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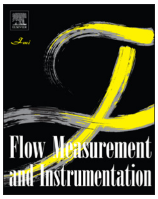

Development of a gas micro-flow transfer standard

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A R T I C L E I N F O

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A B S T R A C T

LNE has ability to calibrate gas micro-flow rates using the dilution method for nitrogen flow rates in the range from 2 μg/s to 200 μg/s or helium ones in the range 0.75–30 μg/s. In addition, a primary constant pressure flowmeter for leak rate measurements from 0.05 μg/s to 35 μg/s is also available. This equipment will be used to validate the dilution method below 30 μg/s. In order to compare these reference facilities, LNE is developing a micro-flow transfer standard (μFTS) in collaboration with ATEQ France, a manufacturer of control equipment for leak testing. The flowmeter consists mainly of an array of three stainless steel capillaries designed to cover the ranges from 0.035 μg/s to 0.35 μg/s, 0.35 μg/s to 3.5 μg/s and 3.5 μg/s to 35 μg/s for nitrogen (0.1–100 ml/h). A dynamic model of the μFTS determines the mass flow rate from the input pressure, the differential pressure of the capillary, the gas temperature, viscosity and density and the length and radius of the capillary. A comparison of both reference methods is carried out with the μFTS from 0.35 μg/s to 35 μg/s.

1. Introduction

The laboratory is currently developing a flow standard with capillaries to measure micro- and nano-gas flow rates between 35 ng/s and 35 μg/s in collaboration with ATEQ, world leader in flow and leak test instruments. The aim is to obtain a relative uncertainty of less than 1% in this flow range. Taking into account the desired technical specifications, it was mandatory to equip the apparatus with high-quality pressure and temperature sensors and to have stainless steel capillaries of low roughness as well as an ultra-tight fluid system.

2. Description of the micro-flow transfer standard

2.1. Principle

The principle of the micro-flow transfer standard (μFTS) consists in measuring a pressure difference at the bounds of several capillaries with a differential pressure transmitter.

The flowmeter is made of the following main elements:

- an absolute pressure sensor upstream from the capillaries (Druck model DPI 142),
- two Pt100 temperature sensors,
- an MKS differential pressure sensor, type BARATRON 120AD, with a measuring range of 0–133 Pa,
- three stainless steel capillaries which dimensional characteristics given by the manufacturer and corresponding flow ranges, listed in Table 1.

The dimensions in mm of the μFTS presented in Fig. 1 are 540 × 240 × 485 (approximately 21 in.).

The block of resin that contains the three capillaries, the two temperature sensors placed upstream and downstream of the capillaries (T_{up} and T_{down}), the absolute pressure sensor (P_{up}) and the differential pressure transmitter (ΔP) are presented in Fig. 2.

2.2. Mathematical modeling

An empirical formula, established from the research of the Van der Waals laboratory [1] and the NIST [2] and based on the Poiseuille equation for capillary geometries and flow ranges similar to those of the μFTS, seems to be the best suited to calculate the mass flow rate. The complete model that gives the value of the mass flow rate through a capillary is:

$$\dot m = \dot m_0 \left(1 + g_{virial}(P_1,P_2) + 4K_{slip}\cdot Kn + \frac{K_{ent}}{16}\cdot\frac{r}{L}\cdot \mathrm{Re} + \left(\frac{K_{exp}}{8}+\frac{K_{therm}}{16}\right)\cdot\frac{r}{L}\cdot\mathrm{Re}\cdot\ln\left(\frac{P_2}{P_1}\right)\right) f_{cent}\left(De, \frac{r}{r_{curve}}\right), \tag{1}$$

with:

$$\dot m_0 = \frac{(P_1 - P_2)\times\rho(T,P)\times\pi\times d^4}{128\times\eta(T,P)\times l} \tag{2}$$

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Table 1
Characteristics of the capillaries.

Characteristics of the capillaries				
Capillary	d (mm)	L (mm)	r_{curve} (mm)	Range (μg/s)
1	0.432	130	–	3.5–35
2	0.278	250	20	0.35–3.5
3	0.193	600	50	0.035–0.35

Fig. 1. A front view of the micro-flow transfer standard (μFTS).

Fig. 2. A view of the components of the μFTS.

and with:

$$K_{therm} = -\left(1+\frac{1}{3}\left(\frac{T}{\eta}\frac{\delta\eta}{\delta T}\right)\right)\left(\frac{R\eta}{M\kappa}\right), \quad (3)$$

where κ is the thermal conductivity of the gas.

This model is implemented in three parts:

- Poiseuille's law for a straight capillary $\dot{m}_0$,
- five correction terms on $\dot{m}_0$ to take into account the different phenomena that occur in the capillary (the terms are in brackets in Eq. (1)),
- the function f_{cent} that corrects the centrifugal effects due to coiling the long capillary.

The contribution of the function f_{cent} is of the order of 10^{-8} for capillary 2 and 10^{-11} for capillary 3. Capillary 1 is not affected since it is straight. This term could be neglected.

The four other correction terms included in the calculation of the mass flow rate were evaluated in the working range of the three capillaries for:

- an average absolute pressure P between 150 kPa and 190 kPa,
- an average temperature T between 296 K and 299 K,
- a pressure difference ΔP between 3 Pa and 55 Pa.

Tables 2–4 give the contribution of the correction terms for the calculation of the minimum and maximum mass flow rates of capillaries 1, 2 and 3 respectively, with the major term in bold.

The values of $K_{\exp}$ and K_{therm} used in the laminar flowmeter model are respectively 9/7 for stainless steel capillary and -0.257 for nitrogen.

Considering the contribution of the correction terms to the mass flow rate calculation, the complete model has been simplified as following:

$$\dot{m}_{simple} = \dot{m}_0\left(1+4K_{slip}\cdot Kn\right) \quad (4)$$

with:

$$Kn = \sqrt{(2RT/M)}\cdot\eta(T,P)/P/r, \quad (5)$$

and K_{slip} is slip coefficient equal to 1

Even though the contribution of the term $(K_{ent}/16)\cdot(r/L)\cdot \mathrm{Re}$ is similar to that of the term $4K_{slip}\cdot Kn$ for the high values of the flow rate of capillary 1, it can be neglected.

In order to implement the simplified model in the flowmeter, we undertook to express directly the mass flow rate in function of the variables P, T and ΔP.

First, the parameters linked to the gas and especially dynamic viscosity (η) and density (ρ) were modeled in function of T and P. The second order polynomial models with interaction adopted for the working range of the flowmeter are:

$$\eta(T,P) = A_{00}+(A_{10}+A_{11}T)P+(A_{20}+A_{21}T)P^2, \quad (6)$$

$$\rho(T,P) = (C_{10}+C_{11}T)P+(C_{20}+C_{21}T)P^2, \quad (7)$$

From Eqs. (2), (4), (5) and (6), Eq. (3) becomes:

$$\dot{m}_{simple} = I\times\left(\frac{G(T,P,\Delta P)+J\times F(T,P,\Delta P)}{H(T,P)}\right), \quad (8)$$

with:

$$I = \frac{\pi\times d^4}{128\times l}, \quad (9)$$

$$J = \frac{4K_{slip}}{r}\cdot\sqrt{\frac{2R}{M}} \quad (10)$$

$$H(T,P) = \eta(T,P) = A_{00}+(A_{10}+A_{11}T)P+(A_{20}+A_{21}T)P^2 \quad (11)$$

$$G(T,P,\Delta P) = (C_{10}P+C_{11}TP+C_{20}P^2+C_{21}TP^2)\Delta P, \quad (12)$$

$$F(T,P,\Delta P) = \begin{pmatrix} (C_{10}A_{10}+C_{20}A_{00})P+(C_{21}A_{00}+C_{10}A_{11}+C_{11}A_{10})TP \\ +(C_{10}A_{20}+C_{20}A_{10})P^2 \\ +(C_{20}A_{11}+C_{21}A_{10}+C_{10}A_{21}+C_{11}A_{20}+)TP^2 \end{pmatrix}\sqrt{T}\Delta P \quad (13)$$