# Establishment of New Flow Standard Technology

Yasuhiro ISOBE

Higher accuracy and reliability for gas flow and liquid material supply are requested with the miniaturization and high integration of leading edge semiconductor device. To meet the requirement, at Kyoto Fukuchiyama technology center, we aim to establish flow standard technologies that are to develop flow standards, to standardize method of process gas flow measurement and to be accredited to ISO/IEC 17025 as a flow calibration authority. In this issue, we introduce our efforts for standardization of flow standard technology.

## Introduction

HORIBA STEC Co., Ltd. manufactures and sells fluid measurement and control devices mainly in the semiconductor manufacturing field. With the miniaturization and high-integration of semiconductor devices, the demands for the enhancement of accuracy and reliability of measurement and control technologies for gas and liquid materials are increasing. Kyoto Fukuchiyama Technology Center was completed (Figure 1) to be dedicated to the research and development to meet such demands in December 2013. Its major research and development elements are the development of flow measurement system traceable to national standards, reconstruction of in-house traceability system, flow measurement of semiconductor process materials, and vaporization test using liquid material vaporizing devices.

In order to improve the accuracy and reliability of fluid measurement and control devices, it is important to compile associated data with high accuracy and reliability as well as to improve the performance of such devices.


Figure 1 Kyoto Fukuchiyama Technology Center

Kyoto Fukuchiyama Technology Center is equipped with up-to-date facilities for fluid measurement and vaporization tests of semiconductor materials, enabling tests with high accuracy and reliability. In addition, for higher reliability, it is preparing to obtain ISO/IEC 17025 accreditation about the calibration of gas flow rates.

## Efforts toward the obtainment of ISO/IEC 17025 accreditation

ISO/IEC 17025 is a standard concerning the general requirements for the competence of testing and calibration laboratories. This standard stipulates the technical requirements for laboratories that perform analysis and measurement and calibration, as well as the requirements for management system as represented by ISO 9001, based on which accredited body determines whether such laboratories have the ability to perform analysis, measurement, and calibration. The laboratories that obtain this accreditation can place a accreditation mark on the Certificates of Analysis and Certificates of Calibration to enhance the reliability of test and calibration results, which are internationally accepted.

HORIBA STEC, Co., Ltd. not only develops flow measurement standards traceable to national measurement institutes such as the National Institute of Standards and Technology (NIST, USA) and the National Metrology Institute of Japan, but also conducts management systems required for accreditation. It is scheduled to obtain accreditation with the international mutual recognition agreement in 2017 by National Voluntary Laboratory Accreditation Program (NVLAP) that NIST administers.

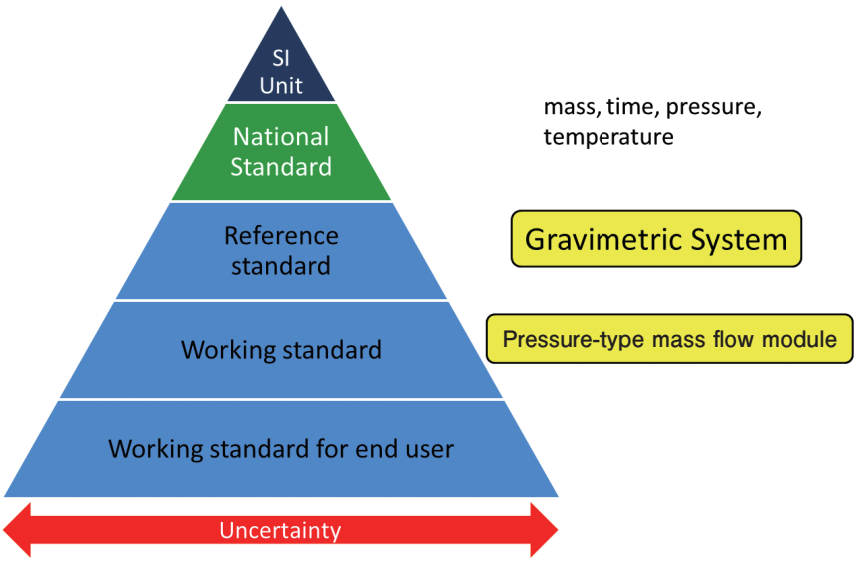


Figure 2 Target flow rate traceability system

Figure 2 shows the outline of the targeted traceability system. Using a in-house weighing flowmeter (gravimetric system) traceable to national measurement institutes as a reference standard, and flow meter based on the technology of the pressure-type mass flow module D500[1] as a working standard, we construct in-house traceability systems so that it can be applied to new products in the future. We are trying to make it possible that the Accreditation of Calibration in which a accreditation mark is placed can be issued at the time of the calibration using a reference standard and working standard.

## In-house development of flow standard

HORIBA STEC, Co., Ltd. is promoting the in-house development of the reference and working standards mentioned above to reconstruct its flow traceability system as well as to enhance its reliability.

### Reference standard (Gravimetric system)

Figure 3 shows the outline of the framework of our gravimetric system under development. The mass flow rate is determined by filling a measurement container with gas and then letting the gas out through a flowmeter to measure the decrease in the mass of the measurement container per time using an electronic weighing machine and GPS Timer. Table 1 shows the major specifications. The calibrated flow range is between 1 sccm[*1] to 50 slm[*1], and the targeted expanded uncertainty is 0.1% or lower.

When calibrating a flowmeter using the gravimetric system, a flow rate is measured using the Gravimetric system and a comparison with the flow output of the calibrated flowmeter is also conducted. The basic technology of the gravimetric system was developed at the Reno office of Horiba Instruments Incorporated and was confirmed to satisfy the targeted specifications as a result of the in-house evaluation test. This technology was introduced to our Kyoto Fukuchiyama Technology Center to obtain the various data to complete the reference standard.

To prove the reliability of the results of calibration performed by a calibration laboratory, a proficiency test in which the calibration result obtained by using the gravimetric system and the one obtained by using the flow standard of NIST are compared by using the after-mentioned working standard is conducted[2]. The proficiency

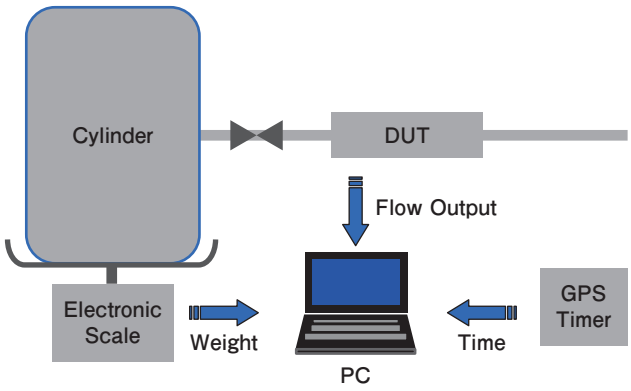


Figure 3 Gravimetric system

Table 1 Target specification for Gravimetric system

| Specification | Description |
|---|---|
| Expanded Uncertainty | 0.1% of set point |
| Flow Range | 0.0012498~62.492 g/min ($N_2$ : 1 sccm~50 slm) |
| Applicable Gases | $N_2$, Ar, $O_2$, $CF_4$, $SF_6$ |

test is scheduled to be conducted after obtaining various data in house.

*1: sccm, slm: Abbreviations for "Standard Cubic Centimeter per Minute" and "Standard Liter per Minute," which are the units representing the gas flow rate (mL/min, L/min) in the standard state (0°C, 101.3 kPa)

### Working standard

We are developing a flowmeter based on the technology of the pressure-type Mass Flow Module D500.[1] Figure 4 shows the structure of the D500. The pressures on the upstream and downstream sides, as well as the gas temperature of a flow restrictor (referring to laminar element restrictor, hereinafter referred to as "restrictor"), are measured and converted into mass flow rate in the internal arithmetic circuit.

A working standard is composed of a restrictor and temperature sensor enclosure and a pressure sensor enclosure based on this technology. Using high-precision sensors for achieving high reproducibility makes it possible to utilize it as a working standard.

This working standard is used in the proficiency test conducted with NIST and calibrating the flowmeters for internal production. Currently, an adjustment and a performance check are performed for use in proficiency test.

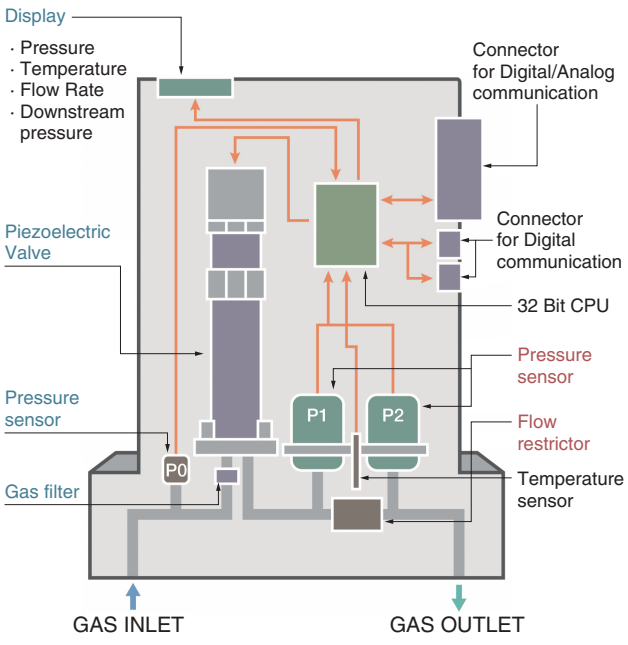


Figure 4 Mass flow module D500 structure

## Efforts toward the standardization of process gas flow rate

The miniaturization of devices and introduction of new materials are promoted for increasing the capacity and enhancing the performance of semiconductor devices. Because of this situation, advanced semiconductor process technologies are required. Also in terms of material supply, the enhancement of precision, speed, and reliability of process gas flow for flow rate measurement and control devices is required.

The flow rate traceability system has already been constructed for inert gases such as $N_2$ and Air. On the other hand, practical traceability systems have not yet been constructed for the flow rate of process gas with corrosive, toxic, and reactive properties.

Mass Flow Controller manufacturers have their own process gas flow rate standards and have the data on the process gas flow rate of the mass flow controllers, which are their products. However, because practical traceability systems for process gas flow rates have not yet been constructed, the process gas flow rate may differ among manufacturers. Therefore, when users change the mass flow controller manufacturer, there are cases where they need to spend efforts to determine the process conditions again. In addition, the reliability of process gas flow data obtained by manufacturers may be unclear. As a result, the need for standardization of the process gas flow rate by mass flow controller manufacturers increased, and the SEMI Standard Technical Committee is currently making efforts to standardize flow rate measuring methods in cooperation with the National Institute of Advanced Industrial Science and Technology.

It is planned to investigate the difference between a national standard and standards of various mass flow controllers manufacturers using a flow meter calibrated at the National Institute of Advanced Industrial Science and Technology, for inert gases first. HORIBA STEC, Co., Ltd. will participate in this investigation using its Rate Of Rise (ROR) System for measuring the flow rate of process gas.[3]

## Conclusion

We introduced our efforts toward the standardization of gas flow rate at HORIBA STEC's Kyoto Fukuchiyama Technology Center and the obtainment of ISO/IEC 17025 accreditation.

HORIBA STEC was formerly named "Standard Technology" at the time of its foundation. This name

contains the belief of establishing concentration standard technologies for gas analysis to contribute to the development of the industry. As a gas flow rate calibration laboratory, obtaining ISO/IEC 17025 accreditation means the realization of the belief at the founding.

As stated in the paragraph on the efforts toward the standardization of process gas flow rate, the SEMI Standard Committee of SEMI JAPAN is making efforts to standardize process gas flow rate under the cooperation of the National Institute of Advanced Industrial Science and Technology. HORIBA STEC, Co., Ltd. will play a central role not only in applying the flow rate standard technology introduced this time to liquid flow standards but also in establishing process gas and liquid flow rate standard technologies, and applying these technologies to the products we develop to contribute to the development of the industry.

## References

[1] K.SHAKUDO, Readout, 43, 53 (2014)
[2] N.ITO, R.MATSUOKA, Readout, 42, 97 (2014)
[3] T.SHIMIZU, Y.ISOBE, M.TERASAKA, Readout, 45, 15 (2015)

Yasuhiro ISOBE
Fukuchiyama Technology Center
HORIBA STEC, Co., Ltd.