AUTOMATED LOW GAS FLOW-RATE CALIBRATOR

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Received April 4, 1989
Accepted April 24, 1989

Dedicated to late Academician Eduard Hála.

A new method of the absolute low gas flow measurement was developed. The method is based on the comparison of the known rate of a piston movement in a calibrated cylinder with the measured gas flow rate. Due to its compensating character, the method is extremely sensitive, and the relative error is given merely by the sensitivity of determining the pressure difference between the cylinder and atmosphere. The method is absolute as the apparatus constant is determined by such operations as weighing and frequency measurement.

Most of the existing devices for measuring low gas flow rates require calibration against a primary standard. Precision bore cylinders equipped with passive or active moving pistons are used as standards. The most popular devices with passive pistons are the soap-bubble flow meter[1] and the Porter flow-rate calibrator[2].

The bubble flow meter, commonly used in gas chromatography, suffers from a series of inherent errors which make this device rather a semiquantitative flow indicator than a precise flow meter (diffusion of the measured gas through the soap bubble, incomplete saturation by water vapor, unknown and variable soap film thickness on the cylinder wall).

The analysis of the disadvantages of the bubble flow meter has led to the design of the Porter gas flow calibrator, where the soap buble has been changed for a mercury sealed plastic piston. Both the mass of the piston and friction manifest themselves in a backpressure and prevent the flow-rate measurement at atmospheric pressure.

A calibrator with an active piston recalling a reciprocating pump has been proposed by van Swaay[3]. Later Noble et al.[4] described a calibrator based on a syringe driven by a motor through a magnetic clutch. A diaphragm with an adjustable switch was located at the syringe inlet and controlled the clutch. The device measured the average volume flow rate as a time interval necessary to fill the syringe by the measured gas. Unfortunately, the transient processes preceding the steady state characterized by a balance between the flow rate and the rate of the piston movement damage the precision of this apparatus.

In order to automate the calibration of the electronic flow controllers[5], a new method of flow measurement based on the active piston has been developed. This method eliminates the influence of the transient processes on the flow-rate measurement by determining the piston steady rate instead of measuring the time interval necessary to fill a known cylinder volume.

Principle of the Method

The method is based on the comparison of the known piston rate with the unknown gas flow rate. Let a piston move steadily in a calibrated cylinder; the volume of the cylinder is then

$$V = V_0 + at ,$$

where V_0 is the cylinder dead volume and a is the rate of the volume increase. Let further a constant gas flow be introduced isothermally into the cylinder; it holds

$$A = \boldsymbol{R}T \frac{\mathrm{d}n}{\mathrm{d}t} = P \frac{\mathrm{d}V}{\mathrm{d}t} + V \frac{\mathrm{d}P}{\mathrm{d}t} = aP + (V_0 + at) \frac{\mathrm{d}P}{\mathrm{d}t} , \qquad (1)$$

where P is absolute pressure in the cylinder, n is the gas flow rate expressed in molar units, $\boldsymbol{R}$ is gas constant and T thermodynamic temperature. By solving Eq. (*1*), assuming that $P = P_0$ at $t = 0$,

$$\frac{A/P_0 - a}{a} = \frac{P - P_0}{P_0} \left(1 + \frac{V_0}{at}\right) . \qquad (2)$$

The left-hand side of Eq. (*2*) expresses the relative difference between the measured flow rate and the piston movement. Evidently, the relative flow-rate difference equals that of pressure, provided that V_0/at is sufficiently small. There is no problem to measure the pressure difference with a high degree of accuracy using present semiconductor pressure sensors; similarly, the rate of piston movement can simply be controlled by a step motor fed by a precise X-tal clock.

Further improvement of the method can be achieved by the introduction of a feedback link between the pressure in the cylinder and the frequency of the step motor. Consequently, the frequency of the step motor is determined by the pressure controller maintaining a constant (atmospheric) pressure in the cylinder. In the steady state the piston moves at the rate corresponding to the flow rate measured; the difference is given merely by the sensitivity of the pressure sensor.

A block diagram of the calibrator–flow controller system is in Fig. 1. The system consists of a pair of controllers of which one, the flow controller, operates independently. The calibrator starts its operation after switching to the gas flow source.

As the properties of the source (flow controller) are given, the properties of the whole system can be changed exclusively by changing the properties of the calibrator.

A detailed diagram of the calibrator is in Fig. 2. The system displays a variable paarmeter V; its change is given by the piston motion. To control pressure in the cylinder, a PS controller has been used. This solution does not optimize the transient phenomena, but it makes the system insensitive to the changes of system parameters.

Figs 3–5 illustrate the main transient phenomena in the system. Fig. 3 represents the pressure response to a unit flow-rate step (a constant difference between the rate of the piston movement and the gas flow rate). Figs 4 and 5 demonstrate the responses of the flow controller valve and of the calibrator pressure sensor, respectively, to

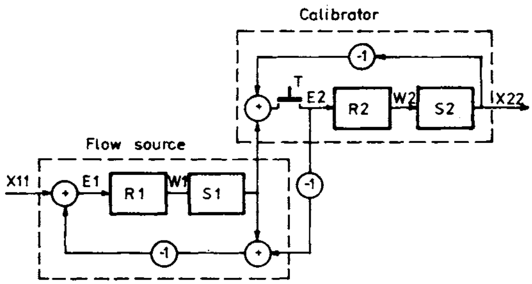

FIG. 1

Block diagram of the calibrator: flow controller system. X11 flow source set value, X22 flow rate generated by the piston, R_1 controller, S_1 controlled system

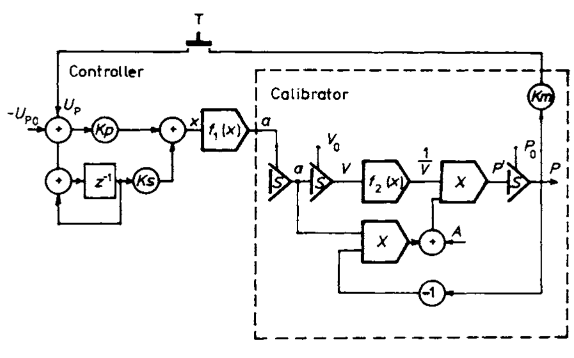

FIG. 2

Detailed diagram of the calibrator. For the controller: $f_1(x) = x$; $x \in \langle 0, x_1)$, $f_1(x) = x_1$; $x \in \langle x_1, \infty)$, $f_1(x) = 0$; $x \in (-\infty, 0)$. For the calibrator: $P' = -(aP + A)/(V_0 + at)$, $f_2(x) =$ $= 1/x$

the instant connection of both apparatus. The large overshoot at he beginning of the control process is due to the discontinuous character of the step motor and/or due to a small initial volume V_0.

It is possible to improve the transient processes by increasing the initial volume of the cylinder; the consequence would be, according to Eq. (2), a decrease in the measurement sensitivity.

The calibrator represents a nonlinear sampled system. To apply the Popov stability theorem to such a system, significant simplifications have been made based on the similarity between the experimentally determined transient characteristics of the calibrator and a PD term. The input signal is the $A/(aP_0)$ ratio; for the stability determination the ratio has been changed for the difference $A/P_0 - a$. The absolute stability conditions for the approximated system are

$$Kp > 0\,, \quad Kp > |Ks|$$

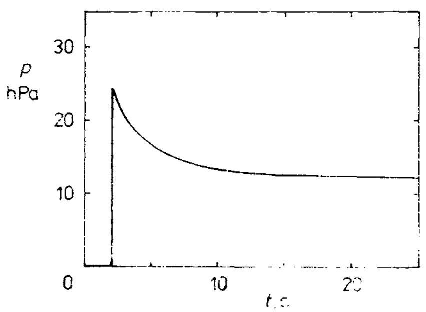

FIG. 3

Pressure response to a unit flow-rate step

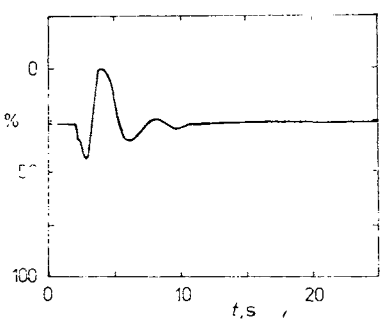

FIG. 4

Response of the flow controller valve to the instant connection of both apparatus

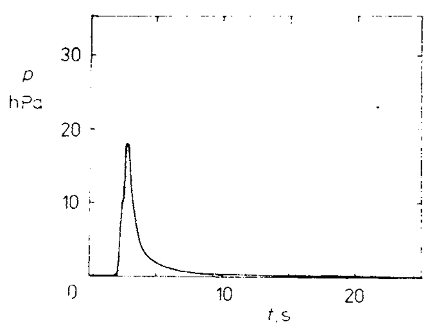

FIG. 5

Response of the calibrator pressure sensor to the instant connection of both apparatus

The results of the absolute stability calculations confirmed the experimental results displaying high stability ot the system.

Calibrator Design

The flow-rate calibrator is formed by a precision bore borosiliate glass cylinder containing a moving piston driven by a step motor. The cylinder is equipped with an inlet tubing, a solenoid valve leading to the atmosphere, and a semiconductor tensometric pressure sensor. At the external cylinder surface a flat platinum thermometer is located and covered by insulation.

The movement of the piston is accomplished by three pull rods; two of them are bridged by a nut holder. The nut is guided by a rotating guide screw supported in ball bearings. The guide screw is driven by the step motor (SMR 300 – 600) through a gear box (1 : 5).

The electronics of the calibrator is built up around the 8080A microprocessor and contains the programmable peripheral interface (8255A) and the programmable interval timer (8253). The analog part is formed by amplifiers normalizing signals produced by the pressure and temperature sensors, by a 2-channel differential multiplexer, by a dual-slope A/D converter (AD2020), and by the necessary circuitry for the step motor control based on the Moore automate (74188 ROM). One of the 8253 counters is wired as a programmable frequency generator and is fed by the processor X-tal clock (approx. 1 MHz).

Flow Rate Measurement

The flow-rate measurement consists of a series of consecutive actions controlled by the microcomputer. At the beginning of each measurement, the inlet valve is opened and the piston is in its initial position marked by a microswitch. The cylinder volume (inclusive the volumes of inlet tubing) is V_0. In this state the offset value of the pressure sensor is stored. After closing the inlet valve the programme of the PS controller is activated. The controller operates with a frequency of 100 Hz and the steady state, characterized by the zero pressure difference between atmosphere and the cylinder, is reached within a few seconds. After a preset time delay, 512 values of the time intervals between individual motor steps (sent to the programmable interval timer) are stored and averaged. The final period starts with the opening of the inlet valve and continues with the piston return to its initial position. During this period the cylinder temperature is measured and stored.

The cylinder volume corresponding to one motor step has been determined by repeated weighing distilled water pushed out of the cylinder by a known number of motor steps. The basic X-tal frequency fed to the timers has been measured by a precise lab counter.

The calibrator programme is written as an assembly subroutine returning flow rate and temperature to the main programme. The main programme, written in FORTRAN, automatically changes the flow controller settings, measures the actual flow rate and calculates calibration constants for each flow controller, prepares a punched tape for EPROMs, and prints full calibration report.

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Translated by the author (S.W.).