until it is worth checking individual tube joints.

Sniffing test is regarded as a localization method, not a quantitative one. Distance from the leak and tilt of the sniffer probe have an influence on the test result. However, one can also achieve quantitative results with sniffer probes.

Main advantages:

- Ability to locate the leak
- The item to be tested does not need to be placed under vacuum
- Easy to perform
- Detector cost only, no tooling

6.5 Integral Test at Atmospheric Pressure

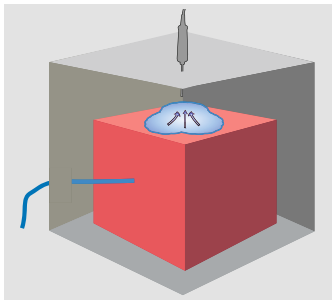


Figure 6-8: Integral test of a pressurized object at atmospheric pressure

The test part is pressurized with tracer gas in a simple accumulation chamber which is under atmospheric pressure. After an accumulation time, the detector analyzes the air inside the chamber and determines if an increase in the tracer gas concentration can be measured.

With hydrogen, helium, and many other tracer gases you always have a natural background signal. So with this accumulation method you need to define the concentration rise you consider as safe for the respective process.

It is important to keep the volume which collects the tracer gas as small as possible. The smaller the volume the safer the measurement and the less time you need. Please see chapter 2 for the theoretical background and calculation examples.

You also need to make sure that the tracer gas escaping from a leak is distributed fast and homogeneously in the collecting volume. This is achieved by circulating the gas with a ventilator. As a rule of thumb, use a fan which has circulated the gas five times before you read the signal on the detector.

Integral test of a pressurized object at atmospheric pressure is a cheap integral method. It can be tracked back to regulations which consider it as a quantitative method. For that reason occasional test of large objects often is made according to this procedure. Make sure that your test equipment does not “ZERO” the measured background regularly which would also eliminate the concentration rise due to leaks. One solution is a non-continuous check of the measured leak rate in certain intervals. This is the only solution with some commercially available hydrogen leak detectors. The other one is continuous measurement with a low-flow sniffer probe which is possible with any helium leak detector.

One should also check the permeation behavior of the cover which is used. Tracer gas loss by permeation through a foil should be minimum one decade less than desired detection limit.

Main advantages:

- Inexpensive tooling
- Easy integration in a production line

6.6 Integral Vacuum Test

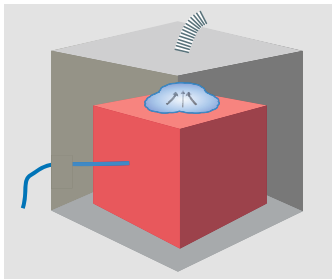


Figure 6-9: Integral test of a pressurized object under vacuum

The test part is placed in a vacuum test chamber and filled with tracer gas. Should a leak be present, the tracer gas will escape from the part into the test chamber and will be measured by the leak detector.

This method normally is much faster than leak testing under atmospheric conditions. Tracer gas velocity in vacuum is much higher than under atmospheric conditions and the response time is determined by the volume to be evacuated and the effective pumping speed. So by minimizing dead volume and maximizing effective pumping speed one can provide for a very fast measurement and short cycle time. For that reason the integral test of a pressurized object under vacuum is the dominating method in industrial leak detection.

Main advantages:

- Very high sensitivity
- High throughput
- Easy integration in a production line
- Easy calibration
- High repeatability
- Can be tracked back to international regulations

6.7 Bombing Test

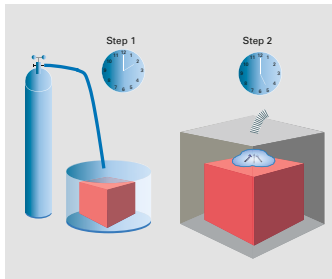


Figure 6-10: Bombing test

The so-called bombing test is applied to parts which are sealed and cannot be evacuated or pressurized by connection to a vacuum system or gas supply. The free volume (which can contain tracer gas – total volume can be bigger) of the parts typically is a few cubic centimeters. Examples are small electronic components, pacemakers, lamp bulbs, airbag gas generators, and food or pharmaceutical packages. Functionality of these components often must be guaranteed over a period of several years. For that reason the leakage rate to be detected can only be measured with a helium leak detector.

The second step of the bombing test is identical to integral test of a pressurized object under vacuum. First, tracer gas has to be admitted into the part to be tested. This can happen either by sealing the object in a tracer gas containing atmosphere or by back-pressurization. The test part is placed in a specific chamber (bombing chamber) and pressurized with tracer gas. Should a leak be present, the tracer gas is forced into the part due to the gas pressure. The amount of gas which is forced into the test object is determined by leak size, volume, and bombing pressure.

After the pressurization step the part is removed from the chamber and exposed to atmospheric conditions over a short period of time. The helium present on the surface or in small pinholes is allowed to desorb.

After that, the part is placed in a vacuum chamber which is evacuated. Any tracer gas that was forced inside the part will now escape and be measured by the leak detector.

Bombing test is not a “what-you-see-is-what-you-get” method. Any displayed leak rate signal on a helium leak detector can either be a large leak or a small leak. Upper and lower detection limit of the method can be calculated and an envelope is determined. The dynamic range of the method can be influenced by parameter selection. Tracer gas loss through a big leak can be so fast, that all tracer gas is lost and the method is blind against massive leaks. For

that reason a second test method must be applied to make sure that no big leak is overseen. This can be done e.g. by bubble test or optical inspection (often automated with a CCD and image data processing). Tracer gas leak test should always be executed first.

Main advantages:

- The only solution to test sealed components with high sensitivity
- High repeatability

6.8 Methods and Equipment

In the paragraphs above we have shown that tracer gas leak detection is a powerful and very versatile non-destructive test method. Pfeiffer Vacuum offers an unrivalled range of leak detectors for many applications.

Recommended leak detectors for the respective test methods are:

| | Spraying test | Sniffing test | Integral Vacuum test | Bombing test | Integral test at atmospheric pressure | Integral test of enclosed parts under vacuum |
|-------------------------------|---------------|---------------|----------------------|--------------|---------------------------------------|--|
| Mini Test | ■ | | | | | |
| ASM 310 | ■ | ■ | | | | |
| ASM 340 | ■ | ■ | ■ | | ■ | ■ |
| ASM 340 D | ■ | ■ | ■ | | ■ | ■ |
| ASM 380 | ■ | ■ | ■ | | ■ | ■ |
| ASM 182 T | ■ | ■ | ■ | ■ | ■ | ■ |
| ASM 182 TD+ | ■ | ■ | ■ | ■ | ■ | ■ |
| ASM 192 T | ■ | ■ | ■ | ■ | ■ | ■ |
| ASM 192 T2D+ | ■ | ■ | ■ | ■ | ■ | ■ |
| ASM 1002 | ■ | ■ | ■ | ■ | ■ | ■ |
| ASI 30 | | ■ | ■ | | ■ | ■ |
| ASM 102 S | | ■ | | | | |
| Quadrupole MS | ■ | | | | | ■ |
| QMS-based gas analysis system | | ■ | | | ■ | |

Table 6-1: Recommended leak detectors for the respective test methods

7 Selection of a Leak Detection Method

The demands of high technology industries and modern manufacturing forced leak detection to become highly specialized. Today, finding leaks in high speed manufacturing lines, in hostile environments or at microscopic sizes is a must.

As pointed out in chapter 1 a leakage rate is a measurable property of the part or system that needs to be tested. A tightness criterion has to be defined according to the functionality and durability of the respective object. Provided that the tightness criterion can be measured, the maximum leakage rate as an “accept / reject” criterion is defined in accordance with technical and economic considerations. Once the tightness criterion is defined, the most appropriate leak detection method can be selected.

7.1 Soaping

Applying a thin film of soapy water to a pressurized part is a traditional and simple method of leak detection. It is also a reliable way to locate large leaks and give some definition to the damage. Some of the drawbacks of this method include, possible damage, corrosion and spoiling the cosmetics of the tested parts. Soaping is also not a very sensitive or easily repeatable method of testing and quantification of leaks is not possible. This method is rather limited for industrial use as it is a qualitative and not a quantitative leak test method and can only detect large leaks.

7.2 Bubble Test

Everybody has been exposed to some basic leak detection. A leaking bike or car tire is a good example of both the dramatic effect of a leak on performance and how to diagnose and repair one. Submerging a pressurized object in water is a traditional and simple method of leak detection. It is also quite a reliable way to locate and give some definition to a large leak. A problem is that it is not always possible to submerge a suspected leak. Doing so also can easily cause damage through corrosion. Moisture also often spoils the aesthetics of the product or machinery. Under some conditions it can take a long time to dry.

Water is not a very sensitive or easily repeatable method of testing. Nor is it suitable for any high technology or fine work.

7.3 Pressure Decay

This method consists of pressurizing a part with a dry gas (most of the time dry air). The part is then disconnected from the pressurized gas source. The pressure inside the part is monitored and if a pressure drop were to occur, it would indicate the presence of a leak. This method is very common and has many uses as it gives the total leakage of a product. But pressure decay cannot locate leaks. It is also mainly used to identify larger leaks as small leaks are hard to identify with this method. Some products are also less suitable for pressure decay testing, such as

flexible materials, large internal volumes and products that are hot or cold or easily vary in temperature. Since a variation in temperature directly influences the pressure, it can easily be misinterpreted as a leak.

7.4 Tracer Gas Leak Detection

At Pfeiffer Vacuum we specialize in tracer gas leak detection. This is because the dominant tracer gas helium is highly sensitive, and detector technology is extremely selective. Helium is a light gas with fast diffusion and permeation times. It behaves consistently and allows testing to be infinitely repeatable which is a key factor especially in manufacturing. It does not use any medium which wets the part to be tested or can cause corrosion. With helium no sample post-treatment like drying is necessary after testing. Helium is a noble gas which does not react with anything. Helium is a 100 %

green gas without any environmental impact on the atmosphere. It is FDA approved and has food additive code number E939.

So why should one use a tracer gas leakage test? From the view of a production engineer the main advantages are:

- You repair only what needs to be repaired.
- You get fast quality feedback upstream in the production process.
- You find leaks that you cannot see.
- You avoid making expensive “over-quality”.
- You avoid having to dry/clean your products after the leak test.
- You can test hot/warm products.
- You may accept less alert operators.

Although tracer gas leak detection is not the least expensive leak test method with yet, this test method delivers a lot of financial benefits:

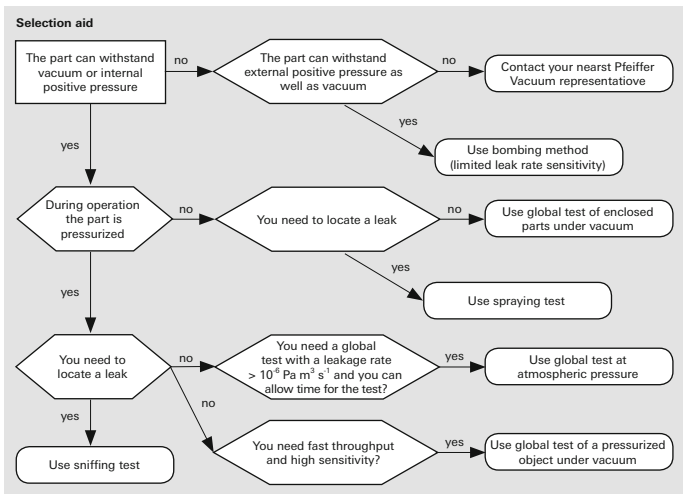


Table 7-1: Selection aid for leak testing methods

- Fewer process steps resulting in lower manufacturing costs per unit.
- Less scrap, faster throughput.
- Lower non-quality costs.
- Higher margins / better competitive edge.
- Less capital tied up in production.
- Better competitive edge through credibility and regulation-conformity.

Helium leak detectors can be used in different ways. Most of the time the selected method will be based on the application requirements. Please see the selection guide in Table 7-1 for more details.

7.5 Selection of Tracer Gases

For practical purpose, helium will be the tracer gas of choice. In some cases, forming gas ($N_2/H_2-95/5$) can also be a possible alternative for gross leaks only.

Pfeiffer Vacuum leak detectors are designed to be used with either or tracer gas.

With any other gas a quadrupole mass spectrometer-based gas analysis system is the instrument of choice.

8 Industrial Leak Detection Systems and Helium Recovery Units

8.1 Leak Detection Systems

Industrial leak detection of many parts can usually be made with standard leak detectors and manual operation. An example is leak-testing of sealed electronic components. The operator loads the samples in a test chamber integrated in the workspace of the leak detector and gets the result by a simple traffic-light style optical signal.

Often there is a requirement which a standard leak detector cannot meet. This can be traceability of a part through the production process, the amount of parts to be tested or in-line implementation of tightness control. High level of automation allows for example optimum reproducibility of measurements with defined background and signal-to-noise

ratio. Another criterion may be the wish to switch from a worker-based test to a fully automated test with automatic data analysis and non-erratic, operator-independent removal of leaking parts. Generally the requirements for an industrial leak detection system can be summarized as follows:

- high throughput of parts to be tested
- high process reliability
- high reproducibility of test results
- high level of automation
- high system uptime
- robustness in industrial production environment
- data analysis and transfer
- self-diagnostics of the system
- remote diagnosis and short-term availability of a world-wide service network

The main challenge in designing a leak detection system is the technical understanding of the part to be tested. Detailed process analysis combined with extensive leak detection and vacuum technology know-how are the key capabilities which are implemented in machine building.

For quantitative results we always need a test chamber. Mainly desired cycle time and tightness specification determine whether this can be an atmospheric chamber or needs to be a vacuum chamber. Short cycle times often require vacuum systems. So in a typical set-up we always have

- part to be tested
- customized adaptation unit
- test chamber
- leak detector
- certified calibration leak
- pumps for pre-evacuation of test object, chamber evacuation and tracer gas removal
- tracer gas supply
- system control
- handling system

Schematically this is shown in Figure 8-1.

Main applications of industrial leak detection systems are:

- Automotive
 - Fuel components: synthetic and metal tanks, carbon canisters, fuel rails and lines, fuel pumps and filters, gas caps
 - RAC (Refrigeration and air conditioning) components: compressors, condensers, refrigerant hose assemblies, refrigerant pumps, heater control valves, thermostat valves, high pressure valves
 - SCR (selective catalytic reduction) components: AdBlue® tanks, AdBlue® level sensors, AdBlue® hose assemblies, supply modules, dosing modules
 - Airbag components: igniters, inflators, gas generators
 - Air suspension systems components: air springs, air storage volumes, compressors, solenoid valves
- Medical Devices: catheters, pacemakers, endoscopes, disposable components like tubes and fittings
- Energy: High and medium voltage switches, surge arresters, solar receivers
- Industry: fire extinguisher, pressure vessels, heat exchangers, filters, pressure and flow sensors, food and pharmaceutical packaging
- Aviation industry: fuel systems, engines, landing gears, ventilation systems

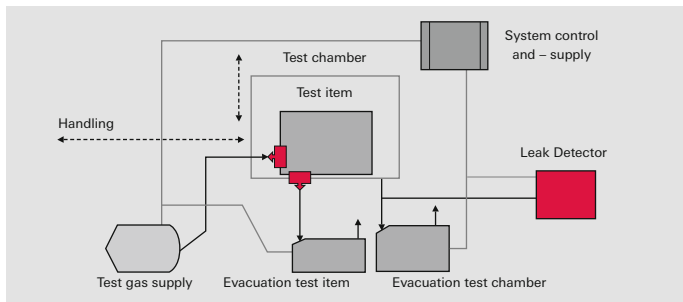


Figure 8-1: Set-up of an industrial leak detection system

8.1.1 Overview

Every leak detection system comprises a leak detector, pumping system, gas supply, and a test chamber. By standardization of these four main functional units one can achieve a low-cost single-chamber or dual-chamber system with manual loading. Customizations occur only regarding interior design of the test chamber and adaptation of the part to be tested.

Pfeiffer Vacuum's entry-of-range system LTS_{eco} is shown in Figure 8-2.



Figure 8-2: Pfeiffer Vacuum LTS_{eco}

This semi-automatic system can be equipped with one or two test stations and chambers with a volume of 50 liters maximum. Test chambers can be exchanged.

The LTS_{compact} series as shown in Figure 8-3 is a fully integrated leak detection system with minimum footprint of less than 2 square meters. With a chamber volume of 3 liters maximum it is designed for small parts. An optional high pressure gas supply unit allows for test pressure of 25 MPa. The automatic chamber cover is equipped with light barrier. The higher degree of automation can be complemented with a handling system.



Figure 8-3: Pfeiffer Vacuum LTS_{compact}

Calibration devices and adaptation tools (see below) are integrated in all Pfeiffer Vacuum leak detection systems. If the requirements cannot be met with LTS_{eco} or LTS_{compact} systems a customized system from the LTS_{clima} or LTS_{fuel} family can be tailored to the respective needs. Often highest throughput with a larger capacity than production rate is the target.

Production flow can be supported by product labeling and marking of good parts or destruction of bad parts still in the machine.

Modular design supports transportation and delivery, system upgrade or workshop re-organization. Modules are e.g. pumping module, chamber module, door module, and electrical cabinet.

In addition to the core task "leak detection" industrial systems can also integrate other functions like

- Mechanical tests
- Gas flow / throughput measurement (e.g. for tubes or lines)
- Electrical tests

Systems can also be designed for maximum flexibility to meet high resolution pressure control over a very wide range for development and industrialization of a new part design.

8.1.2 Adaptation

Adaptation with higher tightness level as the part to be tested is one of the key components of the entire system. Pfeiffer Vacuum relies on top-of-the range commercial products.

If standard tools are not available for the respective part Pfeiffer Vacuum is capable of designing own adaptation tools for all requirements:

- Smooth ends
- Crimped, conical, flared, flanged, or conical tube ends
- Mini diameters
- Internal or external thread

8.1.3 Calibration Leaks

Integrated calibration leaks with a slightly higher value than the defined threshold allow for calibration in the leakage range where the test occurs. Repeatability of the test can be monitored with the calibrated leak. Auto-calibration can be defined in regular time intervals or after a defined number of parts. Comparison of leak value and



Figure 8-5: Example of a calibration leak (capillary leak) integrated in a screw for production or a master part

background signal allows a precise determination of a minimum signal-to-noise ratio and reliability of the measurement. If a pre-defined background is not achieved the system starts with an automatic purge function which ensures optimum process reliability.



Figure 8-4: Example of a Pfeiffer Vacuum helium test leak

Integration of a calibration leak into a test dummy allows for construction of a master part. With a master part both quantitative calibration and integrity of adaptation tools are confirmed in a single test run under real conditions.



Figure 8-6: Example of Pfeiffer Vacuum Special adaptor tools

8.1.4 Application Example: Dual-Chamber System for Refrigerant Lines

The LTS_{clima} KLS/72 integral double-chamber helium leak detection system is employed for testing up to 200 automotive refrigerant lines per hour. The system's smallest detectable leak rate is $8 \cdot 10^{-7} \text{ Pa m}^3 \text{ s}^{-1}$ at a test pressure of 5 MPa and a helium concentration of 100 % (equivalent to a loss of 2 g of R134a refrigerant per year).

Special adaptor tools have been developed for the items to be tested. This was the prerequisite for best efficiency of the test cycle. Up to four identical refrigerant lines can be tested simultaneously. The system offers the option of either determining the total leak rate for the four refrigerant lines or the individual leak rates of each line in sequence, which involves a longer testing period. Moreover, it is also possible to use a hand-

held sniffer to identify the exact location of the leak on an individual line.

By attaching the adaptor tools to interchangeable frames, the entire leak detection system can be converted to handle a new type of refrigerant line in less than five minutes.

Parts which have successfully passed the test are marked, leaking parts are not. Marking is made still in the chamber and there is no need for manual intervention by the system operator after chamber opening. No leaking part can leave the factory.

The machine is controlled by a Siemens S7 PLC (other PLCs are available upon request), with the testing process displayed on a monitor. The monitor also allows simple access to the control system, which additionally stores further data as the process parameters, supplementary statistical data,

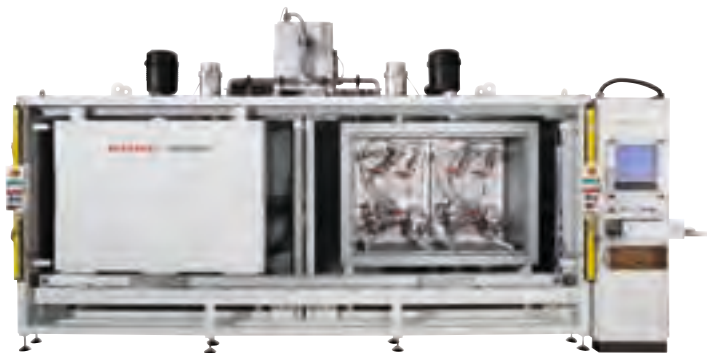


Figure 8-79: Helium leak detection system LTS_{clima} KLS/72 for leak test of refrigerant lines

| Leak Detection System Type | LTS _{clima} KLS/72 |
|----------------------------|--|
| Test item | Refrigerant lines with different geometries |
| Max. test pressure, air | 40 bar |
| Max. test pressure, helium | 50 bar |
| Helium concentration | 100 % (variable) |
| Capacity | 200 refrigerant lines per hour |
| Test chamber dimensions | 1500 x 1000 x 480 mm (W x H x D) |
| Specified leak rate | 2 g/a R134a = $8 \cdot 10^{-7} \text{ Pa m}^3 \text{ s}^{-1}$ at 5 MPa and 100 % helium |
| System dimensions | 5400 x 3100 x 3000 mm (W x H x D) |

Table 8-1: LTS_{clima}-KLS/72 operating parameters

error history, service mode, sniffer mode, individual line testing, etc.

In addition, the system is equipped with internal calibration leaks, which allow regular automated checking of the system for proper operation.

To minimize helium costs, a helium recovery unit (see next chapter) is attached to the leak detection system.

Operating parameters of helium leak detection system LTS_{clima} KLS/72 are summarized in Table 8-1.

8.2 Helium Recovery Units

A helium recovery unit collects the helium emanated from a helium leak detection system (possibly also a cryogenic system using helium as cooling agent) in a storage volume, and then prepares it for re-use. Connected with an leak detection system, helium concentration is constantly monitored by an online sensor, with helium replenishment from a standard gas cylinder if necessary.

Helium recovery systems mainly are used in applications with high tracer gas consumption, in other words when a large amount of large-volume parts need to be tested at high pressures. Recovery rates of more than 98 % can be achieved. An optional purification step allows for even higher gas qualities.

8.2.1 Overview

The parameters of standard helium recovery units are summarized in Table 8-2.

In addition, customized helium recovery units can be delivered. Based on the following parameters Pfeiffer Vacuum can propose dedicated solutions.

- cycle time
- volume of tested part
- parts throughput
- test pressure
- maximum and average gas throughput
- tracer gas concentration

8.2.2 Application Example

A standard HRU can be combined with one or more leak detection systems. The tracer gas used during the test is evacuated into a storage volume.

Let us consider the above-mentioned example for a leak detection system. If we assume four parts with an internal volume of 0.3 liters each, a test pressure of 5 MPa and a throughput of 200 samples per hour we end up at a gas consumption of 1,200 MPa l per hour. A standard 50 liters gas bottle with a filling pressure of 20 MPa contains 1,000 MPa l. With 100 % helium as tracer gas, two-shift operation and 260 working days per year we would need almost 7,000 helium bottles per year. With a helium recovery unit we can save up to 98 % of tracer gas. If we consider just tracer gas costs without electricity costs for the HRU a return on investment can be achieved in far less than one year.



Figure 8-8: Pfeiffer Vacuum helium recovery unit

| Parameter | Balloon | | | Container | | |
|-----------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| Type | CB13-35 | CB11-55 | CB10-200 | CV12-10 | CV16-55 | CV33-55 |
| Max. throughput | 210 NI min ⁻¹ | 180 NI min ⁻¹ | 180 NI min ⁻¹ | 200 NI min ⁻¹ | 260 NI min ⁻¹ | 550 NI min ⁻¹ |
| Max. working pressure | 3.5 MPa | 5.5 MPa | 20 MPa | 1.0 MPa | 5.5 MPa | 5.5 MPa |
| Compression | Oil-sealed | Oil-sealed | Oil-sealed | Oil-sealed | Dry | Dry |
| Recovery rate | < 95 % | < 95 % | < 95 % | < 98 % | < 98 % | < 98 % |

Table 8-2: Overview of standard helium recovery units by Pfeiffer Vacuum

9 Leak Detection Seminars and Practical Trainings

Pfeiffer Vacuum provides a complete training program for all topics related to vacuum technology and leak detection.

Leak detection basics and theoretical background are taught in seminars held by our experts. Operation and good leak detection practice are demonstrated in hands-on trainings under use of our leak detector technology. Users learn preventive maintenance and repair of their leak detectors under supervision of our experienced leak detection service colleagues.

A typical training for operators and maintenance staff of a vacuum system combines leak detection basics with just enough theoretical background to prepare for the job at hand. The various test methods are explained and valued according to the respective application. Systematic practical approach for leak detection is explained and trained. Many tips and hints are given for good leak detection practice.

In theoretical seminars for quality managers or designers of test equipment the backgrounds are explained in detail with application and calculation examples. We can guide you from a function-oriented specification to a quantitative definition of reject level and test recipe. Selection aid is given for the optimum test method in industrial leak detection along with detailed test procedure.

Of course, any of the leak-detection related courses can be combined with other modules for a user-oriented comprehensive training.

There are standard courses and customized trainings. Both can take place either in one of our training centers or at customer's site.

Please do not hesitate to check the Pfeiffer Vacuum web sites for trainings in the respective language. To discuss your individual needs please contact our world wide training manager

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10 Service and Maintenance

For us at Pfeiffer Vacuum, “service” does not only mean “repair” but rather extensive pre-sales and application support.

Pfeiffer Vacuum manufactures all key components of a leak detection solution including detector technology, high vacuum, roughing and backing pumps as well as chambers and mechanical components. For that reason we are not depending on external resources for service and maintenance but can fully rely on our own resources. This includes use of original spare parts and authorized service tools. Genuine Pfeiffer Vacuum parts and tools are created when the product is developed, are tested in extreme situations, and are therefore perfectly matched to every product. Every improvement of a leak detector, pump, or component is automatically incorporated in our genuine parts. Both, new and serviced products have to meet the same strict quality requirements.

The user manual of each leak detector contains recommendations for maintenance intervals. These recommendations are an average value from our installed base which needs to be adapted to the specific use of the instrument. The same applies for leak detection systems. After commissioning and the final acceptance test, the system is running in real production environment for several months. After a defined period, a status analysis is made and maintenance recommendations are provided by our leak detection experts. Wear analysis of the

respective leak detector or system yields recommendations for spare parts to be stored in order to allow fast repair on site. The contents of the spare parts package depend on the needs of the customer. Three service levels enable the adaptation of leak detection system needs, e.g. call-out time and international coverage.

Since education in leak detection is not contained in the majority of careers, we offer a wide range of trainings that are held by our leak detection experts who often are active members of the local NDT (non-destructive testing) societies and associations.

For standard leak detectors, the user can learn how to service and repair the own leak detector under survey of an experienced service technician in the course of a maintenance training.

Pfeiffer Vacuum operates roughly 50 service locations world wide. Therefore, we are able to provide fast repair of any standard component, and talk to operators in their native language. System maintenance during a planned down time is organized in advance in co-operation with operators, local service and – if needed – specialists from our headquarters. You will find a complete overview of our worldwide service locations under “Service > Worldwide Service Contact” on the internet: www.pfeiffer-vacuum.com



11 Addendum

11.1 Tables

11.1.1 Flow Rate Conversion Units

| | Pa m ³ s ⁻¹ | mbar · s ⁻¹ | Pa · s ⁻¹ | Torr · s ⁻¹ | atm cm ³ s ⁻¹ |
|-------------------------------------|-----------------------------------|-------------------------|-------------------------|-------------------------|-------------------------------------|
| Pa m ³ s ⁻¹ | 1 | 10 | 1 · 10 ³ | 7.5 | 9.87 |
| mbar · s ⁻¹ | 1 · 10 ⁻¹ | 1 | 1 · 10 ² | 7.5 · 10 ⁻¹ | 9.87 · 10 ⁻¹ |
| Pa · s ⁻¹ | 1 · 10 ⁻³ | 1 · 10 ⁻² | 1 | 7.5 · 10 ⁻³ | 9.87 · 10 ⁻³ |
| Torr · s ⁻¹ | 1.33 · 10 ⁻¹ | 1.333 | 1.33 · 10 ² | 1 | 1.32 |
| atm cm ³ s ⁻¹ | 1.01 · 10 ⁻¹ | 1.01 | 1.01 · 10 ² | 7.5 · 10 ⁻¹ | 1 |
| lusec (μ Hg · s ⁻¹) | 1.33 · 10 ⁻⁴ | 1.33 · 10 ⁻³ | 1.33 · 10 ⁻¹ | 1 · 10 ⁻³ | 1.32 · 10 ⁻³ |
| sccm | 1.69 · 10 ⁻³ | 1.69 · 10 ⁻² | 1.69 | 1.27 · 10 ⁻² | 1.67 · 10 ⁻² |
| slm | 1.69 | 1.69 · 10 ¹ | 1.69 · 10 ³ | 1.27 · 10 ¹ | 1.67 · 10 ¹ |
| Molecules s ⁻¹ | 3.77 · 10 ²¹ | 3.77 · 10 ²⁰ | 3.77 · 10 ¹⁸ | 2.83 · 10 ²⁰ | 3.72 · 10 ²⁰ |
| Mol s ⁻¹ | 2.271 · 10 ³ | 2.271 · 10 ⁴ | 2.271 · 10 ⁶ | 1.703 · 10 ⁴ | 2.24 · 10 ⁴ |

Table 11-1: Flow rate conversion units

11.1.2 Pressure Conversion Units

| | Pa | bar | hPa | mbar | Torr |
|--------|------------------------|-------------------------|-------------------------|------------------------|-------------------------|
| Pa | 1 | 1 · 10 ⁻⁵ | 1 · 10 ⁻² | 10 | 7.5 · 10 ⁻³ |
| bar | 1 · 10 ⁵ | 1 | 1 · 10 ³ | 1 · 10 ⁶ | 750 |
| hPa | 100 | 1 · 10 ⁻³ | 1 | 1000 | 0.75 |
| mbar | 0.1 | 1 · 10 ⁻⁶ | 1 · 10 ⁻³ | 1 | 7.5 · 10 ⁻⁴ |
| Torr | 1.33 · 10 ² | 1.33 · 10 ⁻³ | 1.33 | 1.330 | 1 |
| micron | 0.133 | 1.33 · 10 ⁻⁶ | 1.33 · 10 ⁻³ | 1.33 | 1 · 10 ⁻³ |
| atm | 1.01 · 10 ⁵ | 1013 | 1013 | 1.01 · 10 ⁶ | 760 |
| at | 9.81 · 10 ⁴ | 0.981 | 981 | 9.81 · 10 ⁵ | 735.6 |
| mm WS | 9.81 | 9.81 · 10 ⁻⁵ | 9.81 · 10 ⁻² | 98.1 | 7.36 · 10 ⁻² |
| psi | 6.89 · 10 ³ | 6.89 · 10 ⁻² | 68.9 | 6.89 · 10 ⁴ | 51.71 |
| psf | 47.8 | 4.78 · 10 ⁻⁴ | 0.478 | 478 | 0.359 |

Table 11-2: Pressure conversion units

| | lusec (μ Hg · s ⁻¹) | sccm | slm | Molecules s ⁻¹ | Mol s ⁻¹ |
|--|--------------------------------------|----------------------|----------------------|---------------------------|-----------------------|
| | $7.5 \cdot 10^3$ | $5.92 \cdot 10^2$ | $5.92 \cdot 10^{-1}$ | $2.651 \cdot 10^{20}$ | $4.403 \cdot 10^{-4}$ |
| | $7.5 \cdot 10^2$ | $5.92 \cdot 10^1$ | $5.92 \cdot 10^{-2}$ | $2.651 \cdot 10^{19}$ | $4.403 \cdot 10^{-5}$ |
| | 7.5 | $5.92 \cdot 10^0$ | $5.92 \cdot 10^{-3}$ | $2.651 \cdot 10^{17}$ | $4.403 \cdot 10^{-7}$ |
| | $1 \cdot 10^3$ | $7.89 \cdot 10^1$ | $7.89 \cdot 10^{-2}$ | $3.535 \cdot 10^{19}$ | $5.87 \cdot 10^{-5}$ |
| | $7.5 \cdot 10^2$ | $5.98 \cdot 10^1$ | $5.98 \cdot 10^{-2}$ | $2.679 \cdot 10^{19}$ | $4.45 \cdot 10^{-5}$ |
| | 1 | $7.89 \cdot 10^2$ | $7.89 \cdot 10^{-5}$ | $3.535 \cdot 10^{16}$ | $5.87 \cdot 10^{-8}$ |
| | $1.27 \cdot 10^1$ | 1 | $1 \cdot 10^{-3}$ | $4.486 \cdot 10^{17}$ | $7.45 \cdot 10^{-7}$ |
| | $1.27 \cdot 10^4$ | $1 \cdot 10^3$ | 1 | $4.486 \cdot 10^{14}$ | $7.45 \cdot 10^{-4}$ |
| | $2.83 \cdot 10^{17}$ | $2.23 \cdot 10^{16}$ | $2.23 \cdot 10^{19}$ | 1 | $1.66 \cdot 10^{24}$ |
| | $1.703 \cdot 10^7$ | $1.34 \cdot 10^6$ | $1.34 \cdot 10^3$ | $6.022 \cdot 10^{23}$ | 1 |

| | micron | atm | at | mm WS | psi | psf |
|--|-------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| | 7,5 | $9.87 \cdot 10^{-6}$ | $1.02 \cdot 10^{-5}$ | 0.102 | $1.45 \cdot 10^{-4}$ | $2.09 \cdot 10^{-2}$ |
| | $7.5 \cdot 10^5$ | 0.987 | 1.02 | $1.02 \cdot 10^4$ | 14.5 | $2.09 \cdot 10^3$ |
| | 750 | $9.87 \cdot 10^{-4}$ | $1.02 \cdot 10^{-3}$ | 10.2 | $1.45 \cdot 10^{-2}$ | 2.09 |
| | 0.75 | $9.87 \cdot 10^{-7}$ | $1.02 \cdot 10^{-6}$ | $1.02 \cdot 10^{-2}$ | $1.45 \cdot 10^{-5}$ | $2.09 \cdot 10^{-3}$ |
| | 1000 | $1.32 \cdot 10^{-3}$ | $1.36 \cdot 10^{-3}$ | 13.6 | $1.93 \cdot 10^{-2}$ | 2.78 |
| | 1 | $1.32 \cdot 10^{-6}$ | $1.36 \cdot 10^{-6}$ | $1.36 \cdot 10^{-2}$ | $1.93 \cdot 10^{-5}$ | $2.78 \cdot 10^{-3}$ |
| | $7.6 \cdot 10^5$ | 1 | 1.03 | $1.03 \cdot 10^4$ | 14.7 | $2.12 \cdot 10^3$ |
| | $7.36 \cdot 10^5$ | 0.968 | 1 | $1 \cdot 10^{-4}$ | 14.2 | $2.04 \cdot 10^3$ |
| | 73.6 | $9.68 \cdot 10^{-5}$ | $1 \cdot 10^{-4}$ | 1 | $1.42 \cdot 10^{-3}$ | 0.204 |
| | $5.17 \cdot 10^4$ | $6.8 \cdot 10^{-2}$ | $7.02 \cdot 10^{-2}$ | 702 | 1 | 144 |
| | 359 | $4.72 \cdot 10^{-4}$ | $4.87 \cdot 10^{-4}$ | 4.87 | $6.94 \cdot 10^{-3}$ | 1 |

11.1.3 Constants

| Bezeichnung | Symbol | Wert | Einheit |
|----------------------------------|-------------------|-------------------------|-----------------------------------|
| Avogadro-Konstante | N_A | $6.022 \cdot 10^{23}$ | mol^{-1} |
| Molvolumen bei Normalbedingungen | V_{mol} | 0.02241 | $\text{m}^3 \text{mol}^{-1}$ |
| Boltzmann-Konstante | k | $1.3807 \cdot 10^{-23}$ | J K^{-1} |
| Gaskonstante | $R = k \cdot N_A$ | 8.314 | $\text{J K}^{-1} \text{mol}^{-1}$ |
| Elementarladung | e | $1.602 \cdot 10^{-19}$ | C |
| Atomare Masseinheit | u | $1.661 \cdot 10^{-27}$ | kg |
| Stefan-Boltzmann-Konstante | s | $5.670 \cdot 10^{-8}$ | $\text{W m}^{-2} \text{K}^{-4}$ |
| Normaldruck | p_0 | 101325 | Pa |
| Normaltemperatur | T_0 | 273.15 | K |
| Normaldichte Luft | ρ_0 | 1.293 | kg/m^3 |
| Dyn. Viskosität Luft | η | $18.19 \cdot 10^{-6}$ | $\text{kg/(m} \cdot \text{s)}$ |

Table 11-3: Major natural constants for vacuum technology

11.2 Helium Leak Detectors – Overview of Series and Applications

| | Analytics | Semiconductors | Coating | Industry | R&D |
|-----------------------------------|---|----------------|---------|----------|-----|
| ■ recommended | Electron microscopy Leak detection Mass spectrometry Surface analysis Plasma monitoring Residual gas analysis Lithography PVD (Physical Vapor Deposition) CVD (Chemical Vapor Deposition) Plasma etching Implantation – source Implantation – beamline Inspection Bonding MBE (Molecular Beam Epitaxy) Load locks, transfer chambers, handling systems LED / OLED Flat Panel Display (FPD) Hard disk coating Photovoltaics Glass coating (PVD) CD/DVD/Blue-ray production (PVD) Optical coating (PVD) Wear protection (PVD, CVD) Hard coating Medical technology Industrial leak detection Electron beam welding Insulation vacuums Bulb and tube manufacturing Heat treatment Vacuum drying Vacuum furnaces Nuclear research Fusion technology Plasma research Particle accelerators Space simulation Low temperature research Elementary particle physics Nanotechnology Biotechnology | | | | |
| Portable | | | | | |
| MiniTest 300 | | ■ | ■ | ■ | ■ |
| ASM 310 | ■ | ■ | ■ | ■ | ■ |
| Multipurpose | | | | | |
| ASM 340 | ■ | ■ | ■ | ■ | ■ |
| ASM 340 D | ■ | ■ | ■ | ■ | ■ |
| High performance - Mobile | | | | | |
| ASM 380 | ■ | ■ | ■ | ■ | ■ |
| High performance - Compact | | | | | |
| ASM 182 T | ■ | ■ | ■ | ■ | ■ |
| ASM 182 TD+ | ■ | ■ | ■ | ■ | ■ |
| ASM 192 T | | | | | |
| ASM 192 T | | | ■ | ■ | ■ |
| ASM 192 T2D+ | | ■ | ■ | ■ | ■ |
| Workstation | | | | | |
| ASM 1002 | | | ■ | ■ | |
| Modular | | | | | |
| ASI 30 | | | | ■ | |
| Sniffing | | | | | |
| ASM 102 S | | ■ | ■ | ■ | ■ |

Table 11-4: Helium leak detectors – Overview of series and applications

11.3 Further Reading

11.3.1 Regulations and Guidelines

| Number | Date | Title |
|---------------------|--------------------|--|
| DIN EN 1330-8 | 1998-07 | Non-destructive testing - Terminology - Part 8: Terms used in leak tightness testing; Trilingual version |
| DIN EN 1518 | 1998-06 | Non-destructive testing – Leak testing Characterization of mass spectrometric leak detectors |
| DIN EN 1779 | 1999-10 2005-02 | Non-destructive testing - Leak testing Criteria for the method and technique selection Correction 1 |
| DIN EN 1593 | 1999-11 | Non-destructive testing - Leak testing Bubble emission techniques |
| DIN EN 13184 | 2001-07 | Non-destructive testing - Leak testing Pressure change method |
| DIN EN 13185 | 2001-07 | Non-destructive testing - Leak testing Tracer gas method |
| DIN EN 13192 | 2002-03 | Non-destructive testing - Leak testing Calibration of gaseous reference leaks |
| DIN EN 13625 | 2002-03 | Non-destructive testing - Leak testing Guide to the selection of instrumentation for the measurement of gas leakage |
| DIN EN ISO 9712 | 2012 | Non-destructive testing Qualification and certification of NDT personnel |
| DGZfP Guideline DP1 | 2005-06 | Guideline on the Selection of a suitable Tracer Gas for Leak Testing according to DIN EN 13185 (including an annex on the selection of a leak testing method according to DIN EN 1779); available in German and English language |
| DGZfP Guideline DP2 | 2009-12 | Richtlinie zur Umrechnung der mit Prüfgasen gemessenen Leckageraten in andere Medien (Gase, Flüssigkeiten), only available in German language |

Table 11-5: Regulations and guidelines

11.3.2 Books

1. Karl Jousten (ed.), Wutz Handbuch Vakuumtechnik, Vieweg 2013, 11th ed.
2. Jobst H. Kerspe (ed.), Vakuumtechnik in der industriellen Praxis, expert Verlag 2003, 3rd ed.
3. Klaus Kutzke, Dichtheitsprüfungen und Lecksuche mit dem Helium-Leckdetektor, expert Verlag 1998
4. Christian Edelmann, Vakuumphysik, Spektrum Verlag, 1998
5. P. O. Moore (ed.), Nondestructive Testing Handbook, Vol. 1, Leak Testing, 3rd ed., American Society for Nondestructive Testing, 1998

11.3.3 Websites

Pfeiffer Vacuum:
www.pfeiffer-vacuum.com

German Society for Non-Destructive Testing (FAQs in German):
www.dgzfp.de/Fachausschuesse/Dichtheitspruefung/FAQ.aspx

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Notes





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