

DEVELOPMENT OF THE PVTt SYSTEM FOR VERY LOW GAS FLOW RATES

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Abstract

The new PVTt system as the standard for gas flow rate less than 5 mg/min has been constructed. This system has three unique aspects for a calibration of gas flow meters. In a PVTt system, the pressure downstream of a device under test (DUT) increases after a flow is diverted to a constant volume tank (CVT) side. It results in a change of a differential pressure working on DUT and a flow rate through DUT changes. Therefore, a flow meter which does not have a function to produce a constant flow rate can not be calibrated by a PVTt system. In this system, by introducing the automatic pressure controller (APC) to control a pressure downstream of DUT and to keep the differential pressure constant, any type of gas flow meters can be calibrated by the PVTt system. Also, as the initial condition in the dead volume uses as a trigger to stop a measurement, the initial and the final conditions in the dead are very close to each other so that the mass correction in the dead volume is not serious and does not need in some cases. And the third aspect is that the mass flow rate through DUT can be directly calculated from the changing rate of the pressure in the CVT at every moment. The CVT that copper wool is stuffed has a characteristics like an adiabatic tank so that the temperature change in the CVT is very small, about 0.02 K/h during a measurement. The relative standard uncertainty of the PVTt system with these new aspects is 0.1 - 0.05 percent on mass flow rates between 0.1 mg/min and 5mg/min for present.

Introduction

The lower the concentration of air pollutant substances is, the lower the concentration of a standard gas required to calibrate a gas analyzer is. The concentration of the calibration standard gas required is considered to be from a few ppm to several tens of ppb in the near future. However, the concentration of a standard gas traceable to the national standard is about several hundreds of ppm at the present time by several reasons, cost, steadiness and so on. A calibration gas with a desirable low concentration required on the calibration of a gas analyzer is prepared from the standard gas with high concentration using a dilution system, which is a device that dilutes a standard gas with high concentration to a gas with desirable low concentration and consists of flow meters.

In Japan, the standard for gas flow rates more than 5 mg/min is already supplied to accreditation laboratories[1], but there has been no standard for lower flow rates than 5 mg/min needed in a dilution system. Resultantly, a concentration of a calibration gas prepared by a dilution system is unreliable, even though it is prepared from a standard gas traceable to the national standard. Therefore, it is required to establish the standard for gas flow rate lower than 5mg/min as well as to develop a reliable flow meter applicable in this flow rate range. NMIJ reported about the calibration

facility based on the dynamic gravimetric method at the 5th IFSS, April 2002[2]. This can provide the standards for gas flow rates less than 1 mg/min. The PVTt system reported here has been developed to cover a flow rate range between 5 mg/min and 0.1mg/min.

The PVTt system for very low gas flow rates

Constant volume tank and vacuum chamber

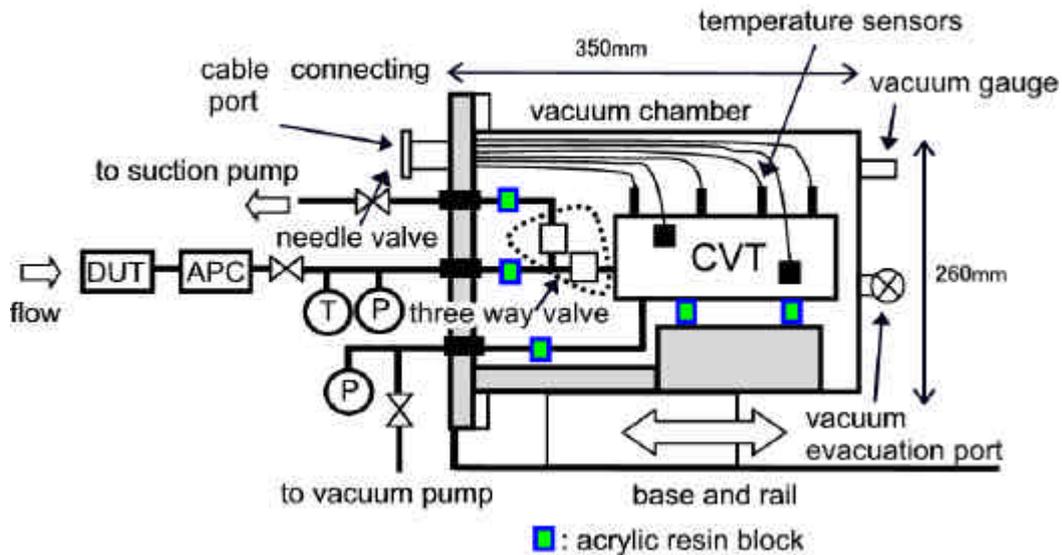


Figure 1 A schematic diagram of the CVT and the vacuum chamber

Figure 1 shows a schematic diagram of the PVTt system. Figure 2 is a picture of the constant volume tank (CVT) where the vacuum chamber is moved backward to see the CVT. Figure 3 is a picture showing the front plate side of the vacuum chamber. The CVT has a cylindrical shape with the volume of about 500ml, in which copper wool is stuffed to uniform temperature distribution in the tank. Four temperature sensors are inserted in the tank and two temperature sensors are putted up outside the tank wall using aluminum tape. It is desirable that the thermal condition of the CVT is not influenced by the temperature change in the circumstance. Therefore, the CVT is set in the vacuum chamber, which is evacuated less than 1 Pa and furthermore the CVT is isolated from the chamber using acrylic resin blocks as shown in Fig.1 to avoid influence by thermal conduction.

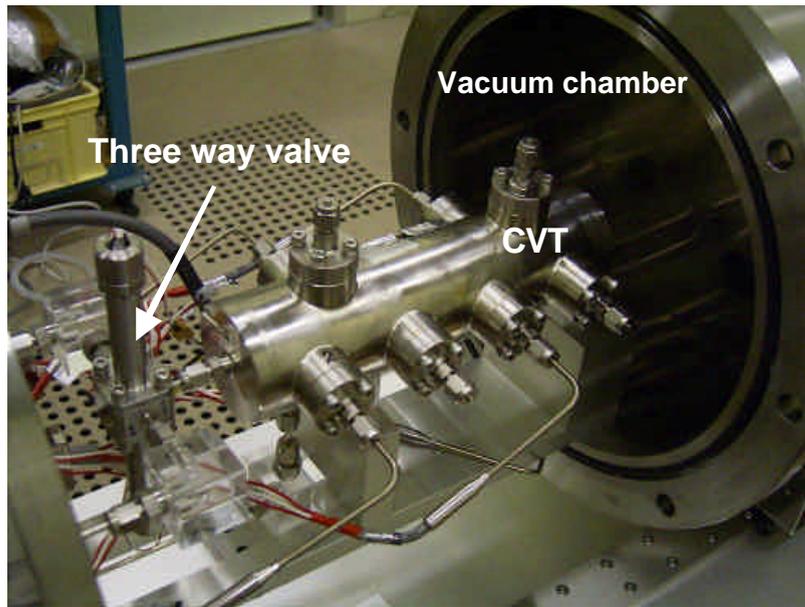


Figure 2 The CVT, the three way valve and the Vacuum chamber

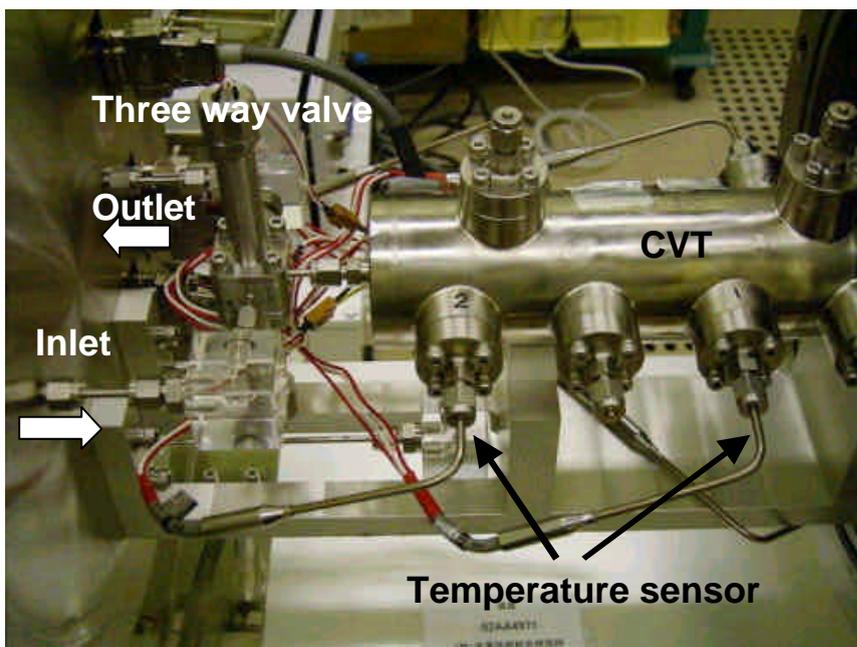


Figure 3 The CVT and the front plate part in the vacuum chamber

The tank, tubes (1/8 inch stainless tube) and cables are fixed to the front plate of the chamber. The pressure gauge to measure the pressure in the CVT is set outside the vacuum chamber. The three way valve, which diverts a flow from the suction pump side to the CVT side and vice versa, consists of two piezo valves. These piezo valves that do not generate any heat is set in the vacuum chamber. All

stainless tubes attached to the chamber wall are cut off in the chamber and the tubes separated are again connected each other through acrylic acid resin blocks as shown in Fig. 1

The leakage of the vacuum chamber is negligible small. The leakage of the CVT and the dead volume are 1 Pa/h and 3 Pa/h respectively. These values are considered to be negligible small on usual collection time, a few hours. However, the CVT and the dead volume will be improved to reduce their leakage for measurements requiring long collection time over one day.

Determination of the collection time

As mentioned above, the three way valve consists of two piezo valves and is specially designed for this system. Three piezo devices are combined in one valve to get enough stroke at the valve opening. The time required to open and to close the valve, t_v , is 0.5 -1 ms. The delay time, t_{int} , the period between the time that the first valve is closed and the time that the second valve is open, can be adjusted in steps 1 ms and it is set to be 2 ± 1 ms to prevent the situation that both valves are open at the same time.

The sequence of the valve operation is shown in Fig.4. Here, the three way valve is used to start and to stop a measurement. As shown in Fig.4, before a measurement, V5 is opened and V4 is closed. The measurement is started by closing V5 and opening V4, and is stopped by closing V4 and opening V5. The measurement of the collection time starts when V4 begins to open and stops when V4 is closed completely. The time related to the valve operation is " $t_{int} + t_v + t_v$ " and is 5 ms at maximum. As the collection time in this system is normally over 20min, it is quite longer compared with the time needed for the valve operation. So that it is not necessary to correct the valve operation time to the collection time, t_c .

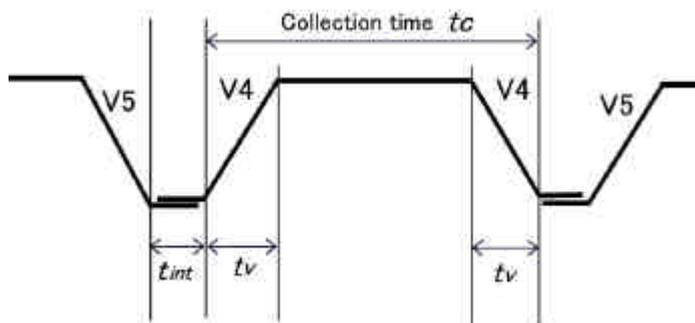


Figure 4 A sequence of the valve operation

Pressure and temperature measurements

Two resonance type absolute pressure gauges with the resolution of 0.01 Pa are used to measure pressures in the CVT and in the dead volume. Their full scale is 250 kPa and 700 kPa respectively. They were calibrated over the range from 30 to 300 kPa by the piston gauge. The deviation between the piston gauge and the NMIJ pressure standard was within ± 10 Pa in this pressure range. The uncertainty of the piston gauge is less than ± 5 Pa, depending on a pressure. And the standard

deviation of the fitted curve of the pressure gauge determined by the piston gauge was within ± 5 Pa in both gauges. Therefore, the standard uncertainty of pressure measurement is less than 12 Pa. The four wire Pt 1000 temperature sensors are used to measure temperature in the CVT and in the dead volume. Two of those temperature sensors are located at about 20mm from the wall of the CVT and other two are located around the axis of the CVT. Furthermore, two of them are put on the wall of the CVT to monitor the wall temperature by using aluminum tape. These sensors are calibrated over the range from 15 to 35 degrees C by the standard temperature calibration facility, which was also calibrated by the JCSS accreditation laboratory. The standard uncertainty of this calibration facility in this temperature range is within 10 mK. The standard deviation of the fitting curve determined was less than 0.05 K so that the standard uncertainty on the temperature measurement was within 0.05 K.

Figure 5 shows the temperature behavior in the CVT when the vacuum chamber is evacuated and the evacuation is finished. Here, the CVT was already evacuated until around 10 Pa. It is found from this figure that the temperature in the CVT quickly goes down by the evacuation of the vacuum chamber and gradually increase after stopping the evacuation. The temperature in the CVT is seemed to be in steady state after about 16 hours. The reason that the temperature slightly fluctuates after that is not clear, but may be influenced from the circumstances by an imperfect evacuation of the vacuum chamber

Figure 6 shows the behaviors of the temperature in the CVT when a gas flows into the CVT. The flow rate is about 1 mg/min and the collection time is about 2 hours. In this figure, the first broken line indicates the diversion of a flow to the CVT side and the second broken line indicates the diversion of a flow to the suction pump side. The temperature slightly changes by the diversion of a flow to the CVT side, but the influence of the second diversion is not clearly appeared on the temperature. It should be noticed that the temperature of four sensors coincide within 0.05K during the gas collection from the start and the temperature change during the collection time is only 0.04 K. The temperature in the CVT continues to decrease at almost the same rate even after the flow is diverted to the suction pump side. The reason of this temperature decrease is not an expansion of flow, but a flowing of low temperature gas into the CVT, which is about 0.15 K lower than that in the CVT. This means that the CVT is not thermally equilibrium and is on transition. On the other hand, as four temperature sensors show the same value, it is reasonable to estimate that there is no serious temperature distribution in the CVT. In other words, temperature in the CVT is not stable, but uniform and a mass flow rate can be calculated from this temperature and the pressure at the same moment. (method 2) Of course, it must be waited for long time to determine the final condition until the temperature is stable in the CVT. (method 1)

The pressure behaviors in the CVT and in the dead volume at the measurement in Fig.6 are shown in Fig. 7. The pressure in the dead volume decreases gradually, not constant initially. After the flow is diverted, its pressure quickly goes down to the value in the CVT and the pressure in the CVT and the dead volume increase at the same rate. After the flow is diverted to the suction pump side, the pressure in the CVT is constant, but the pressure in the dead volume decreases at the same rate as that on the initial condition.

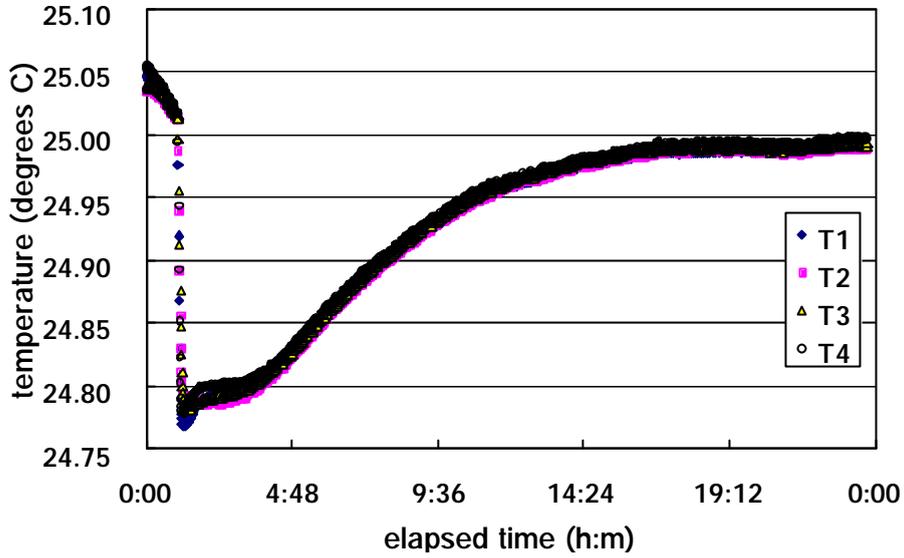


Figure 5 The temperature behaviors in the CVT evacuated when the vacuum chamber is evacuated.

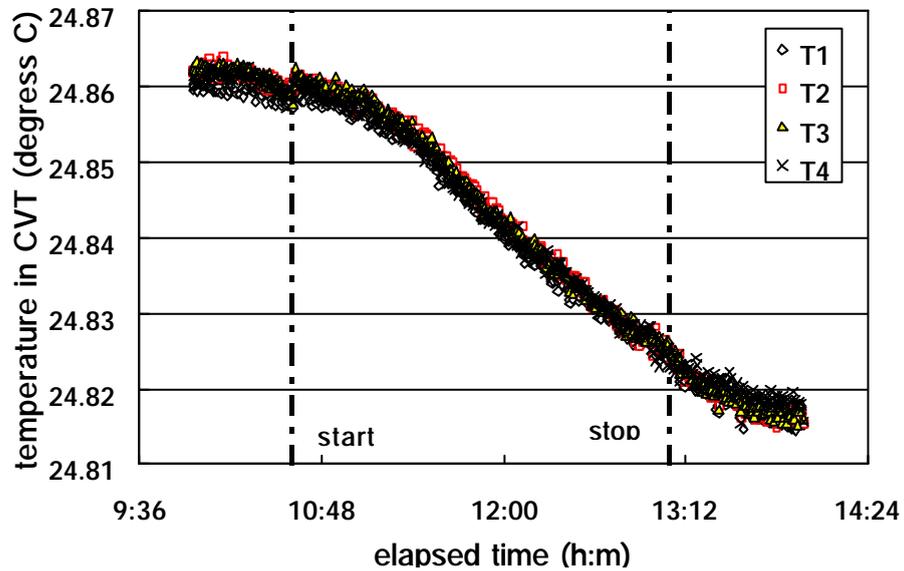


Figure 6 The temperature behaviors in the CVT when flowing a gas in it.
(The flow rate is about 1 mg/min.)

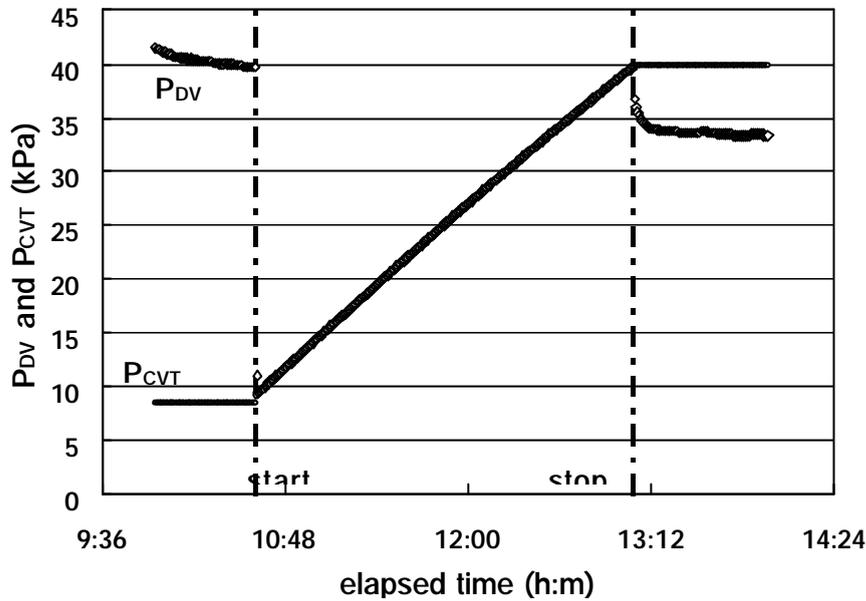


Figure 7 The pressure behaviors in the CVT and the dead volume when flowing a gas in the CVT. (The flow rate is about 1mg/min.)

Automatic pressure controller

When calibrating DUT by a PVTt system, even if a upstream pressure of DUT is kept constant, a downstream pressure of DUT increases with increasing the pressure in the CVT. This means that the output of DUT, except sonic nozzle, changes by the reason that the differential pressure working on DUT changes. In this system, the automatic pressure controller (APC) is installed at the downstream side of DUT to keep its downstream pressure constant. This APC can keep the downstream pressure within 10 Pa of the setting value and the fluctuation of the downstream pressure when diverting a flow to the CVT side is also within 10 Pa. The pressure in the dead volume on measurement can be adjusted at a desirable pressure between 30 kPa and 100 kPa by a needle valve and a suction pump. Here, the dead volume is the section between a DUT and the three way valve. (shown in Fig.8)

Determination of the volume of the CVT and the dead volume

The volumes of the CVT and the dead volume are determined as follows; the mass of the measuring tank (about 10 cm³) pressurized up to 500 kPa with nitrogen gas is measured by the precise comparator with the resolution of 10⁻⁶ g, and then it is connected to the PVTt system where both valves of the three way valve, V4 and V5 in Fig.8, are closed and the dead volume is evacuated less than 1 Pa. The area enclosed by the dotted lone in Fig.8 is called the dead volume. The pressure and the temperature in the dead volume is measured and recorded as the initial condition. The valve of the

measuring tank and V3 are opened to flow the gas from it into the dead volume. After the temperature and the pressure in the dead volume are stable, the pressure and the temperature are measured and recorded as the final condition. And thus V3 and the valve of the measuring tank are closed, and the measuring tank is separated from the system to measure its mass by the precise comparator. The volume in the dead volume, V_{DV} can be calculated from the mass change of the measuring tank, M_{DV} and the initial and the final conditions in the dead volume. V_0 in Eq.(1) , which is shown in Fig.8, was calculated from the pipe size. As this section is a 1/16 inch stainless tube in the length of 50mm, its volume, V_0 , is about 0.4 cm^3 . P_{iDV} in Eq(1) is equal to an atmospheric pressure.

$$V_{DV} (P_{fDV} / T_{fDV} - P_{iDV} / T_{iDV}) = \mathfrak{R} / N_g M_{DV} - V_0 (P_{fDV} / T_{fDV} - P_{iDV} / T_{iDV}) \quad (1)$$

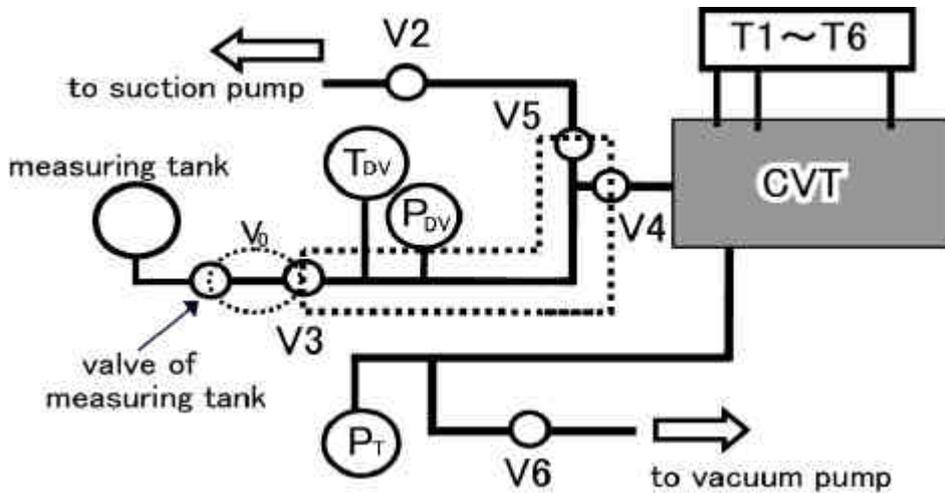


Figure 8 A schematic diagram of the dead volume and the valve positions

The volume of the CVT, V , is determined by the same way. However, as the mass of gas in the measuring tank is too small for the CVT, this operation was repeated over five times to obtain enough pressure rise in the CVT, which is important to determine the volume with low uncertainty. The loss of the mass of the measuring tank is M_k , the final pressure and temperature in the CVT are P_{fk} , T_{fk} respectively. Here, a subscript "k" refers to the k th measurement. The atmospheric pressure is P_a . V_t is the sum of the volume of the CVT and the dead volume and $V_t = (V + V_{DV})$,

$$V_t (P_{fk} / T_{fk} - P_{ik} / T_{ik}) = \mathfrak{R} / N_g M_k - V_0 (P_{fk} / T_{fk} - P_a / T_{ik}) \quad (2)$$

And, after n times are repeated,

$$V_t (P_{fn} / T_{fn} - P_{il} / T_{il}) = \mathfrak{R} / N_g \sum_{k=1}^n M_k + V_0 \sum_{k=1}^n (P_{ak} / T_{ik} - P_{fk} / T_{fk}) \quad (3)$$

The volume of the CVT, V , was calculated from V_t and V_{DV} . The measurements of the volume of the CVT and the dead volume were repeated at least five times and their averaged values were used as the values of V and V_{DV} . As $V=V_t-V_{DV}$, the uncertainty of V includes the uncertainty of V_t and V_{DV} .

Table 1 The volumes of the CVT and the dead volume, and their standard deviation

	Volume (cm ³)	Standard deviation (cm ³)
CVT	457.575	0.10
Dead volume	26.379	0.05

Outline of a calibration of DUT

Before a calibration is performed, the vacuum chamber is evacuated less than 1 Pa and is left for one night as the CVT and the chamber are thermally on equilibrium. The upstream and downstream pressures of DUT are determined as a desirable flow rate can be obtained, and a flow through DUT is produced by the suction pump and the needle valve. After the flow is stable, the pressure and the temperature in the CVT and the dead volume are recorded in the computer, The flow is diverted from the suction pump side to the CVT side and the measurement of the collection time starts. The flow rate is calculated from the pressure and the temperature change in the CVT every moment. When the pressure in the dead volume reaches to the initial pressure recorded there, the flow is diverted from the CVT side to the suction pump side and the measurement of the collection time ends. After the temperature indicated by four sensors in the CVT becomes stable, the pressure and the temperature are recorded as the final condition.

Calculation of the mass flow rate, Q_m

In this PVTt system, two methods can be used to calculate the mass flow rate Q_m through DUT. One is a normal method; calculating the mass flow rate from the final and the initial conditions in the CVT using the state of equation of gas. The other one is to calculate the mass flow rate from the pressure and the temperature changes in the CVT and in the dead volume during the measurement.

(1) the method 1

The mass of gas flowing into the CVT, M , is calculated from the final and the initial conditions and the following equation.

$$M = VN_g / \mathfrak{R} (P_f / (Z_f T_f) - P_i / (Z_i T_i)) \quad (3)$$

where N_g is a molecular weight of gas, V is the volume of the CVT, \mathfrak{R} is the universal gas constant. Z is a compressibility factor. P and T are a pressure and a temperature in the CVT. A suffix "f" and "i"

refer to the final and the initial condition in the CVT respectively. The mass of gas remained in the dead volume, M_{DV} , is calculated by the same way. That is,

$$M_{DV} = V_{DV} N_g / \Re (P_{DVf} / (Z_{DVf} T_{DVf}) - P_{DVi} / (Z_{DVi} T_{DVi})) \quad (4)$$

where a suffix "DV" refers to the dead volume condition. In this system, P_{DVf} is very close to P_{DVi} . The difference between them is within a few tens Pa, although it depends on a flow rate. The temperature in the dead volume is fluctuated by the room temperature and its fluctuation width is within 0.3 K at maximum during the measurement. Resultantly, M_{DV} is quite small and the contribution of M_{DV} to the final mass is negligible. The order of M_{DV} will be evaluated in later. The total mass of gas through DUT, M_t is

$$M_t = M + M_{DV} \approx M \quad (5)$$

and the mass flow rate, Q_m , is calculated by the following equation.

$$Q_m = M_t / t_c = (M + M_{DV}) / t_c \approx M / t_c \quad (6)$$

(2) the method 2

From the state of equation of gas,

$$r = PN_g / (Z\Re T) \quad (7)$$

The mass of gas in a volume V , M , is given as follows;

$$\begin{aligned} PV &= \Re ZT / N_g \times (rV) \\ &= \Re ZT / N_g \times M \end{aligned} \quad (8)$$

By differentiating Eq.(8) with respect to the measurement time t ,

$$VdP / dt = \Re / N_g (ZTdM / dt + TMdZ / dt + MZdT / dt)$$

Here, $Q_m = dM/dt$ and after the simple calculation,

$$Q_m = VP N_g / ZT \Re \times (1/P \times dP / dt - 1/Z \times dZ / dt - 1/T \times dT / dt) \quad (9)$$

If the temperature change in the CVT and in the dead volume is very small, the second and the third terms of Eq.(9) are negligible small compared with the first term. For example, when $Q_m = 1$ mg/min, $T = 300$ K, and $P_{final} = 50$ kPa, dT/dt is about 1.7×10^{-4} K/min and dP/dt is about 0.25 kPa/min. dZ/dt is about 10^{-8} . Therefore, the first term is $O(10^{-2})$, and on the other hand, the second term and

the third term are $O(10^{-6})$ and $O(10^{-8})$ respectively. Therefore, the second and the third terms are negligible small compared with the first term in this case.

Therefore, the mass flow rate Q_m can be calculated from only the pressure change at every moment. As the mass change in the dead volume is calculated by the same way,

$$Q_m = Q_{mCVT} + Q_{mDV} \quad (11)$$

where

$$Q_{mCVT} = V N_g / (Z T \mathfrak{R}) \times dP_{CVT} / dt$$

and

$$Q_{mDV} = V_{DV} N_g / (Z T \mathfrak{R}) \times dP_{DV} / dt$$

The magnitude of the second and the third terms of Eq.(9) are evaluated in the next section.

Uncertainty analysis of the mass flow rate

When calculating the mass flow rate using method 1, Eqs.(3), (4) and (6) are used. From Eq.(6),

$$(dQ_m)^2 = (dM / t)^2 + (dM_{DV} / t)^2 + [(M + M_{DV}) / t^2 dt]^2 \quad (12)$$

and from Eqs.(3) and (4),

$$\begin{aligned} (dM)^2 = & (N_g / \mathfrak{R})^2 \{ [(P_f / (Z_f T_f))^2 + (P_i / (Z_i T_i))^2] \times dV^2 \\ & + \{ V^2 (1 / (Z_f T_f)^2 + 1 / (Z_i T_i)^2) \} \times dP^2 \\ & + \{ V^2 ((P_f / (T_f Z_f^2))^2 + (P_i / (T_i Z_i^2))^2) \} \times dZ^2 \\ & + \{ V^2 ((P_f / (Z_f T_f^2))^2 + (P_i / (Z_i T_i^2))^2) \} \times dT^2 \}^2 \end{aligned} \quad (13)$$

and

$$\begin{aligned} (dM_{DV})^2 = & (N_g / \mathfrak{R})^2 \{ [(P_f / (Z_{DVf} T_{DVf}))^2 + (P_{DVi} / (Z_{DVi} T_{DVi}))^2] \times dV_{DV}^2 \\ & + \{ V_{DV}^2 (1 / (Z_{DVf} T_{DVf})^2 + 1 / (Z_{DVi} T_{DVi})^2) \} \times dP^2 \\ & + \{ V_{DV}^2 ((P_f / (T_{DVf} Z_{DVf}^2))^2 + (P_{DVi} / (T_{DVi} Z_{DVi}^2))^2) \} \times dZ^2 \\ & + \{ V_{DV}^2 ((P_f / (Z_{DVf} T_{DVf}^2))^2 + (P_{DVi} / (Z_{DVi} T_{DVi}^2))^2) \} \times dT^2 \}^2 \end{aligned} \quad (14)$$

Here, the compressibility factor, Z, is given as follows;

$$Z = 1 + PB(T)/(\mathfrak{R}T) \quad (15)$$

where $B(T)$ is the Virial coefficient and is given by the following equation from the table of the reference [5] considering the temperature range of the present measurement condition.

$$B(T) = B(280K) + (B(320K) - B(280K))/(320 - 280) \times (T - 280) \quad (16)$$

and

$$B(320K) = -1.2 \times 10^{-6} \text{ (m}^3/\text{mol)}$$

$$B(280K) = -8.9 \times 10^{-6} \text{ (m}^3/\text{mol)}$$

$$(dZ)^2 = \{B/(\mathfrak{R}T)\}^2 \times dP^2 + \{P/(\mathfrak{R}T)\}^2 \times dB^2 + \{PB/(\mathfrak{R}T^2)\}^2 \times dT^2 \quad (17)$$

The sources of the uncertainty and their magnitudes are listed in Table 2. The dominant term in the uncertainty of Q_m , when the method 1 is used, is the first term of Eq.(12). And the dominant terms of Eqs.(13) and (14), from which the first term of Eq.(12) is calculated, are the second term. If the pressure measurement instrument and the size of the CVT is given, dM does not change so much as a mass flow rate. Therefore, only the way to reduce the uncertainty of Q_m is to make the mass of gas flowing into the CVT, M , large. In this case, longer collection time is needed. The longer collection time is not an essential problem itself, but the collection time of one day or two days is not seemed to be realistic. This is a peculiar problem on low gas flow measurement. From the results in Table 2, the relative standard uncertainty is 0.05 – 0.1 percent on the flow rate range from 5 mg/min to 0.1 mg/min when Q_m is determined by the method 1.

The uncertainty of Q_m obtained by the method 2 is calculated from Eq.(9). That is,

$$(dQ_{mCVT})^2 = (N_g/\mathfrak{R})^2 [(1/(ZT) \times dP_{CVT}/dt)^2 \times dV^2 + (V/(ZT^2) \times dP_{CVT}/dt)^2 \times dT^2 + (V/(Z^2T) \times dP_{CVT}/dt)^2 \times dZ^2 + (V/(ZT))^2 \times d(dP_{CVT}/dt)^2] \quad (18)$$

and

$$(dQ_{mDV})^2 = (N_g/\mathfrak{R})^2 [(1/(ZT) \times dP_{DV}/dt)^2 \times dV_{DV}^2 + (V_{DV}/(ZT^2) \times dP_{DV}/dt)^2 \times dT^2 + (V_{DV}/(Z^2T) \times dP_{DV}/dt)^2 \times dZ^2 + (V_{DV}/(ZT))^2 \times d(dP_{DV}/dt)^2] \quad (19)$$

The magnitude of the uncertainty sources and the results are listed in Table 3. The uncertainty of the pressure gradient in the CVT and in the dead volume was estimated from the pressure measurement uncertainty and the sampling interval of the data, less than 250 ms. All terms in

Eqs.(18) and (19) are almost the same order and again the change of $d Q_m$ is quite small compared with that of Q_m . Therefore, the relative uncertainty of Q_m becomes larger rapidly as Q_m is lower. The relative standard uncertainty is 0.03 - 0.5 percent on the flow rate range from 5 mg/min to 0.1 mg/min on the method 2.

Table 2 Sources of uncertainty and the magnitude of relative uncertainty (method 1)

Source		Uncertainty
Mass in the CVT	M	0.1 - 0.4 g
	$d M$	1.5×10^{-4} g
Mass in the dead volume	M_{DV}	$10^{-8} - 3 \times 10^{-5}$ g
	$d M_{DV}$	10^{-5} g
Pressure	P	30 – 120 kPa
	$d P$	12 Pa
Temperature	T	25±1 degrees C
	$d T$	0.1 K
Compressibility factor	Z	~ 1
	$d Z$	10^{-7}
	$d B$	10^{-8}
Volume of the CVT	V	457.575 cm ³
	$d V$	0.1 cm ³
Volume of the dead volume	V_{DV}	26.379 cm ³
	$d V_{DV}$	0.05 cm ³
Section between V3 and the tank valve	V_0	0.4 cm ³
	$d V_0$	0.1 cm ³
Collection time	t_c	30 – 1200 min
	$d t$	5 ms
Standard uncertainty	$d Q_m$	0.0001 – 0.005 mg/min

Table 3 Sources of uncertainty and the magnitude of relative uncertainty (method 2)

Source		uncertainty
Pressure	P	30 –100 kPa
	$d P$	12 Pa
Temperature	dP/dt	0.02-0.2 Pa/min
	T	25±1degrees C
	$d T$	0.05 K
Compressibility factor	dT/dt	2×10^{-4} K/min
	Z	1
	$d Z$	10^{-7}
Volume of the CVT	dZ/dt	10^{-8}
	V	457.575 cm ³
	$d V$	0.1 cm ³
Volume of the dead volume	V_{DV}	26.379 cm ³
	$d V_{DV}$	0.5 cm ³
Section between V3 and the tank valve	V_0	0.4 cm ³
		0.1 cm ³
		250 ms

$d V_0$	0.0005 – 0.0013 mg/min
Sampling time $d t$	
Standard Uncertainty $d Q_m$	

Summary

The new PVTt system as the primary standard for gas flow rates less than 5mg/min has been developed. This system has the following new and unique aspects; one is to be able to make the mass correction in the dead volume negligible small by matching the final pressure to the initial pressure there. This idea is already introduced in the new NIST system[3]. The second one is that an usual PVTt system can not calibrate only a flow meter with a function to keep a flow rate constant, but this system can calibrate any type of gas flow meters by introducing APC that can keep the downstream pressure of DUT constant. The third one is that the mass flow rate is calculated directly from the pressure change in the CVT and in the dead volume. The CVT that copper wool is stuffed behaves like an adiabatic tank, in which the temperature change is about 0.02 K/h. The determination of the mass flow rate from the pressure change in the CVT could be realized by the adiabatic like CVT and the use of the high response pressure gauge.

The uncertainty of the PVTt system developed is by no means good compared with the target expected. Therefore, the system has been on improvement to make its uncertainty better. However, as discussed in the last section, only the way to improve the uncertainty is to increase the mass of gas flowing into the CVT if measurement tolls for pressure and temperature are given. However, it is needed for long collection time to get enough amount of the mass of gas and quite longer collection time, for example one or two days, is not realistic. The method 2 calculating the mass flow rate from the pressure change in the CVT is a desirable one to short the collection time. However, the uncertainty analysis shows that the uncertainty of the mass flow rate determined by the method 2 does not change so much as a mass flow rate so that the relative uncertainty increases rapidly as a flow rate becomes lower.

The PVTt system developed here has several unique aspects, but unfortunately, the present results verify that it is not easy to apply the PVTt system to lower gas flow measurement than 1 mg/min and to obtain the results with an acceptable uncertainty and a realistic measurement period. Some drastic ideas may be required to overcome them and to calibrate a flow meter with lower uncertainty and not so long collection time .

References

- [1] Nakao,S. and Takamoto, M. , " Development of the calibration facility for small mass flow rates of gases and the sonic Venturi nozzle transfer standard" , JSME International J., Series B, vol.42, pp.667-673, 1999.
- [2] Nakao,S., Terao,Y. and Takamoto,M., " Development of the primary standard for very low gas flow rate ", the 5th IFSS, April 2002

- [3] Wright, J. private communication.
- [4] Ishibashi, M., " Calibration of Super-Accurate Critical Nozzles at Pressurized Condition", The Society of Instrument and Control Engineers, vol.36, No.1, pp1-9, 2000. (in Japanese)
- [5] " A handbook of Chemical Constants", by The Chemical Society of Japan, 1984. (in Japanese)