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ARTICLE *in* ANALYTICAL CHEMISTRY · SEPTEMBER 1974

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To prepare a calibration curve, a measured volume of pure ethylene is transferred into an infusion vial with a gas-tight syringe. Starting from this stock concentration, further dilutions are made in an analogous manner down to the desired minimum concentration. These standard concentrations are introduced into the gas chromatograph as described above.

Gas Chromatographic Conditions:

Instrument: Varian Aerograph, Model 1700
 Detector: Flame ionization detector
 Column: Glass, 100 cm, 2-mm i.d.
 packed with Porapak Q
 Column oven: Room temperature
 Carrier gas: Nitrogen 30 ml/min

Ethylene concentrations in the head space ranging from 0.02 to 2000 ppm (v/v) have been analyzed. If attenuation is adjusted to yield a peak of about half-scale deflection, the relative standard deviation as calculated from 27 duplicate determinations is 0.7% in the range from 0.2 to 2000 ppm (v/v).

ACKNOWLEDGMENT

The kind permission of Varian Aerograph to reproduce Figure 2 is gratefully acknowledged.

RECEIVED for review February 22, 1974. Accepted June 18, 1974.

Constant Pressure Gas Buret

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A constant pressure gas buret is a volume-calibrated container, for the delivery of gas, under a constant pressure, into a vessel or through one or more solutions, with the overcoming of some dynamic and hydrostatic pressures.

A constant pressure gas buret is based upon the principle of the Mariotte bottle. Taylor (1) made use of this when he made a buret for measuring the speed of gas absorption.

The buret shown in Figure 1 and described below, was used in the preparation of gaseous standard solutions (GSS) (2, 3) and also in their use for analytical purposes (4). Air was the gas measured then, but it would be simple to use the buret for other gases.

Let us introduce the following symbols: P : atmospheric pressure; P_b : gas pressure in the buret; P_i : constant hydrostatic pressure imposed by a Mariotte bottle; $P_o = P_b - P$: overpressure of gas in the buret; P_w : sum of the dynamic and hydrostatic pressure drops in the gas flow-line. The constant hydrostatic pressure P_i was achieved between a Mariotte bottle B, filled with a suitable displacement liquid, and an orifice (o) at the top of the buret. In equilibrium, P_o was equal to P_i . Because of this, the displacement liquid cannot descend in the buret. During gas delivery, there was diminution of P_o : the difference in pressure $P_i - P_o$ moved the displacement liquid from the Mariotte bottle to the buret, until equilibrium was restored. The hydrodynamic resistance (i.e., ratio between pressure drop and flow) of the liquid path between the Mariotte bottle and the buret was sufficiently low to give a very small variation of P_o and then of P_b .

Selection and Measurement of the Equilibrium Value of P_o . The Mariotte bottle was placed so that the difference in level between (i) and (o) was such as to produce a P_i somewhat greater than P_w .

Then stopcock 8 was opened (with 1 closed to atmosphere, stopcocks 2, 3, 5, and 6 closed, and 4 and 7 open) and the displacement liquid was made to pass down from B to A until the Mariotte bottle was primed with air bubbling from (i). After that, if the level of displacement liquid in

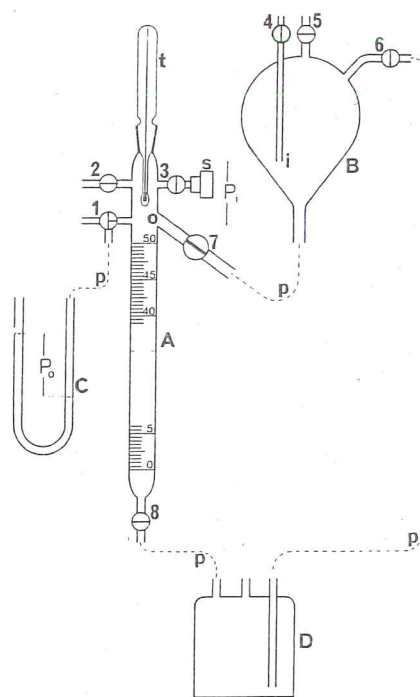


Figure 1. Constant pressure gas buret

A: buret; B: Mariotte bottle; C: open-tube water manometer; D: recovery tank for displacement liquid. 1-8: stopcocks (1-5, all-glass greased stopcocks; 6-8, PTFE-glass stopcocks; 7: stopcock with 15-mm hole length and 4-mm hole diameter, with hydrodynamic resistance to water of 0.02 mm Hg sec/ml); t: thermometer; i: lowest end of the withdrawal tube in the Mariotte bottle; o: orifice for entry of displacement liquid into the buret; s: sleeve with screw for micrometric driving of a gasketed plunger; p: PVC tubes (the tube between B and A was 70 cm long, 0.8 cm in diameter, and had hydrodynamic resistance to water of 0.05 mm Hg sec/ml)

the buret was below zero, by opening stopcock 1 to the atmosphere for a short time, sufficient liquid was made to pass from B in the buret.

The equilibrium overpressure in the buret, $P_o = P_i$, was then accurately read on the open-tube manometer C. The excess of P_o over P_w brought about a pressure drop at stopcock 2 which controls the delivery of gas.

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Zero-Adjusting of the Buret and Setting Up of P_o . Stopcock 7 was closed and stopcock 1 was opened to the atmosphere; then stopcock 8 was opened and the buret was set to 0 line.

To restore the equilibrium condition: $P_o = P_i$, stopcock 1 was closed, stopcock 3 was opened, then the pressure in the buret was increased by operating the gasketed plunger sleeve (s) until the manometer registered that $P_o = P_i$. Stopcock 3 was then closed and stopcock 7 opened. The buret was then ready for use.

Delivery of Gas. Stopcock 2, controlling the delivery of gas, was opened. P_b stayed constant up to a gas flow limit value depending on the hydrodynamic resistance of the tubing between B and A. At the end of delivery the meniscus of the displacement liquid in the buret provided the value of the volume of the gas delivered.

Use of the Buret with Gaseous Standard Solutions. When using the constant pressure gas buret with GSS, gas flow was not controlled by stopcock 2, but by a remote stopcock, not shown in Figure 1. This stopcock was downstream of a solution in which the gas coming from the buret bubbled. In this case, stopcock 2 was opened at the end of selection and measurement of the equilibrium value of P_o . Thus the gas pressure in the gaseous phase above the solution in which the gas bubbled equilibrated with P_i . Stopcock 2 was closed before zero setting to avoid sucking back, and re-opened after P_o had been set-up to the equilibrium value.

Recycling of Displacement Liquid. The displacement liquid could be periodically returned to B from the recovery tank, by closing stopcock 4 and 7 and opening stopcock 6 and sucking in by way of stopcock 5. After this the Mariotte bottle was re-primed. When using the buret with air, a useful displacement liquid was found in a sodium carbonate diluted solution, which avoided the buret walls becoming greasy and reduced errors in measurements.

Performance. With a flow of gas up to 2 ml/sec, the accuracy of the buret was evaluated by measuring the volume of gas delivered by it with the ordinary type of gas buret (5). After correction for difference in pressures, accuracy was proved within the readability of the volume.

The constancy of P_b , with gas flow up to 2 ml/min, was within 0.15 mm Hg.

The constant pressure gas buret, as compared with ordinary burets, has some advantages: possibility of continuous readings of the volume delivered; elimination of having to raise the tank of displacement liquid before each reading; and much easier control of the gas flow. As compared with the Taylor constant pressure gas buret (1), there is the advantage due to the coincidence of the zero of the volume delivered of gas with the zero of the buret. The above described buret has been easier to use with standard gaseous solutions. The constant pressure gas buret enables titrations with GSS to be carried out in open vessels and with continuous readings of gaseous titrant and in this way considerably increases the convenience in using GSS.

The use of the buret for gases other than air would be possible by connecting to a gas source, at a constant known pressure, the openings: of A, by stopcock 1, of B, by stopcock 4, and of D. Before using the buret, the displacement liquid must be saturated with the gas to be measured. With gases having low solubility in water, such as O_2 , N_2 , H_2 , CO , CH_4 , and C_2H_6 , the sodium carbonate dilute solution can be used as displacement liquid. If this does not happen, a suitable liquid must be found (5).

RECEIVED for review January 11, 1974. Accepted April 8, 1974.

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Facile Laboratory Method for Preparation of Weighed Samples of Purified Sodium Metal Sealed under Vacuum

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The reducing properties of the alkali metals, especially when they are dissolved in certain nonaqueous solvents such as liquid ammonia, are well documented (1-5). The purity of the metal, solvent, and cleanliness of the vessels are reported to contribute significantly to the stability of the metal-nonaqueous solutions (6, 7). In our work con-

cerning the reactions of various substrates with sodium in liquid ammonia, it was necessary to devise a facile method to obtain weighed amounts of vacuum distilled sodium metal sealed in glass breakseal tubes (8). Other more elaborate and time consuming procedures have been described elsewhere (9-11).

The apparatus, constructed of borosilicate glass, used in our purification procedure is shown in Figure 1. The main features of this apparatus are a metal still (A), condensing section (E), and tared breakseal tubes. It is constructed in

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