A RAPID METHOD OF DETERMINING CARBONIC ACID IN AIR.

(One Figure in the Text.)

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IN almost all investigations relating to ventilation of inhabited buildings determinations of the carbonic acid present in the air are essential. The method in common use is that of Pettenkofer, or some modification of it. This method has, however, the disadvantage that the determinations necessitate the carrying to and from the laboratory of large bottles, and take a considerable time. To overcome these drawbacks several methods capable of being conveniently applied on the spot have been devised, but their accuracy is hardly sufficient for practical purposes, hence they have not come into general use.

The method now to be described was designed in connection with the work of the Home Office Committee on Ventilation of Factories and Workshops, and the apparatus, the construction of which is shown in the figure, is a simplified form of a gas-analysis apparatus which I described in 1898¹. The practical advantage of the method is that the apparatus can easily be carried about, and is always ready for

¹ *Journ. of Physiol.*, Vol. xxii. p. 465, 1898.

The more important measurements connected with the present apparatus are as follows :—

Internal measurement of case $8 \times 13 \times 3$ inches.

„ „ „, water-jacket $2\frac{1}{2} \times 1\frac{1}{2} \times 7$ inches.

Diameter of bulbs about $1\frac{1}{2}$ inches.

Length of graduated part of burette about 4 inches or 100 mm.

Thickness of wood used throughout = $\frac{3}{8}$ inch.

Weight, including mercury and water, about 6 pounds.

The apparatus has been made for me by Messrs C. E. Muller and Co., 148 High Holborn, London.

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immediate use: that an accurate result can be obtained within about five minutes; and that no calculations are involved.

One-fourth actual size.

The gas burette *A*, which is enclosed in a water-jacket, consists of a wide ungraduated, and a very narrow graduated portion. It holds about 20 c.c. from the tap to the bottom of the scale. The graduated part, which is about 4 inches long, is divided into 100 divisions, each of which corresponds to $\frac{1}{10000}$ th part of the capacity of the burette. The lowest division is marked 0, and the highest 100. Any difference between a reading at or near zero and a second reading is thus shown by the scale in volumes per 10,000.

In using the apparatus the air is first expelled from the gas-burette by opening the three-way tap *B* to the outside, and raising the mercury bulb *C*. The tap is then closed, and the mercury bulb replaced in its stand. On opening the three-way tap again a sample of the air is drawn in, and the level of the mercury falls to near the zero mark. The tap is now opened towards the absorption pipette *D*, which is filled up to a mark at *E* with potash solution, and the sample measured with the precautions to be described below. It is then passed over into the absorption pipette, driven backwards and forwards for a few seconds, and then again measured after the absorption of the carbonic acid. The difference between the two readings gives directly the number of volumes of carbonic acid per 10,000 in the sample of air.

It is evident that the correctness of the analysis depends entirely on the avoidance of errors of various kinds in the two determinations of the volume of the enclosed air. Mistakes might be caused by slight variations in the temperature of the water, or the pressure under which the sample is measured, or in the degree of saturation with moisture of the sample. A variation of 0.1°C . in the temperature of the water in the jacket would, for instance, unless corrected, cause an error of fully $\frac{1}{4}$ volumes per 10,000 in the analysis.

In order to have a sharp index of the pressure under which the air is measured the level, not of the mercury, but of the potash solution in the narrow bore tubing of the absorption pipette, is taken as the index of pressure. At the first measurement the level is accurately adjusted to the mark at *E* by raising or lowering the mercury by means of the rack and pinion arrangement *F*. At the second reading the potash level is again adjusted in the same way. As the potash has a specific gravity of only about a tenth of that of mercury its level is a very delicate index of the pressure. A difference of $\frac{1}{10000}$ th part in the pressure would correspond to a difference of 1 mm. in the pressure of the potash solution, which would be very evident to the eye.

To correct for variations in temperature of the water-jacket a control tube *G* is employed, of a size and shape approximately the same as the gas-burette. The control tube communicates with the potash through the narrow bore glass tube *H*, and before the first measurement is made the level of the potash in *H* is adjusted to the mark by lowering or raising the reservoir *I*, which slides up and down in a loosely fitting cork. At the second measurement the same precaution is taken, so that the air in the control tube occupies exactly the same volume as at the first measurement. As an alteration of

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temperature or of barometric pressure would affect the pressure to an equal extent in the gas-burette and control tube it is evident that the adjustment of the level of the potash reservoir compensates exactly any error which the alteration of temperature or of barometric pressure would cause in the reading of the gas-burette¹.

Before the adjustments of the potash levels are made the water in the jacket is thoroughly mixed by blowing air through it by means of the tube *K*. This manipulation is essential to an accurate result. The tubes *E* and *H* have a bore of about 2 mm. If a narrower bore be employed error may arise through the potash not returning sharply to a perfectly definite level when disturbed.

In order to obviate error due to variations in the saturation of the air both the burette and the control tube are left with a little visible moisture inside. If the burette has once been wetted inside, and as much as possible of the water expelled by raising the mercury, it remains moist for a very large number of analyses, but a little moisture should always be visible. If by any mishap potash should be sucked over into the burette, it, and its connections, must be washed out with dilute acid introduced by the tap.

The accuracy of the graduation can be tested by taking out the burette, filling it with mercury, and weighing what flows out between different points in the graduation. A detached column of mercury should occupy the same number of divisions at all parts of the graduated tube of the burette. The efficient working of the apparatus can easily be ascertained by making an analysis of pure outside air, which should give about 3 volumes per 10,000 (except in dull or foggy weather in towns, where the amount may be a good deal higher.) A further test is to leave a sample of air in the apparatus after the carbonic acid has been absorbed, and see that its volume is not altered after the potash levels have been readjusted for alteration of temperature and atmospheric pressure, or after it has been passed over into the potash pipette, as in an analysis. Any error due to leakage in the

¹ The use of a control tube in gas analysis was first described by Williamson and Russell (*Journal of the Chemical Society*, 1868, p. 238) and afterwards applied in a greatly improved form by Pettersson (*Zeitschr. für analyt. Chemie*, Vol. 25, pp. 467, 479.) The latter along with Palmqvist has successfully applied the same principle to the determination of CO₂ in the air of rooms, and devised for the purpose a special form of gas-analysis apparatus with a narrowed burette similar to that described above, which gives excellent results (*Berichte der deutschen chemischen Gesellschaft*, Vol. 20, p. 2129, 1887).

connections, or blocking of any of them by a drop of liquid would thus be at once revealed.

At the end of an analysis the taps must be turned so as to close the communications between the potash and the burette and control tube: otherwise potash may be sucked in if there is any great fall of temperature or rise of barometric pressure.

The apparatus is so arranged that it can be used either for taking and analysing on the spot examples of air, or for analysing at some convenient place samples which have been collected in small bottles of about 1 oz. capacity. When the former method is preferred a short piece of capillary tubing, which projects through the hole in the cover of the case, is fixed by rubber-tubing on the upper opening of the burette. The burette is filled with mercury, the tap closed, and the mercury reservoir replaced in its stand. The apparatus is then held at the place where the sample is to be taken, and the tap opened, so that the sample is drawn in. During this process the breath should be held so as to avoid any risk of contaminating the sample with expired air. The tap is then turned, and the analysis completed in the ordinary way.

When samples are collected in bottles the latter should be thoroughly clean, and provided with a good cork which has been coated with a thin layer of paraffin wax well melted on. The sample is collected by placing the end of a piece of rubber-tubing in the bottle, and taking in a full breath through the tubing. The bottle is at once firmly corked. For an analysis the piece of bent glass tubing shown at *L* is attached to the opening of the burette and water poured into the glass vessel *M*. The tubing is then filled with mercury by raising the mercury reservoir, the tap closed, and the reservoir replaced. The cork of the bottle is now loosened, the neck immersed in the water, and the cork pushed out under water with the thumb. The bottle is then placed so that the glass tubing projects into it, and the tap opened, so that a sample of the air is withdrawn into the burette. Before turning the tap the bottle is held up, so that the water levels inside and outside of it are equal, and the sample withdrawn is therefore at atmospheric pressure. A second sample cannot be taken from the same bottle, as the presence of the water rapidly affects the percentage of carbonic acid in the air of the bottle.

The following examples give an idea of the degree of accuracy attainable by this method:

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I. Analyses of four bottles full (each bottle held about 30 c.c., or 1 cubic oz.) of air collected in rapid succession in a closed room with gas burning.

Vols. per 10,000.

Bottle <i>A.</i>	15·0	Analysis began 5 minutes after collection.			
Bottle <i>B.</i>	14·8	"	"	10	" " "
Bottle <i>C.</i>	16·2	"	"	20 hours	" "
Bottle <i>D.</i>	15·2	"	"	20 hours	" "

II. Analyses of three bottles of pure outside air, collected simultaneously.

Vols. per 10,000.

Bottle <i>A.</i>	2·8,	Bottle <i>B.</i>	3·6,	Bottle <i>C.</i>	3·2.
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III. Six successive analyses of outside air (night).

Vols. per 10,000.

(1)	3·0,	(2)	3·5,	(3)	3·6,	(4)	3·0,	(5)	3·6,	(6)	3·1.
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These results show that the method is reliable to about 0·5 vols. per 10,000; and this degree of accuracy abundantly suffices for almost all practical purposes.

The manipulations required during an analysis may be recapitulated as follows: (1) Open the tap of the control tube to the air for a moment, and then turn it so as to connect the control tube and potash pressure gauge. (2) Turn the tap of the burette so as to connect the burette and the potash pipette. (3) See that the level of the potash alters sharply and about equally in the tubes when the potash reservoir is raised. (4) Blow air through the water-jacket. (5) Raise or lower the potash reservoir till the potash is exactly at the mark in tube *H*. (6) Raise or lower the mercury reservoir by means of the rack and pinion till the potash in *E* is exactly at the mark. (7) Read off the mercury level on the scale of the burette to ·2 of a division. (8) Raise the mercury to the upper hook, so as to drive the air into the potash bulb, then lower it a little and raise it again twice so as to wash any carbonic acid in the connecting tubing into the potash bulb. (9) Return the air to the burette. (10) Blow air through the water-jacket. (11) Adjust the two potash levels as before and read off the mercury level. The first reading subtracted from the second gives the result in volumes per 10,000. (12) Close the two taps.