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Micro-flow facility for traceability in steady and pulsating flow

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ABSTRACT

This paper describes a primary standard for liquid micro-flow, which covers the flow rate range from 1 ml/min down to 100 nl/min with uncertainties in the range from 0.1% to 0.6% (coverage factor 95%). To realize stable flow rates, METAS applies the principle of generating flow by means of a constant pressure drop over a capillary tube according to the law of Hagen–Poiseuille. The constant pressure drop is mainly possible due to the fact that the relative pressure at the outlet needle remains constant as the outlet needle is positioned just above the beaker collecting the water. The special beaker and the adjustments for the weighing zone to control evaporation will be discussed in the paper as well as measurement results from flow sensors and flow generators, which highlight the repeatability and the reproducibility of the facility.

1. Introduction

Several applications in biotechnology, medical care, sensor technology and process control involve micro-flow wherever mixing, flow or demixing of various fluids or gases are important and where the exact amount of the delivered volume or a stable flow rate is crucial for the efficient operation. METAS develops since 2010 a facility for liquid flow rates from 1 ml/min down to 100 nl/min and participates in the EMRP project "HLT07 Metrology for Drug Delivery" [1,2] which started in June 2012. Based on different methods various facilities have been built in the past years to ensure traceability of calibrations at that low flow rates. One of the possibilities is the gravimetric flow measurement where a sensitive balance is used for measuring the mass as a function of time. The flow rate is determined by the quotient of the mass difference and time difference including some corrections. The water is often collected in a vessel where the outlet of the piping is immersed into the water in the vessel [3–7]. Another possibility is to track directly the meniscus of the waterfront flowing through a capillary as a function of time. This method strongly depends on the traceable calibration of the diameter of the capillary, which is the main contribution to the uncertainty [8]. A third possibility is to use a known expansion of a liquid to realize a flow generator. The flow is produced by a controlled temperature gradient in a given volume [9]. In this paper the dynamic gravimetric method is applied, where the main difference is that the outlet needle is not immersed into water, but placed above a special measurement beaker to produce a very short water stream whose shape is more or less constant in time. The facility itself is described in this paper as well as repeatability and reproducibility measurements of different types of flow sensors and flow generators.

2. Design of the facility

One of the main issues in the development of the facility is not only to generate a very low flow rate but also to ensure good flow rate stability. To realize this flow rate stability METAS applies the principle of generating the flow by means of a constant pressure drop over a capillary tube according to the law of Hagen–Poiseuille [10], (Eq. (1)).

$$Q = \frac{\Delta P \cdot r^4 \cdot \pi}{L \cdot \eta \cdot 8}, \tag{1}$$

where Q is the flow rate, ΔP the pressure drop, r the radius of the capillary, L the length of the capillary, and η the viscosity of the liquid.

In Figs. 1–3, the simplified working principle, the scheme of the facility and a photograph of the facility are shown. A metallic bellow is immerged into a water tank and separates the pressurized air from the pressurized water in the tank to avoid any air absorption in the degased water. To control the water pressure in the water tank, the metallic bellow is expanding or compressing by adjusting the air pressure inside the bellow with a pressure controller. For this part, the air pressure inside the bellow is adjusted according to the signal of a pressure sensor inside the water tank in order to reach the desired relative water pressure in the tank. The stability of the water pressure is guaranteed by the fact that at a constant time interval of several tens of seconds the target pressure is readjusted by the pressure controller by means of a regulation loop controlled by software. The constant pressure

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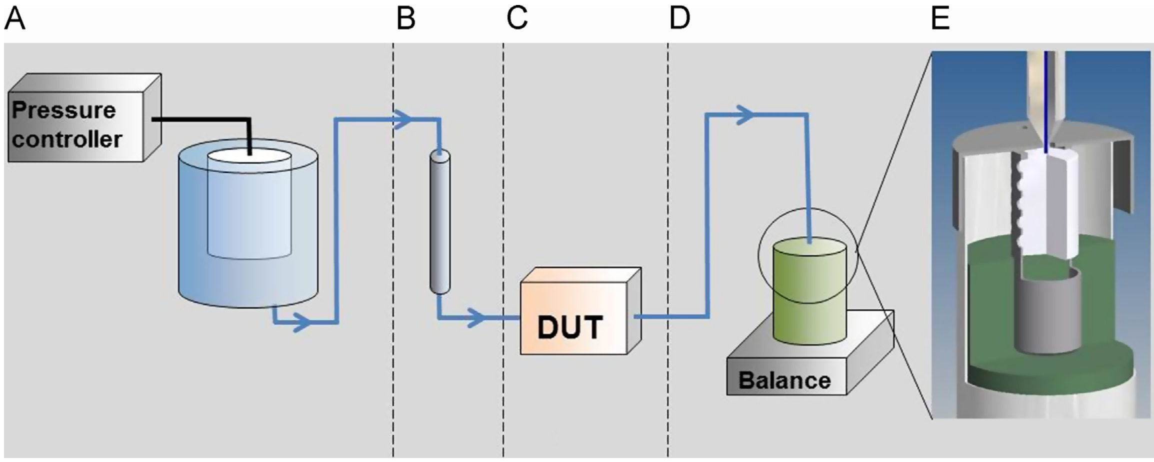

Fig. 1. Simplified drawing of the working principle of the facility. (A) water tank with immerged metallic bellow and pressure controller, (B) capillary tube, (C) device under test (DUT), (D) measurement beaker on the balance, and (E) detailed cross-section of the top part of the measurement beaker.

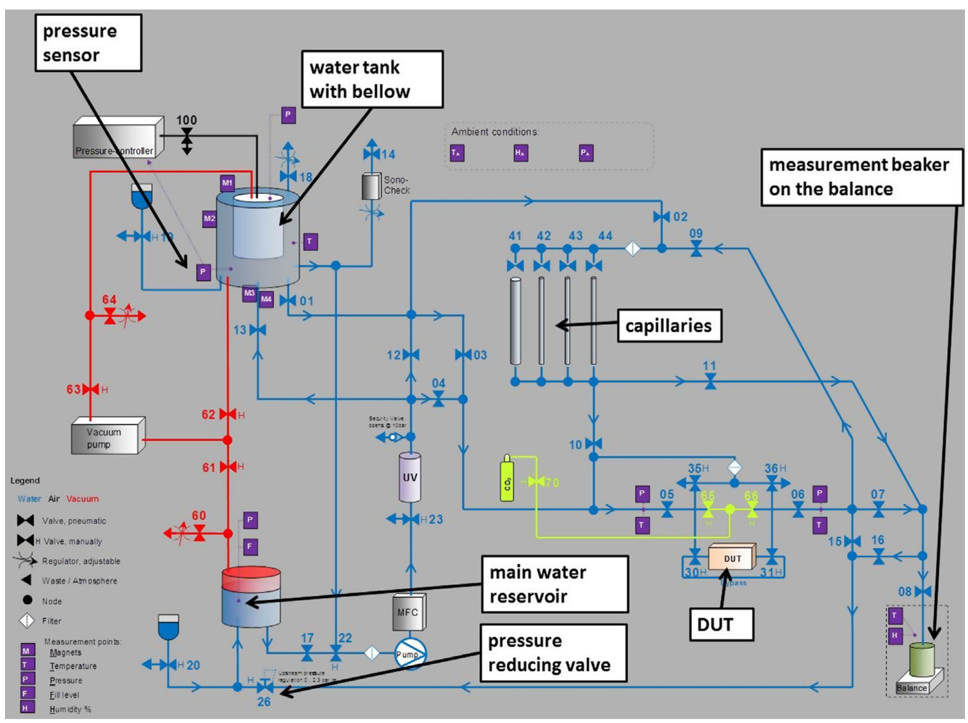

Fig. 2. Scheme of the facility with the main components: water tank with metallic bellow, capillary tubes of 200 mm length and inner diameters varying from 50 μm to 2000 μm, device under test (DUT), measurement beaker on the balance, main water reservoir.

drop from the water tank to the atmospheric pressure at the outlet needle and the size of the capillary tube determine the flow rate and guarantee the steadiness of the flow rate once the stabilization time for a given flow rate is reached. Therefore the flow rate can be set by increasing the water pressure in the water tank up to 2.5 bar and by selecting the appropriate capillary from the set of 4 capillaries with the inner diameters of 50 μm, 80 μm, 150 μm or 2000 μm and a length of 200 mm. It is worthwhile to mention at this point that flow meters for very low flow rates down to several tens of nl/min commonly use capillary tubing with inner diameters of several tens of μm [3,11]. For these cases most of the pressure drop will occur over the flow sensor, as the inner diameter is smaller than our smallest capillary tubing. On the other hand flow sensors for larger flow rates in the range of 1 ml/min down to several μl/min have capillaries or tubing with inner diameter larger than 50 μm and therefore the main part of the pressure drop will occur over the chosen capillary of the facility. At any time, the capillaries of the facility can be changed and different inner diameter used.

The facility is designed in such a way that the water pressure upstream the Device Under Test (DUT) can be varied to some extent. In one mode, the water flows from the capillary tube through the DUT and finally in the measurement beaker on the balance XP6 from Mettler-Toledo [12]. The main pressure drop occurs over the capillary tube and this means that the pressure difference with respect to the ambient pressure upstream the DUT is very small. In a second mode, the water is first guided through the DUT and then through the capillary tube and in the measurement beaker on the balance. In this case the water pressure upstream the DUT is similar to the water pressure in the water tank.

The installation is filled with degased ultrapure water. To hamper growth of bacteria and algae, 50 mg of sodium azide is mixed with