## Title

IMPROVED ACCURACY IN MEASUREMENT OF LOW GAS FLOW RATES WITH A BUBBLE METER



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One of the major factors determining the sensitivity of a gas chromatographic apparatus utilizing thermal conductivity detectors is the flow rate of the carrier gas. As the sensitivity is inversely proportional to carrier gas flow rate, a 1% change in flow rate is reflected in a 1% change in sensitivity in the opposite direction. If control of sensitivity of a gas chromatograph to ±0.1% is to be achieved, the gas flow rate must be measured to better than 0.1%.

Recently Noble, Abel and Cook (4) have described an electromechanical device for absolute measurement of low flow rates, but the usual method of determining low flow rates is by use of a bubble flowmeter and a stopwatch. Levy (2) has discussed the experimental parameters of the bubble method and analyzed the errors inherent in each parameter.

The experimental variables in the bubble meter are 1) the time of rise of the bubble between two fixed marks, 2) the volume between the marks, 3) the temperature of the gas, 4) the pressure of the gas, and 5) the degree of saturation of the gas with water vapor. As analyzed by Levy, by careful design of the apparatus, close control of temperature, accurate measurement of the pressure, and pre-saturation of the gas, each of these variables, except time, can be reduced to an uncertainty of less than 0.02% to 0.04%.

The measurement of time is the most difficult to control. By use of a stopwatch, reproducibility to ±0.2 sec. with one observer is good and with different observers it varies as much as ±0.4 sec. These errors were cut in half when an electric timer (6) with a one second sweep hand and controlled by an external microswitch was substituted for the stopwatch. The basic problem appears to be in making reproducible decisions as to when the bubble crosses the index mark and in achieving reproducible reaction times.

For gas chromatographic use, the time interval of interest is usually between 10 sec. and 120 sec. for displacement of a volume of 50 ml. Thus the error in time varies from 1 to 2% for the short interval to 0.1 to 0.2% for the longer interval. Most of the work we have encountered involves flow rates of 40 to 120 ml./min. where the timing errors are 0.3% to 0.8%.

In order to reduce timing errors to less than 0.02%, an electronic timer has been devised which operates on the principle that the fixed marks on the gas burette are replaced by two 0.25 mm. (10 mil.) diameter light beams across the burette. Interruption of the lower light beam by a bubble turns on a scaler which counts pulses from a 100 KC generator and interruption of the top beam turns off the scaling circuit. Thus times can be measured to ±0.00001 seconds (or ± one count of the 100 KC frequency). Operator bias is also removed since the decision as to when the bubble crosses the index mark is determined by scattering of the light beam by the bubble, and is hence made in a reproducible manner at both the top and bottom index marks.

A block diagram of the apparatus is shown in Fig. 1. The gas entered the thermostated flowmeter (±0.01°) through a thermostated presaturator. A bubble is formed in the usual way by means of a rubber bulb, containing a

bubble forming liquid. "Snoop" (5) was used as the bubble forming liquid as it gave very uniform bubbles which retained the same degree of flatness as they progressed up the tube. At the lower detector, the bubble interrupted the light beam reducing the output of the 2N 2175 photocell. This change in photocell output was amplified and fed to a trigger forming circuit which generated a start pulse. This start pulse activated the electronic switch and gated on the decade scaling circuit. The scaling circuit then counted the pulses coming from a crystal controlled 100 KC pulse generator.

The bubble traveled up the tube and passed the upper light beam. This generated a stop pulse in the same manner as the start pulse was generated. The electronic switch gated the scaler off. The time for the bubble to travel between the two light beams was then read directly off the scaler.

The volume between the light beams was calibrated by 1) the filling with water and weighing the water collected between the interruption of the upper light beam and interruption of the lower light beam, and 2) filling with mercury and weighing the mercury collected in the same manner. The water collection was run as a function of drainage time. For a volume of 44.50 ml., the difference between water and mercury calibrations was 0.10 ml. (1 min. water drainage) and the water volume increased with increase in drainage time. Thus for ultimate absolute accuracy, the volume must be calibrated to take account of the liquid film thickness on the gas burette at various rise times of the bubble, this amounting to approximately a 0.05 ml. difference between the volume collected in 1 minute and 3 minutes drainage times respectively, i.e., about 0.1% difference.

Typical results for a very fast flow, a medium flow, and a low flow are shown in Table I. The fast flow was regulated by the flow from a helium tank fitted with a 2-stage pressure regulator at 40 p.s.i.g., a ballast tank and a temperature insulated Nupro Double Pattern Metering Valve (5). The medium and low flow rates were through a Moore flow regulator (3) (temperature insulated) with the pressure drop controlled by the same type Nupro valve as above. The room was thermostatically controlled to $25\pm0.5^{\circ}$.

The results in Table I show that over most of the range from 20-500 ml./min. the reproducibility is better than 0.01%. The break to greater deviations ~0.1% comes at about 30 ml./min., and this may be a failure in operation of the flow regulator rather than in the bubble flowmeter.

This flowmeter has been used, not as a working measure of the flow rate, but rather as a primary standard to calibrate the working meters, Hastings Flowmeters Model LF-100 (1). With suitable precautions regarding bias and zero settings, the output of these latter flowmeters when measured on a sensitive potentiometer, was found to be reproducible to $\pm0.10\%$ between flow rates of 30 and 250 ml./min., and the calibration has held for a period of six months.

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Table I. Reproducibility of Bubble Meter Measurements Using Electronic Timing

| | Nominal Flow-520 ml/min.* | | Nominal Flow-62 ml/min.* | | Nominal Flow-22 ml/min.* | |
|---|---|---|---|---|---|---|
| | sec./44.50 ml | Δ from average | sec./44.50 ml | Δ from average | sec./44.50 ml | Δ from average |
| | 5.12399 | 0.00077 | 42.9710 | 0.0028 | 118.8041 | 0.2269 |
| | 5.12340 | 0.00018 | 42.9652 | 0.0030 | 119.0687 | 0.0023 |
| | 5.12372 | 0.00050 | 42.9693 | 0.0011 | 119.2625 | 0.1915 |
| | 5.12381 | 0.00059 | 42.9610 | 0.0072 | 119.2847 | 0.2138 |
| | 5.12344 | 0.00022 | 42.9650 | 0.0032 | 119.1149 | 0.0439 |
| | 5.12296 | 0.00036 | 42.9664 | 0.0018 | 118.9133 | 0.1577 |
| | 5.12305 | 0.00017 | 42.9732 | 0.0050 | 118.8973 | 0.1737 |
| | 5.12240 | 0.00082 | 42.9729 | 0.0047 | 119.0092 | 0.0618 |
| | 5.12260 | 0.00062 | 42.9705 | 0.0023 | 119.1758 | 0.1048 |
| | 5.12283 | 0.00039 | 42.9677 | 0.0005 | 119.1793 | 0.1083 |
| Average | 5.12322 | 0.00045 | 42.9682 | 0.0032 | 119.0710 | 0.1324 |
| % Average Deviation | | 0.0088 | | 0.0074 | | 0.110 |
| % Maximum Deviation | | 0.016 | | 0.017 | | 0.224 |
| Flow Calculated | 521.157 ± 0.046 | | 62.139 ± 0.005 | | 22.444 ± 0.025 | |
| Flow STP (dry) | 450.131 ± 0.040 | | 53.670 ± 0.004 | | 19.368 ± 0.022 | |

*10 consecutive measurements at nominal flow.

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(1) Hastings-Raydist, Inc., Hampton, Virginia.

(2) Levy, A., J. Sci. Instr. 41, 449 (1964).

(3) Moore Products Co., Philadelphia, Pennsylvania.

(4) Noble, F. W., Abel, K., and Cook, P. W., Anal. Chem. 37, 1631 (1965).

(5) Nuclear Products Co., Cleveland, Ohio.

(6) Standard Electric Time Co., Springfield, Massachusetts.

FIGURE CAPTION

Fig. 1. Schematic diagram of apparatus for electronically measuring displacement time in a bubble flowmeter.