

METROLOGICAL PERFORMANCES OF MASS FLOW CONTROLLERS FOR DYNAMIC GAS DILUTION

*Alessia Demichelis*¹, *Guido Sassi*^{1,2}, *MariaPaola Sassi*¹

¹ Istituto Nazionale di Ricerca Metrologica I.N.Ri.M., Torino, Italy, a.demichelis@inrim.it

² Department of Material Science and Chemical Engineering Politecnico di Torino, Torino, Italy, guido.sassi@polito.it

Abstract: The dynamic method of generating reference Volatile Organic Compounds (VOC) mixtures at trace level requires the uninterrupted blending of the component flows. The accurate control of the dilution air flows can be realized by Mass Flow Controllers (MFC). Accuracy and stability of MFC are here measured and discussed in order to verify their use in dynamic gas dilutors. Long term stability confirm the necessity of calibrating MFC before each use. MFC accuracy results not pressure drop dependent, but limited by the short term stability for flows below than 70% of MFC full scale and generation time shorter than 5 minutes.

Keywords: Mass Flow Controllers, VOC, dynamic dilutor, reference gas mixture.

1. INTRODUCTION

The dynamic method for the generation of reference Volatile Organic Compounds (VOC) mixtures at trace level requires the uninterrupted blending of the component mass flow rates (VOC flow and dilution air flows) for some specific period of time [1], i.e., the dilution time, generally shorter than 1 hour. Different schemes of dilutor can be adopted, VOC flow can be diluted by one or two stages with fresh air flow and one flow is connecting the stages.

VOC flow rate is controlled by the temperature of the dynamic source, e.g., diffusion or permeation tubes. The stability of the VOC flow rate is mainly dependent on temperature stability while its accuracy depends on temperature variability and weighing accuracy [2] [3].

Air flow rates target uncertainty can be calculated from the uncertainty budget of the generation system [3]. At a broad temperature control, e.g., 0.1 K, the uncertainty of a VOC mass flow rate over 5 $\mu\text{g}/\text{min}$, was calculated to be 1.4% [2] [3]. For a two stages dilutor, an uncertainty less than 0.2% of each air mass flow rate does not affect the concentration uncertainty in the mixed flow. Over 2% uncertainty, the air flow rate becomes the main contributor to the mixture concentration uncertainty. At a narrow temperature control, e.g. 0.01 K, the uncertainty of a VOC mass flow rate over 5 $\mu\text{g}/\text{min}$, was calculated to be 0.4% [2] [3]. For a two stages dilutor, an uncertainty less than 0.1% of each air mass flow rate does not affect the concentration uncertainty in the mixed flow. Over 1% uncertainty, the air flow rate becomes

the main contributor to the mixture concentration uncertainty.

The maximal value that allows to neglect their contribution to the uncertainty of the mixture concentration can be considered as the target uncertainty of the air flow rates. 0.2% and 0.1% are the target uncertainties at narrow and broad temperature control respectively.

The uncertainty of the dilution air flow rate may be composed by composing the accuracy, calibration stability (i.e., long term stability) and operation stability (i.e., short term stability) of the flow control system during the dilution time. Accuracy and calibration stability of the flow control system is calculated by the traceable calibration, whereas the operation stability needs ad-hoc experiments to be calculated.

There are two classes of instruments candidate for the accurate control of the dilution air flows: those based on fixed flow elements (e.g., sonic nozzles) [7][8] and those based on variable flow elements (e.g., Mass Flow Controller, MFC) [4][5][6]. In the both cases the state-of-the-art accuracy of the provided flow rate is 0.1% [9].

The operation stability of the flow rate provided by a sonic nozzle depends on the operation stability of the temperature and pressure of the upstream. It is possible to realize a very stable control of these variables leading to a flow rate operation stability in the order of 0.01% [10]. The calibration stability of the sonic nozzles is given by the stability over time of the orifice geometry, i.e., sonic nozzle may have fouling problems, and the calibration stability of the temperature and pressure measurement systems.

MFC can give a wide range of flow rates (10-100% full scale) allowing to change the composition of the VOC gas mixture rapidly and in a continuous manner. A common drawback of the instruments operating with variable flow elements, is that the flow rate accuracy is limited by the flow rate calibration stability, as reported in [9] and [11]. It follows that it is required a deep understanding of the MFC accuracy and short-term stability, when these instruments want to be used in dynamic dilutors.

In the preparation of gas mixture at trace level with a double stage dynamic dilution, according to ISO 6145-8 standard [12], at least three dilution flows are required to dilute the VOC mass flow, i.e., from diffusion tubes. Two flows correspond to the two main dilution lines, whereas the third connects the two dilution stages.

A pressure drop is necessary from the first stage to the second one to make the MFC properly working. The pressure in the first line must be the lowest possible and cannot exceed 10 kPa to make the diffusion tube properly working. A low pressure drop (2-3 kPa) MFC must be used for the inter-stage flow.

This paper deals with the MFC calibration stability, the accuracy of low pressure drop MFC at very low pressure drop and the quantification of the MFC stability during operation.

The scope is to experimentally verified the use of MFC in dynamic gas dilutors.

The results will lead to a more robust value, not commonly reported, for the MFC flow stability than can be determinant in the choice of the optimal gas control system in the dynamic dilutor. Besides the investigation on the limit gas pressure in the first dilution line of the VOC dilutor system can contribute to the choice of the optimum dilutor geometry allowing also the miniaturizing of the VOC generation system.

2. MATERIALS AND METHODS

Two thermal mass flow controllers at nominal 25 NL/min (model EL-FLOW Select[®], F201CV-20K-AAD-33-V, Bronkhorst HI-TEC) and one mass flow controllers at nominal 100 NmL/min (model LOW- Δ p-FLOW, F-101D-100-AAD-33-V+F-004-AC-LU-33-V, Bronkhorst HI-TEC) were tested in this work. Hereafter they referred as MFC1_A, MFC1_B and MFC2 respectively. Bronkhorst specifications define an optimum gas inlet pressure range, of 2-3 bar.g for MFC1, and 20-30 mbar.g. for MFC2.

Flow rate measurements were performed by the INRIM bell prover (160 liters nominal capacity, 0.2 l/min to 400 l/min) [13] and the INRIM piston prover (3 liters nominal capacity, 0.1 ml/min to 2 l/min) [14].

The experimental set up is reported in Figure 1. (C: Air cylinder; PR: pressure reducer; n: gas connector (1 and 2); BP: bell prover; PP: Piston prover; V: valve). The set up was tested for gas leakages before and after each measurement. Leakages were set lower than 10 μ l/min.

MFC1 were 0.1% calibrated at 10%, 50%, 90% f.s. (full scale) by the bell prover. Temperature and pressure of the tested gas inside the bell prover have been measured at the beginning and at the end of each measurement. MFC2 was calibrated at 20, 60, 80, 100 % f.s. (full scale) by the piston prover. Calibrations were repeated along 6 months from the shipping date of the instruments to evaluate the long-term stability (named “calibration stability” in this work). MFC2 was 0.05% calibrated at 100% f.s. at a pressure drop ranged from 0.08 to 4.5 kPa.

For the quantification of the operation stability MFC1 and MFC2 flow rates were measured as an average on each 10 s along 5 hours. 10%, 50%, 90% f.s. were considered for MFC1, while 100% f.s. was considered for MFC2. The standard deviation of flow rate referred to the average flow rate has been calculated for each time interval inside the 5 hours measurements at different interval length [2], i.e., observation time $\tau = 20$ s -100 min.

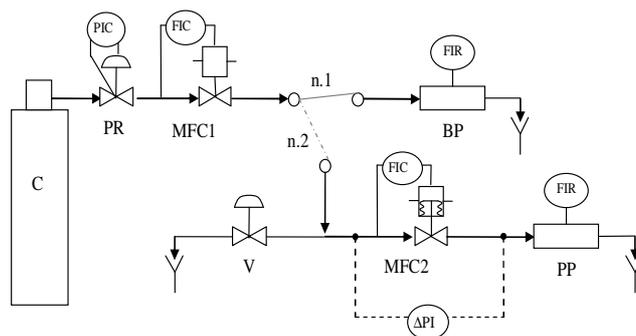


Fig. 1. Experimental setup

3. RESULTS AND DISCUSSION

Calibration stability: The bias between the calibration value, at the working time, and the value of the manufacturer’s calibration certificate, at the shipping time, referred to the nominal flow rate, has been considered to evaluate the calibration stability. Figure 2 reports the bias values for MFC1 at 50% f.s. and at 90% f.s., named B%, in function of the time t. Shipping time has been set as $t=0$. Each value has been calculated as the average of 4 measurements, the repeatability has been lower than 0.04% and the INRIM calibration uncertainty is 0.1% for MFC1 (vertical error bars in Figure 2). The uncertainty of the manufacturer’s calibration certificate for MFC1 at 50% and 90% f.s., are 1.1% and 1.0% respectively.

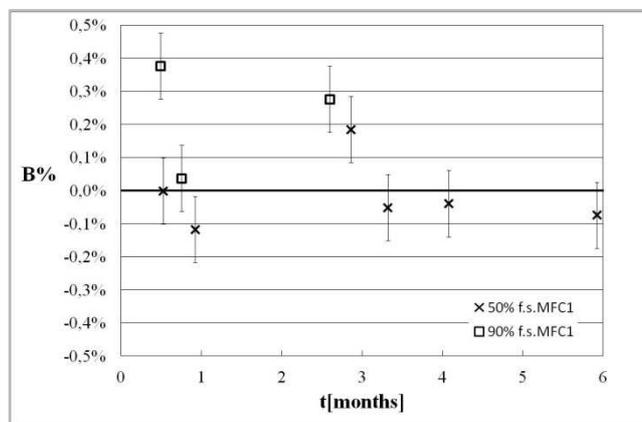


Fig. 2. MFC1 calibration stability

From Figure 2 it is noticeable a “random” calibration variability within 0.5% in a 6 months period and a deviation from the calibration uncertainty (0.1%) just after few days. In spite of this fact the Mass Flow Controllers remains within its manufacturers stated uncertainty (1%) in the tested calibration time. Similar result has been obtained for MFC2. Calibration stability has been checked for 5 hours continuous work of MFC. Calibration variability B% at different observation time has been calculated to be lower than calibration uncertainty. Since the calibration stability exceed the defined target flow uncertainty of 0.1% - 0.2%, the MFC must be calibrated before (and after) each mixture generation.

Low pressure drop effects: MFC2 is recommended to work at 2-3 kPa pressure drop. To reduce pressure in the first stage of the dilutor a lower pressure is more convenient. MFC2 has been calibrated at 80 to 4500 Pa pressure drop. Below 100 Pa pressure drop, the mass flow rate has a 2.5% bias and a 2% variability. In the range 100 to 4500 Pa the variability of average values is similar to the uncertainty (0.03%). Moreover Fisher statistical test on data outside and inside the recommended range, have shown that they belong to the same population with a confidence greater than 97%. It means that above 100 Pa, even outside of the suggested working range (2-3 kPa), the variation of instruments performances is contained in the measurement uncertainty. Consequently pressure drop has no effect on the accuracy of MFC2 for a pressure drop higher than 100 Pa.

Operation stability: The relative standard deviation of flow rate measurements has shown a normal distribution. For each interval length, i.e., observation time t , the maximum observed value and the 98th percentile of the distribution of the relative standard deviation have been calculated to evaluate the flow rate random variability $V\%$ as a measure of the operation stability. Figure 3 reports the operation stability vs. the observation time t , at different mass flow rates F (10%, 50% and 90% f.s. for MF1 and 100% f.s. for MFC2).

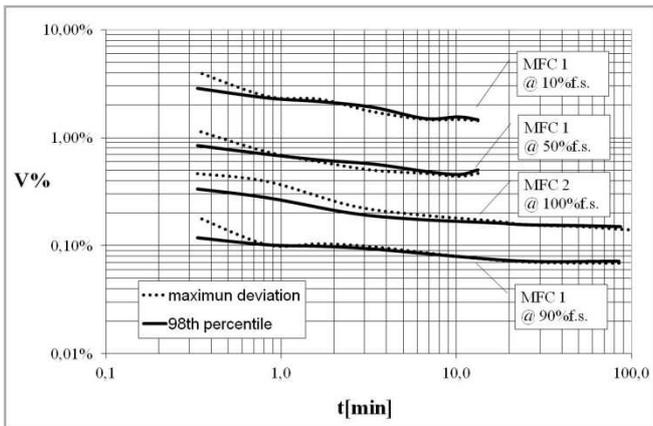


Fig. 3. MFC random variability of the control

Maximum and 98th percentile had similar values when the number of interval is low, but when the number of interval is larger than 2-300 the 98th percentile is more representative than the maximum of the effective variability.

The flow rate random variability slightly decreases as the operation time is longer for all the MFCs at each set point. This means that the longer is the operation time the lower may be the expected mixture concentration variability.

Flow rate variability $V\%$ is minimal at the full scale and approach the 3% at the 10% of the full scale. Random variability has been found to be a logarithmic function of the observation time t and mass flow rate F , as reported in equation (1).

$$V\% = [0.14 \ln(t) + 0.93] \ln(F) + 0.65 \ln(t) + 4.343 \quad (1)$$

MFC2 at 100% f.s. has the same nominal flow rate of MFC1 at 10% f.s. but MFC2 has shown an operation stability around 0.2% versus 2% of MFC1 at the same flow rate. MFC1 and 2 flow rate variability is lower than the flow target uncertainty of 0.1 – 0.2% only around the 100% of full scale.

MFC uncertainty: The uncertainty of flow rate is the combination of calibration uncertainty and operation stability. Following GUM, they can be combined as the quadratic sum of the two contributions. As previously discussed, no pressure effects on MFC accuracy has been considered.

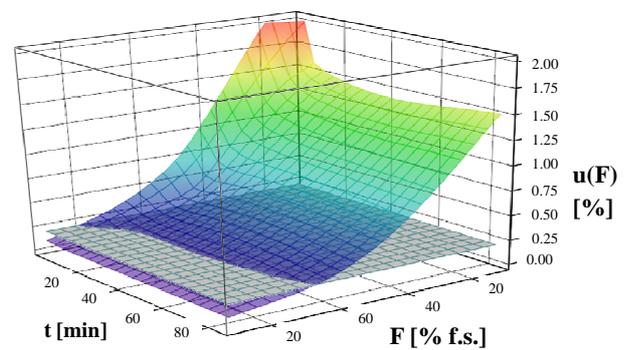


Fig. 4. MFC1 combined uncertainty $u(F)$

In figure 4, the MCF1 combined uncertainty $u(F)$ is reported as a function of the operation time t and of the mass flow rate F from MFC [% f.s.]. Calibration uncertainty gives a constant contribution on the whole ranges, while random variability gives a variable contribution expressed by the square of equation 1.

The grey plateau is at the target uncertainty (0.2%), the target uncertainty is guarantee for flow rates larger than 70% and observation times larger than 5 minutes.

The accuracy threshold is derived from the traceable MFC calibration (time and flow range – invariant) where is considered to calibrate the instrument before each use (in order to eliminate the calibration long term stability contribution).

It can be shown that the evaluated MFC flow uncertainty results strongly dependent on the MFC range (% f.s.) and on the time at lower flows. This totally reflect the behavior of the operation stability which represent the dominant contribution to the total MFC uncertainty.

4. CONCLUSION

In this paper MFC performances have been experimentally investigated to verify their use in dynamic gas dilutors.

From the calibration stability results it has been shown that a normal MFC needs to be re-calibrated before and after each use to guarantee the uncertainty requirement for VOC reference mixture generation. This suggests to provide a handheld flow transfer standard that can be used before each generation. Alternatively, gas control system based on fixed flow element can be employed due to the easier calibration procedure.

It is not possible to state a systematic pressure effect on the provided mass flow accuracy of the low pressure drop MFC, in a inlet pressure range from 100 to 4500 Pa. This means that, in order to not perturb the VOC mass flow from the generator, is possible to construct a dilutor with a limit pressure in the first dilution lineup down to 100 Pa. This allows to miniaturized the VOC generation system with a little design pressure loss.

In this paper a method for the evaluation of the MFC operation stability over time has been proposed. The method, through 98th percentile calculations, allows a conservative evaluation of the maximum observed flow variation in operation period from 1 minute to 100 minutes. Below 1 minute, the maximum deviation estimator seems to be more conservative. It has been found a MFC operation stability strongly flow-dependent and time-dependent at lower flows.

From the investigated MFC performances, a method for MFC uncertainty evaluation has been proposed through the quadratic combination of the traceable instrument calibration result and the operation stability measured. As a result it has been found that the accuracy of the employed MFC flow results to be limited by the flow operation stability at lower flows. Instead for higher flows the operation stability is of the same order of the 0.1% INRIM calibration uncertainty.

The reported target uncertainties for the dilution air flow required for VOC dynamic mixture generation in dynamic dilutors has been calculated to be 0.1% for a narrow temperature control generation and 0.2% for a broad temperature control generation. These target uncertainties can be guarantee by MFC in dynamic dilutors if these flow control systems are calibrated before each mixture generation and if a set-point higher than 70% f.s is used and for a generation time larger than 5 min. Otherwise it may be oriented to more stable flow control systems, e.g., based on fixed flow elements.

5. REFERENCES

- [1] O. G. Nelson, Gas mixtures: preparation and control. Boca Raton, FL : CRC Press, 1992.
- [2] G. Sassi, A. Demichelis and M. Sassi. "Uncertainty analysis of the diffusion rate in the dynamic generation of VOC mixtures", Measurement Science and Technology, vol. 22, no. 10, 2011
- [3] G. Sassi, A. Demichelis, M. Sassi "A dynamic trace VOC generator useful for global climate change study", in Proc. of IMEKO XIX World Congress, Lisbon, Portugal, pp. 2602-2605, 2009.
- [4] S. A. Tison, "A critical evaluation of thermal mass flow meters.", J Vac. Sci. Technol. A, Vol. 14, 1996.
- [5] M. Viswanathan, A. Kandaswamy, S. K. Sreekala S K, K. V. Sajina "Development, modeling and certain investigations on thermal mass flow meters." Flow Measurement and Instrumentation, Vol. 12, pp. 353-360, 2002.
- [6] M. Arlindo Amador de Matos and V. da Silvia Ferreira "Gas mass-flow meters: principles and applications.", Flow Measurement and Instrumentation, Vol. 21. pp.143-149, 2010.
- [7] M. Hayakawa, Y. Ina, Y. Yukoi, M. Takamoto, S. Nakao "Development of a transfer standard with sonic Venturi nozzles for small mass flow rates of gases." Flow Measurement and Instrumentation, vol. 11, pp. 279-283, 2000.
- [8] N Bignell, Y M Choi "Thermal effects in small sonic nozzles." Flow Measurement and Instrumentation, vol. 13, pp. 17-22, 2002.
- [9] P J Brewer, B A Goody, T Gillam, R J C Brown and M J T Milton "High-accuracy stable gas flow dilution using an internally calibrated network of critical flow orifices." Measurement Science and Technology, vol. 21, 2010.
- [10] J. D. Wright, J. P. Kayl, A. N. Johnson, G.M. Kline. "Gas Flowmeter Calibrations with the Working Gas Flow Standard." NIST Special Publication 250-80, 2008.
- [11] ISO 6145 Gas analysis: preparation of calibration gas mixtures - Dynamic volumetric methods, Part 7: Thermal mass flow controller. 2003.
- [12] ISO 6145 Gas analysis: preparation of calibration gas mixtures - Dynamic volumetric methods, Part 8: Diffusion. 2003.
- [13] G. Cignolo, M. Clausen, R. Gorla, H. Baumann, "EUROMET PROJECT 852: A comparison between italian and swiss gas flow standard in the range 0.3 to 25 m³/h," Metrologia, vol. 45, 2006.
- [14] F. Alasia, A. Capelli, R. Gorla, G. La Piana, G. Cignolo, "A primary standard piston prover for measurement of very small gas flows: an update." Sensor Review, vol. 25, no. 1, pp. 40-45, 2005.