Evaluation of Microflow Calibration Rig using Static Weighing System with Flying Start-and-Finish Method

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Abstract

A Liquid microflow calibration rig was designed and evaluated. The developed calibration rig has a static weighing system and improved diverter valves for small flow rates. The weighing system succeeded in reducing an uncertainty due to an evaporation effect by using a detachable weighing tank. Experimental results of evaporation effect were introduced. In this study, two types of devices were introduced so that a Flying Start-and-Finish (FSF) method was implemented into the static weighing system. One of these devices is a set of diverting valves, and the other is a syringe pump with a linear encoder. Estimation tests were conducted. Diverter timing errors for the set of diverting valves were estimated to be about 6 ms.

1. Introduction

Microfluidic devices such a microflow sensor and syringe pump are used in various industries. Calibration rigs are needed as reference flow standards to calibrate these flow devices. Various types of calibration rigs have been developed [1]-[2]. A major weighing method for microflow facility is a dynamic weighing method. In the dynamic weighing method, the flow rate is defined in terms of the change in weight over time. A nozzle tube downstream of a flow sensor is installed under liquid surface in a weighing tank to make a clearance between the nozzle tube and the inlet of the weighing tank. Thus, fluctuations in surface tension must be considered and the evaporation and buoyancy of the immersion tube must be corrected.

To avoid these effects, we developed a detachable system that is placed between a weighing tank and a pipeline [1]. This design is effective to reduce the evaporation effect, but the weighing system becomes a static weighing. Without a special device, the static weighing system must be operated by a Standing Start-and-Finish (SSF) method.

However, it is undesirable to change flow rates at flow sensors under calibration. Some flow meters have quite long dumping time of output value to a changing of flow rate. Some flow meters for micro-flow rates change liquid temperature passed through the flow meter because of its electric power consumption. If flow rate changes, variation of temperature distribution inside tube between the flow meter and the weighing tank. The variation of temperature distribution increases a calibration uncertainty due to changing of liquid mass in the dead volume.

In this study, two types of devices were introduced so that a Flying Start-and-Finish (FSF) method is implemented into the static weighing system. One of these devices is a set of diverting valves, and the other is a syringe pump with a linear encoder.

This paper introduces how to reduce the evaporation effect in the microflow calibration rig Then, the experimental results of these FSF methods are discussed.

2. Detachable system on weighing tank

Figure 1 shows a picture of the improved calibration rig by using the detachable weighing tank system.

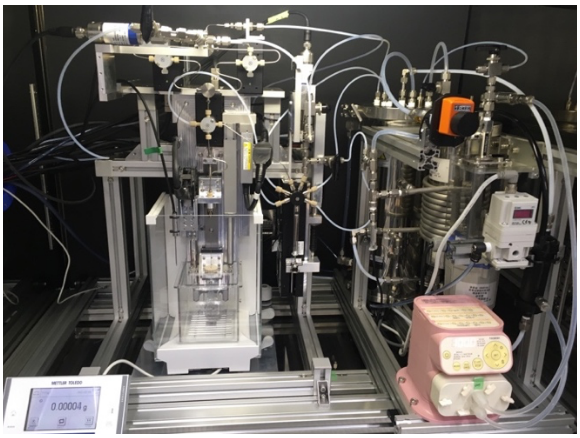

Figure 1: Picture of calibration rig.

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A schematic diagram of the weighing tank and the detachable device is shown in Figure 2. The weighing tank consists of a glass container and a specially designed lid. The glass container is tightly capped by the lid, which has orifices sealed by an elastomer (septum seal). This elastomer orifice seal is a commercial septum seal for gas chromatography instruments. Through the orifices of the lid, a needle nozzle is inserted into the glass container. The diameter of the needle nozzle is 0.63 mm (23 gauge). The needle nozzle is fixed on a rigid frame. The weighing tank can move up or down. The needle nozzle is automatically connected to and disconnected from the weighing tank by motor-controlled lifts. After the weighing tank is disconnected from the nozzle, it is placed on the weighing scale without the application of any tension. This tension-free condition allows accurate mass measurement.

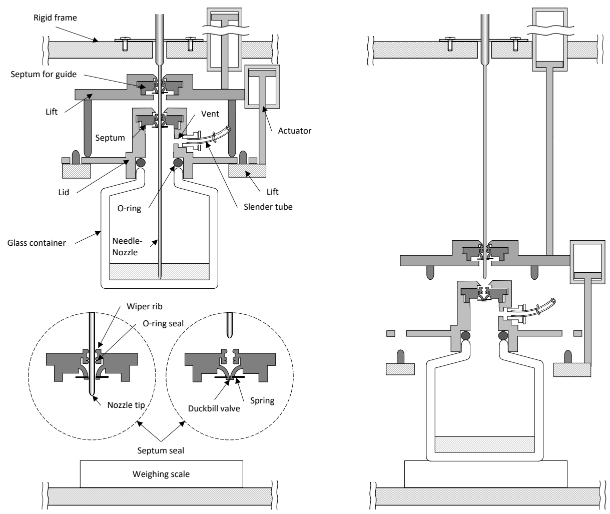

Figure 2: Schematic diagram of calibration rig.

While the liquid is collected, the air pushed out from inside the weighing vessel. If a vent of the weighing tank is widely open, extra vapor could diffuse from inside tank to outside through the vent. Even if the cross section of the vent is small, the resulting evaporation effect could be significant at microflow calibration.

Previous system [1] had a flexible bag at the vent of the weighing tank that collected the air pushed out from inside the weighing vessel. We improved the ventilation system further. The air bag has been replaced to a slender tube. The slender tube is better in handling and is still effective for reducing the evaporation loss.

Figure 3: Photo of the weighing tank with the slender tube.

The liquid mass, M_{DUT}, (or volume, V_{DUT}) that passes through the device under test (DUT) is determined by the gravimetric method, and this reference value is compared to the output signal of the DUT for calibration. The mass of the working liquid collected into the weighing tank, M_{WT}, is calculated based on the difference in scale reading before and after main liquid collection considering the buoyancy correction.

$$M_{WT} = (m_2 - m_1)\frac{M_{STD}}{m_{CAL}}\left(1 - \frac{1.2}{8000}\right)/\left(1 - \frac{\rho_{AIR}}{\rho_{LIQ}}\right) \quad (1)$$

where m_1 and m_2 are the first and second readings of the weighing scale, respectively. M_{STD} is the conventional value of the mass in the calibration certificate for the standard weight. m_{CAL} is the weighing scale reading when the scale is loaded with M_{STD}. ρ_{AIR} and ρ_{LIQ} are the densities of the air and working liquid, respectively. In this study, the working liquid was pure water. Its density was obtained from a previous study [3].

3. Estimation of evaporation effect

3.1 Estimation method for evaporation loss

To evaluate evaporation effect during calibration, the mass change of the weighing tank under no-flow condition, $M_{WT-NOFLOW}$, was measured at the same procedure described in Reference [1]. If the weighing system had an error due to evaporation effect, the reading of m_2 is reduced than m_1 even at no-flow condition.

The loss of weight due to evaporation during collection is a function of period, t_{12}, which is the time period between the first and second readings of the weighing scale. Thus, the evaporation rate, q_{EVA}, can be obtained by differentiating $M_{WT-NOFLOW}(t)$ with respect to period, t_{12}.

$$q_{EVA} = -\frac{\partial\left(M_{WT-NOFLOW}(t)\right)}{\partial t_{12}} \quad (2)$$

3.2 Estimation results of evaporation rate

Figure 4 shows the difference of weights after long period at no-flow condition. The time period t_{12} was varied 1 min to 60 min. Each symbol is average of four repeated measurements and error bar represents standard deviation. The slope of the linear least squares fit represents the evaporation rate, q_{EVA},, under each condition.

The symbols of "Slender tube" are results under the normal condition of this calibration rig as shown in Figure 4. Inner diameter of the slender tube was 1.2 mm and the length was 200 mm. The slope of "Slender tube" indicated that the evaporation rate was about 0.000 6 mg/min. It is suggested that the developed system with the slender tube has an ability to reduce the evaporation rate down to below 0.001 mg/min, which is 0.1 % of 1 mg/min.

"Open duct" means the condition that the slender tube and the fitting on the tank lid side in Figure 2 and 3 was removed. The narrowest duct part inside the lid was of 1 mm inner diameter and 1 mm length. Evaporation rate of "Open duct" condition was 0.004 8 mg/min. This value is equivalent to an error of 0.48 % at a calibration flow rate of 1 mg/min.

These results suggested that the detachable weighing tank system and the ventilation by the slender tube has an ability to reduce the evaporation rate down to below 0.001 mg/min, which is 0.1 % of a calibration flow rate of 1 mg/min.

However, in order to avoid changing of flow rates at flow sensors under calibration, the Flying Start-and-Finish method needs to be introduced.

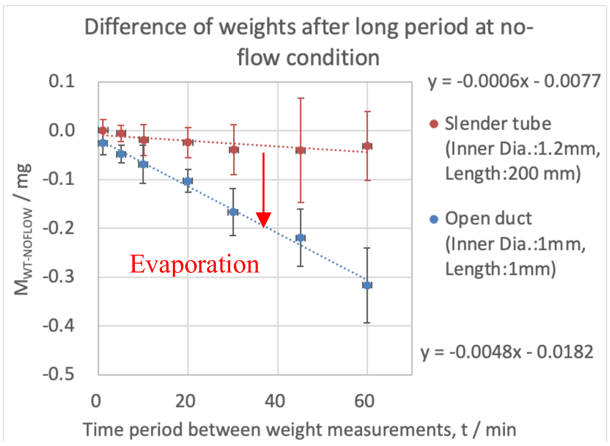

Figure 4: Mass difference of the weighing vessel under no-flow conditions at various times.

4. Flying Start-and-Finish methods

4.1 Synchronized three-way vales (diverter valves)

The diverter valves are consisted of two of three-way valves. The three-way valve is actuated by a motor-drive-positioner. These two of three-way valves are sequentially actuated within a short period. To prevent stopping flow at the flow meters, the valves are operated along to following sequence.

Here, both of the diverter valves are assumed to be set to the bypass-line. The one of the three-way valves is moved firstly from the bypass-line to the weighing-tank-line. The pass of the moving valve is closed for an instant and then opened to the other line. During moving of the first valve, the other valve (the second valve) keeps a flow line to the bypass-line. Around the moment of the opening of the first valve to the weighing-tank-line, the second valve starts moving and closing the bypass-line. The start timing of the second valve is controlled at one millisecond resolution by using a delay time from a pulse signal of the first valve motion. The pass of the second valve is closed for an instant but the pass of the first valve has been opened to the weighing-tank-line. After finishing of motion of the second valve, both of the diverter valves are switched to the weighing-tank-line.

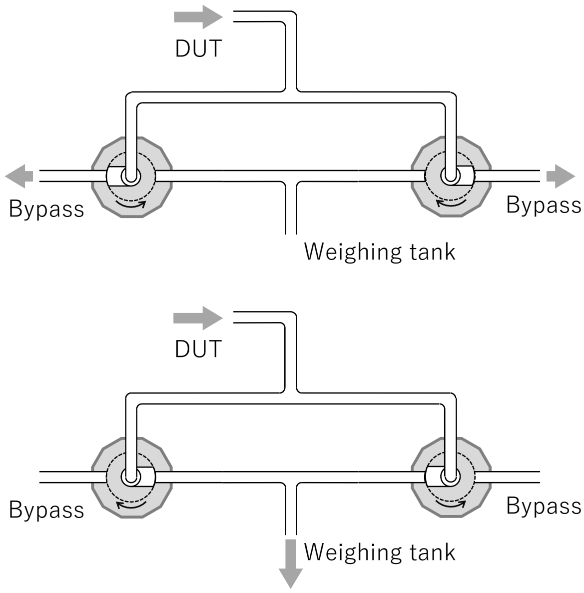

Figure 5: Diverter valve.

In general, it is afraid that this type of diverter valve can change a flow rate at the flow meter due to a changing of pressure drop of the flow line. However, if the ratio of the pressure drop of diverter valves system was low compared with the other pressure drop, the change of total flow rate can be small enough.

4.2 Syringe pump with linear encoder

The other system for FSF is consisted of the syringe pump and the linear encoder with an encoder counter. A portion of the syringe volume is identified via linear encoder positions. The portion of volume is calibrated using the static weighing system with the SSF method. Then, flow meters are calibrated by using the syringe pump as the volumetric flow standard. The output of a flow sensor is accumulated between gate signals from the encoder counter. The gate signals are sent at the moments of passing through the edge positions of the calibrated volume without stopping flow. For the case of mass flow meters, the volumetric flow standard is converted to mass flow value by applying a liquid density value at the syringe pump.

When a syringe pump is used for the flow generator, the stability of the flow rate is important. At this stage, the syringe pump of this rig has a pulsation of flow due to the mechanical system. In the future work, this is going to be replaced to another syringe pump which has smooth motion.

4.3 Comparison of the FSF methods

Figures 6 shows that relative deviations from the reference standard value of a Coriolis flow meter calibrated by using different FSF methods. The minimum collection time in the results of Figure 6 was two min. There was no significant difference from each other. However, reproducibility of flowmeter might not be small enough. In case of the syringe pump with linear encoder, there was flow pulsation during collection. When the diverter timing error was small enough, FSF method by the diverter valves was better in this calibration rig.

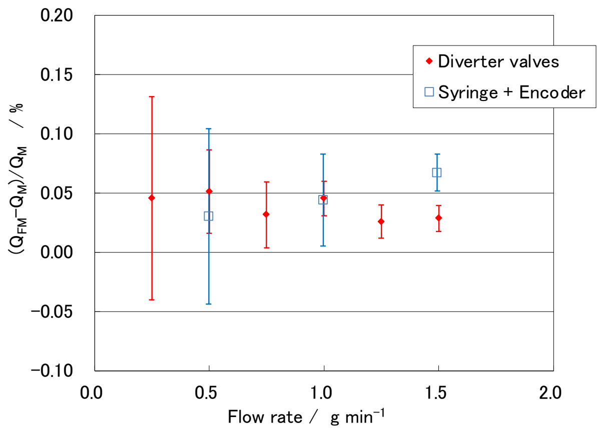

Figure 6: Relative error of Coriolis flow meter.

5. Estimation of diverter timing error

5.1 Estimation equations

The diverter timing error can be evaluated by using the evaluation method described in ISO4185 [4]. Based on the static weighing method, the standard mass flow rate Q_M is given in Equation (3).

$$Q_M = \frac{M_{WT}}{t_C} \tag{3}$$

$$t_C = t_{Id} - \Delta t \tag{4}$$

Here, t_C is the time interval between the starting and stopping signals. The collection time t_C might include the diverter timing error Δt. The time t_{Id} is equivalent to the collection time assuming the diverter is in an ideal condition. This timing error influences the calibration results systematically and cannot be reduced by repeating calibration.

The following test (A. 1.1 Method 1) is one of the estimating methods described in ISO4185. First, the initial weight m_1 is measured. Then the weighing tank is filled up by a single diversion to measure the weight after collection m_2 and the duration of diversion t_{C1}. Next, the total weighing scale $\sum_{i=1}^{n} m_i$ and total time $\sum_{i=1}^{n} t_i$ are measured in a series of n diversions without resetting the timer and the scale. Finally, the error results are obtained from these values by the following formula.

$$\Delta t = \frac{t_{C1}}{n-1}\left(\frac{q_1}{q_n}\cdot\frac{\sum_{i=1}^{n} m_i / \sum_{i=1}^{n} t_i}{(m_2 - m_1)/t_{C1}} - 1\right) \tag{5}$$

Here, q_1 represents the flow rate during a single diversion measured by a flowmeter, and q_n is the average of the flow rates measured by the flowmeter during n diversions.

The diverter timing error is also derived by following expression using Equation (3) and (4).

$$\frac{(Q_{FM} - Q_M)}{Q_M} = \frac{\left(Q_{FM} - \dfrac{M_{WT}}{t_{Id} - \Delta t}\right)}{\dfrac{M_{WT}}{t_{Id} - \Delta t}}$$

$$\approx -\Delta t \cdot \frac{1}{t_C} + \frac{\left(Q_{FM} - \frac{M_{WT}}{t_{Id}}\right)}{\frac{M_{WT}}{t_{Id}}} \tag{6}$$

Here, Q_{FM} represents the flow rate meter reading during time, t_C. This expression means that a relative deviation calibrated by using a calibration rig which has a diverter timing error is a linear function of reciprocal of the collection time, $1/t_C$. The diverter timing error can also be given from the fitting slope of calibrated data varying the collection time.

5.1 Estimation by using ISO4185 method
In order to determine the diverter timing error, the estimating tests was conducted by using the Method 1 in ISO4185. We set the diversion number at n = 10. The flow rate and collection weight were set to be 1 g/min and 2 g, respectively.

The diverter timing error was estimated by using Equation (5). The burst test was repeated 10 times. The average of the diverter timing error was estimated to be 5.5 ms and the standard deviation of ten results was 1.6 ms.

5.1 Estimation by varying collection time
Figure 7 shows the calibrated deviations when the collection time is intentionally shortened. All calibrations were carried out at a constant flow rate (1 g/min). The abscissa presents the reciprocal of collection period, and the ordinate indicates the relative deviation. Each symbol shows the average of six calibration results. The error bars show the standard deviation of the six calibration results for each collection time.

The diverter timing error was estimated to be 6.0 ms by using Equation (6). It is confirmed that the diverter timing errors by two different methods were consistent values each other.

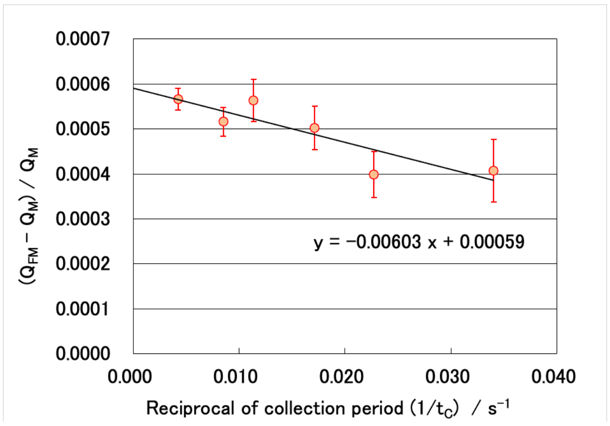

Figure 7: Calibrated deviations varying collection period.

These results show that the FSF methods have possibility to reduce the error timing below 10 ms.

7. Conclusion

A weighing system with low evaporation effects with Flying Start-and-Finish (FSF) method was developed. To avoid evaporation loss, a slender tube was adopted for ventilation of the weighing tank. In the present study, a set of diverting valves was introduced to the weighing system.

By analysing the experimental weighing results under no-flow conditions, the evaporation rate with the slender tube ventilation was estimated to be 0.000 6 mg/min during the collection period. It shows that the weighing system has ability to reduce the evaporation rate during the collection period down to below 0.001 mg/min which is 0.1% of the target minimum flow rate (1 mg/min).

Two different estimation methods to determine the diverter timing error were conducted for the set of diverting valves. The average of the diverter timing error by using ISO4185 method was estimated to be 5.5 ms. In addition, a Coriolis flow meter was calibrated by varying collection time. By analyzing the calibrated data, the diverter timing errors were estimated to be about 6 ms. These results show that the FSF methods has possibility to reduce the error timing below 10 ms.

The presented estimation methods for evaporation and the diverter timing error are applicable to calculate the uncertainty of the calibration rig.

References

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[4] ISO 4185 1980: *Measurement of Liquid flow in Closed Conduits—Weighing Methods*.