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The Haldane Gas Analysis Apparatus

Principles

The apparatus enables one to determine the percentage composition of carbon dioxide and oxygen amongst the permanent gases in the sample. That is to say, considerations of water vapour do not come into the analysis, and since the chief permanent gases we are concerned with are CO₂, O₂ and N₂, the determination of the first two also gives the % of N₂ (by subtraction from 100%).

The fraction of expired air or alveolar air which is made up of the permanent gases depends on what fraction of the atmospheric pressure is exerted by water vapour (this depends on temperature) and what fraction of the atmospheric pressure remains to be exerted by the permanent gases. The pressure exerted by each gas is the pressure exerted by the three permanent gases (i.e. atmospheric pressure less saturated water vapour^{pressure}) multiplied by the percentage composition of that gas expressed as a fraction.

In the apparatus a measured volume of gas (V₁) is first exposed to KOH to remove CO₂, and the volume (V₂) measured again. The gas is then exposed to an O₂-absorbing solution and its volume measured a third time (V₃).

Thus V₂ - V₃ = volume of oxygen removed.

$$\frac{V_1 - V_2}{V_1} \times 100 = \% \text{ CO}_2.$$

$$\frac{V_2 - V_3}{V_1^*} \times 100 = \% \text{ O}_2.$$

* N.B. The volume of oxygen removed must be expressed as a percentage of the original volume V₁.

The detailed technique of analysis is directed to ensuring adequate gas mixing and to avoid errors due e.g. to expansion or contraction of gas volumes due to temperature change.

Method

By reference to the apparatus itself or to the diagram it will be observed that there are four glass bulbs or burettes and five taps.

1. The calibrated or measuring burette can be filled with mercury from a reservoir - the tubing contains a tap (mercury tap) and there is a screw clip for fine adjustments.
2. At the top of the measuring burette is a three-way tap which can either connect the burette to the short sampling tube (through which a sample of gas is admitted) or to the tube leading to the potash and anthraquinone burettes (along which the gas is passed during the absorption of CO₂ or O₂).

It should be realised that the measured volumes of gas are to this tap only. Thus for accurate work there must be no absorbable gas in the tubing leading to the potash or anthraquinone burettes - this is why it is necessary to 'clear' the apparatus by an initial dummy analysis (see later).

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3. The potash burette (clear solution) and anthraquinone burettes are each provided with a flexible U tube leading to a reservoir containing the same respective solution. In the narrow tubing above the bulbs are marks to which the solutions should be adjusted initially with the three-way taps adjusted so that both solutions are open to the atmosphere (i.e. the pressure in the narrow tubes is atmospheric). T_{O_2} and T_{CO_2} in T position.

N.B. Once the levels have been set the oxygen reservoir should not be raised or lowered for the rest of the practical period.

4. To compensate for fluctuations in temperature most Haldanes have (i) a water jacket surrounding the measuring burette, and (ii) an empty burette in the same water jacket which can be connected to a second narrow tube connected to the potash reservoir. This should be opened to the atmosphere initially and the fluid brought to the correct level. T_{temp} in position T.

In the diagram the three levels are labelled a, b and c. The purpose of the extra burette is to indicate any volume change due to temperature fluctuation. Further, any temperature-produced changes can be compensated for by raising or lowering the KOH reservoir to bring the fluid again to level b, and this compensating pressure change will also be exerted on the sample of gas under analysis. This temperature-compensating device is called the thermobarometer.

N.B. Once the fluids have initially been set to their correct levels at atmospheric pressure Taps T_{temp} and T_{O_2} are turned to the \rightarrow position and should remain so for the rest of the practical period.

Dummy Analysis (and Analysis).

5. With T_{sample} connected to the sampling tube, open T_{Hg} and lower the mercury reservoir. Take in a sample of room air to more than the 9.5 ml mark. Close T_{Hg} and hang the mercury reservoir at the TOP OF THE RACK.

N.B. During the course of analyses it is very important always to hang the reservoir at the TOP.

6. See that T_{CO_2} is in \rightarrow position and turn T_{sample} first off for 30 seconds (to allow for temperature equilibration), then turn it so that it connects to the CO_2 absorbing burette. If the level moves from "a" readjust by the screw clip on the mercury tube. Check that level "b" is also correct. Read the gas volume. (Since the KOH solution was previously adjusted to level 'a' at atmospheric pressure, this means the volume of the sample is also being measured at atmospheric pressure.)

7. Now open the mercury tap (T_{Hg}) and allow the mercury to rise into the bulb - the sample will be displaced into the KOH bulb. Take the mercury reservoir and raise and lower it, keeping the mercury rising and lowering in the wide part of the bulb of the measuring burette. This mixes the sample and ensures that it is all exposed to the KOH surface. Raise and lower 12-20 times for CO_2 absorption (20-30 for O_2 absorption as O_2 is less soluble than CO_2).

8. Slowly lower Hg reservoir watching the potash level, and when 1/8" above the mark turn off T_{Hg} and hang the reservoir at the TOP. Adjust level of potash finally with T_{Hg} screw clip. Read volume and repeat last paragraph to see if any further volume is absorbed. In taking volume finally, see that 'a' and 'b' levels are correct.

9. Next turn T_{CO_2} to $\frac{1}{2}$ position so that the sample can pass along to O_2 absorber (sodium hydrosulphite and anthraquinone- β -sulphonate). Open mercury tap (T_{Hg}) and allow mercury to rise in bulb of measuring burette, then proceed as in para.7.

10. After 30 mixings lower the mercury reservoir more slowly, watching the anthraquinone level. (It helps to steady the knuckles of the hand against the rack as the reservoir is being lowered.) When the level is within $\pm 1/8$ of the mark turn off T_{Hg} and hang the reservoir at the top. Adjust finally with the screw clip. Read volume and repeat to confirm no further absorption is taking place. (This is a reading without temperature compensation but is sufficient to ensure that no further absorption is occurring.)

11. With anthraquinone on level 'c' go back to KOH by turning T_{CO_2} to $\frac{1}{2}$ position. Use KOH reservoir to adjust level 'b' if necessary and screw clip on mercury tubing to adjust level 'a'. Then, with levels 'a', 'b' and 'c' all adjusted, make final reading (temp. compensated).

The readings of the 'dummy analysis' are of course inaccurate because atmospheric air in the tubes leading to the burettes would contain O_2 , but at the end of this analysis these tubes are now filled mainly with nitrogen.

So finally tap T_{sample} is connected to the sampling tube and the mercury tap opened and mercury brought carefully to within $\frac{1}{4}$ " of the top of the sampling tube. Tap T_{Hg} is then turned off and the screw clip used to bring the mercury level up to the top of the sampling tube. The mercury reservoir can be hung near the bottom of the rack at this stage, so that by opening the mercury tap a sample of gas will automatically be taken in to a level between 9.5 and 10.0 ml. Once the gas is taken in, T_{Hg} is closed and the mercury reservoir hung at the TOP.

A true analysis follows the above procedures and each analysis leaves the apparatus 'cleared' ready for the next (so long as taps T_{temp} and T_{O_2} remain closed from the atmosphere).

N.B. All important volume readings are made with levels 'a', 'b' and 'c' adjusted. These are the volumes used for calculating the results, i.e. volumes V_1 , V_2 and V_3 referred to in the first section.

Note. For maximal accuracy in research work it is necessary to see that the gas in the narrow tube between the KOH level 'a' and tap T_{CO_2} is cleared of oxygen. One way is to connect to the KOH after the first O_2 absorption has been completed, raise and lower the mercury level to mix this gas with the larger volume of O_2 free gas, reset the KOH level and connect to the anthraquinone burette for a second O_2 absorption. By omitting this procedure the maximum error in a first analysis of e.g. alveolar air is to make the volume of oxygen absorbed too large by about 0.01 ml. Where duplicate and triplicate analyses of the same sample are being carried out this procedure is unnecessary, as the gas between 'a' and tap T_{CO_2} will have the same composition as the beginning and end of a replicate analysis.