A PRACTICAL AND RELIABLE METHOD FOR LARGE VOLUME STANDARD VESSELS CALIBRATION

Valter Y. Aibe, vyaibe@inmetro.gov.br
Estela C. de Farias, ecfarias@inmetro.gov.br
National Institute of Metrology, Standardization and Industrial Quality - INMETRO
Fluid Dynamic Metrology Division - DINAM
Av. Nossa Sra. das Graças, 50
22250-040 Xerém-Duque de Caxias-RJ - Brazil

José M. G.Gouveia, jmgouveia@inmetro.gov.br
National Institute of Metrology, Standardization and Industrial Quality - INMETRO
Fluid Dynamic Metrology Division - DINAM
Av. Nossa Sra. das Graças, 50
22250-040 Xerém-Duque de Caxias-RJ - Brazil

Maria H. Farias, mhfarias@inmetro.gov.br
National Institute of Metrology, Standardization and Industrial Quality - INMETRO
Fluid Dynamic Metrology Division - DINAM
Av. Nossa Sra. das Graças, 50
22250-040 Xerém-Duque de Caxias-RJ - Brazil

Abstract. *The calibration of volume standard vessels should be made under controlled environmental conditions. However, the capacity, size, model or installation mode of the volume standard vessel, sometimes can be a limitation to calibrate it inside a laboratory, where there would be guarantee of controlled environmental conditions. In special, for large volumes, it is very common to make a calibration* ***in loco*** *and, depending on the features of the local where the calibration needs to take place, a traditional calibration method, as the gravimetric, cannot be used. As the calibration needs to be reliable and to ensure the achievement of measurements uncertainties levels according to the tolerance levels allowed in the Standardization or Regulations for fieldwork calibration, in this paper is proposed a practical and reliable procedure to calibrate large volume standard vessels at fieldwork, based on Batch System. This method was compared to the gravimetric method applied to volume calibration, and the results has shown a good agreement between them. Nonetheless, it was found that the time to collect a batch of large size influences in the calibration results.*

Keywords*: volume standard vessel, fieldwork calibration, volume totalizer, measurement uncertainty*

1. INTRODUCTION

The calibration of standard facilities for volume measurement usually demands several steps in calibration apparatus preparation, which depends on the capability and location of the facility to be calibrated. Gravimetric and volumetric calibration methods are more frequently used because both of them provide short ranges of measurement uncertainties. However, the first method provides smaller uncertainty range than the second, when both methods are compared for a calibration under the same laboratory environmental conditions. Schoonover (1974) investigated the equivalence of gravimetric and volumetric methods for vessels calibration and evidenced a little difference between the uncertainties levels attained by using these methods.

It is known that mass, temperature and pressure of the work fluid, such as environmental temperature and pressure are strongly influential quantities on the final results for volume measurement uncertainties. The task of controlling those quantities becomes easy when the calibrations are made under monitored and controlled laboratory conditions. However, depending on the volume standard vessel capacity and size or how and where it is installed, it is not possible to convey it to a laboratory for calibration. In this way, it is necessary to make a calibration *in loco,* but to carry the gravimetric method of calibration out, in which the balance is the basic equipment, could be very difficult or unfeasible, due to some obstacles to install or maintain a balance on the calibration place if it was not appropriate. So, in order to bypass such limitation for calibration on fieldwork, a mobile standard apparatus was projected, built and tested. The experimental results acquired using that equipment were compared to the results of the calibration carried out with gravimetric method.

2. APPARATUS

The apparatus, a practical and mobile standard to volume calibration, is composed of a volumetric flowmeter (oval gear), a centrifugal pump, a resistance thermometer Pt-100 well-shielded and hoses to flow fluid from the reservoir (cistern) until the receiver (tank or vessel) which will be calibrated. It has a panel, where the flow rate or the amount of liquid to be past through the flowmeter can be set by a digital controller and the instantaneous flow rate, fluid temperature downstream the flowmeter and total volume delivered to the receiver can be displayed. The total volume can be automatically or manually controlled (by previously setting its value or by turning off the pump at any time, respectively). During the calibration , a computer registers all data.

The system is 1090 mm length , 700mm width and 1580mm height. Figure 1 illustrates schematically the Mobile System and the facilities to calibrate it.

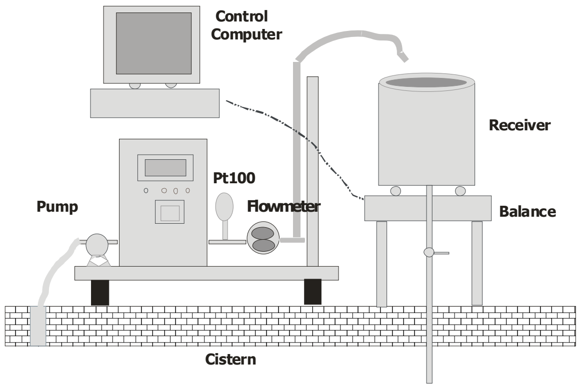

Figure 1 - Calibration of the Mobile System

3. METHOD AND MEASUREMENT PROCEDURE

The gravimetric method was used to calibrate the Batch System, as shown in Fig. 1. At this picture, the receiver figures any recipient to be placed on the balance, which also could be a calibrated volume standard vessel. Summarizing, basically the calibration procedure consisted of fixing on a fluid flow rate and a total volume to be past through the totalizer, after comparing the last one with both the total volume in fact indicated on the Batch System panel and its correspondent volume collected in the receiver. So, the reliability of the apparatus was investigated.

The complete calibration procedure includes the preparation for measurement and the calibration in fact, as follows. Firstly, the centrifugal pump has to be turned on, in order to set up a flow rate by handling a valve downstream the centrifugal pump, while the current flow rate value can be visualized on the digital panel. After that, with the valve overture fixed to permit such flow rate, the pump is turned off and a total volume to be past through the flowmeter has to be set up on the digital control panel. Then, after to register the indicated mass of the empty receiver or to tare the balance with the receiver on it, finally, the pump is turned on again, conducting the fluid to the receiver. The fluid flows at the flow rate that was fixed, until the total volume past through the flowmeter has been obtained. As the procedure aims a calibration, in this point the system turn off the pump automatically. The mass of the collected fluid is registered and, since the specific mass is known, the collected volume of fluid can be calculated. The pressure and temperature of the fluid (in the tubing and in the recipient) and also of the environment, have to be registered during the measurements due to corrections for density and buoyancy be made. For each different flow rate level, the procedure of pumping the respective fixed total drained volumes to the receiver has to be repeated several times. In the presented article, are analyzed the results for two different flow rates, each one for two different values of totalized volume.

The main goal of the Batch System calibration was to determine the correction factor K of the equipment, since this information takes account all influential quantities and variables related to the calibration method Also, it can inform on the usage limitations of the apparatus to work on field.

4. DATA REDUCTION

After data collection, each total volume into the recipient was determined, as well as the K factor of Batch System. The volume calculation was based on the ISO/TR 20461, as follows:

$$V_G(T_L) = \frac{X}{(Q - S)} Z \tag{1}$$

in which

$V_G(T_L)$ = volume of fluid determined by gravimetric method

$$X = M_C - M_V + M_E \tag{2}$$

where
M_C = balance indication when fluid stops to go into receiver + correction value given on the calibration certificate of the balance
M_V = balance indication when:
- the recipient is empty or
- after to tare the balance or
- before the next running

These values need to be added to a correction value given on the calibration certificate of the balance
M_E = balance indication of evaporated mass + value of correction indicated on the calibration certificate of the balance

In Eq.(1), Z is calculated according to

$$Z = \left(1 - \frac{\rho_{ab}}{\rho_b}\right) \tag{3}$$

where
ρ_{ab} = Specific mass of air during the balance calibration
ρ_b = Specific mass of standard weight used to calibrate the balance

S is given by

$$S = \left(\frac{k_1 \cdot P_a - \varphi \cdot (k_2 \cdot T_a - k_3)}{T_a + k_4}\right) \tag{4}$$

being
k_i = Polynomial parameters to calculate the specific mass of the air
P_a =Atmospheric pressure
φ = Air humidity
T_a = Air temperature

And

$$Q = A_4.(T_L+\Delta T)^4 + A_3.(T_L+\Delta T)^3 + A_2.(T_L+\Delta T)^2 + A_1.(T_L+\Delta T) + A_0 + \delta\rho_L \tag{5}$$

where
$\delta\rho_L$= Systematic error on specific mass of water
A_i = Polynomial parameters to calculate the specific mass of the water
T_L = Water temperature inside volumetric meter
ΔT= Systematic error on temperature water determination at the volumetric meter

Then, the correction factor K for the Volume Totalizer is calculated as

$$K = \left[1 + \frac{V_G(T_L) - V_M + \delta R_M}{V_M + \delta R_M}\right] + \delta A \tag{6}$$

where
V_M = total volume indicated on the panel of the Batch System
δR_M = Error due to volume totalizer resolution
δA =Repeatability and reproducibility of calibration

After K value was determined, its associated uncertainty was evaluated according to ISO/GUM 1995 and EAL-R2.

5. RESULTS

5.1. K factor of the Batch System and related calibration results

In Table 1 are shown, for each volumetric flow rate, the total volume (V_M) of fluid past trough the flowmeter and its respective value determined by gravimetric method (V_L), the temperature (T_L) of the liquid downstream the flowmeter, the K factors and its associated uncertainty, the degrees of freedom and coverage factor (k). In this table, the values of flow rate, V_M , V_L and T_L are averaged of a sequence of 10 measurements at the nominal total volume of 20L. At the nominal total volume of 100L, eight corresponding measurements were performed.

In calibrations at nominal flow rates of 20L/min and 30L/min, the balance was tared only once, just at the beginning of the series, when the receiver was empty. Thus, in the successive measurements of these series, after each nominal batch of 20L was added to the receiver, the pump was turned off and the measured quantities were registered. This procedure was repeated until complete the 10th measurement point of the sequence. Otherwise, the receiver was emptied before to receive each batch of fluid.

The results were:

Table 1. Correction Factor for the Batch System

Flow rate [L/min]	**V_L [L]**	**T_L [^{0}C]**	**K**	**Expanded Uncertainty of K**	**Expanded Uncertainty of K %**	**Coverage factor k**	**Degrees of Freedom**
19.5	20.01	26.75	0.99486	0.001032	0.100	2.140	19.02
19.4	99.58	26.59	0.99567	0.001949	0.196	2.869	4.09
30.1	20.04	26.76	1.00156	0.001295	0.129	2.284	10.81
29.8	100.23	26.63	1.00218	0.000745	0.074	2,869	4.67

Table 2. Standard deviations

Flow Rate [L/min]	**Standard Deviation [L/min]**	**V_M [L]**	**V_M Standard Deviation [L]**	**V_L [L]**	**V_L Standard Deviation [L]**	**V_L Standard Deviation %**	**V_L - V_M [L]**
19.5	0.02	19.91	0.003	20.01	0.007	0.035	0.1
19.4	0.05	100.01	0.002	99.58	0.069	0.069	-0.43
30.1	0.10	20.01	0.002	20.04	0.009	0.045	0.03
29.8	0.21	100.01	0.000	100.23	0.025	0.025	0.22

6. DISCUSSION

According to Tab. 1, the greater the flow rate, the greater the correction factor K. It is observed, at totalized nominal volume of 100L, that the associated uncertainty to the correction factor K is higher at nominal flow rate of 20 L/min than at 30 L/min. Added to that, the difference between V_M and V_L is higher for flow rate 20 L/min than for 30 L/min (see Tab.2). As the method to collect that totalized volume was the same for both flow rates, based on table 2, on which is shown that the standard deviations of total volumes are a little high for flow rate 20L/min, it is suggested that the time interval to collect a batch can affect the results. During a large time interval for sampling, the equipment could detect substancial scattering of the responses, being necessary more measurement data in order to minimize masked results.

Analyzing the results for small batches (nominal 20L), at both flow rates tested, the range of standard deviation for V_L is shorter than that for 100L. Besides that, the difference between V_L and V_M is smaller in 30L/min than in 20L/min for nominal totalized volume 20L, indicating, again, that a large time interval to get a sample at a fixed flow rate is recommendable and provides more representative results, because it would be possible to pick variations or instabilities up of the system. So, if the flow rate is high and the time duration to get a sample is quite short, the results are not representatives to calibration. This can be verified also by observing the standard deviation of V_L at nominal totalized volume 20L and the uncertainties for the correction factor K .

7. CONCLUSION

A mobile and practical system to calibrate vessels on field had been proposed. A prototype was built and calibrated in two different flow rates and size of batch, using the gravimetric method. The results show how important is the time interval duration for sampling and, for the built equipment, the flow rate 20L/min was considered to be better than 30L/min for the measurements, although the lowest flow rate requests more measurement data for large size batches.

More investigations on the performance and limitations of the Batch System here presented are recommended, in order to draw optimized parameters for usage of this mobile standard, i. e., the best combination of nominal flow rate and totalized volume. These parameters shall be found for a range of nominal totalized volume that is feasible when the system is used at field. However, the analyzed results show that the equipment is an adequate alternative to vessels calibrations at field.

8. REFERENCES

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