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Development of small weighing system for liquid micro-flow

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Abstract

A prototype small weighing system for liquid micro-flow from 10 mL/min down to 1 µL/min has been constructed. The calibration method was the static weighing with standing start and stop. The weighing system was set in a constant temperature and humidity chamber. At small flow rates, evaporation effect comes to be a significant error source. In order to reduce evaporation error, a detachable system between a weighing tank and a pipe line has been developed. The detachable device prevents pipeline tension to the weighing scale and is able to keep the pipeline closed while collection. Performance of the weighing system was evaluated by analysis of calibration factors to a syringe pump. At a preliminary test, the collected liquid was from 0.2 g to 2 g of water. In the preliminary test, the evaporation error was estimated to be below 0.1 % down to 2 µL/min of water. The system is aiming to use at calibrations of liquid micro-flow meter for the semiconductor industry and the medical field.

1. Introduction

A purpose of this study is to minimize evaporation errors in weighing system. The goal is to reduce the evaporation error to less than 0.1% in the flow rate range from 1 µL/min to 10 mL/min. For this goal, a detachable system between a weighing tank and a pipeline was developed. By using this mechanism, a calibration facility for micro flow sensors is constructed. By providing a means to evaluate micro flow sensors using this calibration system, we aim to contribute to the micro / nano technology, semiconductor industry, and medical fields.

NMIJ has expanded the range of flow standards to smaller flow rates to cope with fuel flow meters in automotive industry [1][2]. On the other, micro flow sensors are also used in semiconductor industry and medical fields. Upstream of the vaporizer for semiconductor equipment, a liquid mass flow meter measures and controls small flow rates (down to several µL/min) of various of liquids. In pharmaceutical and chemical industry, microflow chemical synthesis using microreactors instead of batch processes is being developed. Micro flow sensors and syringe pumps will play an important role in the flow chemical synthesis system.

The weighing method is the main calibration method for micro flow calibration. The syringe pump also needs to be calibrated using the weighing method. At micro flow rates, the evaporation effect causes significant errors in the weighing method [2]. Several approaches can be used to reduce the evaporation error, including trapping evaporation with a saturated vapor atmosphere

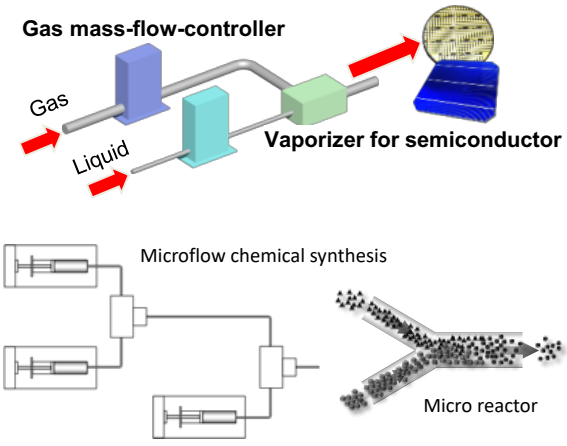
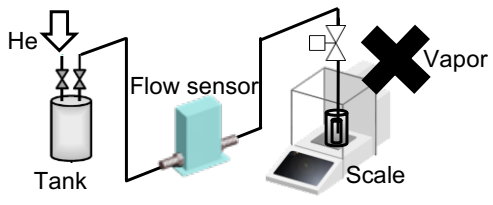


Figure 1: Micro flow sensors and devices.



(Error: Vaporization, Surface tension, Buoyancy)

Figure 2: Conventional weighing system.

[2-4] and applying a cover layer of a lower-density liquid [5,6], such as oil, which has a low vapor pressure.

In this study, a novel detachable device was adopted. A prototype small weighing system for liquid micro-flow from 10 mL/min down to 1 μL/min has been developed. Preliminary test has been conducted to evaluate evaporation error. This paper reports the results.

2. Weighing system

2.1 Outline of calibration facility

The facility is set in a constant temperature (usually 23 deg. C) and humidity chamber. The calibration method is the static weighing with standing start and stop. The working liquid is water and can be collected up to 10 g at one calibration. The weighing scale has a resolution of 0.01 mg. To remove bubbles, an inline degasser equipped with a vacuum pump is installed in a circulation line. The flow is generated by a pressurized tank or syringe pump.

2.2 Weighing tank and detachable device

The weighing tank and the detachable device are shown in figure 3. The weighing tank is capped by a lid having an orifice sealed with elastomer. A needle-nozzle from a flow meter or syringe pump can penetrate the orifice. The needle-nozzle is fixed with a frame, and the weighing tank is moved up and down by a motor-controlled lift. The weighing tank is automatically connected and disconnected with the needle-nozzle by the motion of the lift. While the liquid is collected, evaporation from the weighing tank is prevented because of the orifice seal between the lid and the needle-nozzle. The air pushed out from inside the weighing tank is accumulated into an air bag. Then, after disconnected from the nozzle, the weighing tank is placed on a weighing scale at free from any tension.

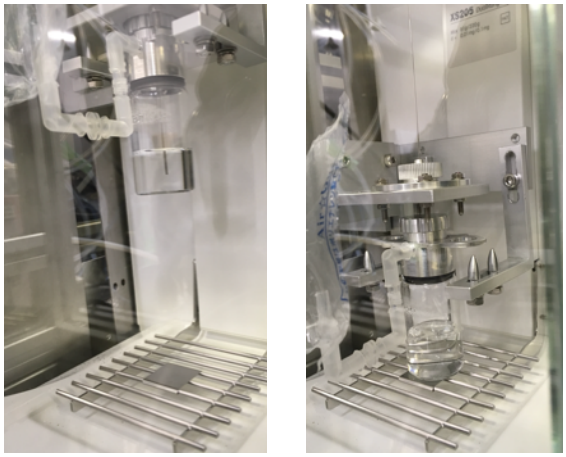


Figure 3: Detachable system between the weighing tank and needle-nozzle.

2.3 Syringe pump as working standard

The syringe pump is shown in figure 4. The syringe pump can be used to calibrate a flow meter as a working standard. A portion of the syringe volume identified by the pulse address is calibrated by using the primary weighing system. The syringe pump is also used to drain the working liquid in the weighing tank. As a preliminary test to evaluate the evaporation effect, the pulse rate per volume of the syringe pump was calibrated at different flow rates. The syringe pump has a resolution of approximately 19,200 pulses per mL when using a 2.5 mL syringe. In case of 0.1 mL syringe, the syringe pulse factor is to be 480,000 (pulse/mL).

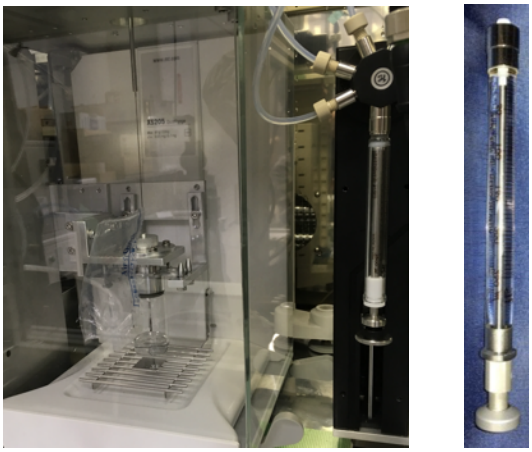


Figure 4: Syringe pump.

At this stage, the motor specification of the syringe pump is a stepping type, but the syringe pump will be replaced with a servomotor type to improve flow stability.

3. Results and discussion

3.1 Analysis model using calibrated syringe pulse factor

The syringe pulse factor is the reciprocal of the volume per stroke of the pulse in the calibration region of the cylinder. It is thought that the syringe pulse factor should be constant for different flow rates. However, the calibrated value could be change if the weighing system or syringe system have something error source. For instance, if the calibration system has an evaporation error, the syringe pulse factor increases as the flow rate decreases [2]. Of course, other errors might affect it. if air bubbles are generated in the connection pipe between the syringe and the weighing tank, the factor decreases as the flow rate decreases. Analytical models are plotted in figure 5. Assuming that the other errors are smaller than the evaporation error, the evaporation rates can be estimated by using fitting.

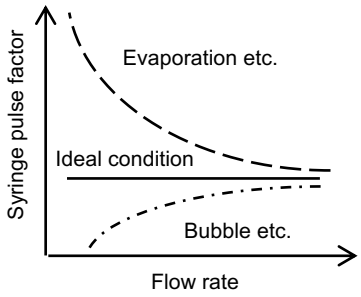


Figure 5: Analysis model using calibrated syringe pulse factor

3.2 Calibration result of 2.5 mL syringe

The syringe pulse factor is calibrated at different flow rates to evaluate the evaporation error of a weighing system equipped with the detachable mechanism. When a 2.5 mL syringe is attached to the pump actuator, the minimum flow is 25 µL/min. At the minimum flow rate, the pump actuator creates a pulsating flow due to the nature of the stepping motor. It is not suitable for flow meter calibration, but there is no problem in evaluating evaporation effect.

Figure 6 shows the calibration factor for a 2.5 mL syringe. The small spread of the plot shows that the detachable mechanism has sufficient repeatability to a 2.0 g collected weight. It has been shown that evaporation is prevented as the calibrated factors do not change significantly for different flow rates.

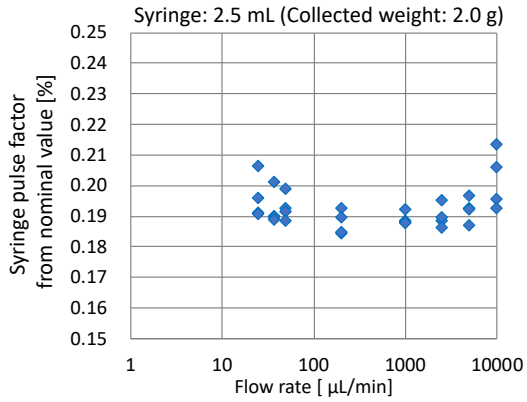


Figure 6: Calibration factor for 2.5 mL syringe.

3.2 Calibration result of 0.1 mL syringe

The installed syringe was replaced to a smaller size to investigate the evaporation effect at lower flow rates. For a 0.1 mL syringe, the minimum flow rate becomes 1 µL/min and the collected weight is 0.08 g. Figure 7 shows the calibration factor for the 0.1 mL syringe. The open vent condition (without the air bag) was also calibrated in order to confirm the phenomenon of evaporation error to the calibrated syringe pulse factor. The spread of the plot became large because the relative value of the repeatability of the detachable mechanism to the collected weight increased. For the sealed weighing tanks (normal condition), the calibration factor did not increase as the flow rate decreased. This means that this weighing system is not affected by evaporation down to 1 µL/min. On the other hand, for the open vent case, the calibrated syringe pulse factor increased as the flow rate decreased, and the evaporation rate was estimated to be 4 nL/min to 8 nL/min.

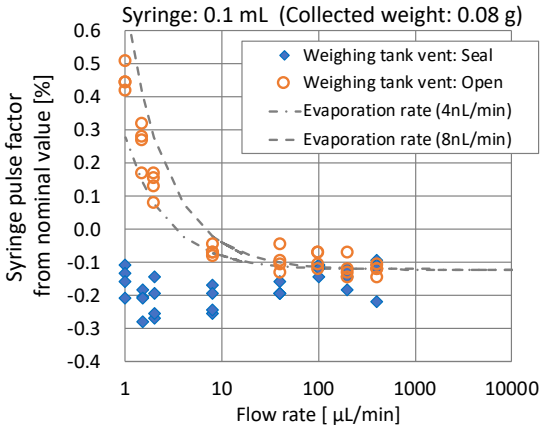


Figure 7: Calibration factor for 0.1 mL syringe.

7. Conclusion

A weighing system for liquid microflow from 10 mL/min to 1 µL/min has been constructed. In order to prevent evaporation errors, a novel detachable system was adopted between the weighing tank and the pipeline. Two size of syringes (syringe pumps) have been calibrated using the weighing system. The ability of the detachable system has been confirmed by analysis of the syringe pulse factor. It was confirmed that the evaporation error was suppressed at flow rate down to 1 µL/min.

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