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DESIGN OF HELIUM PERMEATION REFERENCE LEAKS WITH GENERATED FLOWS BELOW 10^{-14} mol/s

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Abstract – The calibration of helium permeation reference leaks below 10^{-14} mol/s is presented. The reference leak is based on the known construction with a He reservoir and a permeable glass tube leak element. The leak rate is determined *in situ* using the rate of pressure rise method. The reference leak is equipped with a small calibration volume containing a miniature Ti-getter pump to reduce the residual pressure rise due to outgassing and a spinning rotor gauge (SRG) to measure the rate of He pressure rise. In vacuum metrology, the SRG has been recognized as a transfer standard due to its inertness, high resolution and excellent long-term stability. The He leak rate ranging from $4{,}0\times10^{-17}$ mol/s to $1{,}0\times10^{-14}$ mol/s, measured at the reference leak temperature of 23°C, is linearly dependent on the He fill pressure ranging from 4,6 Pa up to 1120 Pa. Multiple He flow measurements in the 10^{-15} mol/s range show that a measurement uncertainty less than 2% can be achieved.

Keywords: gas permeation, gas flow, spinning rotor gauge, helium reference leak

1. INTRODUCTION

He permeation leaks are based on the permeation of He from a He reservoir through a glass membrane into vacuum. At NIST, USA, small He permeation flows down to 10^{-14} mol/s were generated and calibrated against the primary leak standard in dependence on the He reservoir concentration, with a measurement uncertainty up to 5% [1].

1.1. Definition of a gas flow

Gas quantity n in (mol) can be obtained from the ideal gas law equation:

$$n = \frac{PV}{RT}. \tag{1}$$

The derivation of (1) with respect to time gives for a gas flow dn/dt in (mol/s) at T = const. the following equation:

$$\frac{dn}{dt} = \frac{1}{RT}\left(P\frac{dV}{dt} + V\frac{dP}{dt}\right). \tag{2}$$

1.2. Rate of pressure rise method

The rate of pressure rise method has been selected to determine an unknown gas flow dn/dt in spite of reported disadvantages [2]. The method is based on the measurement of a linear pressure rise $\Delta P/\Delta t$ in the evacuated volume V at T = const as obtained from (2):

$$\frac{dn(T)}{dt} = \frac{V}{RT}\frac{\Delta P}{\Delta t}. \tag{3}$$

2. DESCRIPTION OF A He REFERENCE LEAK

2.1. Construction of a He reference leak

The cross-section of a He reference leak is presented in Fig. 1. The main features of this construction (compared to the standard He permeation test leaks) are as follows:

- ultrahigh vacuum (UHV) all-metal construction
- fill valve enabling the refilling of the reservoir with He
- demountable leak element enabling the use of different glass tube materials and dimensions
- demountable calibration volume enabling *in situ* calibrations using the rate of pressure rise method
- miniature Ti-getter pump which is used to reduce the residual pressure rise in the calibration volume
- ball/flange assembly of the SRG, which is used for He pressure rise measurements in the calibration volume and is chosen due to its inertness, high resolution (10^{-6} Pa) and excellent long-term stability (< 1% / year)

2.2. Gas flow of a He reference leak

The molar flow dn/dt of a He permeation leak has been found empirically to obey the following relationship [3]:

$$\frac{dn(T)}{dt} = ATe^{-B/T}. \tag{4}$$

The He pressure P in the reservoir is proportional to the absolute temperature T, therefore (4) can be rewritten as follows [4]:

$$\frac{dn(T)}{dt} = C(T)P, \tag{5}$$

where *C(T)* is linearly proportional to permeability and area of the leak element and inversely proportional to its thickness.

According to (5), the molar leak rate at the fill temperature T_f amounts to:

$$\frac{dn(T_f)}{dt} = C(T_f)P_f\,, \tag{6}$$

where P_f is the fill pressure.

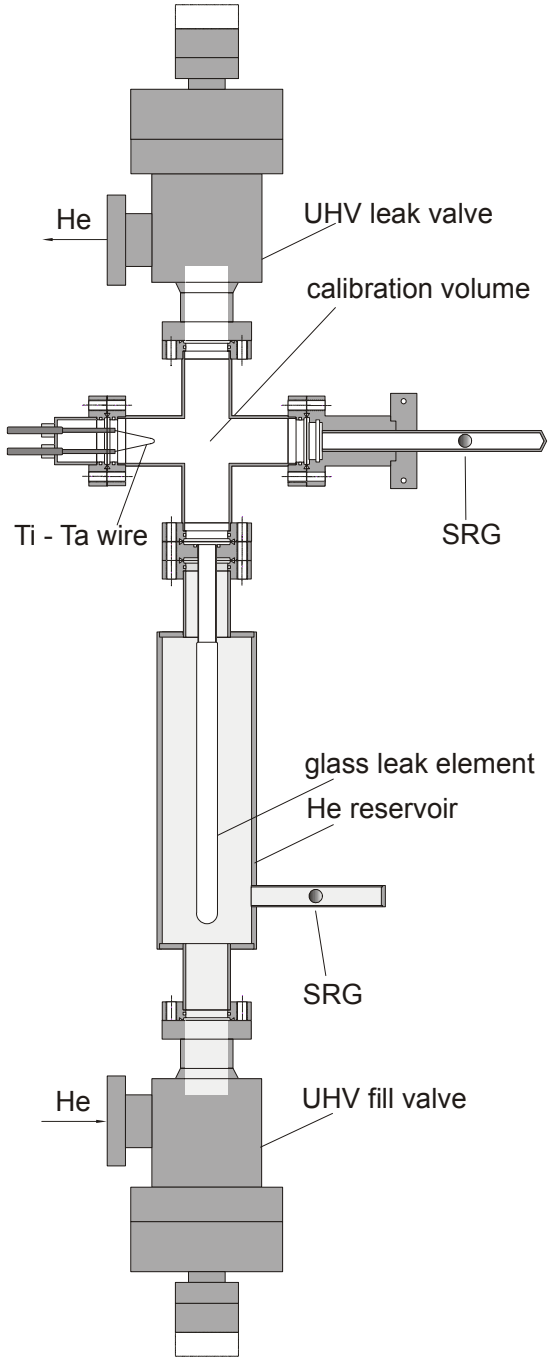

Fig. 1. Cross section of a reference He permeation leak

3. RESULTS

First a residual pressure increase was measured and the result is shown in Fig. 2. Large deviations from the linear pressure increase were caused by the variations of the room temperature (±2°C). The equivalent He flow due to the outgassing of inner surfaces of $(1{,}7\pm0{,}6)\times10^{-18}$ mol/s was calculated from the measured slope amounted to $(3{,}7\pm1{,}3)\times10^{-7}$ Pa(He eq.)/hour and the volume V = $(40{,}0\pm0{,}2)$ cm^3. In the subsequent He flow measurements better control of the ambient temperature around the measurement set-up was assured (±0,5°C).

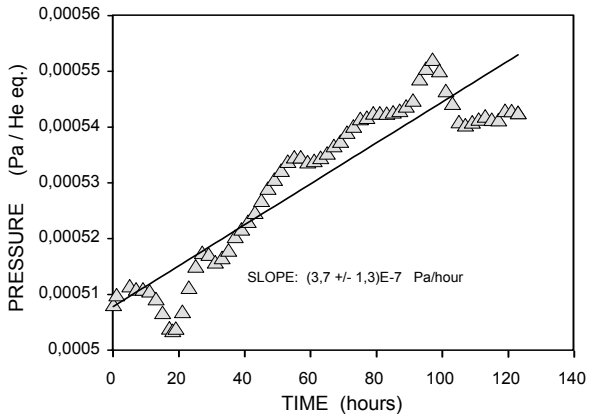

Fig 2. Residual pressure increase at a flow of $1{,}7 \times 10^{-18}$ mol/s due to the outgassing of inner surfaces

At the temperature of 23°C, the He reservoir was filled at four fixed pressures ranging from 4,6 Pa up to 1120 Pa which were measured using either a SRG or a capacitance diaphragm gauge (CDG), starting from the lowest He fill pressure. After each fill we had to wait at the fill temperature for 10 days to establish the steady state flow. The results of the flow measurements are summarized in Fig. 3 and Fig. 4. After filling the reservoir with He at the

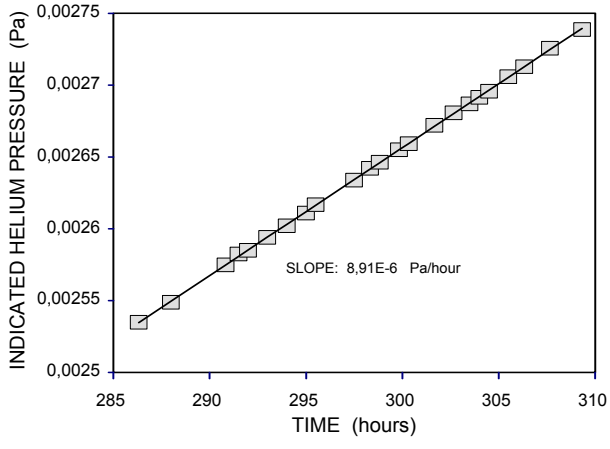

Fig. 3. Measurement of the He pressure increase with a slope of $8{,}91\times10^{-6}$ Pa/hour resulting in a calculated He flow of $4{,}0\times10^{-17}$ mol/s

fill pressure of 4,6 Pa (measured with the SRG) and establishing the steady state He flow, the He pressure increase with a slope of $8{,}91\times10^{-6}$ Pa/hour was measured for about 1 day which is shown in Fig. 3. The calculated He flow amounted to $4{,}0\times10^{-17}$ mol/s. After filling the reservoir with He at the fill pressure of 1120 Pa (measured with the CDG) and establishing the steady state He flow, the He pressure increase with a slope of $2{,}22\times10^{-3}$ Pa/hour was measured for about 3 hours which is shown in Fig. 4. The calculated He flow amounted to $1{,}0\times10^{-14}$ mol/s.

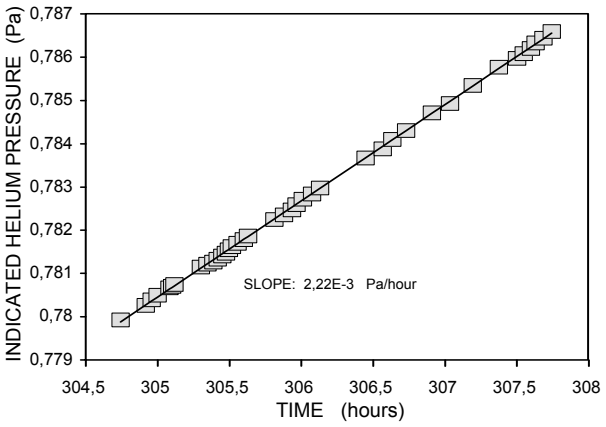

Fig. 4. Measurement of the He pressure increase with a slope of $2{,}22\times10^{-3}$ Pa/hour resulting in a calculated He flow of $1{,}0\times10^{-14}$ mol/s

The dependence of the He leak rate, ranging from $4{,}0\times10^{-17}$ mol/s to $1{,}0\times10^{-14}$ mol/s, on the He fill pressure is linear as is shown in Fig. 5. The data points were fitted with a line using the least square method. The intercept of the linear fit was forced through zero.

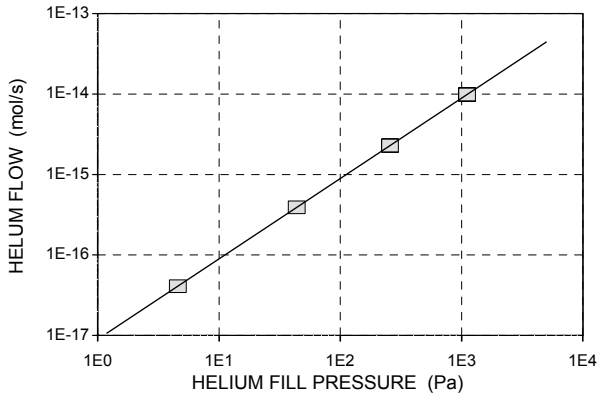

Fig 5. He flow dependence on the He fill pressure measured at the fill temperature of 23°C (the intercept of the linear fit was forced through zero)

The residuals (difference of the measured from the predicted value) of the fit of the He flow to the He fill pressure are shown in Fig. 6.

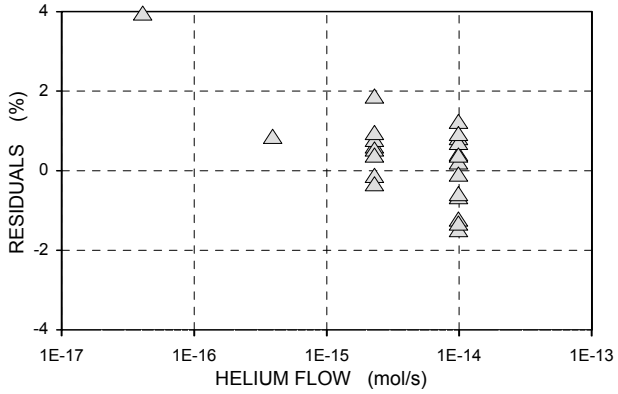

Fig 6. Residuals of a linear fit of the He flow to the He fill pressure

4. CONCLUSIONS

Multiple He flow measurements at $1{,}0\times10^{-14}$ mol/s and $2{,}3\times10^{-15}$ mol/s indicated that a measurement uncertainty as low as 2% can be achieved. The rate of pressure rise method for the calibration of the helium permeation reference leaks using the spinning rotor gauge exhibits thus the measurement uncertainty which is comparable to those of other methods developed at national standards laboratories.

The work on the evaluation of the presented calibration will continue and a detailed analysis will be done when we will gain more information on the stability over the entire measuring range.

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