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Air flow rate thermal control system at low pressure drop

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ABSTRACT

Low pressure drop thermal Mass Flow Controllers are generally thought to fulfill needs concerning the realization of a dynamic reference gas mixture generator for accurate gas analysis. A small air flow rate at low pressure drop must be controlled in a stable and precise way in the generator. True operative pressure drop limits, set point reproducibility, calibration needs and flow rate stability during operations were investigated for a low pressure drop thermal Mass Flow Controller. The flow rate bias due to late calibration and flow rate short-term stability were measured and discussed. The Allan method was used to calculate stability during operation. Calibration uncertainty, bias for late calibration, stability and set point reproducibility were composed to calculate the total uncertainty of the flow rate as a function of the operation time. Results show that it is possible to operate below the target uncertainty stated for a dynamic generator of gas mixtures down to 100 Pa pressure drop. Stability gives the main contribution to total uncertainty at very short operation times, while calibration uncertainty gives the main contribution to total uncertainty at normal operation times. The calibration uncertainty at 0.1% is low enough to assure the target uncertainty for operation times over 10 s. Daily verification of calibration enhances the reliability of the measurement. An accurate voltmeter is necessary for the reproducibility of the set point.

1. Introduction

Reference mixtures of volatile organic compounds (VOC) at trace level for accurate gas analysis can be prepared in a dynamic reference gas generator. The VOC mass flow, e.g. from diffusion tubes [2], is continuously diluted by air flows in serial stages connected by inter-stages lines. Reference gas generators require, for a specific period of time [1], the uninterrupted blending of component mass flows, i.e., the VOC flow, air flows to dilution stages and mixture flow in the inter-stage line. Operation times in the range of 1–100 min are generally used for direct supply either to a gas-chromatograph or spectroscopic instrument or sorption tube charging system. Measurement instruments with small flow cells usually need lower operation times. Often a double stage dilution system is used in reference gas generators, where two main dilution lines are connected by an inter-stage line, the first stage is directly connected to the diffusion cell and the pressure in the diffusion cell affects VOC mass flow [3]. A significant over-pressure in the first dilution line can introduce gas leakage problems in the diffusion cell and inaccurate pressure correction for the VOC diffusion rate [3]. The mass flow of the reference gas mixture coming out of the last dilution stage is usually supplied at atmospheric pressure or higher for gas analysis applications. The discharge pressure of the inter-stage line can be reduced by a Venturi tube at the second stage. To ensure a very accurate mass flow rate and short-term stability a flow rate control system is necessary in any case. It follows that the design specification for the inter-stage flow control system of the reference gas generator must be at a low pressure drop.

The target uncertainty of the inter-stage mass flow rate, typically in the range of 1–100 Sml min^{-1}, is 0.2% ($k=1$) is a consequence of the target uncertainty of the concentration of the trace level reference VOC mixture [3,4]. Short-term stability of the inter-stage flow rate, in the typical range of observation times 1–100 min, must be compatible with the total flow rate uncertainty, to avoid affecting concentration stability and uncertainty of the sampled reference mixture.

Accurate flow control systems which are most commonly used, e.g., either thermal Mass Flow Controllers (MFC) or the stable sonic nozzles, operate with a significant pressure drop, larger than 1 bar. A thermal MFC at low pressure drop was specifically designed by Bronkhorst to operate at 0.1–500 kPa absolute working pressure in the downstream line and 2–3 kPa pressure drop [9]. It can work for the present purpose because thermal mass flow control technology has good accuracy, the working pressure drop is low and mass flow tuning is highly versatile. No evidence is reported either for their calibration bias over time or short-term

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control stability. The manufacturer reports 80 Pa pressure drop in the measuring part of the controller [9].

The scope of this work is to verify the usability of a "Low pressure drop MFC" for the control of the inter-stage flow in a dynamic gas dilutor of a reference mixture generator. A comprehensive metrological characterization of this instrument is here given, focusing on true operative pressure drop limits. The flow rate bias for both late calibration and flow rate short-term stability in operative conditions were calculated. The Allan's variance [6] were used to analyze the short term stability.

Results will contribute to overcoming the engineering problem of the construction of a double stage dynamic gas dilutor to prepare highly accurate reference trace VOC mixtures. The metrological characterization given provides a method for the verification of instrument performance, not always supplied by the manufacturer, and also provides a guideline for the calibration of this flow instrument class and defines procedures to ensure target flow rate uncertainty. The characterization parameters here applied (reproducibility, operative limits, stability, calibration) are also applicable to other instrument models and classes [10].

2. Materials and methods

A Mass Flow Controller (MFC) at 100 ml min^{-1} nominal flow rate at Standard condition (model LOW-Δ*p*-FLOW, F-101D-100-AAD-33-V+F-004-AC-LU-33-V, Bronkhorst HI-TEC) was tested. Hereafter it is referred to as F101. The experimental setup adopted for F101 characterization is reported in Fig. 1, a schematic cross-view of F101 is highlighted. Bronkhorst specifications define an optimum pressure drop in the range of 2–3 kPa. Synthetic air cylinders (Air Liquide, air Alphagaz 1) with a declared purity of 99.999% without any gas filter were used to supply the controller. The instrument was switched on and conditioned before measurements for at least 1 h at the set flow rate. Flow rate measurements were performed by the INRIM piston prover (3 L nominal capacity, 0.1 ml min^{-1} nominal flow) [5]. The set up was tested for gas leakages before and after each measurement; leakages were kept lower than 10 μl min^{-1} at standard condition. Flow rate readings and regulation were performed by a 6½ digit multimeter. Flow rates are here reported in Sml min^{-1}, i.e., ml min^{-1} at standard conditions: 1 bar, 0 °C. F101 was calibrated at 20%, 60%, 80%, 100% of maximum flow rate by the piston prover, calibration standard uncertainty by piston prove is 0.05% [5]. Calibrations were repeated over 30 months since shipping to evaluate the calibration bias over time. F101 was calibrated at maximum flow rate at a pressure drop range from 80 to 4500 Pa. The pressure drop was changed by varying the upstream pressure and keeping a constant downstream pressure (atmospheric pressure) as the dilutor configuration requires. The Fisher and *t*-Student statistical tests were performed to compare the data populations inside and outside the recommended pressure working range, i.e., 2–3 kPa. The F101 flow rate at 100% of maximum flow rate was measured over 5 h every 10 s to quantify the short-term stability by the Allan deviation [6]. Allan stability data are here reported in function of the observation time, i.e., the operation time, in order to analyze flow rate stability.

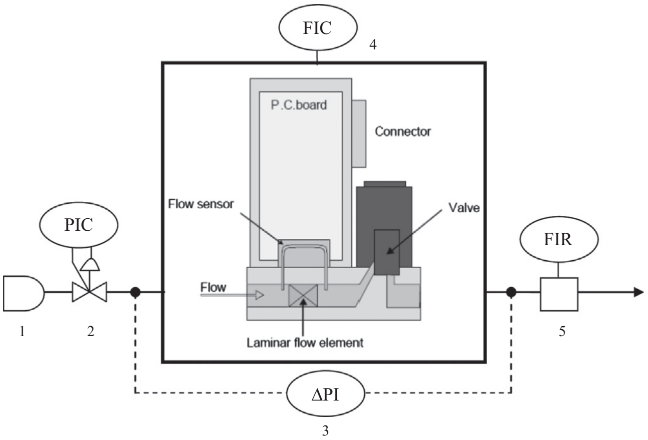

Fig. 1. Experimental setup for F101 characterization. (1) Pure air cylinder, (2) pressure reducer; (3) differential pressure measurement, (4) Mass Flow Controller (MFC) under test, and (5) Piston prover. PIC, pressure indication and control; FIC, flow rate indication and control; FIR, flow rate indication and registration; ΔPI, differential pressure indication.

3. Results and discussion

3.1. Low pressure drop effects

The F101 was calibrated in the pressure drop range of 80 to 4500 Pa. Data and mean value over the range 100–4500 Pa are reported in Fig. 2; vertical error bars refer to the standard calibration uncertainty. Observed variability of flow rate values is within calibration standard uncertainty (0.05%) for pressure drop between 100 and 4500 Pa. The mass flow rate at 80 Pa pressure drop had −2.5% mean bias from the mean value over the range 100–4500 Pa (data are not reported in Fig. 2). The performance variation of F101 is lower than the measurement uncertainty above 100 Pa, even outside of the suggested working range (2–3 kPa). The population of pressure drop data inside and outside the recommended range were compared by Fisher and *t*-Student statistical tests. The statement that the populations are different were refused with a confidence of more than 98%. Consequently pressure drop had no effect on the calibration standard uncertainty of F101 for a Δ*p* higher than 100 Pa. The minimal pressure limit for the use of the Mass Flow Controller F101 is then 100 Pa.

3.2. Bias for late calibration

The mass flow controller needs to be re-calibrated when it overcomes the target uncertainty. The target uncertainty depends on the application that the mass flow controller is devoted to. The calibration uncertainty and the bias for late calibration may be composed as defined by GUM, i.e., the contribution of each source is the square of its relative value [7]. The calibration contribution was calculated as the square of the MFC calibration uncertainty (0.05%) and the bias contribution was calculated as the square of the relative shift from the last calibration. Two threshold limits

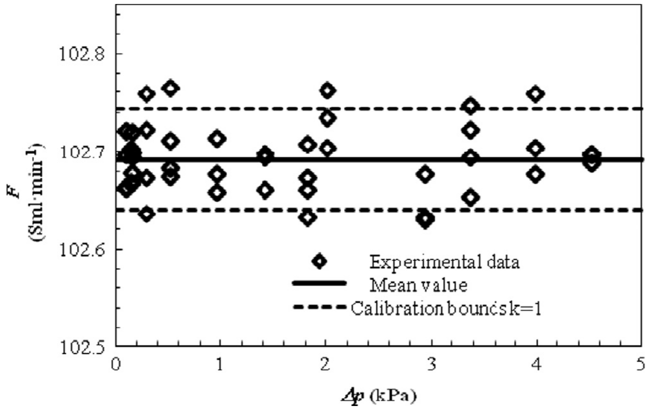

Fig. 2. Pressure drop Δ*p* effect on F101 calibration standard uncertainty at the maximum flow rate. Mean value with standard calibration uncertainty ($k=1$) of 0.05%.