Notes

The design of an automatic absolute flow meter for gas chromatography

The accurate measurement of gas flow and gas flow rates may be regarded as one of the outstanding technical problems in gas chromatography. Absolute values are needed when retention data are to be converted into partition coefficients or other thermodynamic data, and the accurate reproduction of flow rates is a vital requirement for the identification of components by means of retention indices. In large-scale chromatography, the programming of the trapping cycle on the basis of gas flow, rather than time, appears attractive.

This paper concerns the design of a flow meter which is essentially an automatic gas burette*.

The gas pressures at the inlet and outlet of the burette are maintained equal by a feedback loop consisting of a differential manometer and a servo amplifier and motor (Fig. 1). When the outlet of the burette is open to the atmosphere, the complete system acts as a metering pump, pumping gas into the atmosphere without changing its pressure. Two outputs are available:

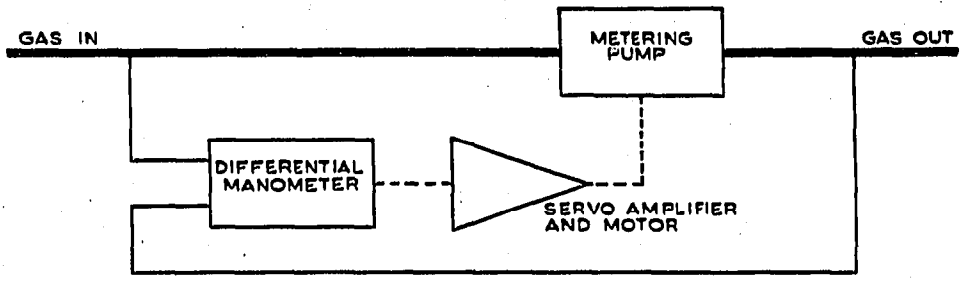

Fig. 1. Block diagram of flow meter.

(1) The number of revolutions of the motor shaft is proportional to the volume pumped through; this will be called the integral output.

(2) The rotational speed of the motor shaft is proportional to the flow rate of the gas; this will be called the differential output.

Description of the instrument

A practical design of the instrument is shown schematically in Fig. 2. The burette consists of a reciprocating piston moving in a cylinder with a net gas volume of, *e.g.*, 50 ml. Movement of the piston is effected by rotation of the drive shaft, which is threaded so that one stroke of the piston corresponds to 100 revolutions of the drive shaft. The revolutions are counted by a counter actuated by a switch and cam on the drive shaft, and the decade transfer on the counter is used to reverse the operation

* Construction is under progress of a prototype instrument, with which the author hopes to be able to obtain actual performance data.

of the burette. The gas connections are changed by means of the two 3-way valves shown, and the drive direction of the piston is changed by means of the bevel gears and the magnetic clutches. Thus the motor always runs in the same direction, and its speed may be used as a differential output.

The manometer is a differential capacitor with a movable centre plate. Similar manometers have been described in the literature[1-3]; these were powered by high-frequency energy, and the movable plate was maintained in the centre position by application of a d.c. potential. The applied d.c. potential is then a measure of the pressure difference across the membrane. The associated electronic equipment can be

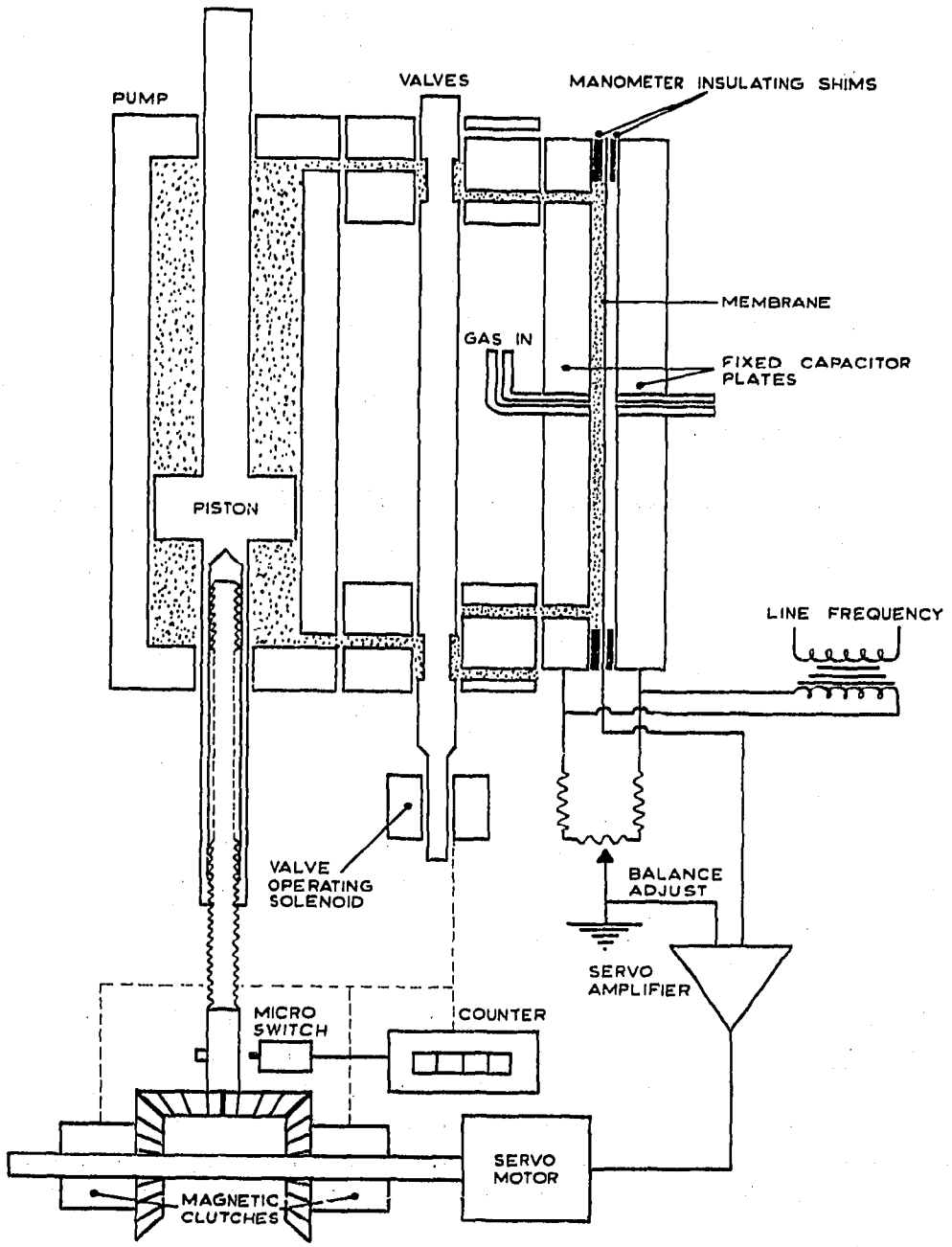

Fig. 2. Schematic representation and lay-out of flow meter.

stable and reliable, and such manometers have a high sensitivity. A different circuit, however, is indicated in Fig. 2. An attractive feature of this circuit is that the signal at the transducer has a frequency, amplitude and phase angle which can be accepted as input by the power amplifier. Proper shielding is necessary, because the source impedance of the transducer is of the order of 20 MΩ, and a simple preamplifier is needed when a servo system with low input impedance (*e.g.* the Honeywell-Brown continuous balance system) is used.

Drift and random errors

When care is taken that the servo system operates in the proportional region, *i.e.* that the amplifier is not driven to saturation, the main causes of error are temperature fluctuations of the burette and frictional changes in the drive system.

The Honeywell-Brown continuous balance system is driven to saturation with an a.c. input of about 500 μV on the grid of the first tube. Calculations have been given for the actual capacitance changes for a properly flexed membrane[1]. For the present purpose this is not necessary; we may obtain an estimate of the output signal by assuming that 10 % of the surface has the maximum displacement calculated by means of the equations given by ROARK[4], and that the remaining surface stays at the central position. With this approximation we may calculate that a pressure difference of 1 mm H_2O across a stainless steel membrane of 100 μ thickness and 40 mm diameter produces an output voltage of 50 mV, when the fixed plates are mounted with 75 μ spacing and driven at 100 V. Thus a pressure change of 10 μ H_2O will produce saturation of the amplifier. The change in volume of the manometer corresponding to this pressure amounts to *ca.* 1 μl; this is the maximum random error in the integral output caused by frictional changes in the drive system.

Temperature fluctuations will produce random errors with a magnitude $V\Delta T/T$, where T is the absolute temperature, and V the gas volume contained in the burette. At room temperature this amounts to 0.16 ml/°C, when the burette volume is 50 ml. This value is reduced to about 0.1 ml/°C at 250°.

Drift in the integral output can be caused by leakage of gas past the piston or through end seals and valves. For the equipment under consideration, greaseless bearings of metal on metal can be made with clearances of 10 μ or less. If the piston has a length of 1 cm and a circumference of 10 cm, the leak rate will be 10^{-4} ml H_2/h at the maximum pressure difference that may exist in practice. Smaller values are obtained for the end seals and the valves, so that drift caused by leakage should be entirely negligible. A change in the temperature would of course result in a change in the integral output. Changes of the outlet pressure do not affect the volume indication of the instrument, but the weight of the gas delivered per stroke will depend on the pressure.

The switching at the end of a stroke may produce transients in the integral output, but the system will "catch up" with the gas flow in a very short time, after which the output will again represent the true integral.

Noise in the differential output can be derived from the noise in the integral output; it depends, of course, on the frequency of the noise sources and the pass band of the feedback system.

Systematic errors can arise from nonlinearity of the thread on the drive shaft, and deviations from the nominal volume of the burette will give rise to a systematic

absolute error. Both errors depend entirely on mechanical factors; they can be held to very low values, and corrections may be made by means of calibration.

Use of the flow meter as a detector

It is interesting to consider the potential usefulness of the flow meter as a gas chromatographic detector. When the system is connected to a gas chromatograph, the integral output curve may have the shape shown in Fig. 3. When a sample is injected, there will be a peak, as the sample vaporizes and then partly dissolves in the stationary phase. This peak may be partly or completely damped out by the filtering action of the column. Under conditions of linear chromatography the volume

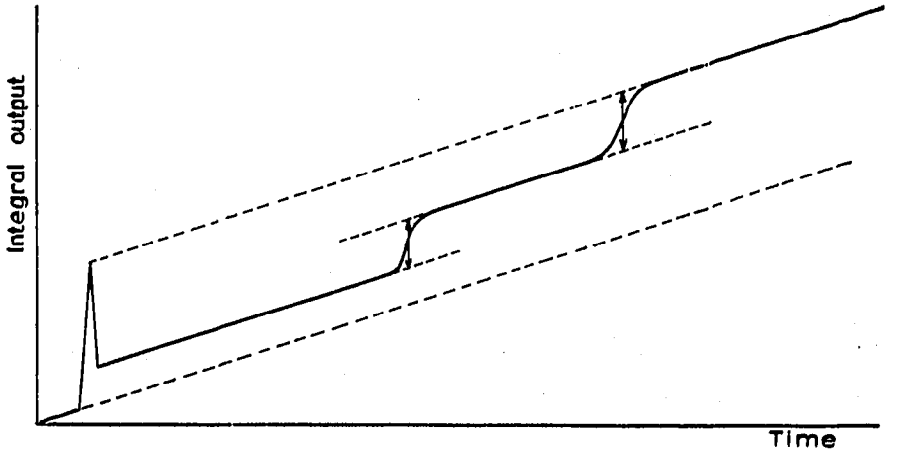

Fig. 3. Theoretical response of flow meter detector for a 1:1 mixture of 2 components having capacity ratios of 2 and 4.

of the sample plug in the gas phase then remains constant as the sample moves through the column. At the column exit, however, the dissolved fraction of the sample re-enters the gas phase, causing a step in the curve with a height proportional to the amount of sample multiplied by $k'/(1 + k')$, where k' is the capacity ratio.

The entire sample should remain in the gas phase while it passes through the flow meter; this condition is met when the instrument is heated to the temperature of the column, and when no adsorption or absorption takes place in the system. As was stated earlier, it is technically feasible to construct the essential parts out of metal, without need for lubrication. The proper choice of alloys will depend on the type of sample to be handled.

The precision and sensitivity of the instrument should be adequate for detection of samples emerging from conventional packed columns. The flow of gas through the column may vary, however, because the presence of the sample affects the viscosity of the gas. In addition, there may be random fluctuations in the flow of carrier gas. Both effects will cause noise in the detector output, which may limit the sensitivity attainable in practice. Where this limit will lie depends to a large extent on the amplitude and frequency of the noise, neither of which can be easily estimated from theoretical considerations.

By means of the arrangement shown in Fig. 4, an output may be obtained which directly represents the sample content of the gas entering the system. Two flow meters are connected in series, with a trap mounted between the two. The first flow meter is

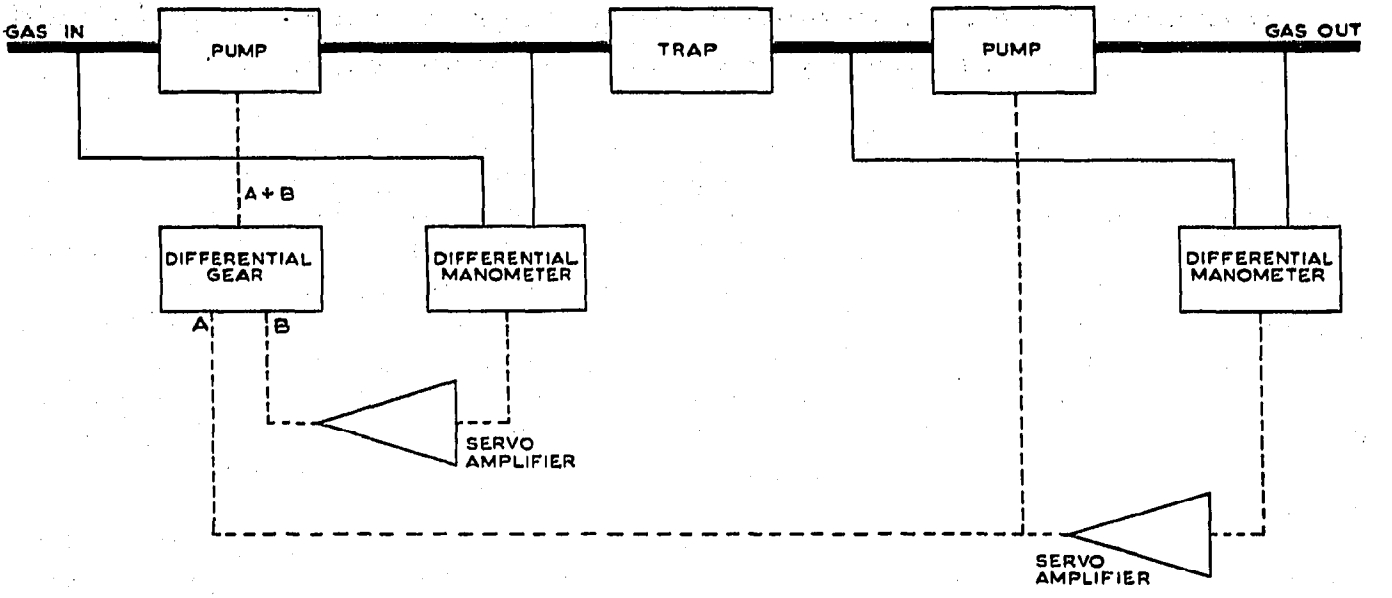

Fig. 4. Schematic diagram of a detector with an integral output directly proportional to the number of moles of sample contained in the incoming gas. Integral outputs: A = pure carrier gas; B = total sample, A + B = total gas flow.

driven via a differential gear, one input of which is driven with a speed corresponding to the flow of the pure carrier gas. When the incoming gas contains a sample component, the first flow meter will lag behind by an amount corresponding to the volume of the sample vapour.

This lag is sensed by the associated manometer, which will cause a correction signal to be given to the flow meter via the second input of the differential gear. Three integral outputs are now available: the flow of pure carrier gas is represented by the signal at A, the sample content at B, and the total gas flow at A + B. It should be noted that this arrangement represents an in-line detector with truly integral characteristics. In addition, the response should be a linear function of the number of moles of sample, when ideal mixing in the gas phase is assumed.

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