

cm) were used with the Kawerau apparatus. The $M/5$ test solutions of SnCl_2 and SbCl_3 for the experiments given in Tables I–IV were prepared as follows:

1.89 g of SnCl_2 and 2.28 g of SbCl_3 crystals were dissolved in 15 ml of conc. HCl separately and the solutions were then diluted to 50 ml with distilled water. The $M/50$ and $M/100$ test solutions were prepared by subsequent dilution with 30% HCl. Two spots (approx. 0.0014 ml) of each of the two test solutions were applied to the test paper with a thin glass capillary. The cations were detected by hydrogen sulphide gas. The room temperature was $26^\circ \pm 4^\circ$.

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- ¹ E. LEDERER AND M. LEDERER, *Chromatography*, 2nd Ed., Elsevier, Amsterdam, 1957, pp. 495–496.
² I. I. M. ELBEIH, J. F. W. MCOMIE AND F. H. POLLARD, *Discussions Faraday Soc.*, 7 (1949) 183;
³ F. H. BURSTALL, G. R. DAVIES, R. P. LINSTAD AND R. A. WELLS, *J. Chem. Soc.*, (1950) 516.
 S. HARASAWA, *J. Chem. Soc. Japan, Pure Chem. Sect.*, 72 (1951) 423; *C.A.*, 46 (1952) 1917.
⁴ G. STEFANOVIĆ, T. JANJIĆ AND R. CRNOJEVIĆ, *Bull. soc. chim. Belgrade*, 20 (1955) 343; *C.A.* 53 (1959) 18727.

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A simple apparatus for keeping the final pressure in gas chromatographs constant

In the chromatographic analysis of gases with the aid of katharometer detectors both the peak height and the retention time depend on the flow rate of the carrier gas; the peak height moreover depends on the pressure in the detector cell. The factors which affect the flow rate of the gas are the pressure before the column, the resistance of the column, and the pressure after the column. The pressure before the column and the resistance of the column can be easily kept constant by using suitable regulating valves and by thermostating the column. The pressure after the column, however, depends on the barometric height. In serial tests, in which the calibration curve is not checked after each measurement, the change in barometric height over a few hours or days may cause considerable deviations from the calibration curve; according to our experience, the peak height varies by about 0.1% upon 1 mm change in barometric height. A diagram of a simple apparatus for keeping the pressure after the column constant is shown in Fig. 1.

The exit of the detector cell 1 is provided with a needle valve 2. A differential pressure gauge 3, which measures the pressure difference between the points 1 and 4, is fitted between the cell and the valve. 4 is a glass bulb with a capacity of approx.

100 ml, which is filled with an oxygen-free gas under a pressure higher than the barometric height to be expected. The limbs of the manometer 3 contain an amount of mercury. When the pressures in 1 and 4 are equal, the platinum contacts 5A and 5B are just above the mercury surface. The contacts are electrically connected by means of two signalling lamps 6A and 6B.

When the pressure in 1 changes, one of the lamps lights up, after which the needle valve can be turned in the corresponding direction of rotation. In this way it is possible, even during fluctuations in barometric height, to maintain a final pressure which does not deviate more than 1 to 2 mm from the pressure in 4.

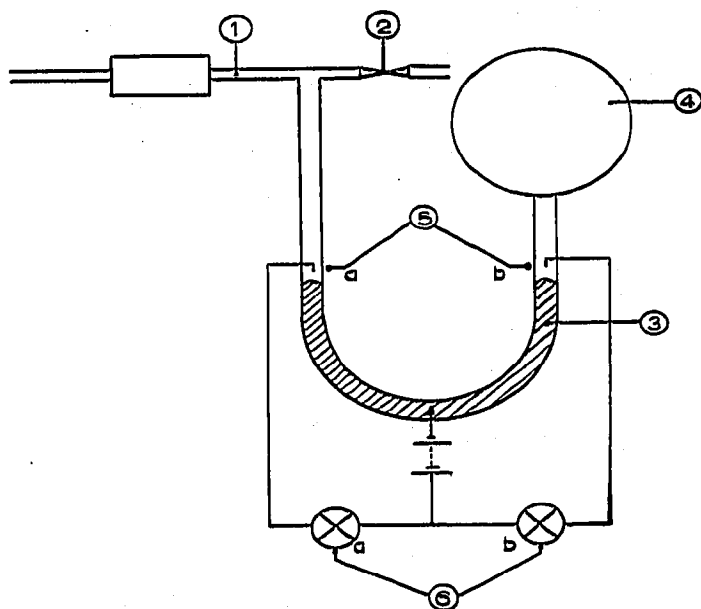


Fig. 1.

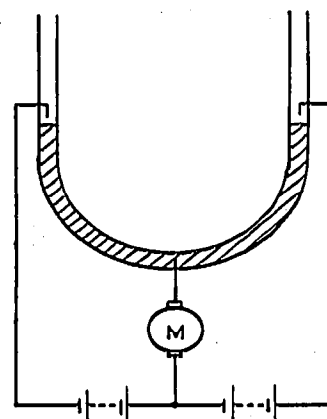


Fig. 2.

Pressure corrections should be made before or after the analysis, because during the analysis the pressure tends to fluctuate owing to surge and viscosity effects. It is obvious that vessel 4 should be accommodated in the thermostated column compartment.

The apparatus could be easily made automatic by connecting it to a reversible d.c. motor in the way shown in Fig. 2. This motor can control the needle valve via a high-ratio reduction gear. By making the adjustment of the valve take place very slowly, the influence of surge and viscosity effects on the valve adjustment can be minimized.

However, there is little point in using this extension in the non-automatic gas-chromatographic analysis, as the apparatus can be quickly and reliably controlled by hand.

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