1. Introduction

Definitions of some important terms should be given first of all.

The following are defined in DIN 28400 page 3, 1972:

2.3 Leak

Leaks in a vacuum system are holes or pores in the walls on the vacuum system or on connection points (welding seams, vacuum flanges, etc.), caused for example by material or processing faults or incorrect handling of sealing elements.

Other characteristic sizes are given in DIN 28400 part 8, 11.1997.

4.3 Outgassing rate of a vacuum system

Amount of gas, which is desorbed by all internal surfaces of the vacuum system for each unit of time (given as pV flow).

Note: In practice, outgassing inside a vacuum system can often simulate a leak. In this case, it is termed a “virtual leak”.

4.4 Vacuum system leak rate

The amount of gas flowing into the vacuum system for each unit of time given as pV-flow.

4.5 Pressure rise rate of a vacuum vessel

The pressure rise rate of a vacuum vessel (disconnected from the pump system) is the pressure increase per unit of time at a constant temperature.

2. Leak test using pressure rise method

The leak test is used to determine the leak rate, and not the outgassing rate.

Using this leak test method, the system – usually with its own pump system – is brought as near as possible to the ultimate pressure (vacuum), then the valves to the pump system are closed and at least two pressure measurements are taken at a set interval (downtime). The pressure increase method measures the amount of gas flowing into the vacuum chamber from the pressure through real leaks in the vacuum system and the pressure increase caused by the amount of gas given off by the surface of the vacuum system. This outgassing can therefore cause faulty readings.

2.1 Outgassing rate

The outgassing from the walls initially decreases approximately 1/t (t = time). The following example explains this:

The outgassing after 10 hours' pumping time amounts to only 1/10 of the outgassing after one hour of pumping time, and after 20 hours' pumping time only half of the value after 10 hours' pumping time, or 1/20 of the value after one hour of pumping time.

For this reason a pumping time of at least 10 hours is recommended if the part of the outgassing on the pressure rise measured should be kept low.

Fig 1 shows the outgassing rates (qG) to be expected after pumping times of 1, 10 and 100 hours related to the volume of the system V.

2.2 Measuring with cold traps

The disturbing outgassing rate is usually approx. 90% due to water vapour. This large amount of water vapour can be retained by a cold trap. On the other hand, air entering through leaks from the exterior contains only a small percentage of water vapour. With 1013 mbar (= 760 mm Hg), 20°C and 100% relative humidity, the partial pressure of the water vapour is about 23.4 mbar (= 17.5 mm Hg) or 2.3 %. The actual leak rate determined is therefore not influenced by the use of a cool trap.

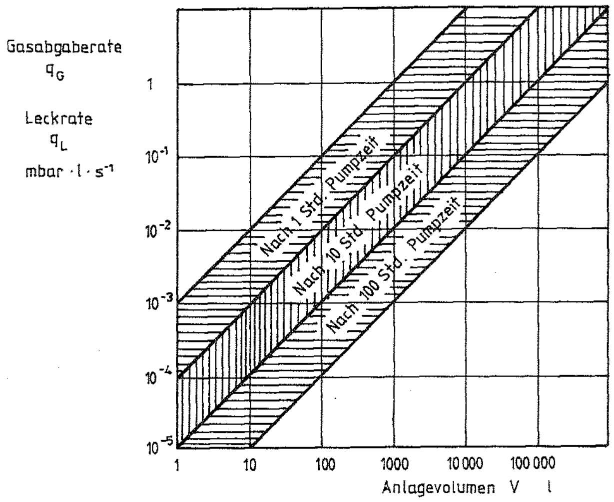
3. Check process and detection limits

The pressure rise method is a simple and comfortable test process that is used to determine the leak rate (gL) of a vacuum system. However because of strong influence of the outgassing of walls and components within the system, it can only be used for high leak rates – in the relation to the size of the system. As only the integral value, i.e. the total of all leaks, is always measured, it is not possible to localize a single leak in this way.

The detection limit of the leak rate is equal to the expected outgassing rate corresponding to the pumping time; it varies considerably depending on the type and size of the system (fig. 1).

After ten hours' pumping time, for example, leak rates can be detected of approximately

qL = 10 ^-5 and 10^-4 mbar * l * sec^-1 (system volume V in l).



Legend

Gasabgaberate qG	Outgassing rate qG
Leckrate qL	Leak rate qL
Nach 1 Std. Pumpzeit	After 1 hour pumping time
Nach 10 Std. Pumpzeit	After 10 hours' pumping time
Nach 100 Std. Pumpzeit	After 100 hours' pumping time

Fig. 1 Expected outgassing rate (qG) and leak rate detection limits (qL) related to system volume V.

For example: System volume V = 10000 l,
Leak rate detection limits between
qL ≥ 10^-5 * 10000 = 0,1 mbar * l * s ^-1 und
≥ 10^-4 * 10000 = 1,0 mbar * l * s ^-1 .

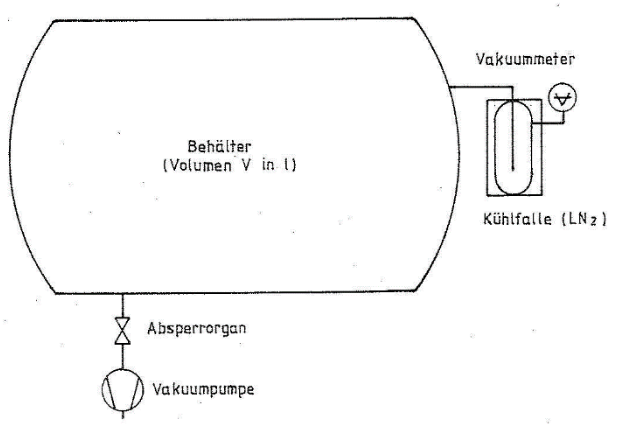
When using cold traps in front of the measuring device, the detection limit can be improved considerably. For example, after 10 hours' pumping time it lies between

qL ≥ 10^-6 * V and ≤ 10^-5 * V (in mbar * l * s ^-1).

Air and gas leaks are assumed. If there are leaks in the water cooling system, there may be larger differences.

4. Pressure rise in closed vacuum vessels

The principle schematic of a vacuum system with pressure measuring set-up is shown in fig. 2.



Legend

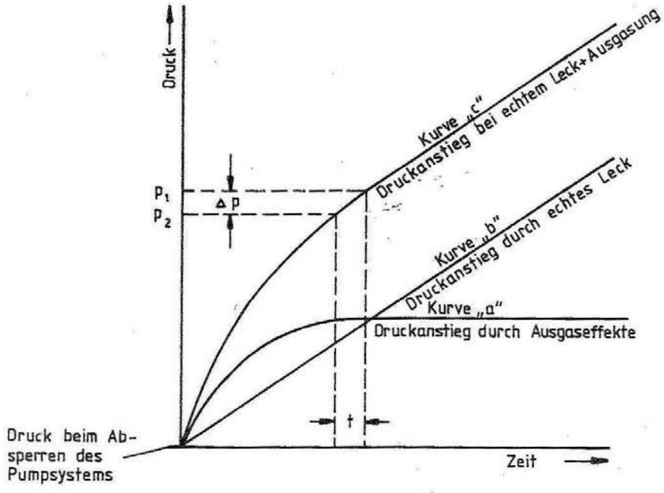
Behälter (Volumen V in l)	Vacuum vessel (volume V in l)
Absperrorgan	Stop valve
Vakuumpumpe	Vacuum pump
Vakuummeter	Vacuum meter
Kühlfalle (LN_2)	Cold trap (LN_2)

Fig. 2 Principle schematic of a vacuum system with pressure measuring set-up

The basic pressure rise as a result of the gas flow amounts caused by leaks and outgassing in the vacuum system is shown in fig. 3.

If the pressure rise is caused only by outgassing, the curve is generally proportional with the integral 1/t, i.e. logarithmic function of t, as shown in curve “a” in fig. 3. This applies as long as the saturated pressure of water vapour has not yet reached 23 mbar at room temperature (20°C). Depending on the size of the vacuum system, this can take quite a long time, e.g. with an outgassing rate qG = 0.1 mbar*l*s^-1 and a volume V = 10000 l, the time is t = 2.3 *10^6 s H 1 month.

However a leak results in a linear development of the pressure rise as shown by curve “b” in fig. 3.



Legend

Druck	Pressure
Druck beim Absperren des Pumpsystems	Pressure when sealing off the pump system
Kurve “C” Druckanstieg bei echtem Leck + Ausgasung	Curve “c“ – Pressure rise through real leak + outgassing
Kurve “b” Druckanstieg durch echtes Leck	Curve “b“ – Pressure rise through real leak
Kurve “a” Druckanstieg durch Ausgaseffekt	Curve “a“ – Pressure rise through outgassing
Zeit	Time

Fig. 3 Pressure rise in a vacuum vessel after sealing off the pump system (schematic)

Generally, however, the pressure rises analogue to curve “c”, which is a combination of the curves “a” and “b”.

The curve “a” shows the pressure rise after a relatively short pumping time and therefore the outgassing rate is still high at the start of the test. This disturbing part of the pressure rise is to a large degree switched off when the pressure rise is measured after longer pumping times, e.g. 10 hours or more, i.e. when the outgassing is considerably smaller or almost negligible.

Only the linear part of the curve “c" shall be used to determine the leak rate (qL). This gives the total leak rate (qL) approximated to:

$$qL = \Delta p * V / t \quad \text{in mbar} * l * s^{-1} ,$$

whereby Pressure rise between 2 measurements: $\Delta p = (p1 - p2)$ in mbar .
System volume: V in l
Time between 2 measurements: t in s

5. Pressure measuring devices

Generally, the pressure rise is measured in the vacuum system using a built-in measuring device. If this device is not accurate enough, an ionization vacuum meter, for example, shall be used. If the leak rate limit is approximately in the region of the expected outgassing rate (qG) according to the specification (see fig. 1), then a cold trap with liquid nitrogen, for example, shall be installed in front of the vacuum meter.

6. Test personnel

The leak test shall be carried out by the vacuum test personnel. The preparation, carrying out and evaluation of these tests shall be done by personnel who are qualified and have the necessary training and experience.
The test supervisor shall check all procedures and is responsible for ensuring the maintenance of approved technology.

7. Preparing for the leak test

The interior of the system to be tested shall be clean, dry, and free of rust, burnt residue, oil and other materials that produce gases. Components, which are not included in the leak test, such as holders, cages, cover plates, crucibles, etc. shall be dismounted. Fingerprints shall be avoided due to outgassing, wear protective gloves.

8. Carrying out the leak test using pressure rise method

It is not possible to provide a single applicable, detailed guideline for all systems. The following routine can however be followed when testing most systems:

a.) Pump down vacuum system to ultimate vacuum; pumping time at least 10 hours.

b.) Close valves to pump system.

c.) Measure pressure rise for approx. 0.5 hours, whereby the pressure should rise to at least double, i.e. factor 2.

d.) The measurement can be repeated after a short pumping time to check the values measured.

e.) When measuring with cold traps, the pressure rise shall first be measured before the trap is cooled and again when the trap is cool. (If there is little or no change in the rate measured, there is an air leak). The greater the rate change, the larger the portion which is caused by outgassing.

f.) Leaks in the water circuit are determined when the trap is not cooled, e.g. if a change in the water pressure is detected.

g.) To localize individual leaks, other suitable methods shall be used, e.g. leak test using "spray test" (5.550-6046) or over pressure method using foaming spray (5.550-6047).

9. Assessment and acceptance report

The outgassing and leak rates allowed shall be taken from the drawing or specification.
The specified values shall be recorded in the test report:

-- Volume V in l
-- Measuring time t in s
-- Total pumping time h (Desorption time)
-- Ultimate pressure p1 in mbar
-- Starting pressure p2 in mbar and
-- Differential pressure $\Delta p = (p1 - p2)$ in mbar .

The outgassing and leak rate (q) shall be calculated from these values in accordance with the formula:

$q = \Delta p * V / t = (p1 - p2) * V / t$ in mbar * l*s^-1 .

The leak rate determined must be smaller than that required; it can be larger than, or as large as, the leak rate detection limit (see fig. 1).

If the leak rate determined is larger than the value required, the measurement shall be repeated and the reason for the high leak rate determined.

The responsible tester shall enter the measured results and their evaluation in the test protocol of the system and also in the quality test certificate.

Note:

The system specification shall state the method which shall be used to determine the leak rate and how large it may be. The leak rate cannot lie under the detection limits, i.e. under the outgassing rate expected for the type and size of system and the pumping time, if this is determined according to the pressure rise method.

For example, it shall be entered in the specification as:

Leak rate $q \leq 1 * 10^{-3}$ mbar * l / s ,

determined by the leak test using the pressure rise method (factory standard 5.550-6048) after 10 hours pumping time.

This ensures that the method of measurement and evaluation is clearly stated. This will prevent any misunderstandings and disagreements between customers and Leybold Optics concerning leak rates promised and reached.