Validation of a Microflow Calibration System based on a Weighing Method with Pneumatic Control

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Abstract To calibrate micro flow sensors and as well flow controllers for liquid handling in bio-medicine diagnoses, a traceable system that considers a weighing approach, virtual piston approach, and a velocity-based approach was proposed at CMS. Firstly, a pneumatic push-and-pull pressure control mechanism for steadily driving fluid was verified and estimated of its measurement uncertainty. The capability of the system depends on weighting of the collection bag, the stability of pressure difference, the time interval and the variation of ambient temperature. Due to the low flowrate, weight of collected fluid was measured within a time interval that was pre-determined to keep required accuracy and uncertainty from potential environmental influences. Tests of liquid flow in channels of different dimensions showed that the present system can measure flowrate from 0.1 μL/min to approximately 10 mL/min with an uncertainty < 1.8%. Deviation of repeated measurements indicated feasibility of the estimation; and comparison with commercial micro liquid sensors indicated practicability of the weighing method.

Keywords: Micro flowrate, Gravimetric analysis, Uncertainty analysis

1. Introduction

Facing the needs from the IC-industry, ultra low gas flowrate has been implemented of its measurement standards at CMS in 2000. The traceability was based on a gravimetric system to measure collected gas. For the emerging biomedical applications, ultra-low liquid flowrate measurement is also essential and many devices have been developed[1-5], indicating the ecessity of standard systems for micro flowrate measurements.

Accompanying with the development of micro flow sensors, some calibration facilities have designed as well. Tai *et al.*[6] applied an idea similar to volumetric prover for gas flowrate; and Bornhop[7] applied an optical velocity-based approach. Based on the existed technique for micro-scale flow measurement at CMS, micro liquid flowrate measurement began with the use of micro-resolution PIV. Considering the accuracy and traceability, however, gravimetric approach is still essential to compare with velocity-based approach and prover-like volumetric approach. Though time consuming, the use of high resolution, high performance mass balances and frequency traceable timer, uncertainty can be accurately estimated. That is, gravimetric flow measurement is the basis for ultra-low mass flowrate calibrations.

In this paper, a calibration system that the fluid was pneumatically driven was implemented to transfer gravimetric determinations to flowrate by micro liquid meters. The tiny increment of weight and the time interval was recorded in the calibration in which the pressure was precisely controlled. The system was proved of its capability of calibrating flowrate from 0.1 μL/min to 10 mL/min; and was estimated of its measurement uncertainty to be less than 1.8%. Besides, repeated measurements and flowmeter tests were applied to verify the practicability. In the near future, a piston-like method and a velocity-based method is to be introduced to determine the proper solution for micro flowrate calibration.

2. Gravimetric Calibration System

Gravimetric approaches are widely adopted for flowrate standards. Conceptually, both dynamic mass determination and static mass determination are available for ultra-low liquid flowrate. To avoid long-time idling in expelling gas to the pipeline during calibration, dynamic mass flow measurement was adopted in our design.

The system architecture is shown in figure 1, which mainly comprises a reservoir, pipelines, liquid collection bag, a mass balance, pressure regulation module, temperature sensor, and operation software. To drive liquid in microchannel, steady water head has been widely used; however, the determination of water head inherently results in considerable uncertainty. Therefore, we employed a pressure control loop to steadily generate micro liquid flow. The pressure control module utilized a two-stage pressure valve module and a precision pressure regulator to stabilize the driving pressure, so that the Gauge pressure was thus reduced of its variation in flowrate measurement ranging from 0.1 μL/min to 10 mL/min. A Si-based microchannel that was embedded between the reservoir and flow meter was used for microfluidic study and velocity-based microflow standard, and was also helpful to stabilize the liquid pressure when further decreasing the flowrate. The collector was firstly made of a gas-sealed aluminum tank, and now was replaced by a transfusion bag on considering the evaporation of working fluid to the air in the tank.

The calibration is performed based on a flying-start -and-flying-stop operation. This means that when the flowrate reaches stable, the time and mass of liquid are recorded synchronously. The dynamic range is 10^5 so that the stop of calibration by time or by mass depends on the flowrate. In addition, the system is operated under a strict temperature control environment to reduce the variance of fluid density which significantly affects the uncertainty of collected fluid quantity.

The working liquid is driven from the reservoir to the collector on the precision balance that was interfaced with a computer to synchronize the mass and time measurements. Since the mass of the bag assembly is much greater than that of the increased mass, the resolution of the balance becomes a significant uncertainty component in addition to pressure control. An automatic control interface written in Labview commanded the calibration process and recorded all the measurement parameters. The change of weight, the time log, the temperature log, and information from pressure regulator are then employed to determine the standard flowrate. If microflow meter under calibration is inserted in the pipeline in serial, calibration can be accomplished by comparing the output of flowmeter with standard flowrate

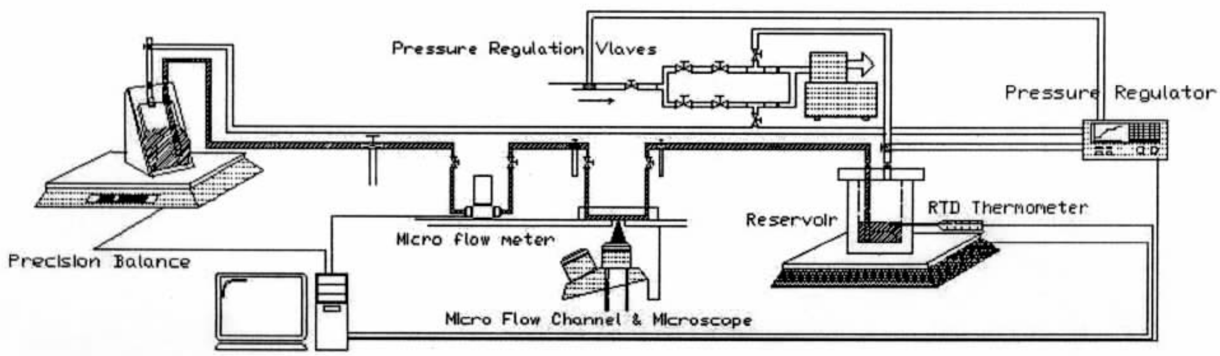

Fig. 1 System architecture for microflow calibration

3. System Evaluation

3.1 Uncertainty Analysis

According to the principle of mass conservation, the mass of the fluid, when measured by the flow measurement facility, is given as

$$m = V\rho_f(1+P\times f)+V_{cp}(\rho_{f,0}-\rho_{f,1})(1+P\times f) = (W_1-W_0)B \quad (1)$$

where

m : Mass of the working fluid that flows through the pipeline and is delivered to weighing bag

V : Volume of the working fluid that flows through the flow meter under calibration

ρ_f : Density of fluid at atmosphere

P : Gauge pressure (Pa)

f : Compressibility of the working fluid

W : Readout of weighing scale

B : Buoyancy correction factor, and is expressed as

$$B=1+\rho_a(1/\rho_f-1/\rho_w) \quad (2)$$

where ρ_a, ρ_f and ρ_w are the densities of the air, line fluid and calibration weights, respectively

cp (suffix) : Pipeline between the flow meter and the weighing bag.

f (suffix) : Line fluid

0 and 1 (suffix): Conditions at the beginning and end of the calibration, respectively.

Because the properties of fluids are influenced by environment, the fluid mass in the pipeline between the calibrated flow meter and the weighing bag might change during the calibration. The change is considered in Eq. (1), where the variance of fluid quantity is deduced from the density difference ($\rho_{f,0}-\rho_{f,1}$). Since the variance of pressure is not significant, the compressibility of air are listed here for reference. Therefore, the fluid volume that flows through the micro flowmeter can be deduced as

$$V = (W_1-W_0)B/[\rho_f(1+ P\times f)]-V_{cp}(\rho_{f,0}-\rho_{f,1})/ \rho_f = (W_1-W_0)B/[\rho_f(1+ P \times f)]-\Delta V_{cp} \quad (3)$$

where ΔV_{cp} is the change of the fluid quantity in the pipeline between the flow meter and the weighing bag from the beginning to the end of test and is equal to $V_{cp}(\rho_{f,0}-\rho_{f,1})/\rho_f$. While the calibration was engaged under constant ambient circumstances, the change of density of working fluid and of collection fluid quantity can be neglected. Therefore, the volume flow rate of the meter under test in a specific time interval can be deduced from Eq. (3) and expressed as

$$Q_v = V/t=[(W_1-W_0)B/[\rho_f(1+P\times f)]]/t \quad (4)$$

where Q_v is the volumetric fluid rate and t is the duration of the calibration.

The system can calibrate various meters as long as the flowrate can be stabilized. From Eq.(1), the initial and final weight, buoyancy correction factor, driving pressure difference, fluid control volume and compressibility coefficient are influential factors in the microflow calibration system. Referring to ISO-5168[7], the calibration system can be expressed of its measurement uncertainty as

$$u(V) = [(\frac{\partial V}{\partial W_1}u(W_1))^2 + (\frac{\partial V}{\partial W_0}u(W_0))^2 + (\frac{\partial V}{\partial B}u(B))^2 + (\frac{\partial V}{\partial P}u(P))^2 + (\frac{\partial V}{\partial \rho_f}u(\rho_f))^2 + (\frac{\partial V}{\partial f}u(f))^2 + (\frac{\partial V}{\partial \Delta V_{cp}}u(\Delta V_{cp}))^2]^{1/2} \quad (5)$$

For convenience, the above measurement uncertainty can be expressed in a relative form as follows,

$$\frac{u(V)}{|V|} = [(\frac{\partial V}{\partial W_1}u(W_1))^2 + (\frac{\partial V}{\partial W_0}u(W_0))^2 + (\frac{\partial V}{\partial B}u(B))^2 + (\frac{\partial V}{\partial P}u(P))^2 + (\frac{\partial V}{\partial \rho_f}u(\rho_f))^2 + (\frac{\partial V}{\partial f}u(f))^2 + (\frac{\partial V}{\partial \Delta V_{cp}}u(\Delta V_{cp}))^2]^{1/2}/|V| \quad (6)$$

The buoyancy correction factor B is a function of ambient temperature, pressure, humidity, and fluid temperature; while ΔV_{cp} is mainly influenced by fluid temperature. Assuming the independence of the above influential factors, the uncertainty of B should be expressed as the combination of uncertainties of ambient temperature, pressure, humidity and the fluid temperature. Meanwhile, the uncertainty of ΔV_{cp} is a function of the uncertainty of fluid temperature. However, because the buoyancy correction factor can be regarded as a constant and the variance of the fluid volume is negligible, the uncertainties of buoyancy correction B is obtained from table and volume variation ΔV_{cp} is artificially estimated.

The buoyancy correction factor, fluid density, and the quantity variation in the pipeline are partially interdependent because all of them relate to fluid temperature. However, in the evaluations of sensitivity analysis, the above three variables are considered independent because two of them are assigned values. Therefore, combining the uncertainties of the three variables by means of root-sum-squares is feasible.

3.2. Sensitivity Analysis

The sensitivity coefficients that derived from Eq.(2) and Eq.(6) are shown in Eq.(7),

$$\begin{aligned}\frac{u(V)}{|V|} &= [c_{W1}u^2(W_1) + c_{W0}u^2(W_0) + c_B u^2(B) \\ &+ c_P u^2(P) + c_{\rho_f} u^2(\rho_f) + c_f u^2(f) \\ &+ c_{\Delta V cp} u^2(\Delta V_{cp})]^{1/2} / |V|\end{aligned} \quad (7)$$

where

$c_{w1} = \partial V / \partial W_1 = B/[\rho_f(1+P\times f)] \approx 1/\rho_f$

$c_{w0} = \partial V/\partial W_0 = - B/[\rho_f(1+ P \times f) \approx -1/\rho_f$

$c_B = \partial V / \partial B = (W_1-W_0)/[\rho_f(1+ P \times f)] \approx (W_1-W_0) / \rho_f$

$c_P = \partial V/\partial P = -f(W_1-W_0)B/[\rho_f(1+P\times f)^2] \approx -f(W_1-W_0)/\rho_f$

$c_{\rho f} = \partial V/\partial \rho_f = -(W_1-W_0)B/[\rho_f^2(1+P\times f)^2] \approx -(W_1-W_0)/\rho_f^2$

$c_f = \partial V/\partial f = -P(W_1-W_0)B/[\rho_f(1+P\times f)^2] \approx - P(W_1-W_0) / \rho_f$

$c_{\Delta V cp} = \quad \partial V/\partial \Delta V_{cp} = -1$

The relative uncertainty of determined micro flow rate can be combined from Eq.(4)and (7) as follows,

$$\begin{aligned}\frac{u_c(Q_V)}{|Q_V|} &= [(\frac{\partial Q_V}{\partial V}u(V))^2 + (\frac{\partial Q_V}{\partial V}u(t))^2]^{1/2} \\ &= \{(u(V)/t)^2 + (\frac{V}{t}u(t)/t)^2]^{1/2}\}/|Q_V| \\ &= [(u(V)/V)^2 + (u(t)/t)^2]^{1/2}\end{aligned} \quad (8)$$

and the effective degrees of freedom is expressed as

$$\nu_{eff,QV} = u(Q_V)^4/[(u(V)/t)^4/\nu_{eff,v} + c_t^4 u^4(t)]/\nu_t] \quad (9)$$

All considered uncertainty sources in the measurement were quantified and expressed either by Type A or Type B evaluations. The uncertainty components are combined using the method of root-sum-squares and then multiplied by a coverage factor to give an expanded value at 95% confidence level. Table 1 summarizes the sensitivity analysis of the current architecture.

4. Results

The microflow calibration system utilized D. I. water as working fluid under constant temperature of 25 ± 0.01 °C. All parts of the pipelines are made of 1/8" stainless steel tube and connected by Swagelok. The Silicon-based microchannels utilized in our system comprised of channel sectors and a glass cover, which were fabricated by Deep-RIE and packaged by Anodic bonding. Figure 2 shows the chips with microchannels in the experiments. By dosing fluorescent particles into the working fluid, the transparent side of the glass cover enables the integration of the calibration system and the μ-PIV measurement.

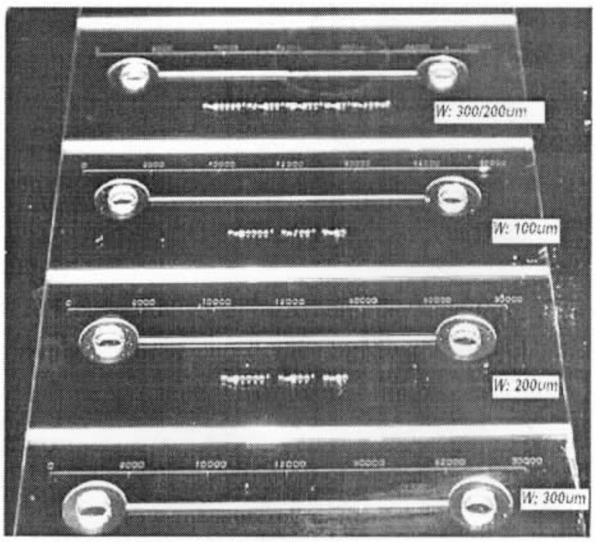

Fig. 2 Microhannels in chip-level (Length: 20000 μm, Height: 45 μm ± 2 μm)

Table 1. Summary of the sensitivity analysis in current microflow calibration system

Uncertainty Component $u(x_i)$	Source of uncertainty	Type	Possibility Distribution	Standard Uncertainty $u(x_i)$	Sensitivity Coefficient c_i	D.O.F. ν_i
W_1	Final weight			0.06 mg	1.003	20
	Calibration					
	Reproducibility	B	Normal	$1\sim2\times10^{-9}$		9
	Readout	B	Rectangular	0.029		∞
	Reading timing error	B	Rectangular	0.06		12
W_0	Final weight			0.06 mg	1.003	20
	Calibration	B	Normal	$1\sim2\times10^{-9}$		9
	Reproducibility	B	Rectangular	0.029		∞
	Readout	B	Rectangular	0.06		12
	Reading timing error					
B	Buoyancy Correction factor Env. & Fluid condition	B	Rectangular	0.00012	0.01 ~10.028	∞
ρ_f	Density			0.0000476 g/cm^3	-0.01 ~ -10.056	9
	Regression equation	B	Normal	0.0270		12
	Reproducibility	A	Normal	0.0100		24
	Fluid temperature	B	Rectangular	0.0378		4
P	Pressure Pressure transducer	B	Rectangular	8×10^{-9} ~ 1.7×10^{-5} g/cm^2	3.9×10^{-18} ~ 7.6×10^{-12}	10
f	Compressibility Coefficient Fluid temperature	B	Rectangular	~0	-0.0030 ~ -5114.32	∞
ΔV_{cp}	Variation of fluid volume Variation of temperature	B	Rectangular	0.0000185 cm^3	-1	12
t	Recorder	B	Rectangular	0.0207sec	2.78×10^{-5} ~2.78×10^{-10}	4

Three microchannels of different dimensions were tested in the experiment; each was repeated 10 times. In addition to mass measurement, temperature, pressure, density, and time must also be monitored and recorded for system validation. Figure 3 shows the average flow rates through three channels of different widths from 100 μm to 300 μm under different driving pressures with uncertainty < 1.8%. The uncertainty analysis in this experiment is discussed in the next section.

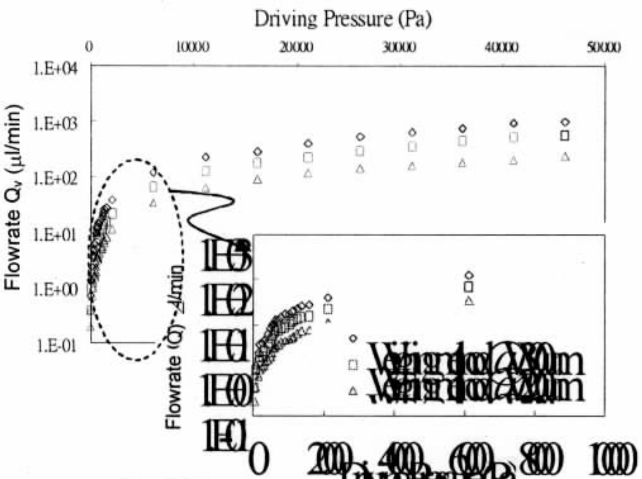

Fig. 3 Flowrate vs. driving pressure

5. Discussions

Instinctively, increasing the collection of working liquid to the bag can reduce the measurement uncertainty based on heavy weight and long time interval. However, long-time calibration is not efficient and would induce extra error due to environmental influence. For instance, mass loss is possible due to evaporation and seepage through the bag and connecters; also, the variation of temperature and controlled pressure are possible to influence the calibration. Considering the acceptable uncertainty for calibrating marketed microflow sensors and the mentioned measurement limits, the minimum collected weight depended on the flowrate, and thus lower flowrate resulted in higher uncertainty. According to experiments and analysis as shown in Table 1, the system was evaluated of its measurement uncertainty to be less then 1.8% for all flowrate rages as listed in Table 2, where the relative expanded uncertainty, coverage factor, and the effective degree of freedom at 95% confidence level depend on desired range of the flowrate. Particularly, the measurement uncertainty is better then 0.2% for all except the case of 0.1 μL/min $\leq Q_V <$ 1 μL/min due to the resolution of mass balance. This means there still remains rooms for improvement.

Table. 2 Estimated uncertainty and efficient degree of freedom of flowrate measurement

Range Q_V (μL/min)	Minimum collected weight (g)	$U_{95}/\lvert Q_V\rvert$ (%)	$k_{95,QV}$	$\nu_{eff,QV}$
$0.1 \leq Q_V < 1$	0.01	1.79	2.06	26
$1 \leq Q_V < 100$	0.1	0.19	2.05	27
$100 \leq Q_V < 1000$	1	0.03	1.97	208
$1000 \leq Q_V \leq 10000$	10	0.03	1.97	346

To verify the stability and accuracy of the calibration system when calibrating various micro flow meters, a long-term check and control procedure was applied. Figure 4 shows the comparison results of the experimental deviation of 10 repetitions (vertical axis) with the above estimations (horizontal axis) under the same environmental conditions. Obtained from different flowrate and through different channels, the uncertainties in figure 4 were indicated in liquid volume (μl). Ideally, the measurement deviation should approach the estimation uncertainty as indicated as the dash line in figure 4. Most of the indicated points were around the dash line, which show the feasible measurements were performed. More points were below the dash line, indicating that the deviation of experimental results were smaller than the previous estimations according to the sum of root squares of each affecting factor for the calibration system.

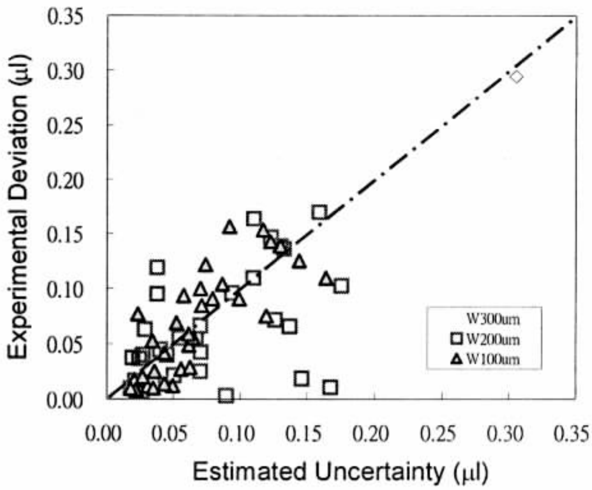

Fig. 4 Uncertainty comparisons of the microflow calibration system

Furthermore, marketed microflow meters were tested in this phase for comparison to test the applicability of calibration system. Three flowmeters for different ranges of flowrate from 0.1 μl/min to 1000 μl/min were employed. Figure 5 shows the result of Rheotherm TU sensor that has larger flowrate than the other two. The comparisons shown in figure 4 and 5 implied the rationality of the uncertainty analysis; however, more detailed long-term experiments for system validation were also required.

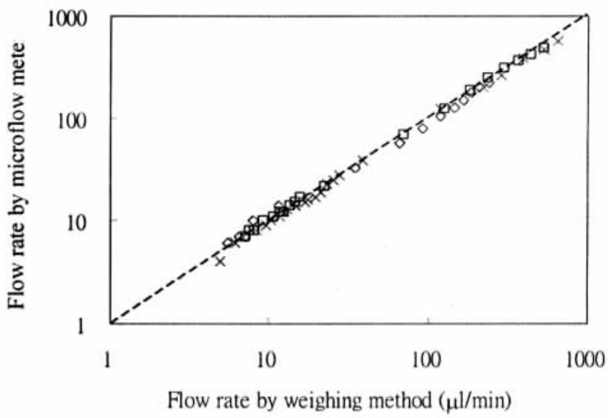

Fig. 5 Comparison of flowrate measurement by using weighing method and a microflow meter

6. Conclusion

A calibration facility for micro liquid flowrate was designed and implemented because of the needs from bio-medicine applications. To drive the fluid in the microchannels stably, a pneumatic driving mechanism was adopted with precisely pressure control. The system was evaluated to calibrate flowrate from 0.1 μL/min to 10 mL/min; and was also estimated of its relative uncertainty of < 1.8% for Q<1μL/min and < 0.2% for Q < 1μL/min based on measurement parameters and the capability of environmental control. In addition, both comparisons with repeat measurements and demonstration by using micro liquid sensors manifested the applicability of the operation. The results also implied that long-term verification was still necessary for improving the measurement capability.

Acknowledgment

The authors are grateful to the Bureau of Standards, Metrology and Inspection (BSMI/MOEA), Taiwan China, for support of this research under project code 93-1403-31-H-00-00-00-24.

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