Development of a gas micro flow transfer standard

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Abstract: LNE has ability to calibrate micro gas flow rates using the dilution method in the range from 2 µg/s to 200 µg/s of nitrogen or 0.75 µg/s to 30 µg/s of helium. In addition, a primary constant pressure flowmeter for leak rates measurements from 0.05 µg/s to 35 µg/s is also available. In order to compare these reference facilities and to validate the dilution method below 30 µg/s, LNE is developing a micro flow transfer standard (µFTS) with the collaboration of ATEQ France, manufacturer of control equipments for leak test. 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 of nitrogen (0.1 ml/h to 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, the gas properties (viscosity and density) and the dimensional parameters of the capillary (length and radius). The comparison of both reference methods was carried out with the µFTS from 0.35 µg/s to 35 µg/s.

Keywords: Micro flow; Leak rate; Dilution method; Capillary

1. Introduction

The laboratory is developing a micro flow transfer 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 expanded uncertainty of less than 2 % in this flow range. Taking into account the targeted technical specifications, it was mandatory to equip the equipment with high-quality pressure and temperature sensors and to have stainless steel capillaries of low roughness as well as an ultra-tight fluid system. This equipment will be used first to validate the dilution method and next as travelling standard for interlaboratory comparison.

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:

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

Table 1 – Characteristics of capillaries

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

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

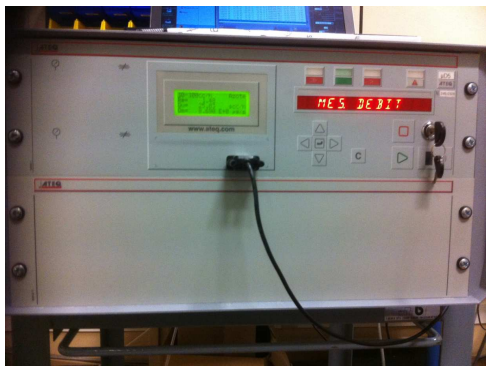

Figure 1 – A front view of the micro flow transfer standard (µFTS)

The block of resin that contains the three capillaries, the two temperature sensors placed upstream and downstream of the capillaries, the absolute pressure sensor and the differential pressure transmitter are presented in Figure 2.

Figure 2 – A view of the components of the µFTS

2.2 Mathematical modeling

An empirical formula, established from the research of the Van der Waals laboratory [3] and the NIST [4] and based on the Poiseuille equation for capillary geometries and flow ranges similar to those of the µFTS, seems the best suited to calculate the mass flow rate.

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

The complete model that gives the value of the mass flow

$$\dot{m} = \dot{m}_0 \left(1 + g_{virial}(P_1,P_2) + 4K_{slip}.Kn + \frac{K_{ent}}{16}.\frac{r}{L}.\mathrm{Re} + \left(\frac{K_{\exp}}{8} + \frac{K_{therm}}{16} \right).\frac{r}{L}.\mathrm{Re}.\ln\left(\frac{P_2}{P_1}\right) \right) f_{cent}\left(De, \frac{r}{r_{curve}} \right) \quad , (1)$$

rate through a capillary is:

$$\text{with : } \dot{m}_0 = \frac{(P_1 - P_2)\times\rho(T,P)\times\pi\times d^4}{128\times\eta(T,P)\times l} \quad , (2)$$

Table 2 – Contribution of correction terms for capillary 1

Correction terms					
$\dot{m}$ (µg/s)	$g_{virial}(P_1,P_2)$	$4K_{slip}.Kn$	$\frac{K_{ent}}{16}.\frac{r}{L}.\mathrm{Re}$	$\left(\frac{K_{\exp}}{8}+\frac{K_{therm}}{16}\right).\frac{r}{L}.\mathrm{Re}.\ln\left(\frac{P_2}{P_1}\right)$	$f_{cent}(De,\delta)$
3.7829	-2.172E-04	7.673E-04	-7.428E-05	-4.189E-09	-
34.781	-2.172E-04	7.673E-04	-6.833E-04	-3.545E-07	-
Contribution to the mass flow rate calculation					
3.7829	21 %	**72 %**	7 %	0 %	-
34.781	13 %	**46 %**	41 %	0 %	-

Table 3 – Contribution of correction terms for capillary 2

Correction terms					
$\dot{m}$ (µg/s)	$g_{virial}(P_1,P_2)$	$4K_{slip}.Kn$	$\frac{K_{ent}}{16}.\frac{r}{L}.\mathrm{Re}$	$\left(\frac{K_{\exp}}{8}+\frac{K_{therm}}{16}\right).\frac{r}{L}.\mathrm{Re}.\ln\left(\frac{P_2}{P_1}\right)$	$f_{cent}(De,\delta)$
0.4050	-2.172E-04	1.192E-03	-4.133E-06	-2.797E-10	-
3.5196	-2.950E-04	1.139E-03	-3.573E-05	-1.909E-08	-
Contribution to the mass flow rate calculation					
0.4050	15 %	**84 %**	0 %	0 %	-
3.5196	20 %	**78 %**	2 %	0 %	-

Table 4 – Contribution of correction terms for capillary 3

Correction terms					
$\dot{m}$ (µg/s)	$g_{virial}(P_1,P_2)$	$4K_{slip}.Kn$	$\frac{K_{ent}}{16}.\frac{r}{L}.\mathrm{Re}$	$\left(\frac{K_{\exp}}{8}+\frac{K_{therm}}{16}\right).\frac{r}{L}.\mathrm{Re}.\ln\left(\frac{P_2}{P_1}\right)$	$f_{cent}(De,\delta)$
0.03922	-2.172E-04	1.717E-03	-1.667E-07	-1.128E-11	-
0.3409	-2.950E-04	1.641E-03	-1.441E-06	-7.700E-10	-
Contribution to the mass flow rate calculation					
0.03922	11 %	**89 %**	0 %	0 %	-
0.3409	15 %	**85 %**	0 %	0 %	-

Considering the results, the model has been simplified as following:

$$\dot{m}_{simple} = \dot{m}_0\left(1+4K_{slip}.Kn\right) \quad , (3)$$

with : $Kn = \sqrt{(2RT/M)}.\eta(T,P)/P\ /r$, (4)

and K_{slip} : slip coefficient equal to 1

Even though the contribution of the term $\frac{K_{ent}}{16}.\frac{r}{L}.\mathrm{Re}$ is close to that of the term $4K_{slip}.Kn$ 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* et *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 adopted models in the working range of the flowmeter are:

$$\eta(T,P) = A_{00} + (A_{10}+A_{11}T)P + (A_{20}+A_{21}T)P^2 \quad , (5)$$

$$\rho(T,P) = (C_{10}+C_{11}T)P + (C_{20}+C_{21}T)P^2 \quad , (6)$$

From equations (2), (4), (5) et (6), equation (3) becomes:

$$\dot{m}_{simple} = I\times\left(\frac{G(T,P,\Delta\mathrm{P})+J\times F(T,P,\Delta\mathrm{P})}{H(T,P)}\right), (7)$$

with :

$$I = \frac{\pi\times d^4}{128\times l}, (8)$$

$$J = \frac{4K_{slip}}{r}.\sqrt{\frac{2R}{M}} \quad , (9)$$

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

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

$$F(T,P,\Delta\mathrm{P}) = \begin{pmatrix}(C_{10}A_{10}+C_{20}A_{00})P+(C_{21}A_{00}+C_{10}A_{11}+C_{11}A_{10})TP\\ +(C_{10}A_{20}+C_{20}A_{10})P^2\\ +(C_{20}A_{11}+C_{21}A_{10}+C_{10}A_{21}+C_{11}A_{20}+)TP^2\end{pmatrix}\sqrt{T}\Delta\mathrm{P} \quad (12)$$

The terms which contribution is below 10 % were not taken into account in the calculation of equation 12 (constant term, in *T*, T^2P, T^2P^2 and P^3).

Table 5 shows the coefficients of the equation of the simplified model of nitrogen mass flow rate. The equation applies to the range of temperatures between 296 K and 299 K, pressures between 0.15 MPa and 0.19 MPa and pressure differences between 3 Pa and 55 Pa.

Table 5 – Coefficients of the simplified equation of the mass flow rate

ij	Aij	Cij
00	1.77615E-05	-7.3974E-06
10	-1.92493E-04	2.26509E+01
11	6.47469E-07	-3.80688E-02
20	6.58396E-04	3.31932E-01
21	-2.21307E-06	-1.0419E-03

An example of calculation of the maximum mass flow rate for the three capillaries with the two models is given in Table 6. This example was obtained from pressure and temperature values read on the flowmeter and with the geometrical characteristics of the capillaries

Table 6 – Flow calculation with equations (1) and (7)

Data given by µFTS			
	T (K)	ΔP (Pa)	P (Pa)
Capillary 1	296.3	46.29	176904
Capillary 2	296.2	52.98	179296
Capillary 3	297.6	53.04	182508
Mass flow rate			
	$\dot{m}$ (µg/s)	$\dot{m}_{simple}$ (µg/s)	Relative difference (%)
Capillary 1	34.549	34.567	0.052
Capillary 2	3.5778	3.5786	0.022
Capillary 3	0.3499	0.3501	0.077

The relative differences observed between the flow rate values obtained from the complete model and the ones obtained from the simplified model (equation 7) are between 0.004 % and 0.08 % for the three capillaries. So the simplified model is relevant and can be used and implemented in the flowmeter.

A software that operates the µFTS measurements (see Figure 3) allows to record as data frames the absolute pressure upstream of the capillary, the pressure difference, the upstream and downstream temperature of the capillary, the mass flow rate, volume flow rate and the Reynolds number.

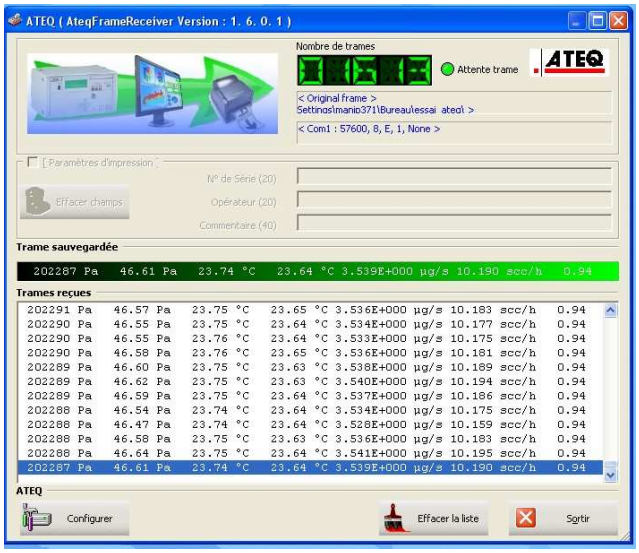

Figure 3 – Screenshot of the window from the data frames writing software

The stabilities of the µFTS different parameters during the acquisition time of approximately 15 minutes for the traced gas mass flow rate ($q_{m,gt}$) are presented in Table 7.

The obtained results prove the good functioning of the device during the acquisitions of the traced gas flow rate. Relative stability of the µFTS mass flow ranges is between 0.01% and 0.3%. The difficulty to regulate a 0.35 µg/s flow rate with Capillary 2 as well as the very small measured pressure difference (5 Pa) are the reasons for the value of 0.7 %.

3. Calibration of the micro flow transfer standard

3.1 Experimental set-up

A mass flow controller Bronkhorst type EL-FLOW controls the gas flow rate between 0.35 µg/s and 35 µg/s of pure nitrogen flowing from a 10 liters reservoir through the micro-flow transfer standard (µFTS). The calibration facility is connected downstream the MFC (Figure 4). The upstream pressure in the reservoir is maintained at (180 ± 10) kPa during the calibration sequences. The pressure downstream the MFC depends on the calibration method, 110 kPa for the dilution method and atmospheric pressure with the constant pressure flowmeter.

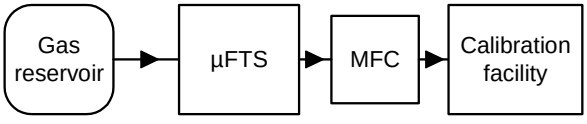

Figure 4 – Schematic diagram of the experimental set-up

3.2 Calibration with the dilution method

The µFTS is calibrated directly with the dilution method between 0.35 µg/s and 35 µg/s of nitrogen (1 ml/h to 100 ml/h). A previous paper [3] described the dilution method but for a better understanding, the equations used to calculate the micro mass flow rate ($q_{m,gt}$) called "traced gas" are given below.

$$q_{m,gt} = \frac{M_{gt}}{M_{gv}} \cdot \frac{X_{gt,mix}}{1 - X_{gt,mix}} \cdot q_{m,gv} \quad (13)$$

where

$$X_{gt,mix} = \frac{\bar{S}_{gt,mix}}{(\bar{S}_{1gt,ref} + \bar{S}_{2gt,ref})/2} \cdot X_{gt,ref} \quad (14)$$

- M_{gt} is the molar mass of the traced gas and M_{gv} is the molar mass of the dilution gas,
- $X_{gt,ref}$ is the mole fraction of the gravimetric reference gas mixture,
- $q_{m,gv}$ is the main mass flow of the dilution gas,
- $\bar{S}_{gt,mix}$ is the average peak areas of the dynamic mixture,
- $\bar{S}_{1gt,ref}$ and $\bar{S}_{2gt,ref}$ the average peak areas of the reference gas mixture measured by the chromatograph.

The reference gravimetric gas mixtures are pressurized cylinders with different concentrations of nitrogen in argon used to calibrate the chromatograph to cover the flow range from 0.35 µg/s to 35 µg/s. Table 8 gives the list of the nominal flows of nitrogen and argon measured and the amount fractions of the gravimetric reference gas mixtures.

Table 7 – Stabilities of the µFTS different parameters for the three capillaries

$\dot{m}$ (µg/s)	σ_{sta} (µg/s)	P_{amont} (Pa)	σ_{sta} (Pa)	ΔP (Pa)	σ_{sta} (Pa)	T_{amont} (°C)	σ_{sta} (°C)	T_{aval} (°C)	σ_{sta} (°C)
Capillary 1									
3.6252	0.0040	178614	8	4.848	0.006	24.56	0.01	24.44	0.01
34.549	0.004	181648	51	45.41	0.01	24.44	0.01	24.32	0.01
Capillary 2									
0.3797	0.0025	182319	1	5.57	0.04	24.50	0.01	24.36	0.01
3.5220	0.0020	180328	6	52.22	0.03	24.35	0.01	24.22	0.01
Capillary 3									
0.3572	0.0010	181674	2	54.81	0.15	24.59	0.01	24.46	0.01

Table 8 – Nitrogen and argon mass flow rates and amount fractions of reference gas mixtures

Nitrogen flow ($q_{m,qt}$) µg/s	Argon flow ($q_{m,qv}$) mg/s	Amount fraction ($X_{qt,ref}$) µmol/mol
0.35	2.7	200
0.85	6.3	
1.7	2.5	1000
8.7	12.5	
17	5.7	4000
35	11.5	

Figure 5 shows the experimental set up used for the measurement of the micro mass flow rate ($q_{m,gt}$). The nitrogen micro mass flow uncertainty is calculated by propagating the uncertainties of argon mass flow and nitrogen amount fraction. The dependence of the uncertainty $u(q_{m,gt})$ with the flow is presented in Table 9.

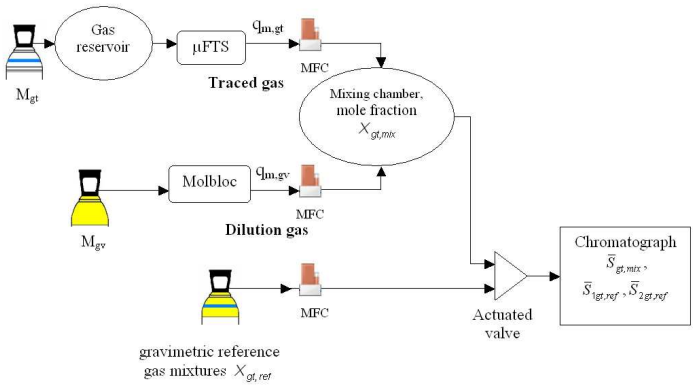

Figure 5 – Experimental set-up used for micro flow calibration

Table 9 – Standard uncertainties of the nitrogen flow measured by the dilution method

Nitrogen flow ($q_{m,gt}$) (µg/s)	Argon flow uncertainty $u(q_{m,gv})$	Nitrogen amount fraction uncertainty $u(x_{gt,mix})$	Nitrogen flow uncertainty $u(q_{m,gt})$	Nitrogen flow uncertainty $u(q_{m,gt})$ (µg/s)
0.3474	0.15 %	0.45 %	0.48 %	0.0017
0.8337	0.15 %	0.45 %	0.48 %	0.0039
1.1810	0.15 %	0.45 %	0.48 %	0.0055
1.7368	0.15 %	0.17 %	0.23 %	0.0039
2.6052	0.15 %	0.17 %	0.23 %	0.0059
3.4736	0.15 %	0.17 %	0.23 %	0.0083
8.684	0.15 %	0.17 %	0.23 %	0.020
17.368	0.15 %	0.11 %	0.19 %	0.033
26.052	0.15 %	0.11 %	0.19 %	0.049
34.736	0.15 %	0.11 %	0.19 %	0.066

3.3 Calibration with the primary constant pressure flowmeter

The constant pressure flowmeter is a primary device widely used in vacuum laboratories of National metrology institutes to carry out the continuous expansion method for low absolute pressure measurements and also for small leak flow rates calibrations, with reference to vacuum. More recently, this principle has shown its ability to calibrate leak rates flowing at atmospheric pressure [4][5]. The measured quantity is the so-called throughput q_{pV}, in $Pa \cdot m^3 \cdot s^{-1}$, given by the following definition:

$$q_{pV} = \frac{\delta(pV)}{\delta t}, \quad (15)$$

at a constant temperature T_0.

In this system, the pressure is constant and equal to p_0 over the measurement. If the volume variation is ΔV during Δt we have:

$$q_{pV} = p_0 \frac{\Delta V}{\Delta t}, \quad (16)$$

The gas which flow rate has to be measured is introduced in a volume where a piston of well known surface S is displaced in order to vary the capacity of this volume and keep the pressure constant (Figure 6). At the initial instant t_1 the position of the piston is x_1 and at the end of the measurement, the piston's position is x_2 at the instant t_2.

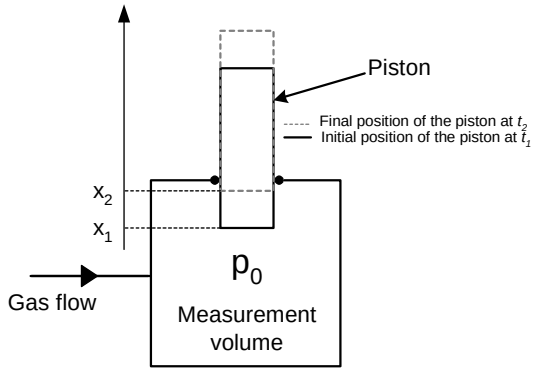

Figure 6 – Measurement principle with the constant pressure flowmeter

Finally, the throughput q_{pV} is:

$$q_{pV} = p_0 \cdot S \frac{(x_2 - x_1)}{(t_2 - t_1)}, \quad (17)$$

The piston's displacement is measured by mean of an optical absolute transducer and the time interval (t_2-t_1) by the computer's clock.

The LNE's constant pressure flowmeter has two pistons (respective diameters of 5 and 20 mm), and is ranging from $1 \cdot 10^{-6}$ to $2 \cdot 10^{-3}$ $Pa \cdot m^3 \cdot s^{-1}$, for gas flow rates referred to atmospheric pressure. Correspondence with mass flow rate of nitrogen $q_{m,CPF}$ is obtained by deriving with time the perfect gas law:

$$q_{m,CPF} = q_{pV} \cdot \frac{1}{M_{N2}} \cdot \frac{1}{RT_0}, \quad (18)$$

where M_{N2} is the molar mass of nitrogen, R the molar gas constant and T_0 the gas temperature inside the measurement volume.

The calibration standard uncertainty $u(q_{m,CPF})$ on the mass flow rate measured with the constant pressure flowmeter is estimated to be:

$$u_{ref}(q_{m,CPF}) = 0.0010 \times q_{m,CPF} + 0.0021 \ \mu g/s.$$

3.4 Calibration results

The calibration of the transfer standard µFTS was performed between 0.35 µg/s and 35 µg/s on the ranges corresponding to the three capillaries (Table 1), both with the dilution method and the constant pressure flowmeter.

The standard calibration uncertainties for the dilution method and the constant pressure flowmeter take into account the short-term reproducibility of the transfer standard u_{rep}(µFTS). This contribution, given in the Table 10, was estimated by performing additional measurements with the constant pressure flowmeter.

Table 10 – Estimated short-term reproducibility u_{rep}(µFTS) of the transfer standard for each capillary

Capillary	u_{rep}(µFTS)
1	0.00020 x q_m + 0.0010 µg/s
2	0.00020 x q_m+ 0.00058 µg/s
3	0.00020 x q_m+ 0.0022 µg/s

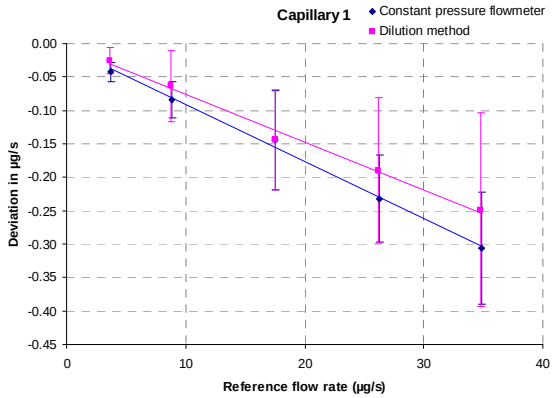

Figure 7 – Calibration results for the capillary 1 with associated regression line for each method; uncertainty bars at k = 2

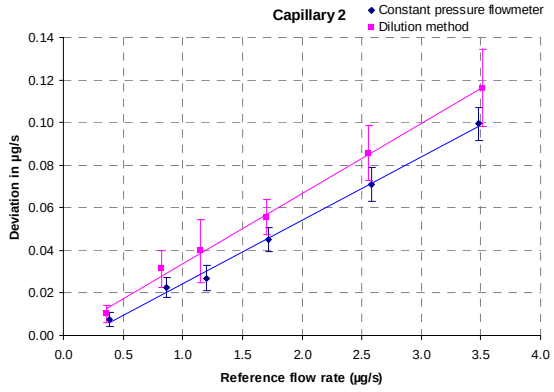

Figure 8 – Calibration results for the capillary 2 with associated regression line for each method; uncertainty bars at k = 2

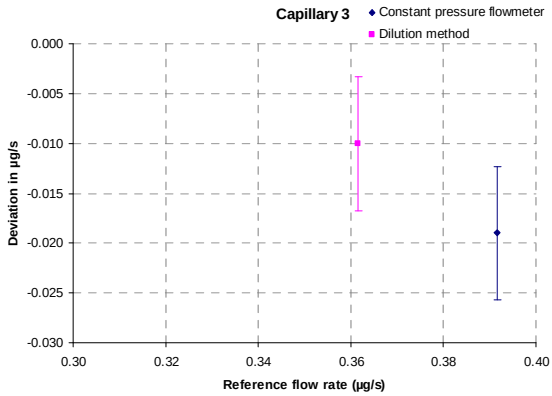

Figure 9 – Calibration results for the capillary 3; uncertainty bars at k = 2

Figures 7 to 9 show the calibration results for each method and for capillaries 1, 2 and 3 respectively. A regression line is plotted for capillaries 1 and 2.

To compare the results obtained with the dilution method and those obtained with the constant pressure flowmeter, the reference value of the deviation is calculated as the weighted mean of the deviations, as recommended in [6]. The calculated standard uncertainty on this reference value is then combined with uncertainty of each method to give the final uncertainties attributed to the deviations from the reference value. Results are given in Tables 11 to 13 and illustrated on the Figures 10 to 12 (the represented plots for deviation correspond to the same reference flow rate for both calibration methods; they are slightly shifted on the graphs to facilitate reading).

Table 11 – Comparison results for capillary 1

Reference Flow rate	Relative deviation from the reference value		Relative uncertainty on the deviation	
	Dilution method	Constant pressure flowmeter	Dilution method	Constant pressure flowmeter
µg/s	%	%	%	%
3.668	0.30	-0.15	0.54	0.39
8.806	0.19	-0.05	0.61	0.30
17.526	0.00	0.00	0.43	0.42
26.211	0.12	-0.04	0.42	0.25
34.821	0.12	-0.04	0.41	0.24

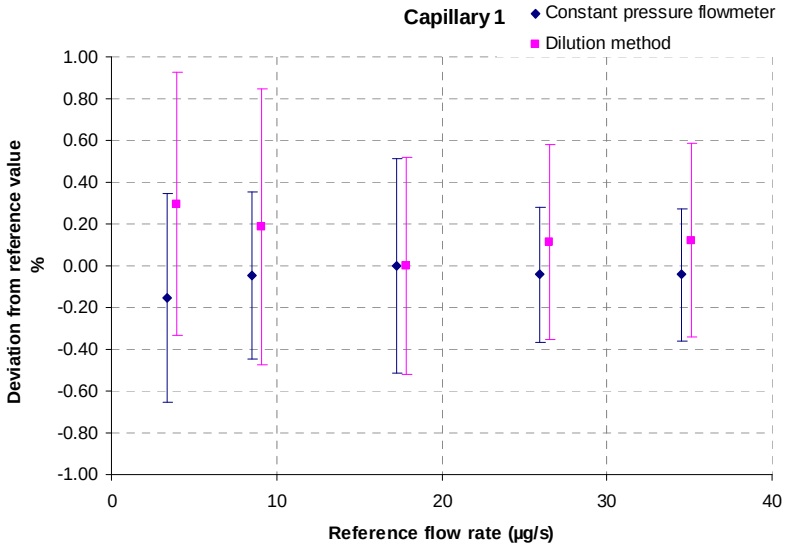

Figure 10 – Comparison results for capillary 1; uncertainty bars at $k = 2$

Table 12 – Comparison results for capillary 2

Reference Flow rate	Relative deviation from the reference value		Relative uncertainty on the deviation (k = 2)	
	Dilution method	Constant pressure flowmeter	Dilution method	Constant pressure flowmeter
µg/s	%	%	%	%
0.3761	0.32	-0.63	1.1	1.5
0.8425	0.67	-0.50	1.0	0.87
1.1753	0.90	-0.31	1.3	0.74
1.7100	0.27	-0.38	0.47	0.56
2.5728	0.30	-0.31	0.50	0.51
3.4970	0.31	-0.14	0.52	0.35

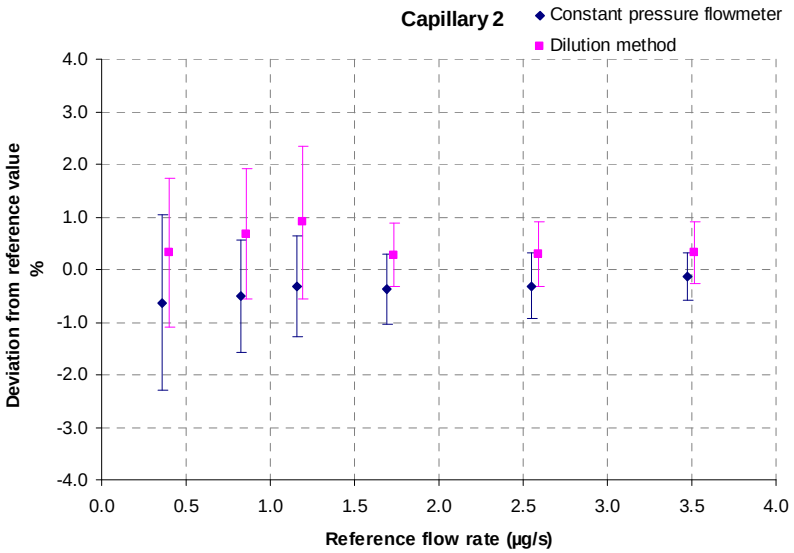

Figure 11 – Comparison results for capillary 2; uncertainty bars at $k = 2$

Table 13 – Comparison results for capillary 3

Reference Flow rate	Relative deviation from the reference value		Relative uncertainty on the deviation (k = 2)	
	Dilution method	Constant pressure flowmeter	Dilution method	Constant pressure flowmeter
µg/s	%	%	%	%
0.3766	1.01	-0.98	1.8	1.8

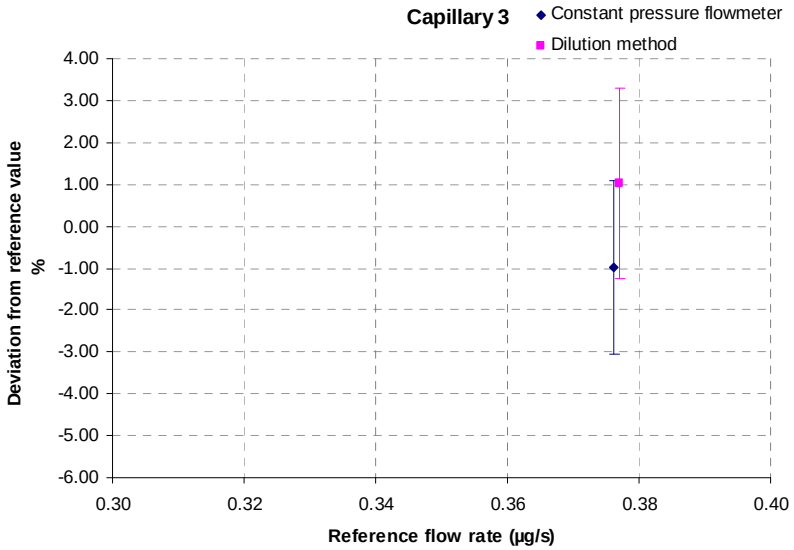

Figure 12 – Comparison results for capillary 3; uncertainty bars at $k = 2$

5. Conclusion

The work presented here shows the performance offered by the dilution method for measuring small gas flows of nitrogen in the range of 0.35 µg/s to 35 µg/s with an relative expanded uncertainty between 1.8 % and 0.4 %.

The calibration results of the µFTS with the dilution method have shown deviations of -0.7 % for capillary 1, 3.3 % for capillary 2 and -2.8 % for capillary 3. Concerning the primary constant pressure flowmeter, deviations of -0.9 % for capillary 1, 2.5 % for capillary 2 and -4.9 % for capillary 3 have been observed. These results will lead to calculate new dimensional parameters of the capillaries (length and radius).

It can be seen that results on reference flow rates with the constant pressure flowmeter are systematically below the results obtained with the dilution method for a same indication of the transfer standard, however always within the respective expanded calibration uncertainties. So the dilution method is validated below 30 µg/s.

Moreover, very few National Metrology Laboratories implement methods and standard facilities in this flow range and no intercomparison was performed between these laboratories. The use of the micro flow transfer standard as travelling standard will enable to demonstrate the equivalence between the methods used and to validate the announced uncertainties.

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