FLOW LEAKS RENORMALIZATION

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Abstract

Flow leaks are small devices that generate a well-determined flow when subject to a pressure differential (feed pressure).
Such devices are widely used in the industry for the easy generation of flows, which can be used for several applications. In order to be correctly included in a Quality Management System (QMS), they need to be calibrated against a reference flow. Such calibration depends on the feed pressure and on the fluid density through a complex relation which can be derived from the modified Darcy law, therefore results of a calibration performed in a given condition are not necessarily valid when the leak is used in different conditions (e.g. different atmospheric pressure, ambient temperature).
In the present paper we will show how to obtain a correct renormalization of the calibration results, which, if applied in use, allows to compute precisely the actual flow rate generated by the leak.
The renormalization is based on the modified Darcy law, and therefore requires the determination of the leak permeability.
A mathematical description of the renormalization will be presented.
Additionally, a method for the experimental determination of the permeability will be discussed
The effect of the renormalization on the output of the leak will be demonstrated through a set of example cases, obtained in various environmental conditions within our laboratory.
It will be shown that, first the calibration uncertainty can be reduced dramatically by applying the correct normalization, and second that the in-use uncertainty can be brought to be of the same order of magnitude as the calibration uncertainty.

1. Introduction

Flow leaks are small devices aimed at regulating the quantity of gas that flows through them (flow rate) by changing the pressure difference to which they are subject; since they allow to easily generate a well-defined flow and they are very stable, they are widely employed in several industrial fields, ranging from checks of leaks in flow rate devices to chemical applications, to food processing, textile permeability checks and so on.

The flow range of these devices is extremely large, ranging from fractions of an SCCM (Standard Cubic Centimetre per Minute) to hundreds of SLM (Standard Litres per Minute), depending on the size and fabrication process of the leak.

Two main technologies are applied for the manufacturing of the leaks active element: for small flow rates, the permeable element is produced by high-pressure sintering of ceramic or metallic materials, while for larger flow rates calibrated holes are drilled through very hard materials (synthetic rubies or similar); the active element is then inserted into a holder provided with standard gas connections which allows to insert the leak into the flow circuit.

Of course, the nominal flow rate through the leak is determined by design, but due to the uncertainties in the production process the actual value of the flow at a given pressure difference may vary for different leaks of the same model; for high-accuracy applications it is therefore necessary to calibrate the individual leak against a reference flow; also, such devices are often included within a Quality Management System (QMS), which again requires periodical calibration of the instruments.

In the present paper we will focus on the calibration of sintered leaks; an accurate analysis of the response of these devices shows that the flow rate depends not only on the pressure difference, but also on the feed pressure and on the fluid density, therefore a correct calibration, and a correct employment of its results, requires to take into account such influences.

To do this, we will describe the theoretical analysis of the renormalization of the calibration results, which when applied will allow to precisely compute the actual flow rate through leaks both in calibration and in use.

The analysis, fully described in Par. 2, is based on different forms of the Darcy law; such equation includes several parameters which are difficult, if not impossible, to know a priori, therefore an experimental determination of the coefficients is necessary. We will describe the experiments that we performed to this aim in par. 3, while in Par. 4 we will present the data analysis alongside to a few application examples.

2. Mathematical Formulation

2.1 Background and notation

The sintered block that constitutes the active element of the leak can be considered as a microporous element, i.e. a conglomeration of very small channels through which the gas flows and, due to the fluid dynamical resistance it encounters, undergoes a pressure drop; of course at steady state the pressure drop (i.e., the pressure difference across the leak) is in equilibrium with the flow, hence the working principle of the device. The analysis of the flow resistance through microchannels can be performed by the Darcy Law, which describes the flow of a fluid through a porous medium; it was originally formulated for liquid flows, but it was later extended to gases in the form called compressible Darcy Law (par. 2.2). When the size of the channels is extremely small, molecular interaction between the walls and the flowing gas becomes important, and must be taken into account; this can be done through the Knudsen modification of Darcy Law (par. 2.3).

The analysis presented here is based on the one performed in the paper by Carrigy et al. [1], slightly modified to adapt it to our needs; we will use the following symbols to describe the various quantities:

A_{ex} is the exit section of the leak;
B_v is the viscous permeability;
D^k is the Knudsen diffusivity;
d_p^{eff} is the effective pore diameter;
η is the gas dynamic viscosity;
L is the length of the porous medium (active length of the leak in our case);
M is the molar mass of the gas;
N is the molar flux;
Q_V is the volumetric gas flow;
Q_{SCCM} is the gas flow in SCCM;
ρ is the gas density;
R^* is the universal gas constant;
T is the temperature;
p is the pressure;
x is the spatial coordinate taken as positive;

the subscripts $_1$ and $_2$ refer to the conditions upstream and downstream of the leak, respectively, while the subscript $_{ref}$ refers to the reference conditions of interest.

2.2 Compressible Darcy Law

Assuming one dimensional gas flow, the differential form of Darcy's Law is given by [2]:

$$N = -\frac{B_v}{\eta}\frac{p}{R^*T}\frac{dp}{dx} \quad (1)$$

Assuming that the flow is isothermal and steady, the operations described in [1] can be performed with some adaptations; in particular, since the flow rate in our case is measured downstream, we will write that:

$$pv = p_2 v_2 \quad (2)$$

The (average) velocity downstream of the leak is:

$$v_2 = \frac{Q_V}{A_{ex}} \quad (3)$$

one thus obtains the following equation, which is equivalent to eq. (8) in [1], as reformulated for the aims of the present work:

$$Q_V = \frac{A_{ex}B_v}{2\eta L}\left(\frac{p_1^2 - p_2^2}{p_2}\right) \quad (4)$$

Now, since the flow rate in SCCM is:

$$Q_{SCCM} = Q_V \frac{\rho}{\rho_{ref}}$$

one gets:

$$Q_{SCCM} = \frac{p_2 T_{ref}}{p_{ref} T_2}\frac{A_{ex}B_v}{2\eta L}\left(\frac{p_1^2 - p_2^2}{p_2}\right)$$
$$= \frac{T_{ref}}{T_2}\frac{A_{ex}B_v}{2\eta L}\left(\frac{p_1^2 - p_2^2}{p_{ref}}\right) \quad (5)$$

It can be seen from this equation that the flow rate in SCCM depends on the difference of the squares of the pressures upstream and downstream of the leak, on the flowing gas through its viscosity, on geometrical parameters (A_{ex} and L) and on the viscous permeability of the leak B_v, which can be assumed as a property of the leak.

2.3 Knudsen's expression

Darcy's law (and therefore eq. (5)) was derived in the assumption of continuum regime and no-slip condition for the interaction between the gas and the leak walls; though, in the case of leaks with very low permeability, the effects of microscopic interaction between the gas and the microchannels constituting the leak cannot be neglected; a more complex formulation must therefore be adopted. This formulation stems from the expression proposed by Knudsen for predicting gas flows in all regimes, as explained in [2], where it is also shown

how, for sufficiently high pressures, Knudsen's expression can be simplified to the form:

$$N = -\left(\frac{R^2}{8\eta}\frac{p_1+p_2}{2} + D^k\frac{c_1^k}{c_2^k}\right)\frac{1}{R_gT}\frac{p_2-p_1}{x_2-x_1} \quad (6)$$

From this equation and performing developments similar to the ones described in [1], one gets to the equation (18) of [1] which, when expressed as a function of Q_V, becomes:

$$Q_V = \frac{A_{ex}B_v}{2\eta L}\left(\frac{p_1^2-p_2^2}{p_2}\right)$$
$$+ 0.89\frac{d_p^{eff}}{3L}\sqrt{\frac{8R^*T}{\pi M}}\left(\frac{p_2-p_1}{p_2}\right) \quad (7)$$

And, when expressed as a function of the flow rate in SCCM:

$$Q_{SCCM} = \frac{T_{ref}}{T_2}\frac{A_{ex}B_v}{2\eta L}\left(\frac{p_1^2-p_2^2}{p_{ref}}\right)$$
$$+ 0.89\frac{T_{ref}}{T_2}\frac{d_p^{eff}}{3L}\sqrt{\frac{8R^*T_2}{\pi M}}\left(\frac{p_1-p_2}{p_{ref}}\right) \quad (8)$$

Notice that the first term in eq. (8) is the same as eq. (5), so that its second term can be considered as a correction, which should become vanishing as the slip effects decrease.

2.4 Determination of the constants
It can be observed that, with the exception of the thermodynamic conditions and the gas properties, both in eq. (5) and in eq. (8) all values are either constants or properties of a given leak (possibly depending on the thermodynamic conditions). It will then be possible to reformulate these equations as follows (where the reference conditions were included in the values α', α and β):

$$Q_{SCCM} = \frac{\alpha'}{\eta}\left(\frac{p_1^2-p_2^2}{T_2}\right) \quad (5b)$$

$$Q_{SCCM} = \left[\frac{\alpha}{\eta}\left(\frac{p_1^2-p_2^2}{T_2}\right) + \beta\sqrt{\frac{R^*T_2}{M}}\left(\frac{p_1-p_2}{T_2}\right)\right] \quad (8b)$$

If the thermodynamic conditions and the flow rate are measured, it will then be possible to determine the values of α and β for a specific leak.
Specifically, in the case of eq. (8b), it will be possible to write:

$$\frac{Q_{SCCM}T_2}{p_1-p_2} = \frac{\alpha}{\eta}(p_1+p_2) + \beta\sqrt{\frac{R^*T_2}{M}} \quad (8c)$$

In order to isolate the value β, we will write:

$$\frac{Q_{SCCM}T_2}{\sqrt{\frac{R^*T_2}{M}}(p_1-p_2)} = \frac{\alpha}{\eta\sqrt{\frac{R^*T_2}{M}}}(p_1+p_2) + \beta \quad (8d)$$

For uniformity, we will also reformulate eq. (5b) as follows:

$$\frac{Q_{SCCM}T_2}{\sqrt{\frac{R^*T_2}{M}}(p_1-p_2)} = \frac{\alpha'}{\eta\sqrt{\frac{R^*T_2}{M}}}(p_1+p_2) \quad (5c)$$

The use of eqs. (5c) and (8d) in calibration and application of the leaks will be discussed in the following paragraphs. Notice that similar expressions could be obtained also for the volumetric flow rate Q_V, but we preferred to work with the standardized flow rate Q_{SCCM} due to its wide use in practical applications.

3. Experimental setup

3.1 Dataset
Test were performed using four ATEQ flow leaks (type A, type 5, type D and type E), which were mounted in series upstream of the reference test rig. The upstream pressure was regulated by a Druck PACE 5000 pressure regulator, which allows to obtain relative pressures up to 7 bar with a stability of approximately 10 Pa; the downstream pressure was set at the ambient pressure (see par. 3.2). Leaks were tested at various nominal differential pressures as reported in table 1: all measurements were performed considering a reference temperature of 20 °C (293.15 K) and a reference pressure of 101325 Pa (1 atm) for the definition of the standard flow rate.

Table 1: Test pressures.

Test #	Nominal differential pressure (mbar)	Leak tested			
		A	5	D	E
1	100	NO	X	X	X
2	300	X	X	X	X
3	500	X	X	X	X
4	850	X	X	X	X
5	1000	X	X	X	X
6	2000	X	X	X	NO

Unfortunately, the present preliminary setup did not allow to obtain reliable measurements in some cases, which were therefore excluded from the analysis, due to excessive pressure losses or insufficient pressure stability; we are currently preparing an improved setup which was designed to overcome such limits.

3.2 Reference Measurement
The flow rate from the leaks was measured using INRIM high accuracy piston prover *MICROGas*, fully described in previous works [3,4]. The rig is a piston of the plunger type, whose movement is controlled by a feedback system programmed to keep the internal pressure of the piston to a predetermined level; in the present work, this pressure level was set at ambient pressure. Due to the very low flow rates employed, pressure losses

in the tubing are negligible, thus the pressure at the leak exit can be assumed to be equal to the pressure inside the piston. The test rig measurement capability ranges between 0.1 and 1200 SCCM, with an uncertainty of 0.05%.

The piston is in a laboratory whose temperature is controlled to within 0.1 K, while the temperature inside the piston is kept constant to within 0.02 K; both temperatures were set to 20 °C nominal for the experiments described in the present paper.

Calibration measurements were performed by measuring the flow rate provided by the leak under test at the various pressures, and repeating every measurement three times. On the other hand, elaboration was performed using various calibration variables, that were computed based on the calibration results (see par. 4).

4. Calibration Results

Based on Eqs. (5c) and (8d), data collected during calibrations were plotted on Cartesian graphs using the following variables:

$$X = \frac{(p_1 + p_2)}{\eta\sqrt{\frac{R^*T_2}{M}}} \; ; \; Y = \frac{Q_{SCCM}T_2}{\sqrt{\frac{R^*T_2}{M}}\,(p_1 - p_2)}$$

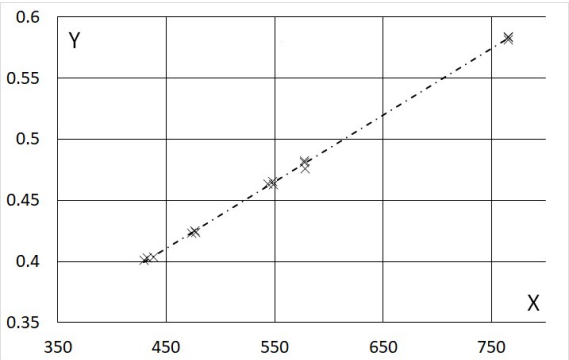Results thus obtained are presented in Figg. 1 (a) to 1 (d).
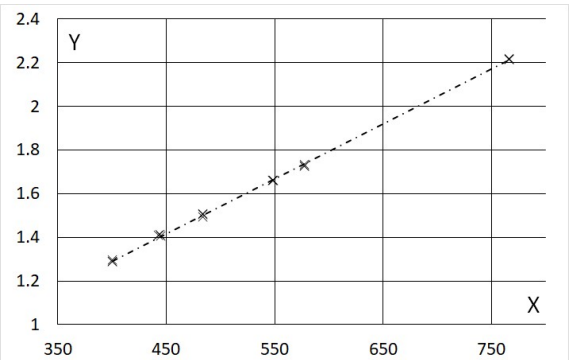

Figure 1(a): Calibration of Leak A.

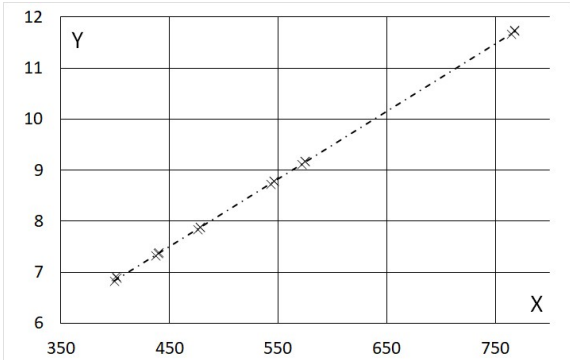**Figure 1(b):** Calibration of Leak 5.
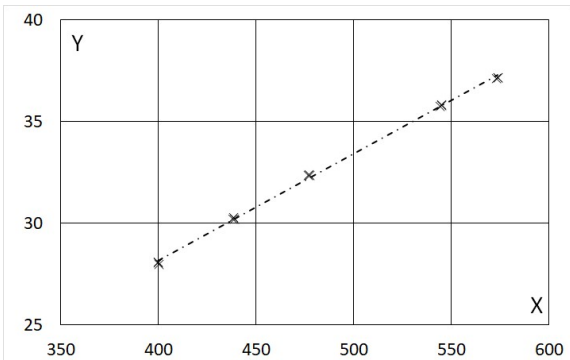

Figure 1(c): Calibration of Leak D.

Figure 1(d): Calibration of Leak E.

4.1 Elaboration according to Eq. (8d)

As can be observed in Figg. 1 (a) to 1 (d), data are aligned along straight lines. This is in accordance with the theoretical predictions of Eq. (8d), which has the general form $Y = \alpha X + \beta$. It is therefore possible to determine the values of α and β through a simple linear regression analysis on the data. The resulting parameters for the four leaks under test are reported in Table 2:

Table 2: Regression equations for the four leaks.

Leak	Equation
A	$Y = 0.0005446X + 0.1657507$
5	$Y = 0.002513X + 0.286205$
D	$Y = 0.013234X + 1.555687$
E	$Y = 0.052447X + 7.186322$

It can be observed that the values of α and β display a growing behaviour as the leak nominal flow rate increases, which is coherent with the expected result.

4.2 Elaboration according to Eq. (5c)

When data are analysed according to Eq. (5c), it must be observed that this equation assumes a

direct proportionality between the values of X and Y; this means that every calibration point must be analysed separately in this case, to determine the ratio $\alpha' = Y/X$. We will then plot graphs of the value of α' as a function of X (Figures 2 (a) to 2 (d)), from which it will be possible to deduce the evolution of the proportionality ratio as a function of the pressure level. Physically, the result that the value of α' is not constant expresses the fact that the simple compressible Darcy law is not exactly valid, i.e. that in the range analysed here the leaks employed undergo the effect of the slip condition described in 2.3.
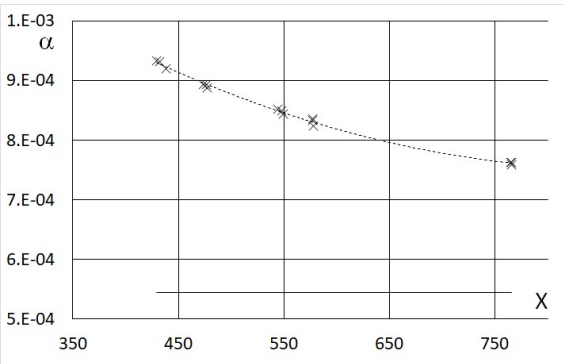

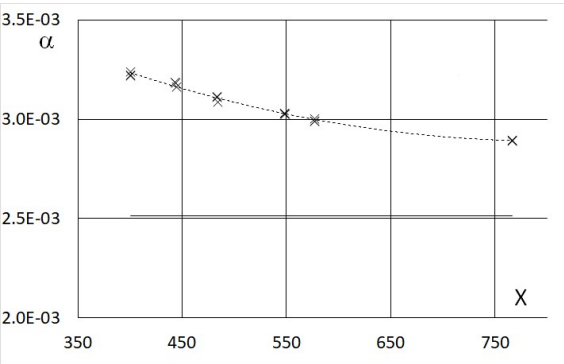**Figure 2(a):** Comparison of Eq. (5) to Eq. (8), Leak A.

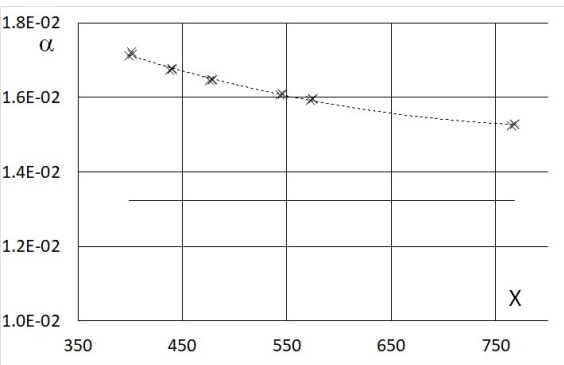**Figure 2(b):** Comparison of Eq. (5) to Eq. (8), Leak 5.

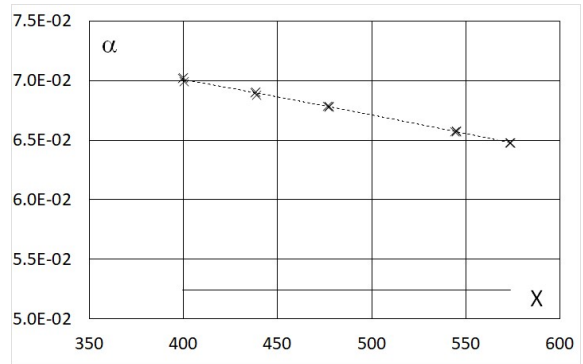**Figure 2(c):** Comparison of Eq. (5) to Eq. (8), Leak D.

Figure 2(d): Comparison of Eq. (5) to Eq. (8), Leak E.

Figures 2 (a) to 2 (d) also include an horizontal line, which represents the value of α obtained in par. 4.1, and is of course constant with the pressure.

It can be observed that, in all cases, the behaviour of α' tends to the value of α following a clear and repeatable trend, which can therefore be interpolated through regression analysis. This implies that it is also possible to apply Eq. (5) for the determination of the flow rate, with a method similar to what will be shown in Par. 4.3, by computing α' from the relevant interpolation equation. We do not feel, though, that this approach should be recommended, because the small reduction in complexity for the elaboration in the results would be more than offset by the requirement of more calibration datapoints, necessary for a correct determination of the functions in Fig. 2, and the further complexity in the determination of the interpolation equation.

It is also possible to notice that the relative differences between α' and α tend to diminish as the nominal flow of the leak increases; this fact is coherent with the observation that the leaks with lower flows imply smaller passages for the gas and therefore an increase of the molecular effects.

4.3 Application in use

In order to check the validity of the calibration of the leaks, we performed a few tests of application; specifically, we applied various differential pressures (different from the calibration pressures, but within the interpolation range) to leak D, in slightly different ambient conditions than the ones encountered in calibration.

Since the relevant conditions (pressures, temperatures) were also measured, it was possible to determine the value of X and, by applying the equation for leak D reported in Table 2, to compute the value of Y; inverting then the definition of Y, it was possible to compute the flow rate in SCCM.

By measuring the actual flow rate as described in par. 3, we could compare this forecast flow rate to

the actual one; it was therefore possible to compute the percent difference between these flows. Results of such tests are reported in Table 3.

Table 3: in-use tests, leak D.

Nominal differential pressure (mbar)	Measured flow (SCCM)	Computed Flow (SCCM)	Difference, %
200	1.429	1.427	0.17
400	3.066	3.065	0.05
900	8.033	8.029	0.05
1500	15.663	15.657	0.04

It can be observed that, in all cases, the difference is very small, thus confirming that the proposed rescaling of the quantities of interest allows to obtain an excellent accuracy in the computation of the flow delivered by the leak.

5. Conclusions and future work

An improved way of rescaling the flow delivered by a flow leak was proposed; it was shown that the proposed model allows to obtain excellent calibration curves, which in turn lead to a very good accuracy in the computation of the delivered flow for applications.

The setup used in the present work was preliminary, therefore it had some limitations which we expect to overcome with a new setup that we are presently building; this is expected to allow larger measurement ranges with the same accuracy.

There are still several questions not answered, that we are going to investigate in future works. First of all, the assumption that the temperature of the leak is the same as the one of the gas collected in the reference test rig is quite strong and can be considered valid only in laboratory applications at low flow rates, where the ambient is at a constant temperature and the friction heating of the leak is negligible; we intend to investigate such effects by measuring directly the temperature of the leak. Of course, this would still imply the assumption of isothermality of the leak, but this is a much weaker assumption.

Another important point is the effect of the working gas. According to the mathematical analysis presented in par. 2, this should be accounted for through the molar mass and the viscosity of the gas, but an experimental verification must be performed. The analysis discussed in the present paper will be the basis for the design of a transfer standard (TS) for flow comparisons, which we expect will provide very good stability properties.

References

[1] N. B. Carrigy, L. M. Pant, S. Mitra, and M. Secanell, "Knudsen Diffusivity and Permeability of PEMFC Microporous Coated Gas Diffusion Layers for Different Polytetrafluoroethylene Loadings", *Journal of the Electrochemical Society*, **160**, F81-F89, 2013.

[2] R.E. Cunningham and R.J.J. Williams, *Diffusion in Gases and Porous Media* (New York, Plenum Press), 1980.

[3] G. Cignolo, F. Alasia, A. Capelli, R. Goria, G. La Piana: "A Primary Standard Piston Prover for Measurement of Very Small Gas Flows", *Proceedings of the 5th ISFFM*, Arlington, Va. (USA), p. 10, 2002.

[4] P. G. Spazzini, A. Piccato, P. Pedone: "Reduction of a Gas Prover Uncertainty", *Proceedings of the 10th ISFFM*, Querétaro. (Mexico), 2018.