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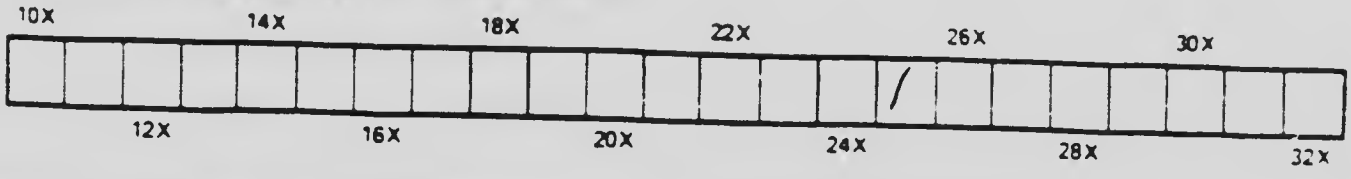

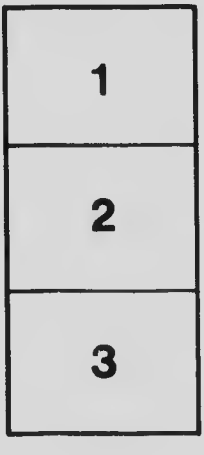

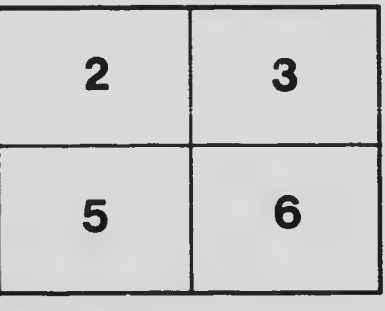

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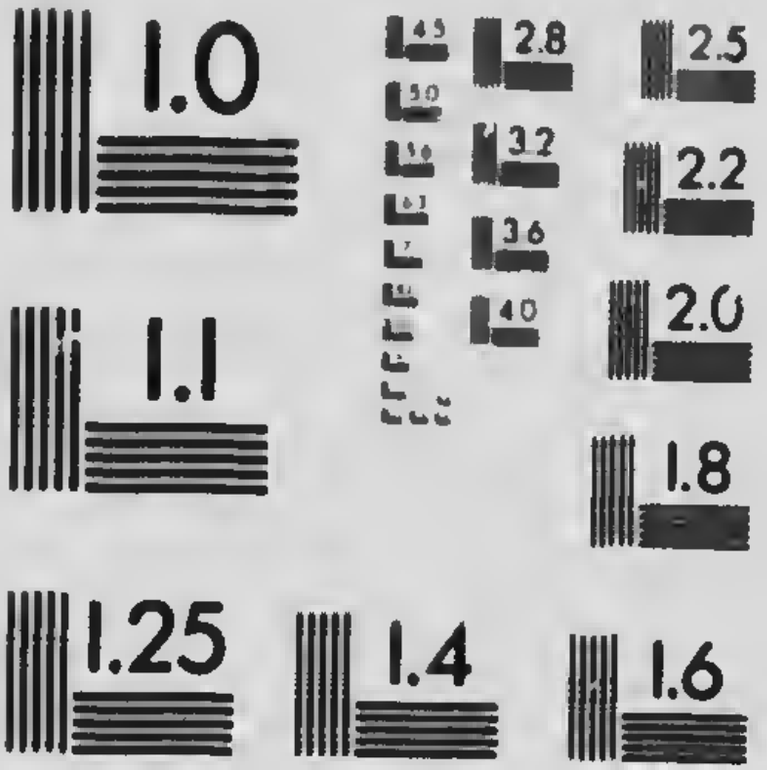

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No. 19: SIMPLIFIED GAS ANALYSIS, BY PROFESSOR J. J. R. MACLEOD

(REPRINTED FROM THE JOURNAL OF LABORATORY AND CLINICAL MEDICINE, VOL. IV)

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SIMPLIFIED GAS ANALYSIS

By J. J. R. Macleod, M.B., Toronto, Canada

NO. II. BURETTE WITHOUT STOPCOCKS FOR GAS ANALYSIS

FOR purposes of teaching, as well as for occasional use in clinical practice, the usual types of gas burette are unsuitable because of the care and attention that has to be given them in order to prevent "freezing" of the stopcocks. Other objections to stopcocks are that they are liable to leak unless well ground, and they add considerably to the cost of the apparatus. The increasing necessity of gas analysis in medical diagnosis and in research makes it important that simple and reliable apparatus be available.* Such an apparatus can be constructed by using screw clips in place of stopcocks, provided some means be taken to adjust the gas pressure in the burette after the screw clips have been tightened. This can be accomplished by the use of the "pressure adjuster" described below.

Another difficulty which the inexperienced constantly meet with in using gas burettes of the usual pattern (Haldane's) is in preventing the strong alkaline solution from running up the narrow tubing which connects the burette and absorption bulbs. This accident delays the analysis, since all traces of the alkali must be removed from the burette and mercury before proceeding with the analysis, and moreover the alkali, if not removed, eats its way around the stopcock, and causes etching of the glass and "freezing." In the following apparatus this danger is guarded against by inserting a small bulb on the connecting tubing. The adjustment of the mercury—very little of which is required—is simplified by using a screw clip and pinchcock on the tubing connected with the leveling burette.

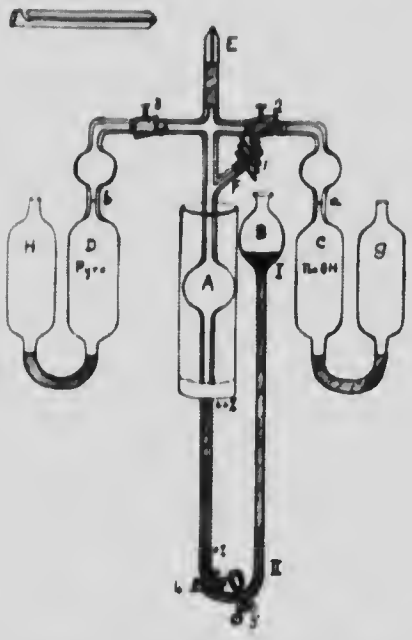
DESCRIPTION OF THE APPARATUS

The gas burette (*A*) is 10 c.c. capacity from the end of the oblique side tube (*E*) to the lowest graduation on the narrow portion. The uppermost graduation corresponds to 2.2 c.c. from the lowest, the distance between the two being divided into c.c. and 1/50th c.c. The bulb part of the stem to just above the 2.2 mark is surrounded by a water jacket. Above the side tube (*E*) the burette is continued into a narrow-bored tube (narrower than represented in the diagram), with two arms at right angles to each other. These are connected by thick walled pure-gum tubing, with the absorption bulbs (*C* and *D*) containing re-

*It is not suggested that the apparatus herein described should be employed by those accustomed to the use of the standard burettes or where expense is no object.

spectively a 20 per cent solution of NaOH and a 10 per cent solution of [illegible] with 10 per cent pyrogallic acid dissolved in it.*

Each bulb is connected below by rubber tubing with the overflow (*G* and *H*). A small bulb (relatively smaller than represented in the diagram) is blown on the stem of each absorption bulb to serve as a trap preventing solutions from passing over into the burette. The vertical tube above the bulbs is closed by the *pressure adjuster*, which consists, as shown in the side sketch in the diagram, of a glass rod beveled at one end and bored part of the way down the center. At a distance of about 3 mm. from where the beveled portion joins the tube a lateral hole is bored to meet the channel in the center. The adjuster is connected by pure gum tubing with the capillary tube of the burette, the beveled end of the former being in contact with the [illegible]



of the latter. When the rubber tubing is pinched up opposite the side tube the burette is brought into communication with the outside and the pressure in it remains undisturbed when the tubing is allowed to fall back into place.

The lower end of the burette is connected by thick-walled rubber tubing with the reservoir (*E*), and on this tubing are a pinchcock (*5*) and screw clip (*4*). About 15 c.c. of mercury suffices to fill the apparatus. The reservoir is hung by a loop of wire around the neck on hooks placed on a wooden upright stand, the higher one being in such a position (marked I†) that the mercury stands at the end of the side tube (*E*), and the lower one (marked II) so that it stands exactly at the mark 0% on the burette.

*NaOH may be substituted for KOH, but is not so satisfactory because of its viscosity. The solution is made by dissolving pure NaOH (electrolytic, if possible) in an equal weight of water, diluting 10 c.c. of this solution with 4 c.c. water, and dissolving 10 gm. pyrogallic acid in the mixture. Shipley: Jour. Am. Chem. Soc., 1916, xxxviii, 1687.

†The position I should be at a higher level than is shown in the figure.

TECHNIC OF THE ANALYSIS

The first step is to fill the tubing between the side tube (*F*) and the absorption bulbs with nitrogen. This is accomplished by taking a sample of air into the burette by opening screw clip 1, placing the mercury reservoir so that it stands opposite the lower end of the burette (at II), and with the screw clip 3 open, cautiously opening the pinchcock (*5*) so that air enters the burette. The screw clip 2 is then opened and the reservoir (*B*) raised to position I. By cautiously opening the pinchcock (*5*) the air is made to pass into *C*, and with 3 open the reservoir is raised and lowered several times so that all traces of CO_2 are removed from it by the alkaline solution, after which the reservoir is replaced in position II and the pinchcock, previously closed, opened until the NaOH stands at the mark (*a*) on the stem. Screw clip *2* is then closed, *3* opened, and the oxygen removed from the air by repetition of the same procedure. Screw clip *3* is then closed. This preliminary filling of the burette with nitrogen is unnecessary if a previous analysis has been made.

The sample of gas for analysis is now collected in an all glass (Luer) syringe with the piston well lubricated with vaseline. With the screw clips (1) open the burette is filled with mercury up to the end of the side tube (*F*), and the nozzle of the syringe is inserted in the rubber tubing of the side tube. While making this connection the piston of the syringe should be gently pressed so that no air may be allowed to become entrapped in the rubber tubing on *F*. To separate the sample into the burette the reservoir is placed in position II and the pinchcock *5* opened, gentle pressure being meanwhile maintained on the piston of the syringe. After the gas has been transferred, the mercury meniscus should stand about 1 mm. below the 0 graduation on the burette, this being accomplished by gentle pressure on the piston of the syringe. This leaves the gas in the burette under a slight positive pressure.

The pressure in the burette must now be made equal with that of the atmosphere. For this purpose the pinchcock (*5*) and all screw clips except 1 are opened, and the tubing on the adjuster (*E*) is pinched opposite the bevel. Being under a slight positive pressure, some of the gas in the burette escapes through the adjuster, and it is during this procedure that a slight error is incurred because some of the sample of gas mixes with the nitrogen above it. The error thus incurred is, however, negligible for most purposes. The menisci of mercury should now stand exactly at the 0% mark.

Screw clip *3* is now closed. This slightly compresses the gas in the burette and lowers the menisci at *a* and *b*, the new level at *a* being marked on the glass by a glass pencil. The reservoir (*B*) is slowly raised and lowered several times (with 4 and 5 open) so that the gas passes in and out of *C*, in which the CO_2 is absorbed. In doing this, care must be taken that the mercury does not rise above the level of *F*, and that the NaOH solution does not rise higher than the mark *a*. Four movements usually suffice to absorb all of the CO_2.

To determine the volume of the remaining gas, the pinchcock (*5*) is closed, the clip (*1*) half way screwed down, the reservoir placed in position II, and the pinchcock then cautiously opened until the NaOH solution is as nearly

as possible brought back to its original position at *a*. The fine adjustment of this level to the pencil mark is finally effected by means of screw clip *1*, and reading on the burette opposite which the mercury now stands gives in cubic centimeters the amount of CO_2 in 10 c.c. of gas. To make certain that all the CO_2 has been absorbed the above procedure should be repeated.

To determine the oxygen, screw clip *2* is closed and *3* opened. The level of the menisci of the pyrogallic acid solution is marked with a glass pencil at *b*. The gas is then moved in and out of D several times, until all the oxygen is absorbed as determined by no further shrinkage. This takes considerably longer than for CO_2. The final volume of gas as read on the burette, less the volume of CO_2, gives the oxygen.

After completion of the analysis the apparatus should be left filled with nitrogen and with the screw clips all closed. A short piece of rubber tubing should also be connected with the upper end of the reservoir (*1*) and closed by pinchcocks. This is to prevent undue oxidation of the pyrogallic solution.

As above remarked, a slight error is incurred in using the above apparatus during the adjustment of the pressures. With a little practice, however, this becomes very small. It is very important to make certain that all the rubber unions are perfectly tight, which is best insured by wiring the tubing.

The following analyses for CO_2 made by Mr. Shen will serve to illustrate the degree of accuracy of the method.

1. Mixture of nitrogen and CO_2 from stock bottle: CO_2, 1.78, 1.79, 1.81, 1.76; average, 1.785. Another mixture: 1.91, 1.99, 1.95, 1.95; average, 1.950.

2. Samples of alveolar air taken in all-glass syringe by using the Haldane tube:

a. Deep expiration after a normal inspiration: 6.03, 5.88, 6.04, 6.09, 5.96; average, 6.00.

b. Deep expiration after a deep inspiration: 5.42, 5.38, 5.33, 5.23, 5.54; average, 5.38.

3. Samples of air aspirated from the mouth after a deep expiration. This was done by placing a short piece of rubber tubing attached to an all-glass syringe at the back of the mouth, closing the lips around it after making a quick forced expiration, and quickly withdrawing the piston, the "dead space" of the tubing having been filled with alveolar air by a preliminary trial of the procedure.

a. Deep expiration after a normal inspiration: 5.76, 6.07, 6.09, 6.19; average, 6.00.

b. Deep expiration after a deep inspiration: 5.42, 5.31, 5.44; average, 5.39.

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