A NEW CALIBRATION APPARATUS FOR ATMOSPHERIC HELIUM AND HYDROGEN LEAK STANDARDS

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Motivation

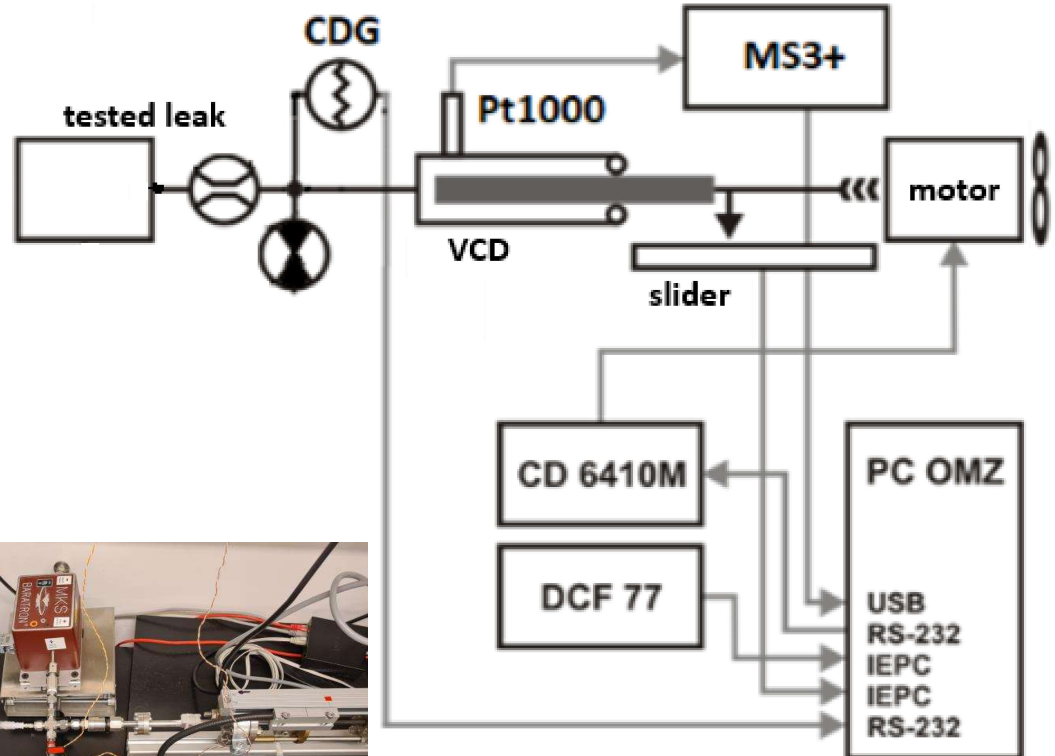
- The Czech Metrology Institute routinely provides the calibrations of *vacuum* helium leak standards. However, the calibrations of *atmospheric* helium leak standards (for sniffer mode applications) are also increasingly requested.
- A leak detector in sniffer mode sucks the air from the ambient atmosphere at a constant flow rate and feeds it into a turbomolecular vacuum pump, which is used to pump the mass spectrometer. It is necessary to use a source of known helium flow into the atmosphere to calibrate the detector in this mode. The standard helium leaks with output to the atmosphere serve for this.

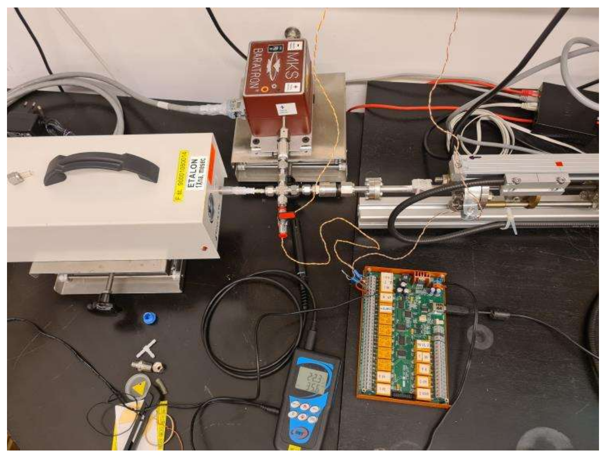
Motivation

- The standard atmospheric leaks consist of a helium reservoir with a control pressure gauge, a pressure reducing valve to set a small constant gauge pressure (up to a few kPa) and a capillary, the conductivity of which is chosen so that the helium flow at the given gauge pressure meets the needs - typically about $2 \cdot 10^{-5}$ mbar·L/s.
- However, applications of hydrogen as a tracer gas are also emerging, where a safe mixture of hydrogen and nitrogen (usually, 5% hydrogen and 95% nitrogen) is used as tracer gas. The sought-after traceability solution should therefore ideally also cover the possibility of ensuring traceability for the hydrogen standard leaks.

Volume Calibration Device

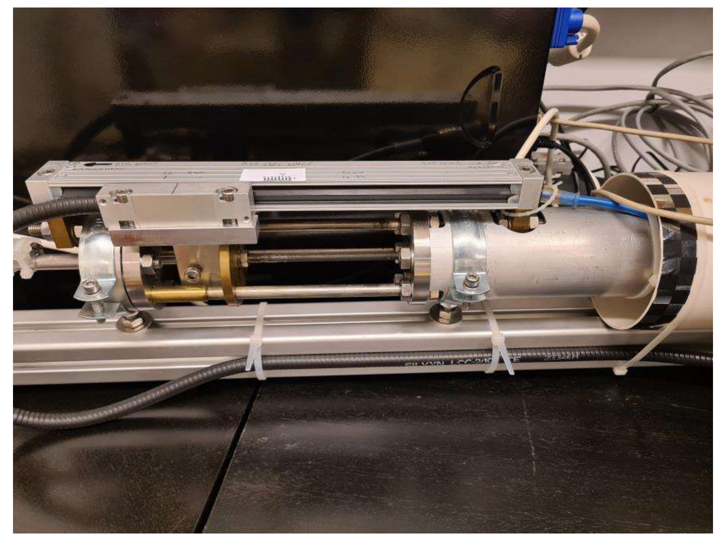
- A Volume Calibration Device (VCD) was originally developed and used to determine the variable volumes of the bellows of a constant pressure – variable volume flowmeters.
- The base of the VCD is a precisely dimensionally calibrated piston, connected to a stepper motor, which ensures its defined extraction from the VCD body according to a pressure gauge signal.
- An output of a calibrated leakage can be connected to the VCD instead of the bellows.





VCD

- The calibrated leak output is connected to the VCD by an element with a minimal internal volume.
- The VCD piston is connected to a stepper motor via an M6×1 threaded rod, which ensures a defined piston extraction from the VCD body. The stepper motor is cooled by a fan to minimize the thermal influences.
- The piston extraction position is detected optoelectronically (ESSA - SL126-170-LP/40/S-2,0OH-CA9) and recorded using an IEPC USB card in a controlling PC.
- A Pt1000 temperature sensor is connected to the VCD body, read by a Comet MS3+ data logger connected to the PC via a serial bus.
- The CD 6410M stepper motor electronics is connected to the PC via RS-232 communication line. A pressure gauge (either absolute or differential) is connected likewise.



Operation

- The VCD allows the use of any non-agressive gas. Hydrogen, helium, nitrogen and dry air were used for system tightness tests.
- After starting the program, the control computer starts moving the piston and automatically stops when the desired position is reached. The piston returns to the starting position with the valve open.
- During the piston movement, especially during the relatively rapid return of the piston to the starting position, friction generates the heat that negatively affects the stability of the entire system. For this reason, it is necessary to wait for the system to stabilize before further measurement (judged from the temperature measurement record).
- Further, it was necessary to develop a method of using this volumetric standard as a leak rate standard. An algorithm was created, combining in one procedure both a determination of the actual dead volume of the system (necessary for optimal evaluation) and a control loop maintaining a pressure difference between the internal volume and the atmosphere within several pascals.

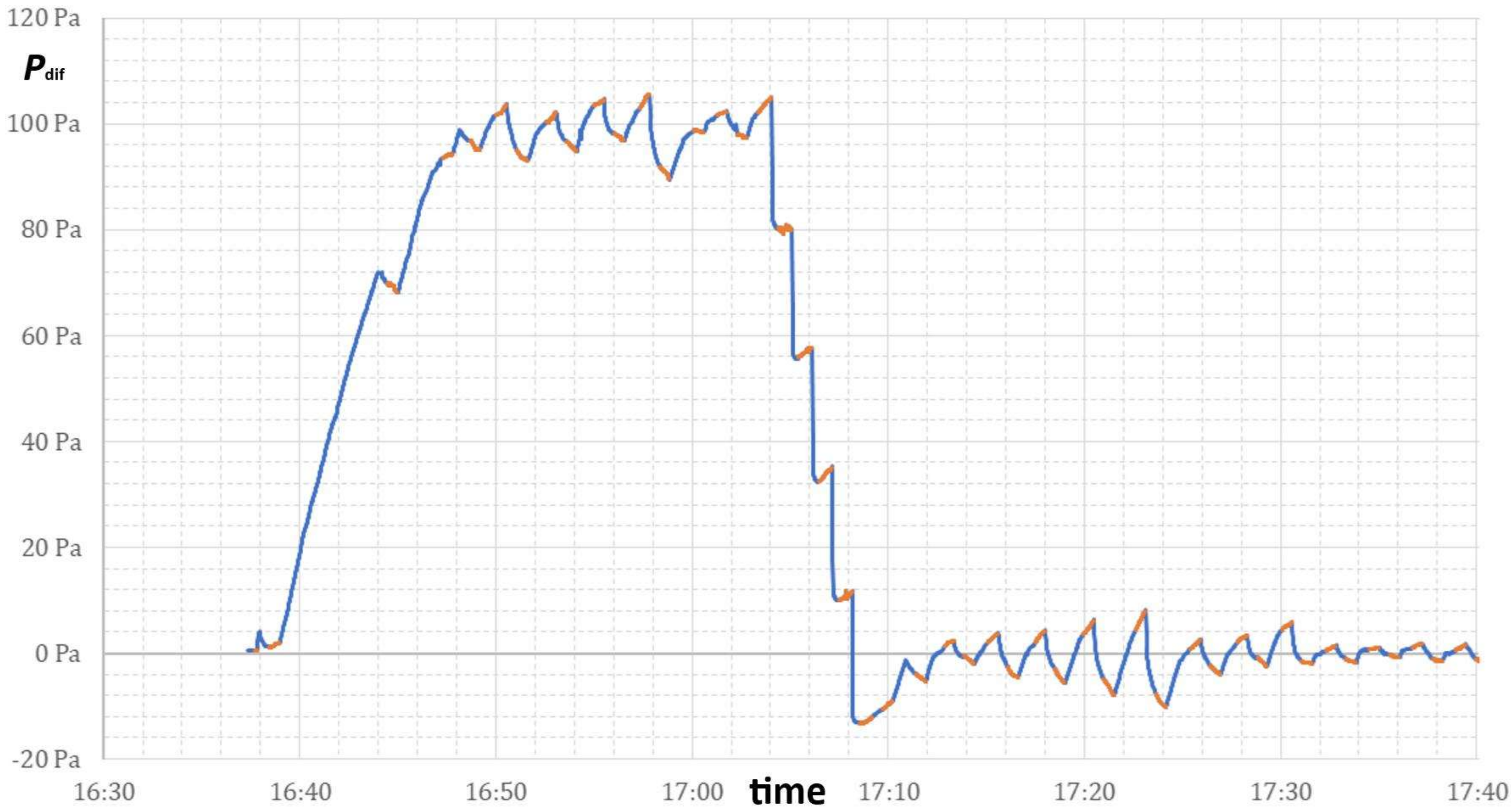
Measurement procedure

The measurement procedure is controlled by a program in the FreeBasic environment and includes the following steps:

1. Initialization of the stepper motor.
2. Opening the VCD valve - connecting the volume to the atmosphere.
3. Inserting the piston.
4. Reading the zero of the differential gauge.
5. Closing the valve - separating the volume from the atmosphere.
6. *Alternative determination of the dead volume estimate using several step changes in the piston position; first, a slightly higher gauge pressure in the system is intentionally set (100 Pa), the piston displacement is started so that the pressure is kept constant, and a step change in volume is performed five times. From the system reaction, it is possible to estimate the current dead volume.*
7. Starting the regulation - actual measurement of the leak rate, usually takes place until the piston reaches its extreme position.
8. Opening the VCD valve - connecting the volume to the atmosphere.
9. Returning (inserting) the VCD piston.

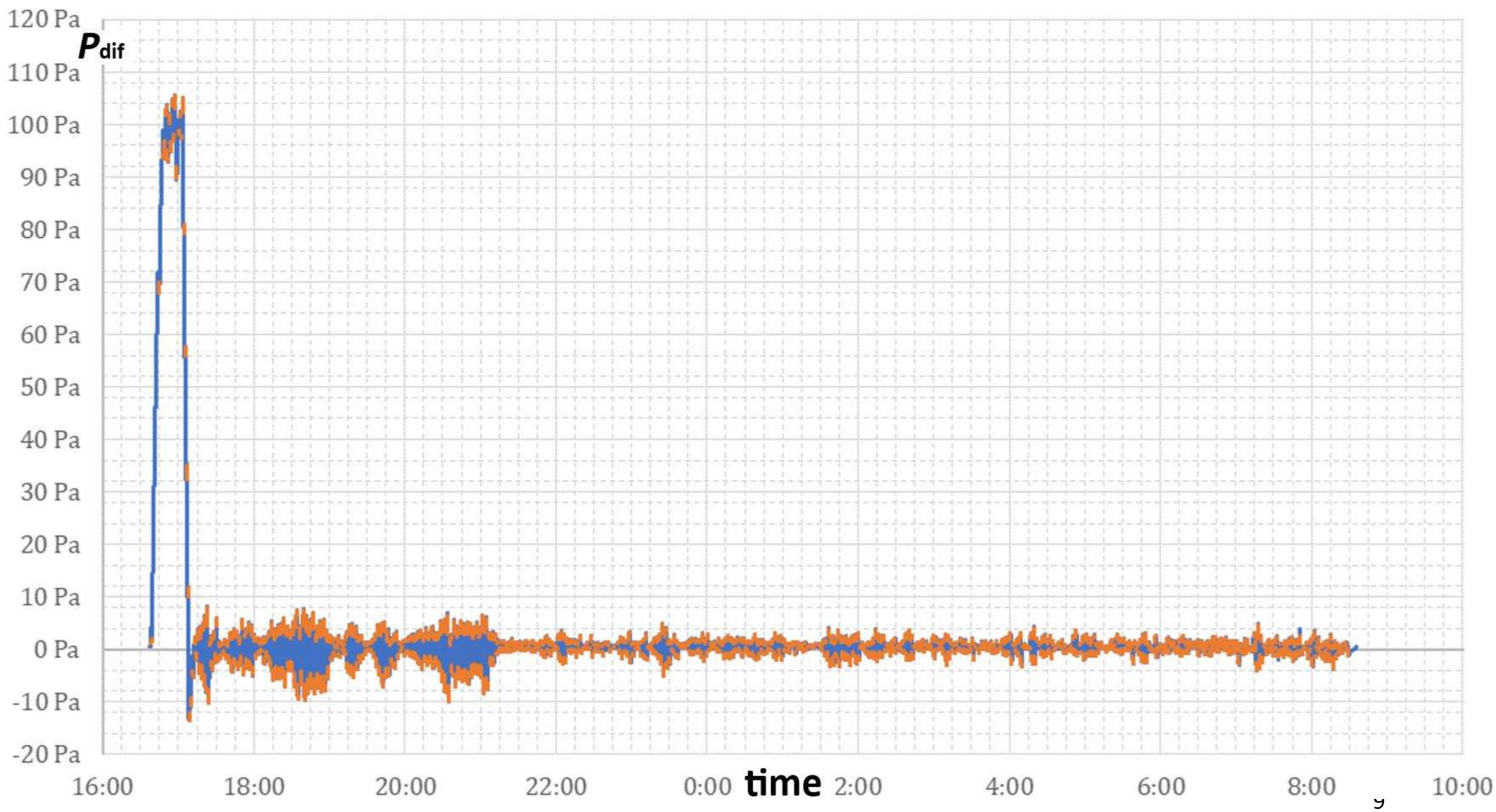
Measurement procedure

Differential pressure gauge indication during a start of a leak calibration: setting the gauge pressure to 100 Pa, regulation, five step changes in volume and regulation around 0 Pa.



Measurement procedure

Differential pressure gauge indication during the entire calibration. This is the same recording as in the previous image, but on a wider time scale. The selected regulation maintains the gauge pressure in the volume within a range of up to a maximum of 10 Pa, in the later part of the recording up to several Pa.



Flow rate determination

- Range (2·10^{-6} to 2·10^{-3}) Pa·m³/s is given by a time needed for one piston run over its entire stroke from (1 d 15 h 16 min) to (2 min 21 s).
- Gas volume within the closed space recalculated to the standard conditions is:

$$V_{\mathrm{STD}} = [V_{\mathrm{D}} + Sx_{\mathrm{i}} + S(x - x_{\mathrm{i}})] \cdot \frac{p}{T} \cdot \frac{T_{\mathrm{STD}}}{p_{\mathrm{STD}}}.$$

- We neglect the compresibillity changes. Gas flow at the standard conditions (in this field usually 1 atm or 1 bar abs. and 23 °C) is for time interval Δt given as:

$$q_{\mathrm{STD}} = \frac{1}{\Delta t} \cdot \frac{T_{\mathrm{STD}}}{p_{\mathrm{STD}}} \cdot \left([V_{\mathrm{D}} + Sx_{\mathrm{i}} + S(x_{\mathrm{e}} - x_{\mathrm{i}})] \cdot \frac{p_{\mathrm{e}}}{T_{\mathrm{e}}} - [V_{\mathrm{D}} + Sx_{\mathrm{i}}] \cdot \frac{p_{\mathrm{i}}}{T_{\mathrm{i}}} \right).$$

$$q_{\mathrm{STD}} = \frac{S \cdot x_{\mathrm{e}}}{\Delta t} \cdot \frac{T_{\mathrm{STD}}}{p_{\mathrm{STD}}} \cdot \frac{p_{\mathrm{e}}}{T_{\mathrm{e}}} \cdot \left(1 + \frac{x_{\mathrm{D}}}{x_{\mathrm{e}}} - \frac{x_{\mathrm{i}} + x_{\mathrm{D}}}{x_{\mathrm{e}}} \cdot \frac{p_{\mathrm{i}}}{T_{\mathrm{i}}} \cdot \frac{T_{\mathrm{e}}}{p_{\mathrm{e}}} \right).$$

$$q_{\mathrm{STD}} = \frac{S \cdot x_{\mathrm{e}}}{\Delta t} \cdot \frac{T_{\mathrm{STD}}}{p_{\mathrm{STD}}} \cdot \frac{p_{\mathrm{e}}}{T_{\mathrm{e}}} \cdot C.$$

Uncertainty determination

- Relative type-B uncertainty is given as:

$$u_{\mathrm{Br}}(q_{\mathrm{STD}}) = \sqrt{u_{\mathrm{r}}{}^2(S) + u_{\mathrm{r}}{}^2(x_{\mathrm{e}}) + u_{\mathrm{r}}{}^2(\Delta t) + u_{\mathrm{r}}{}^2(p_{\mathrm{e}}) + u_{\mathrm{r}}{}^2(T_{\mathrm{e}}) + u_{\mathrm{r}}{}^2(C)}\,.$$

- Typical input values are:

$u_{\mathrm{r}}(C) = 1.6 \cdot 10^{-3}$, dominated by $u_{\mathrm{r}}(V_{\mathrm{D}}) = 0.1$

$$u_{\mathrm{r}}(S) = 8 \cdot 10^{-4}$$

$u_{\mathrm{r}}(p_{\mathrm{e}}) \leq 1 \cdot 10^{-4}$, but adding compressibility changes $u_{\mathrm{r}}(p_{\mathrm{e}}) \leq 3.2 \cdot 10^{-4}$

$$u_{\mathrm{r}}(T_{\mathrm{e}}) \leq 1.5 \cdot 10^{-4}$$
$$u_{\mathrm{r}}(\Delta t) = 5 \cdot 10^{-5}$$
$$u_{\mathrm{r}}(x_{\mathrm{e}}) = 2.5 \cdot 10^{-5}$$

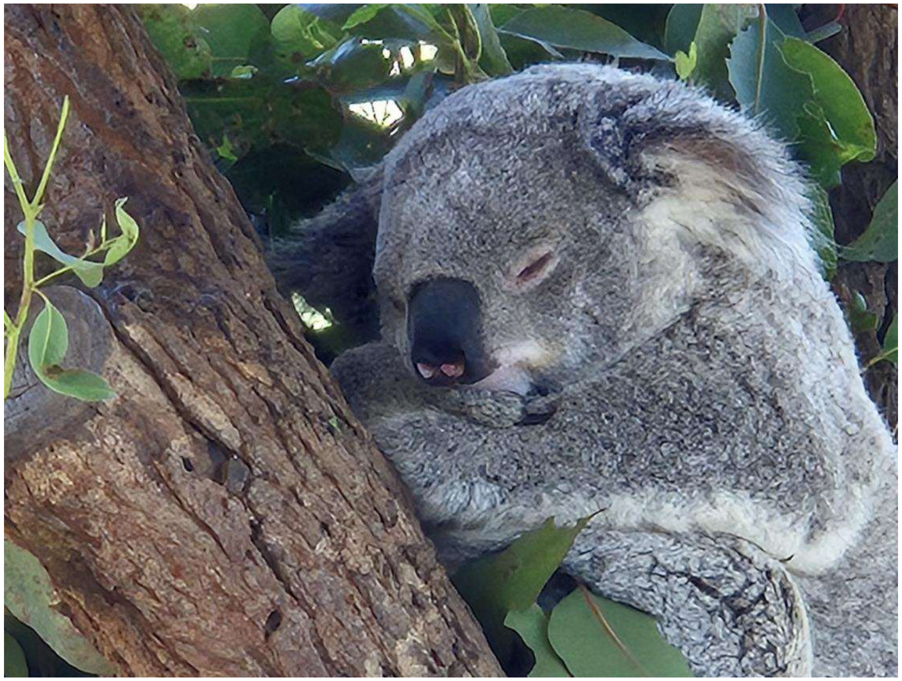
To the resultant $u_{\mathrm{Br}}(q_{\mathrm{STD}}) = 0.18\ \%$, we add a typical dispersion of one measurement (piston run) 1 % and repeatability (several runs) again cca 1 %, resulting for ($k = 1$): $u_{\mathrm{C}}(q_{\mathrm{STD}}) = 1.4\ \%$. (For high flows – a start-stop error cannot be neglected.)

Results

- In this way, the leak rates of Inficon type 12238-TL5 (helium, resultant value $1.86 \cdot 10^{-5}$ mbar·dm³/s, manufacturer's value $2.1 \cdot 10^{-5}$ mbar·dm³/s) and Pfeiffer Vacuum type CL004/BG447705-T (hydrogen, resultant value $2.64 \cdot 10^{-5}$ mbar·dm³/s, manufacturer's value $2.8 \cdot 10^{-5}$ mbar·dm³/s) were determined. Both values are lower than the values stated by the manufacturer, by 11% and 6% respectively. However, the manufacturer's values have a relative uncertainty of 15%, so a good agreement can be stated.
- The method was also tested on Inficon type 12240 TL134 with working medium R134a (1,1,1,2-tetrafluoroethane), leak rate $7.28 \cdot 10^{-5}$ mbar·dm³/s (9.48 g/a R134a). During the measurements, we observed a decrease of several percent in the measured values in successive measurements. Such a systematic difference was not observed with helium or hydrogen.
- A possible explanation is the absorption of R134a in the material of some part of the VCD volume (sealing of piston). The phenomenon is faster and more intense, the higher the concentration of the medium in the measured volume. After purging with pure nitrogen, the measured value “returns” to its original level in the next measurement. For routine use, we seek to eliminate it by using suitable components and materials. (For He a H_2 polyamide seems OK, but problems with polyurethane and silicone. For R134a polyamide acceptable but teflon better.)

Conclusion and future

- The calibration method for atmospheric helium and hydrogen leak standards was established over a range of $(2 \cdot 10^{-6}$ to $2 \cdot 10^{-3})$ Pa·m³/s with an uncertainty of about 3-5 % (the nominal values usually state 15 %).
- After research into material compatibility and appropriate modification of the apparatus, the method is also planned for the atmospheric leak standards for refrigerants (such as 1,1,1,2-Tetrafluoroethane).
- Because $2 \cdot 10^{-3}$ Pa·m³/s equals 1.2 sccm (standard cm³/min) it could allow a comparison with our gravimetric low gas flow standard that goes down to 1 sccm with expanded uncertainty cca 0.7 %.
- An interlaboratory comparison with other national metrology institutes.
- Experiments in a vakuum leak set-up (piston insertion).



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THANK YOU VERY MUCH FOR YOUR ATTENTION

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