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Method to test linearity of quadrupole mass spectrometers by use of a flowmeter and a standard leak

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ABSTRACT

A linearity test of the quadrupole mass spectrometer signal can be performed by using the pressure generated by the flow rate of a secondary leak as a fix point and varying the flow rate by a primary gas flowmeter around this value. Applying this method, we have investigated three different quadrupole mass spectrometer in a range of helium partial pressures between 10^{-9} Pa and 10^{-4} Pa, corresponding to flow rates of 10^{-7} Pa L s^{-1} to 10^{-2} Pa L s^{-1} in our system. Our preliminary results indicate significant non-linearities for even modest partial pressure changes.

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1. Introduction

When quadrupole mass spectrometers (QMS) are used for quantitative measurements e.g. for the calibration of standard leaks by using a calibrated set of standard leaks [1,2], it has to be assumed that the signal of quadrupole mass spectrometer has a linear response to partial pressure and flow rate through a vacuum system. Also when comparing outgassing rates this has to be assumed.

Non-linearity has been defined in ISO 14291 [3] as extent to which the change in ion current is not proportional to the corresponding change in partial pressure. The non-linearity is equal to the change of sensitivity in a given range. According to ISO 14291, the linear response range of a QMS is the partial pressure range over which the non-linearity is within a specified limit.

Investigations have shown [4–6] that the linear response range may not only depend on the partial pressure but also on the total pressure and gas mixture. This was not the focus of our investigation and we also did not investigate which parameters of the QMS settings influence the linearity as it was done e.g. in Ref. [4]. Instead, the focus of our investigation was the development of a method which allows an accurate test of linearity of a QMS. It is a by-product of our calibrations of standard leaks by a primary gas flowmeter. Standard leaks are leak elements which emit a constant flow of gas [7,9], usually of only one gas species which is helium. They may be of the permeation type [1,7,8], where the gas is diffusing through a solid (e.g. helium though quartz glass), or of the capillary type [1,9,10], where the flow is determined by the conductance of the capillary or a crimped part of it.

Our experimental method will be described in the following section; Section 3 discusses the influences of the methodology which may contribute to the uncertainty of the non-linearity, Section 4 will give preliminary results for three types of QMS that were in usage on our laboratory before we draw some conclusions.

2. Experimental set-up and method

The experimental set-up of our calibration system for standard leaks is shown in Fig. 1. The calibration is carried out by a direct comparison of the unknown flow rate from the standard leak with the known flow rate from the primary gas flowmeter. The QMS signal serves to compare the two flow rates. For this, the secondary leak and the flowmeter are mounted at equivalent places on the vacuum system with respect to the QMS. The length of the tube which is about 1 m from the gas source to the QMS, the diffuser and the position of the QMS, ensure that equal flows from both sources generate the same signal on the QMS.

The flowmeter is a primary measurement device and was described in Ref. [11]. It generates known gas flow rates from

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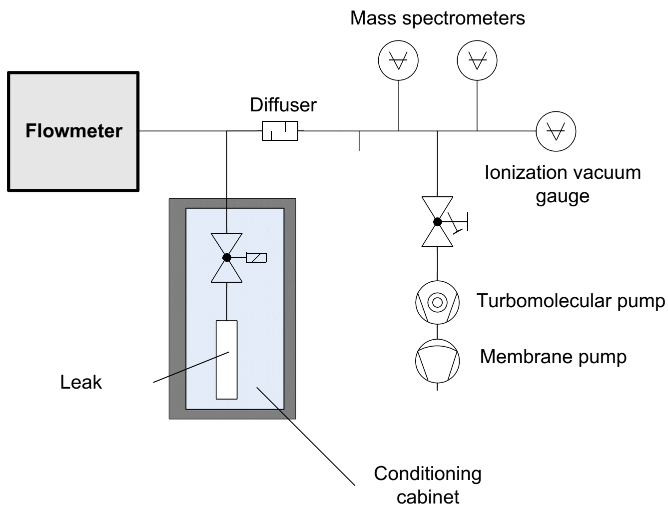

Fig. 1. Scheme of the experimental set-up of the calibration system for standard leaks.

10^{-7} Pa L s^{-1} to 10^{-2} Pa L s^{-1} at 23 °C. A very similar flowmeter in our laboratory was described in Ref. [12] with an extended measurement uncertainty discussion of the flow rate. The flowmeter was modernized since the publication [11], but the measurement method remained the same.

The standard leak is installed in a temperature-controlled cabinet, since the flow rate from a standard leak depends on temperature, particularly for permeation type standard leaks. The cabinet may accommodate three standard leaks at a time and shows a temperature drift of less than 0.005 K/min.

The QMS is mounted on a 6-way DN63CF cross. Two QMS may be installed on this at equivalent positions. The cross is pumped by a 180 L/s (for helium) turbomolecular pump with Holweck stage backed by a membrane pump (Balzers Typ TCM180). The conductance to the DN63 cross is 100 L/s, so that the effective pumping speed for helium is about 64 L/s.

To understand the following section, we will briefly describe the principal measurement method of the flowmeter.

In the upper range of the generated flow ($>10^{-5}$ Pa L s^{-1}) the flowmeter is used in the constant pressure mode. The gas flow exits via a leakage with a conductance of about 10^{-6} L/s. The pressure decrease can be compensated by changing the volume by squeezing a bellows displacer volume. The pressure is measured by a differential capacitance diaphragm gauge with respect to a constant pressure reference volume. The volume change ΔV by the displacer to keep the pressure constant within a measured time interval gives the conductance of the leakage at the prevailing pressure p in the flowmeter:

$$C = \frac{\Delta V}{\Delta t} \tag{1}$$

The flow rate is obtained by multiplying C with the pressure p.

In the lower range of the generated flow ($\leq 10^{-5}$ Pa L s^{-1}) the flowmeter is used in the constant conductance mode. Here, the flow through the leakage is molecular and the conductance independent of pressure which is below 80 Pa. The conductance is measured at higher pressure and the fill pressure is reduced to give the desired flow rate.

In both measurement modes, the molar gas flow rate of the flowmeter $q_{\nu,\mathrm{FM}}$ is determined by

$$q_{\nu,\mathrm{FM}} = \frac{pC}{RT_{\mathrm{FM}}} \tag{2}$$

where R denotes the molar gas constant and T_{FM} the temperature in the flowmeter.

In a calibration of a standard leak, the unknown flow rate $q_{\nu,\mathrm{SL}}$ from the standard leak can be determined by

$$q_{\nu,\mathrm{SL}} = q_{\nu,\mathrm{FM}} \cdot \frac{I_{\mathrm{SL}} - I_0}{I_{\mathrm{FM}} - I_0} \tag{3}$$

where: I_{SL} is the signal of the QMS for the relevant gas species, when this is exposed to the unknown flow from the standard leak, I_{FM}, when it is exposed to the known flow from the flowmeter of the same gas species, and I_0 the offset at residual pressure. If the ratio

$$Z = \frac{I_{\mathrm{SL}} - I_0}{I_{\mathrm{FM}} - I_0} \tag{4}$$

is different from 1, any non-linearity of the mass spectrometer will affect the measurement results. For this reason $q_{\nu,\mathrm{FM}}$ is varied around $q_{\nu,\mathrm{SL}}$, so that Z varies around 1 by typically ±20% (Fig. 2) and $q_{\nu,\mathrm{SL}}$ can be determined for $Z = 1$ by linear interpolation of the values $q_{\nu,\mathrm{SL}}(Z)$. The final value

$$\bar{q}_{\nu,\mathrm{SL}} = q_{\nu,\mathrm{FM}}(Z = 1) \tag{5}$$

is not affected by the non-linearity of the mass spectrometer. Typically, 5 measurements are taken with different $q_{\nu,\mathrm{FM}}$.

The methodology to evaluate the non-linearity of the QMS signal is as follows: The ratio

$$S' = \frac{I_{\mathrm{FM}} - I_0}{q_{\nu,\mathrm{FM}}} \tag{6}$$

can be identified as sensitivity S' of the QMS at flow rate $q_{\nu,\mathrm{FM}}$. In principle, the non-linearity could be simply determined by changing $q_{\nu,\mathrm{FM}}$ and observing the corresponding change of $(I_{\mathrm{FM}}-I_0)$. In this case, however, a timely change of the sensitivity or of the pumping speed in the system could be confused with a non-linear response. The always constant flow from the standard leak $q_{\nu,\mathrm{SL}}$ gives us the possibility to eliminate this effect.

If the sensitivity of the QMS signal changes in time, the ratio

$$S = \frac{I_{\mathrm{SL}} - I_0}{q_{\nu,\mathrm{SL}}} \tag{7}$$

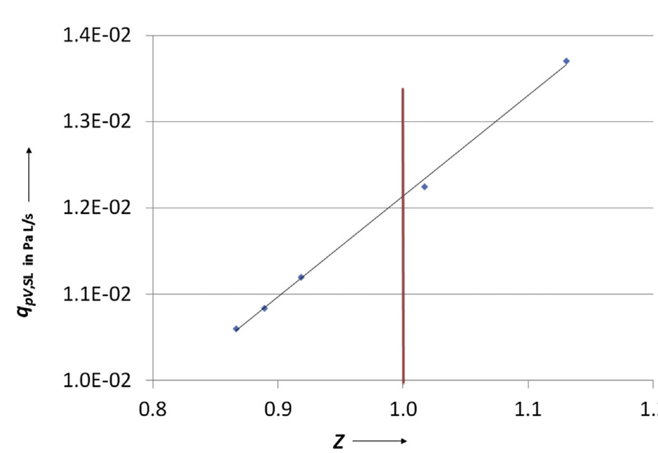

Fig. 2. Example for a measurement of leak rate $q_{\nu,\mathrm{SL}}$ (Z) with QMS A. Linear interpolation for $Z = 1$ yields $\bar{q}_{\nu,\mathrm{SL}}$ (red line). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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