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A FLOW METER FOR PRECISE AND ACCURATE MEASUREMENT OF GAS FLOW USING A TRAVELLING MERCURY DROP

JAN ÅKE JÖNSSON
Department of Analytical Chemistry, Chemical Centre, University of Lund, S-220 07 Lund 7 (Sweden)

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SUMMARY

A flow meter for the accurate measurement of gas flow and for the calibration of other flow meters is described. It is analogous to the soap-film flow meter, but the use of mercury instead of soap solution eliminates many of the problems encountered with soap-film flow meters. Thus, accuracy and precision are improved; the over-all uncertainty is calculated to be 0.07%.

INTRODUCTION

A high-precision gas chromatograph that has been constructed in our laboratory[1] utilizes a thermal flow sensor (Brooks Instrument, Emerson Electric, Veenendaal, The Netherlands; Model 5910), which, in order to attain high accuracy, must be frequently recalibrated. The classical instrument for measuring gas flow in gas chromatography is the soap-film flow meter, the accuracy of which has been evaluated by Levy[2] to be 0.25% (with certain precautions). This figure could perhaps be improved if an electro-optical system actuating an electrical timer were substituted for the observer's eye and stopwatch. However, the question of water vapour saturation poses severe problems[2].

The gas to be measured is usually dry, but, in a soap-film flow meter, it becomes more or less saturated with water; thus, when the pressure of the gas is determined, a correction for the partial pressure of water must be applied. At room temperature, this pressure is about 24 mmHg (about 3% of the total pressure) for pure water. However, as the water is not pure, but contains some "soap", its partial pressure is reduced. Additionally, the gas must be saturated with water vapour; procedures for achieving this have been described by Levy[2].

A second problem arises from the possibility of there being a film of water inside the tube; this film can change the volume actually occupied by the gas from that originally determined by a weighing procedure. Additonally, Czubryt and Gesser[3] have presented results indicating that the soap film is, in some instances, permeable to the gas.

These problems, although perhaps possible to overcome, can be by-passed by using mercury instead of a soap solution. The vapour pressure of mercury, at room temperature, is about $2 \cdot 10^{-3}$ mmHg, which is negligible; further, mercury does not wet glass, so that no film will be deposited inside the tube.

APPARATUS

Our instrument consists of an almost horizontal glass tube, approximately 1 m long and of I.D. 4 mm, provided with a thermostating jacket (see Fig. 1). Two "light reflection transducers" (Farchild FPA 103) define the ends of the measuring volume.

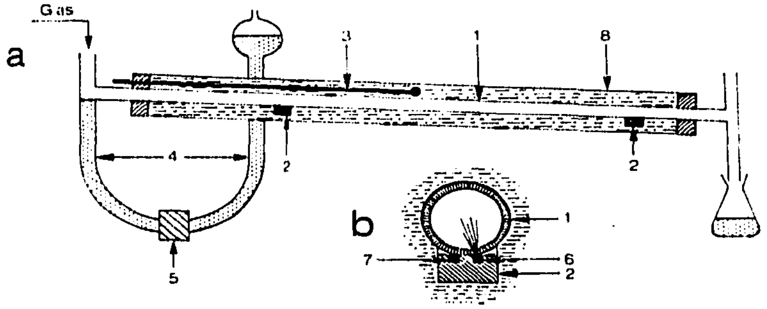

Fig. 1. (a) General view of mercury flow-meter. (b) Section through the inner tube at one of the sensors. 1 = Glass tube; 2 = photoelectric sensors; 3 = thermometer; 4 = tube containing mercury; 5 = solenoid valve; 6 = light-emitting diode; 7 = phototransistor; 8 = thermostatically controlled heating jacket. (Electrical connections are not shown).

Each of these components contains one light-emitting diode and one phototransistor to receive reflected light, and two such components are cemented to the outer wall of the tube approximately 800 mm apart. The tube is painted black around each sensor, and an insulated electrical lead is connected, the connections being covered with Araldite to provide insulation from the water in the jacket. The electrical connections are shown in Fig. 2.

A drop of mercury is brought into the tube by means of a solenoid valve when the computer issues the signal B in Fig. 2; a simple timing circuit determines how long the valve will stay open. The drop travels through the tube, forced forward by the gas,

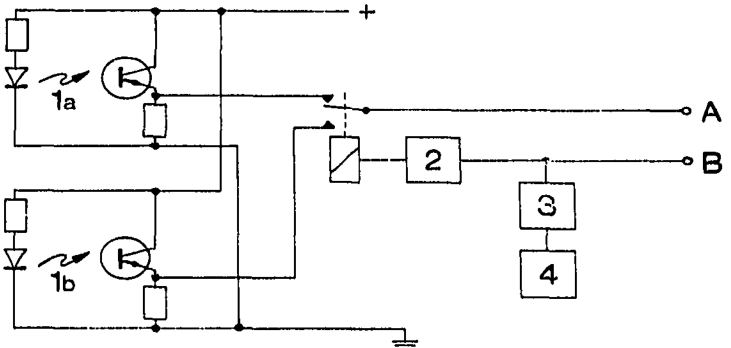

Fig. 2. Electrical connections. 1a, 1b = Photoelectric sensors (FPA 103); 2 = relay driver; 3 = timing circuit; 4 = solenoid valve for mercury admission.

and when it covers the first light-emitting diode, the light is reflected and a signal is produced by the phototransistor (see Fig. 3a). As the mercury drop passes through the tube, the process is repeated at the other sensor, and the drop is finally collected in a receiver.

In our equipment, the signals from the photoelectric sensors are fed to a computer, which calculates the time for the drop to pass through the measuring volume, the computer program defining the start- or stop-point as the inflexion point of the tailing edge of the signal. The function of this program can be checked by means of an oscilloscope, as the computer generates a special signal during the time-measurement period (see Fig. 3b). The error in time measurement is estimated to be 1 msec.

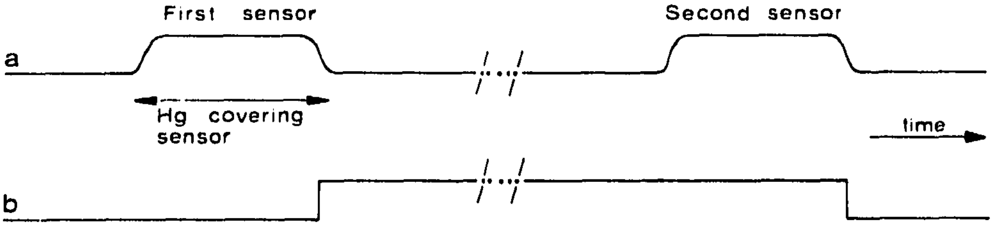

Fig. 3. Electrical signals.

Temperature and pressure are measured by means of a platinum-resistance thermometer and a capacitive pressure sensor (Rosemount Engineering, Bognor Regis, Great Britain; Model 830A-7). The measurement is governed by the computer, which takes a reading every 4 msec and derives the respective means after the run (for details of this process see ref. 4).

When the drop travels through the tube, the pressure is increased by friction. This effect can be compensated for if the tube is not exactly horizontal, but has a small downward slope. A U-tube manometer can be inserted to find the slope, which corresponds to minimal pressure difference across the mercury drop. With a flow of about 50 ml/min, the difference is about 1 mmHg if the tube is horizontal, but it can be reduced to less than 0.1 mmHg by tilting the tube. The size of the mercury drops apparently has no significant effect on the pressure difference. In our work, making a continuous measurement of pressure, small variations in pressure are accounted for.

CALIBRATION OF VOLUME

The volume of the tube between the two sensors is calculated by weighing mercury. The tube is placed vertically, and an arrangement of two vacuum-stopcocks is connected to the lower end. One of the stopcocks connects the tube to a mercury container, from which it can be filled, and the other stopcock acts like the stopcock on a conventional burette. A voltmeter is connected to the signal output from the phototransistors, and the tube is filled with mercury covering the photoelectric sensor; a high voltage is observed. If the mercury is lower than the sensor, a smaller voltage is observed, and, with care, the mercury level can be so adjusted that the observed voltage is the mean of the two previous readings. In this way, very sharp "end-points" are obtained. The mercury contained between the two sensors is collected and weighed, and the volume of the tube can then be determined in the usual way. The uncertainty in volume measurement is estimated to be less than 0.04%.

CALCULATION OF ERROR

The mass flow, F, of gas through the tube is given by

$$F = \frac{p \cdot V}{t \cdot R \cdot T} \tag{1}$$

where p is the pressure, V is the volume of the tube, t is the time to pass through this volume, R is the gas constant, and T is the absolute temperature. The systematic errors of the various parameters in a typical instance are summarized in Table I. In this way, the systematic error in F is estimated to 0.05% $[(0.02^2 + 0.04^2 \cdot 0.01^2 + 0.02^2)^{\frac{1}{2}}]$.

TABLE I

SYSTEMATIC ERRORS

Parameter	*Unit*	*Typical value*	*Absolute error*	*Relative error (%)*	*Reference*
p	mmHg	760	0.15	0.02	4
V	ml	10	0.004	0.04	Present work
t	sec	10	0.001	0.01	Present work
T	°K	300	0.05	0.02	4

To evaluate the precision of the device, it must be appreciated that an instrument of this kind will make some disturbances in the flow when the mercury drop is introduced, thereby making it difficult to obtain a sufficiently stable flow-rate. In our system, the reading from the mercury meter is compared with the reading from the thermal flow sensor, and the following measurements refer to the quotient between mercury-meter reading and thermal-sensor reading. Repetitive measurements show a standard deviation of 0.03%. Thus, for 10 determinations, the over-all uncertainty will be 0.07%, which is the systematic error plus the half the width of the 95% confidence interval for the mean $(0.05 + 0.03 \cdot 1.96/\sqrt{10})$.

DISCUSSION

This work was restricted to a flow meter for use with flow-rates normally encountered in gas chromatography, *i.e.*, 10–100 ml/min; for other flow-rates, tubes of other sizes must be used. We have not investigated the maximum and minimum diameters for adequate functioning outside the flow-rate range of interest to us. To use the principle of weighing mercury as described above, it is convenient that the tube be straight and not much more than 1 m in length.

As mentioned above, our instrument forms an integral unit for the calibration of a thermalflow sensor in a larger system. We believe that it is possible to connect this instrument to an electrical timer and to use a conventional barometer to measure the pressure without seriously affecting the accuracy. If the instrument is used for direct measurement of flow-rate, flow disturbances caused by the flow meter will decrease the accuracy. Experiments with the flow meter connected to a conventional gas chromatograph resulted in a standard deviation of 0.1%. The effect of the mercury drop on the flow-rate gives rise to a systematic error, and this depends on the tilt of the flow-meter tube. It is easy to adjust the tilt so that the systematic error is less than 0.05%.

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