Establishment of Micro Liquid Flow facility at NIM

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Abstract

In order to meet the needs of micro-flow measurement in industrial, biological and medical fields, the National Institute of Metrology (NIM) has established a micro liquid flow facility based on dynamic weighing method, with the flow rate range of 100nL/min ~ 150mL/min. The facility is composed of two sets of high accuracy electronic balances as the main standard, and three Coriolis mass flowmeters and two sets of independent pipeline systems according to different flow rate ranges. It has been designed a special degassing, a filter and a pipe air exhausting system, as well as a group of weighing containers and syringes of different specifications. An acquisition method of weighing value based on the fluctuation frequency is adopted to effectively reduce the uncertainty introduced by dynamic reading. A 'dual time' method is used to realize the synchronization between weighing system and meter under test by the "time stamp" with resolution of 1ms. The uncertainty sources of balance that came from weighing accuracy of mg magnitude, short-term drift and long-term stability are evaluated by experimental method. In addition, the uncertainty introduced by liquid evaporation and capacity change of middle pipe are also analyzed. The results show that the facility uncertainty is better than 1.5%(k=2) at 100nL/min flow rate, and the uncertainty is better than 0.1%(k=2) when the flow rate is above 1mL/min.

Keywords: liquid flow; micro flow; dynamic gravimetric method; uncertainty budget

1. Introduction

The micro liquid flow generally refers to the flow rate of less than 100 mL/min, even as low as nL/min magnitude. In the field of biology, pharmacy and chemical industry, micro flow is used for catalyst addition, nucleic acid detection, and others quantitative liquid control. The accurate measurement of micro liquid flow can reduce the experimental scale, thus saving the development cost and reducing the risk of experimental failure. In the medical field, it is an important control parameter for syringe pump, infusion pump and anesthesia machine, that is directly related to the safety and health of patients.

In recent years, in order to meet the needs of scientific research and flow meter traceability, many countries or economies have established micro liquid flow facilities, and most of which adopt the principle of dynamic gravimetric method, and different technical methods are used in the key processes of facilities such as water supply system, evaporation reducing and degassing method. The main information and uncertainty of these facilities are shown in Table 1.

Table 1: Characteristics of micro flow facility in different organizations

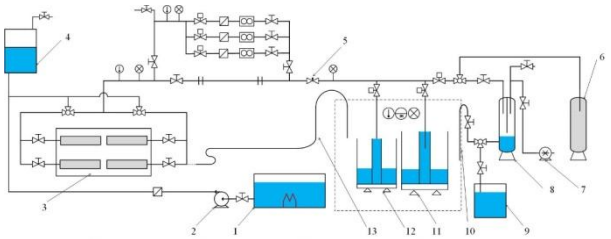
Organization	Principle	Evaporation Reducing	Liquid Temperature(℃)	Degassing Method	Uncertainty (k=2)
METAS[1]	dynamic weighing method	humidity trap	22	ultrasonic degassing	0.07%~3%
LNE[2,3]	dynamic weighing method	oil film covering	10~50	negative pressure	0.1%~0.6%
AIST[4]	static weighing method	humidity trap	20±0.08	online degassing	0.066%~0.07%
IPQ[5]	dynamic weighing method	humidity trap	room temperature	heating and ultrasonic degassing	0.1%~0.5%
CMS	static/dynamic weighing method	oil film covering	15~27	/	0.2%~2.0%
DTI[6]	dynamic weighing method	oil film covering	21±1	ultrasonic degassing	0.1%~2%

NIST[7]	dynamic weighing method	humidity trap	room temperature	vacuum pump de-bubbler	0.04%~4.5%
VSL[8~9]	dynamic weighing method	humidity trap	room temperature	vacuum degassing	0.1%~0.3%
CMI[10]	dynamic weighing method	humidity trap	0~50	online degassing	0.2%~0.6%

2. Design and implementation

2.1 Overall design and system division

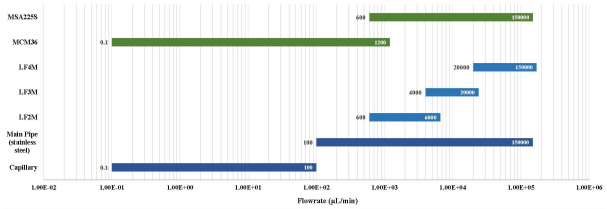
After the investigation of existing micro-flow facilities and the requirements, NIM has developed a micro-flow facility based on dynamic gravimetric method (DGM) with a flow-rate range of 100nL/min to 150mL/min. The facility is mainly composed of water pretreatment system, syringe pump water supply system, master meters system, pipeline system, weighing system, pipeline air exhausting system and measurement and control system, and its process flow is shown in Figure 1.



1-heating and ultrasonic degassing water tank; 2-pump; 3-syringe pump; 4-storage bag; 5-regulating valve; 6-CO_2 vessel; 7-vacuum pump; 8-air and liquid isolation tank; 9-waste liquid tank; 10-drainage pipe；11-balance: MSA225S; 12-balance: MCM36; 13-capillary pipe

Figure 1: Process flow diagram of micro liquid flow facility

The facility can be divided into several measurement subsystems with different flow ranges according to the difference of the standards or the pipe systems. Two high accuracy balances are used as the main standards, the flow rate range are separated to 100μL/min~1.2mL/min (balance: MCM36) and (0.6~150) mL/min (balance: MSA225S) for the use efficiency and uncertainty. In order to improve the flexibility of the facility, three Coriolis mass flowmeters as the master meters are used as the secondary standard, which can cover the high flow rate of (0.6~150) mL/min. The experimental pipe system consists of two kinds of pipe, that are the main pipe made by stainless steel with inner diameter of 4mm and the capillary (or peek) pipes with inner diameter of 50μm~0.8mm. The flow rate range of main pipe is (0.6~150) mL/min, that is suitable for the installation and test the tube type flowmeters, such as Coriolis mass flowmeter and ultrasonic flowmeter. However, for lower flow rate, it could be a main uncertainty source due to the large capacity of main pipe and small bubbles inside that are difficult to remove. The capillary pipes are more flexible to connect. The flow-rate ranges of these subsystems are shown in Figure 2.



MCM36、MSA225S - main standard;
LF4M、LF3M、LF2M - master flowmeter.

Figure 2: The flow range of different measuring subsystems

2.2 Weighing system

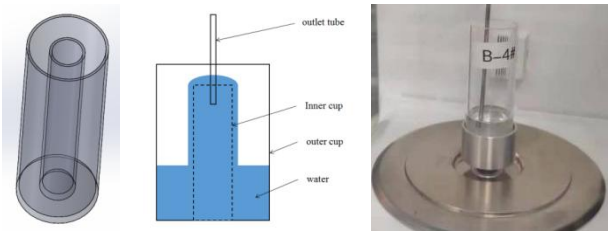
The balance is one of the key devices of facility. Two high accuracy balances with resolution of 1μg and 10μg respectively are used as the main standards, and their main technical parameters are shown in Table 2 :

Table 2: Electronic balance parameter

Items	Parameters	
Model	MCM36	MSA225S
Maximum Weighing	31 g	220g
Resolution	1 μg	0.01mg
Repeatability	<±0.002mg	≤±0.025mg
Linearity	<±0.015mg	≤±0.1mg
Partial Load Error	15 μg	0.15mg
Minimum Initial Weight	0.82mg	20mg

Due to the actual effective quantity of weighing is very small, the accuracy of weighing could be affected by evaporation and dripping during the test, so the weighing container is also the key component. In order to avoid discontinuous flow, needles or capillaries are used as outlets and immerse in the liquid of the weighing container. The evaporation rate of water is usually below about 170μg/min under tested laboratory conditions, so the evaporation reducing method should be used when the flow rate is below 100μL/min. Based on the comparison of some evaporation reducing methods and different kinds of oil film coverage effects, paraffin oil is finally used as the medium for coverage whose thickness is about 3mm[11]. When the flow rate is high (such as above 1mL/min), the liquid level in the weighing container rises rapidly. The weighing container is constructed as an inner and outer double beaker, as shown in Figure 3 (a). The inner beaker keep overflowing all the time during the measurement, that is in order to avoid causing the change of flow

rate and liquid surface tension[7] by the change of liquid level. But it should be noted that when the flow rate is below 1mL/min, discontinuous overflow could occur due to the influence of surface tension. When the overflow state is changed, the indicator value of weighing will fluctuate greatly. Therefore, it is not suitable to adopt inner overflow beaker for the low flow rate.



(a) (b) (c)

a）stucture of double beakers; b）liquid overflow; c）picture of the weighing container

Figure 3: Schematic diagram of weighing container

The 'dual time' synchronization method is used to improve the accuracy of the DGM. The standard time base is crystal oscillator of 11.0592 MHz. When the measurement and control system receives the command of 'Test Start' (such as pulse rising edge sent by photoelectric switch that is triggered by the syringe pump, as shown in Figure 4 ~ Figure 6), it starts respectively to monitor and mark the latest dynamic weighing value of balance and the first whole periodic signal (such as pulse rising edge) of meter under test (MUT) with 'time stamp'. When the measurement would be finished, the procedure is same. Therefore, the accurate measurement time of the weighing and the MUT can be obtained respectively, and the accumulated flow rate of standard and MUT can be converted to the same time scale(the calculation model is shown in Formula (2)). The schematic diagram of dual-time synchronization method and the schematic diagram of flow rate test process are as follows Figure 4 and Figure 5.

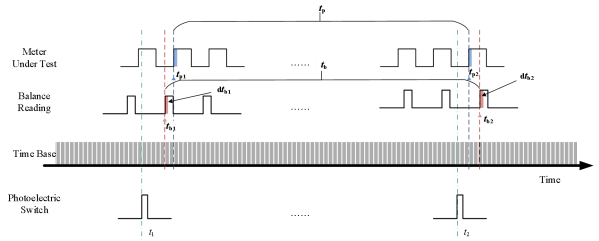


Figure 4: Schematic diagram of signal synchronization in 'dual time' method

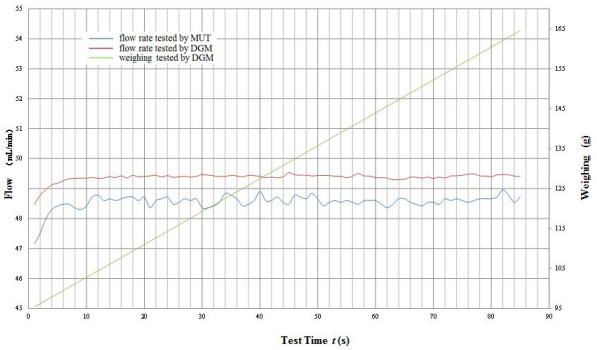


Figure 5: Schematic diagram of test process

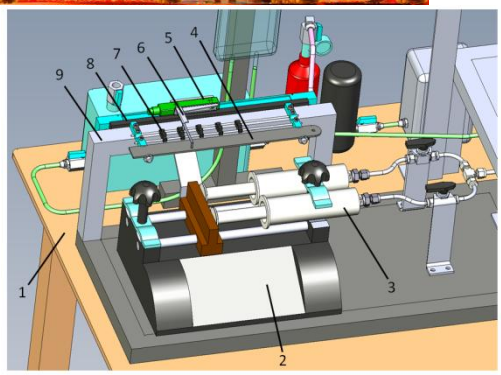
2.3 Liquid supply system

A syringe pump with ultra-high stability is used for the main liquid supply of facility. The flow rate can be controled from pL/min to mL/min by matching syringes of different specifications, and the pump can be operated with a maximum linear thrust of 75lbs. A microprocessor-controlled stepper motor with minimum stepper angle drives the screw and push rod forward, and its specific parameters are shown in Table 3.

Table 3: syringe pump parameters

Project	Parameters
Stability	±0.25%
Repeatability	±0.05%
Minimum	0.5μL
Maximum	140mL
Minimum Velocity	1.56pL/min
Maximum Velocity	215.8 mL/min
Straight Line of Thrust	34kg（75lbs）
Distance Forward of Motor at Each Step	0.082μm/ step

In order to obtain the realtime instantaneous flow, a set of auxiliary measurement system for syringe pump is designed, which is also used for the accurate calibration of syringe pumps. The main component of the system is a grating ruler, with a resolution of 4μm and active length of 200mm. The reading head of grating ruler is connected with the syringe pump by a guide rod, the motor can drive them to move synchronously. A metal sheet is fixed on the reading head to trigger the photoelectric switch at the specified position, which can accurately mark the actual measurement section, as shown in Figure 6.



1-vibration isolation optical platform; 2-syringe pump; 3-syringe; 4-steel plate ruler; 5-grating ruler reading head; 6-metal sheet for trigger; 7-photoelectric switch; 8-grating ruler slide rail; 9-grating ruler bracket.

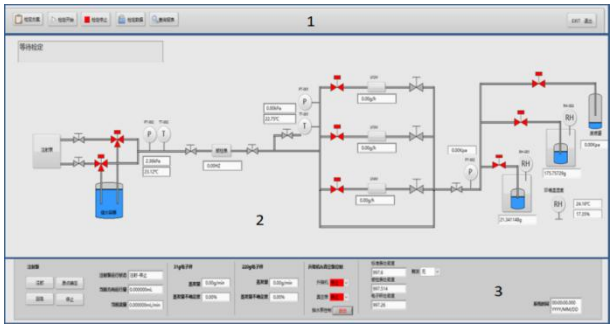
Figure 6: Schematic diagram of syringe pump measurement auxiliary system

The maximum distance of syringes travel is about 120mm, and different volumes of syringes can be selected according to flow rate. Most of syringes are glass, and some special ones were made by stainless steel and ceramic for experiment.

2.4 Auxiliary system

The auxiliary system of facility included experimental water pretreatment system, pipe air exhausting system and measurement and control system. The experimental liquid is ultra pure water, which needs to be degassed by heating and ultrasonic oscillation before use. After it cools down, it can enter the storage bag of facility through a three-stage filter. For removing the air from the pipe as far as possible when filling water into the facility, the vacuum pump exhausting and carbon dioxide replacement method are used at the end and high position of the entire pipe system.

The measurement and control system is mainly composed of I/O control module, analog data acquisition module, frequency signal acquisition module, serial port communication module, standard time base module and measurement software. The schematic diagram of the software interface is shown in Figure 7.



1-menu area; 2-operating area;3-auxiliary commands and display area

Figure 7: Main interface of measurement software

3. Experiment and uncertainty analysis

3.1 Mathematical model

Taking a MUT being calibrated by DGM as an example, the accumulated mass flow rate measured by the weighing system can be expressed as follows:

$$Q_{\mathrm{m}} = m_{\mathrm{s}} - \Delta m_{\mathrm{D}} - \Delta m_{\mathrm{P}} - \Delta m_{\mathrm{Va}} - \Delta m_{\mathrm{O}} \qquad (1)$$

Where, Q_{m} is accumulated mass flow rate; Δm_{D} is the mass deviation introduced by the synchronization deviation between weighing system and MUT, Δm_{P} is the mass deviation introduced by the variation of the capacity of the middle pipes, Δm_{Va} is the mass deviation introduced by medium evaporation at the weighing container, and Δm_{O} is other factors affecting mass measurement (including the changes of surface tension at the weighing containers); m_{s} is the standard mass value obtained from the balance and converted to the corresponding measuring time of the measured meter, as described in section 2.2, the balance and the measured meter adopt the 'dual time' to synchronize, and m_{s} can be obtained by Formula (2) :

$$m_{\mathrm{s}} = \left(m_2 - m_1\right) \cdot \frac{t_{\mathrm{p}}}{t_{\mathrm{b}}} \cdot C_{\mathrm{ba}} \cdot C_{\mathrm{bp}} \qquad (2)$$

Where, m_1 is the first value of weighing after 'test strart' command, m_2 is the first value of weighing after 'test finish' command; t_{p} is total test duration of MUT, t_{b} is actual time corresponding to m_1 and m_2; C_{ba} is air buoyancy correction coefficient, the calculation model is shown in Formula (3).

$$C_{\mathrm{ba}} = \frac{0.99985}{1 - \dfrac{\rho_{\mathrm{a}}}{\rho_{\mathrm{w}}}} \qquad (3)$$

Where, ρ_{a} is air density, ρ_{w} is experimental liquid density; C_{bp} is insert tube buoyancy correction coefficient, the calculation model is shown in Formula (4).

$$C_{\mathrm{bp}} = 1 - \frac{A_{\mathrm{p}}}{A_{\mathrm{b}}} \qquad (4)$$

Where, A_{p} is cross-sectional area of the insertion tube (needle), A_{b} is effective cross-sectional area of weighing container (beaker).

The accumulated volume flow rate can be expressed by the following formula[13]:

$$Q_v = \frac{Q_m}{\rho_w} \quad (5)$$

Where, Q_v is standard accumulated volume flow under the state of MUT.

Due to the weak correlation, the main uncertainty components are combined by square and root method. For an example of 1mL/min, the uncertainty budget of the volume flow rate is shown in Table 4.

Table 4: Evaluation of uncertainty at 1mL/min

Symbol	Main Sources of Uncertainty		Standard Uncertainty $u_{rel}(x_i)$	Sensitive Coefficient $c_{rel}(x_i)$	$u_{rel}(x_i)\cdot c_{rel}(x_i)$	Fractional Uncertainty Synthesis
m_s	accumulated mass measurement	standard weight	0.000%	1	0.000%	0.001%
		balance resolution	0.000%	1	0.000%	
		dynamic reading	0.000%	1	0.000%	
		repeatability	0.000%	1	0.000%	
		accuracy	0.000%	1	0.000%	
		short-term stability	0.000%	1	0.000%	
		long-term stability	0.000%	1	0.000%	
C_{ba} /C_{bp}	buoyancy correction	air density	2.9%	1.20×10^{-03}	0.003%	0.017%
		liquid density	0.003%	-1.20×10^{-03}	0.000%	
		inserted tube buoyancy correction	0.013%	1	0.013%	
		surface tension	0.010%	1	0.010%	
Δm_p	pipe capacity change	temperature change of pipeline and liquid	0.036%	1	0.036%	0.043%
		pipe micro-deformation	0.000%	1	0.000%	
		pipe gas holdup	0.023%	1	0.023%	
Δm_D	synchronization	dual time method timing	0.001%	1	0.001%	0.001%
Δm_{Va}	evaporation effect	evaporation rate of liquid	0.019%	1	0.019%	0.019%
t	time	timer	0.002%	1	0.002%	0.002%
ρ_w	liquid density	density calculation formula	0.001%	1	0.001%	0.003%
		liquid temperature	0.002%	1	0.002%	
		liquid pressure	0.000%	1	0.000%	
$u(q_v)$	uncertainty synthesis				0.05%	
	expanded uncertainty (k=2)				0.10%	

See Section 3.2-3.7 for specific analysis methods of major uncertainty components.

3.2 Mass measurement

(1) The weights for the balances calibration

The weights used in the calibration are all E_1 grade, with the minimum of 1mg and the maximum of 100g. According to the different measurement mass and corresponding weight specifications, the maximum permissible error can be obtained from the corresponding technical regulation[14], and the uncertainty introduced by it can be calculated according to the uniform distribution.

(2) The accuracy and repeatability of balance

After conventional calibration of balance, combined with the characteristics of facility, the normal weighing in measurement was used as the increment to test the relative error and repeatability relative to the increment at different tare weights. Experimental conditions were:

A. MCM36: increment of 1g or 2g, tare range of (10~28) g;

B. MSA225S: increment of 10g, 20g or 30g, tare range of (10~200) g.

The experiment was repeated 6 times at each test. The experiments of group A and B showed that the relative error of indicated value was not beyond ±0.005%, and the repeatability was better than 0.003%, and the balance showed good basic performance. In order to further determine the

lower limit of the use of the balance, the increment value was further reduced to mg magnitude. Three standard weights of 5mg, 50mg and 500mg were loaded on the MCM36 balance with three different tare weights of 10g, 15g and 25g respectively, and each weight loading at each test were also repeated 6 times. The experimental results are shown in Table 5:

Table 5: Test results of MG grade measurement performance of MCM36 electronic balance

No.	Increment Weight (mg)	Tare Weight (g)	Average Error (μg)	Relative Error	Repea-tability
1	5	10	0.0	0.00%	0.14%
2	5	15	2.6	0.05%	0.07%
3	5	20	4.1	0.08%	0.09%
4	50	10	4.1	0.01%	0.01%
5	50	15	2.2	0.00%	0.01%
6	50	20	2.1	0.00%	0.01%
7	500	10	7.2	0.00%	0.00%
8	500	15	6.5	0.00%	0.00%
9	500	20	4.3	0.00%	0.00%

As can be seen from the experimental results, the relative error and repeatability of the balance are significantly worse under 5mg test. For the lower limit flow of the facility, if the uncertainty requirement is also decline to about 1%~2% (k=2), the mg magnitude also can be used. Several mg magnitude can greatly reduce the measurement time to less than 1h under minimum flow (such as 100nL/min), which can avoid or reduce the impact of environmental condition changes and balance drift.

(3) The short-term stability test of balance

Under the low flow rate condition, the single measurement time is long that can reach 30min to 1h, and the effective weighing is relatively small. The short-term drift characteristics of balance should be considered as a uncertainty source, that can be tested by experiments. A standard weight was placed on balance, and the indicator was recorded every 1s and continuously for about 8h. For instance, an experiment data of MCM36 balance is shown in Figure 8.

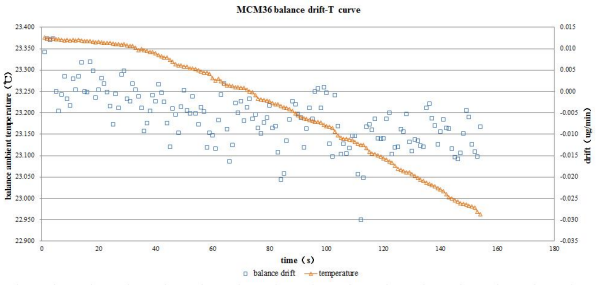


Figure 8: The relationship between drift of MCM36 balance and ambient temperature

After several tests, it was found that the maximum drift rate of MCM36 balance was less than 0.1μg/min, and that of MSA225S balance was less than 0.5μg/min. It also could be found between this drift rate and ambient temperature and humidity has certain relevance. But it is difficult to establish mathematical model to correct this drift, much more experiments and research should be necessary. So the maximum drift rate is an uncertainty with uniform distribution into account.

(4) The dynamic reading test of balance

The DGM requires a balance with high sensitivity and dynamic characteristics, and its operation mode should be set to 'filling mode'. In this mode, the balance outputs real-time weighing data every 0.2s that has not been averaged and filtered, so the fluctuation is relatively large and needs to be tested. A 20g E_1 grade weight was loaded, and balance value was automatically recorded each 0.2s by a computer. The experimental results are shown in the blue line in Figure 9.

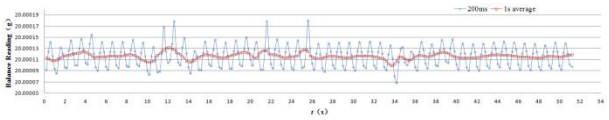


Figure 9: Comparison of dynamic reading modes

The experimental results show that the fluctuation of readings is large that reaches 50μg~80μg. That means relative uncertainty introduced could be more than 1% for the minimum weighing (3mg). The spectrum analysis results obtained by FFT (Fast Fourier Transformation) for the experimental data of the blue line in Figure 9 are shown in Figure 10.

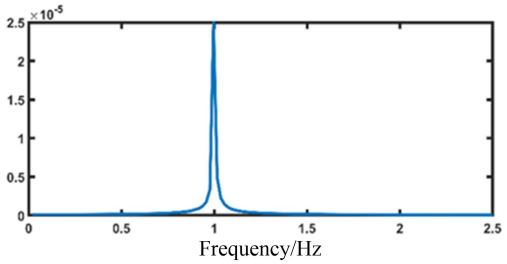


Figure 10: Dynamic reading spectrum analysis results

It can be seen from Figure 10 that the dynamic reading of MCM36 balance has an obvious periodic fluctuation with a frequency of 1Hz, which could be some oscillation mechanism of the balance. In view of this feature, the starting and final value of weighing reading are changed from single value to moving average values (by the latest 5 times measurement data). As shown in the red curve in Figure 9, the fluctuation decreases

significantly, and the influence of the dynamic reading is reduced to less than 20μg.

(5) The Long-term stability of balance
It is found that the maximum relative variation of the two balances is both 0.003% by comparing the calibration data at a six-month interval. They have a good long-term stability. Based on the above main sources, the uncertainty of weighing can be obtained at different flow rates. An example of 100nL/min is shown in Table 6.

Table 6: Uncertainty of balance weighing at 100nL/min

Items	Unit	Numerical	Distribution Coefficient	Introduced Uncertainty	Using Value	Relative Uncertainty
resolution	mg	0.001	3.46	0.0003	3	0.01%
dynamic reading	mg	0.020	1.73	0.0115	3	0.38%
repeatability	mg	0.005	1	0.0050	3	0.17%
accuracy	mg	0.005	1	0.0050	3	0.17%
short-term drift	nL/min	0.1	1	0.1000	100	0.10%
long-term stability	mg	0.001	1.73	0.0006	3	0.02%

3.3 Signal Synchronization
As shown in Figure 6 and Formula (2) in Section 2.2, the 'dual time' method is adopted. The resolution of the timing system is better than 1ms, and considering the fluctuation of flow rate, the maximum deviation introduced by synchronization is conservatively estimated to be 5ms. The relative uncertainty of the flow introduced by synchronization is related to the measurement time, which directly affects the repeatability of the measurement results. This estimation can be verified by the experimental results of the Coriolis mass flowmeters (master meters). Under the flow rate of 10mL/min, the experiment duration was set to 100s and 300s, and the repeatability of the measurement results was 0.01% and 0.00% respectively. They were very small and no significant difference. At the maximum flow rate of 150mL/min, the measurement duration was only 21s and the repeatability was 0.02%, which also met expectation.

3.4 Pipe capacity change
Based on the continuity equation, at the beginning and end of measurement, the change of liquid mass in the pipeline between the MUT and weighing system will affect the measurement results. During the experiment, three main factors that will cause the change of pipe capacity: (1) The temperature varied will cause the expansion (or contraction) of pipe and the density change of liquid. For capillary pipes, since the temperature of the liquid in the pipe cannot be measured directly, it can only be estimated by monitoring the temperature change of ambient and the water storage bag. According to the experimental results, the maximum variation is estimated to be less than 0.2℃ during the longest measurement duration of about 30min for minimum flow, a large relative uncertainty will be introduced. (2) For capillary pipelines, the position and state should be kept unchanged as far as possible in the experimental process. As a conservative estimate, the shape change is of 0.002% of the total volume of the pipe, which may introduce 0.19% relative standard uncertainty under the condition of the minimum flow rate (3mg weighing). (3) For the main pipe(stainless steel), the effect of deformation can be ignored. But due to a number of joints and valves, the air inside of pipes is not easy to remove entirely, that effect needs to be into account. If the pipe pressure changes at the test start and end, it causes the change of air volume. For the estimation of uncertainty, some assumed conditions has been presented such as the volume ratio between residual air and middle pipelines air less than 3% and the change of pressure less than 1% based on experiments and observations. This uncertainty could be the one of main sources of uncertainty under the condition of high flow rate. Table 7 shows the uncertainty budget introduced by pipe capacity changes at typical flow rates.

Table 7: Uncertainty introduced by changes in pipe capacity

Pipe System	Capillary Pipeline		Main Pipeline	
flow rate (μL/min)	0.1	100	1000	100000
pipes / liquid temperature change	0.41%	0.003%	0.036%	0.002%
pipeline deformation	0.19%	0.001%	0.000%	0.000%
residual air	0.01%	0.046%	0.017%	0.017%
relative uncertainty	0.45%	0.046%	0.040%	0.017%

3.5 Buoyancy correction
The uncertainty of liquid and air density should be considered in buoyancy correction. The calculation method of experimental liquid density is shown in Section 3.7, and air density is calculated from ambient temperature, pressure and humidity[15]. Since the sensitivity coefficients are both only about 1.2×10^{-3}, even if conservative estimation is made, these uncertainty can still be ignored (as shown in Table 4).

According to Formula (4), it can be seen that the buoyancy of the inserted tube also needs to be corrected, that uncertainty could be analyzed under the typical experimental configuration and flow rate. The specific budget results are shown in Table 8.

Table 8: The uncertainty introduced by insert tube buoyancy correction

Balance	Nominal Flow Rate (μL/min)	Beaker Number	Effective Area of Beaker (mm^2)	Needle Number	Cross Sectional Area of Needle (mm^2)	Diameter Error of Beaker	Cross Section Error of Needle	Introduced Uncertainty
MCM36	0.1	B-4#	314.15	N-1	1.5	5%	5%	0.013%
MSA225S	1000	B-2#	1924.42	18G	0.60	5%	5%	0.002%

3.6 Evaporation Effect

Based on the investigation of the existing facilities and the experimental comparative study of different evaporation suppression methods[11,16], paraffin oil is used cover the liquid surface at the weighing container during the test of low flow rate. Under the laboratory conditions, the maximum evaporation under the paraffin oil film cover is less than 0.3nL /min. Since it is difficult to make a correction model of evaporation, the maximum evaporation is used as one source of uncertainty. To simplify the experimental operation, the oil film cover are not in used for flow rate above 100μL/min, and the uncertainty can be estimated by maximum evaporation of water measured under laboratory conditions (as said in Section 2.2). The uncertainty introduced by evaporation at typical flow rates are shown in Table 9.

Table 9: Uncertainty introduced by evaporation

Flow Rate (μL/min)	Balance	Minimum Weighing Value (mg)	Minimum Measuring Time (s)	Oil Film	Uncertainty
0.1	MCM36	3	1800	Yes	0.12%
1	MCM36	10	600	Yes	0.01%
10	MCM36	50	300	Yes	0.00%
1000	MSA225S	2700	162	No	0.012%
10000	MSA225S	17000	102	No	0.001%
100000	MSA225S	60000	36	No	0.000%

3.7 Liquid density

The facility uses ultra pure water as the experimental medium, and its density is calculated by Tanaka Formula[12]. Its uncertainty mainly comes from the measurement of liquid temperature. For main pipe, the liquid temperature is measured by a platinum resistance thermometer with maximum permissible error of ±0.2 ℃ , so this uncertainty item can be ignored. Since liquid temperature cannot be measured directly for capillary pipes, the temperature of ambient and water storage bag are used for estimation. The maximum deviation is ±1 ℃ as a conservative estimation, and the relative standard uncertainty introduced by density measurement is 0.012%. In addition, the uncertainty introduced by the measurement of liquid pressure is negligible.

3.8 *Distribution of uncertainty under different flow rate conditions*

The flow measurement system could be composed of different subsystems on different flow rates, so the uncertainty is evaluated respectively. The curve of the relationship between the uncertainty of facility and the flow rate is shown in Figure 11. The uncertainty obviously decreases as the flow rate increases, and it becomes steady and reaches the level of 0.1%(k=2) when the flow rate is greater than 10μL/min.

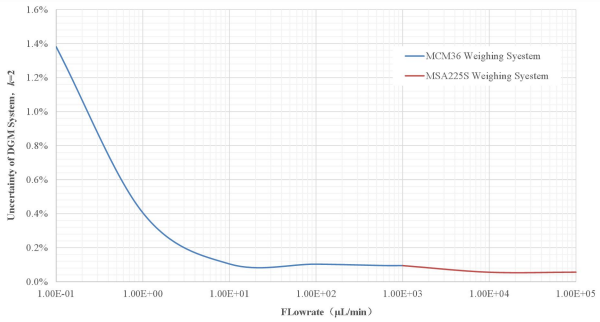
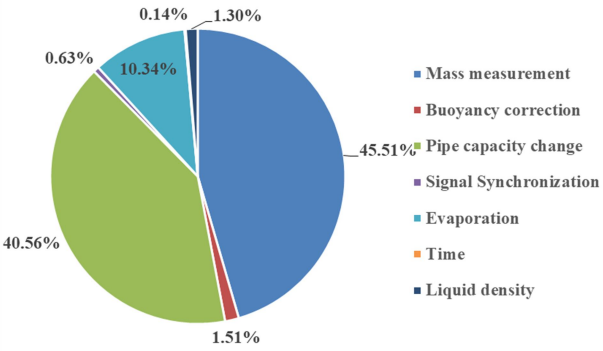
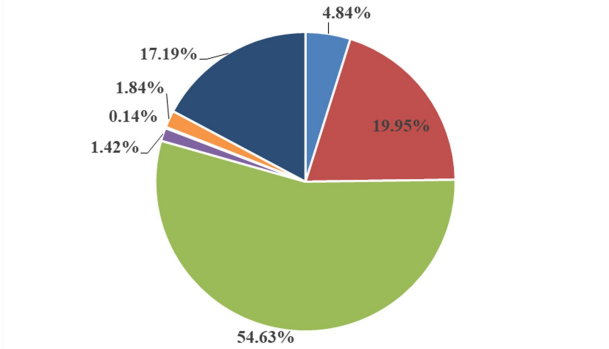


Figure 11: Schematic diagram of the relationship between uncertainty and flow rate

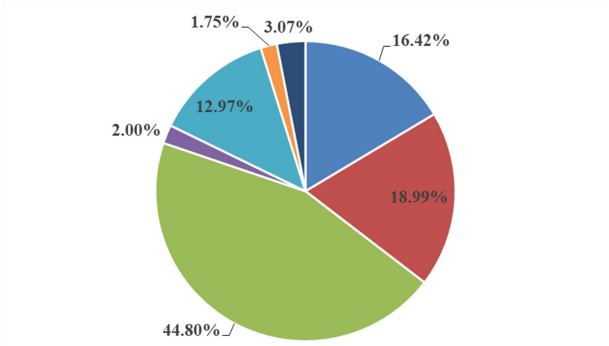
Under different typical flow rates, the contribution ratio of major uncertainty varies greatly, which is also a characteristics of micro flow facilities, as shown in Figure 12.



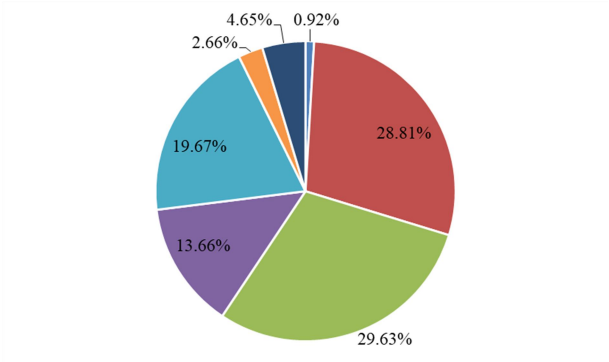
a) comparison of contribution rates of uncertainty components at 100nL/min



b) comparison of contribution rates of uncertainty components at 100μL/min



c) comparison of contribution rates of uncertainty components at 1mL/min



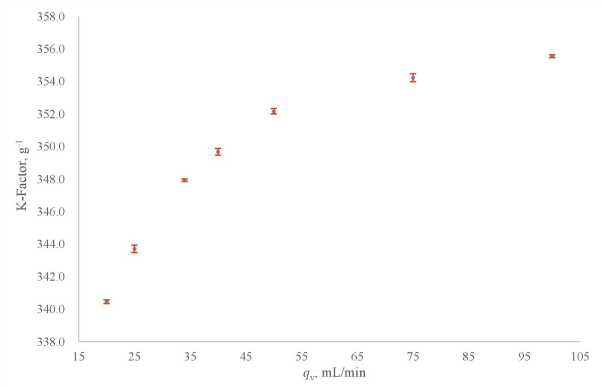
d) comparison of contribution rates of uncertainty components at 100mL/min

Figure 12: Schematic diagram comparing contribution rates of uncertainty components under different flow rates (the legend meanings in Figure b), c) and d) are the same as those in Figure a))

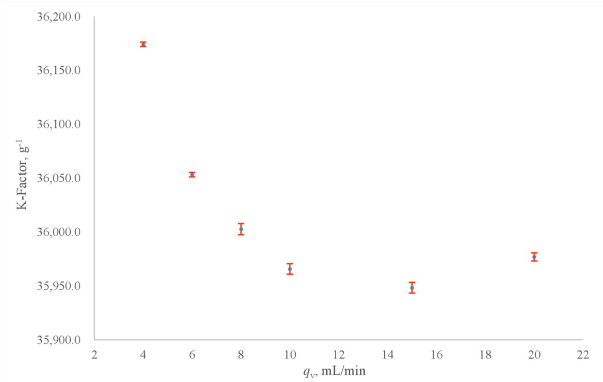
4. The experimental test

4.1 Test experiment of MAS225S balance weighing system

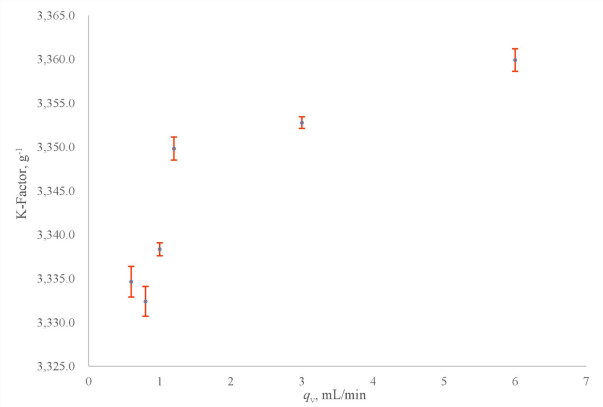
DGM was used to test three Coriolis mass flowmeters (master meters) respectively. The experimental results of MSA225S balance are shown in Figure 13, the blue circle represents the *K*-factor of the meter at different flow rates, and the length of red vertical line of each circle represents the measurement repeatability at each flow rate. The experimental results show that the repeatability of the measurement results is good, that is in the range of 0.01%~0.07%.



a) *K*-factor of LF4 master meter at different flow rates



b) *K*-factor of LF3 master meter at different flow rates



c) *K*-factor of LF2 master meter at different flow rates

Figure 13: *K*-factor of three master meters at different flow rates

4.2 Test experiment of MCM36 balance weighing system

The master meter of LF4M was tested by MCM36, the results were shown in Figure 14. The repeatability is also good and within 0.01%~0.06%. The syringe pump of this facility was measured at 100nL/min~100μL/min, and the repeatability was 0.09%~0.63%, as shown in Figure 15. In addition,

the repeatability gets worse obviously at low flow rate, but it is still in an acceptable range. The repeatability of all experiments can be partially verified the uncertainty budget of facility.

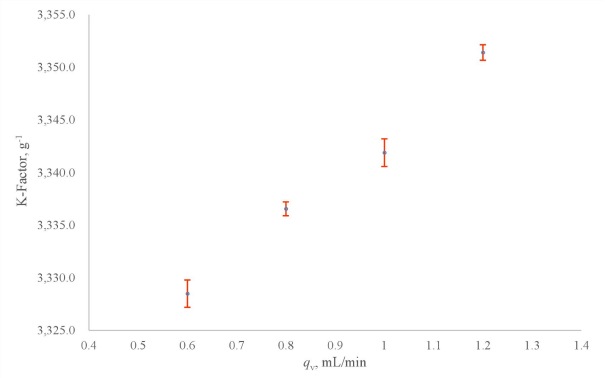


Figure 14: *K-factor of LF4 master meter under different flow rates*

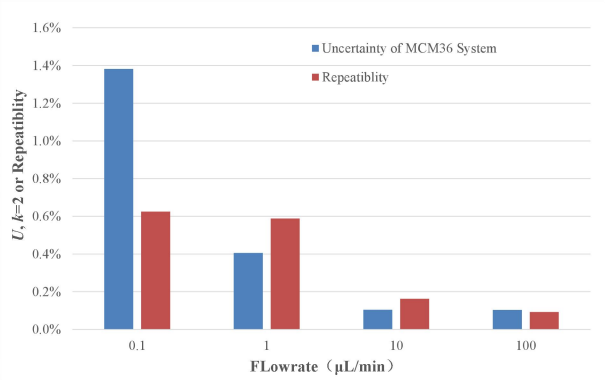


Figure 15: *Uncertainty of MCM36 weighing system and repeatability of measurement*

5. Summary and prospect

Based on DGM the liquid micro flow facility with a wide range of seven orders of magnitude was established. The accuracy of the facility was improved by the 'dual time' method and the optimized dynamic reading mode of balance. The uncertainty sources that include weighing, pipe capacity variation and evaporation and so on were analyzed by experiments. According to the difference of weighing systems and pipe systems, the uncertainty of different typical flow rates is evaluated respectively. The uncertainty of the facility can reach or better than 0.1% (k=2) at the flow rate from 10μL/min to 150mL/min, When the flow rate is less than 10μL/min, the uncertainty gradually get bigger with the decrease of flow rate, and the uncertainty is about 1.5% (k=2) under the condition of 100nL/min.

From the distribution of uncertainty main sources, the pipe system still has a large room for improvement, such as controlling the volume and temperature of the capillary, improving the air exhausting method of the main pipe, that can reduce the effect of the pipe capacity change in the experiment. In addition, the overall uncertainty needs to be verified by comparison between facilities in the future.

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