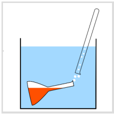

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Eudiometry (18th century analysis of the percentage of O2 in the atmosphere)

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1 Works for me dx.doi.org/10.17504/protocols.io.ba7aihie

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ABSTRACT

Reproduction of a XVIII century procedure for the analysis of the volumetric percentage of Oxygen in the atmosphere. The procedure uses polysulfides as an oxygen-consuming reactive. The change in volume of a sample of atmospheric air is measured before and after oxygen has been consumed.

GUIDELINES

This protocol is an approximation to an original XVIII century experiment. We have interpreted as closely as possible the work of the Spanish scientist Antoni Marti i Franques. His original description of the article can be found in:

Marti i Franques, Antoni de (1801). “Memoir on the Quantity of Vital Air in the Atmosphere, and the different Methods of measuring it”, *The Philosophical Magazine*, London, Vol. 9. pp. 250-262.

The history of the experiment and a detailed discussion on the likely reactants and reaction mechanisms can be found in Catalan, Spanish and English in:

Marti i Franques, Antoni de, Josep Grau Bové, and Josep Bonet i Avalos (eds.), *La química de l'aire*. Publicacions URV, 2011.

We consider this interpretation likely similar to the original experiment, but by no means a final and exact reproduction. The original work has aspects that others may interpret differently, or which could change with further historic research. We welcome any feedback.

MATERIALS

NAME	CATALOG #	VENDOR
Calcium Oxide		
Sulfur		

Preparation of the reactants

1 In order to prepare 500 ml of polysulfide solution (sufficient for 4-5 measures), mix 391.5 g of distilled water with 74.5 g of S and 34 g of CaO. 10m

If larger volumes are required, take care to maintain the same percentages by weight (78.3 of water, 14.9 of S, and 6.8 of CaO)

2 Bring the mixture to the boil, and boil during one hour. Replenish the water level to compensate for evaporation, keeping the volume constant at 500 ml. 60h 10m

The appearance of the final solution should be a deep orange, close to red.

Eudiometric measure

3 **Measure 100 ml of atmospheric air.** 5m

This can be achieved in many ways. We propose using a long volumetric tube (A, Figure 1.1), with a volume larger than 100 ml, closed at one end. Submerge tube A with the open side down into a water bath and let air scape until the final volume of air inside the tube is exactly 100 ml.

Naturally, any known volume would be useful. The advantage of using 100 ml is that any reduction is equivalent to the volumetric percentage change.

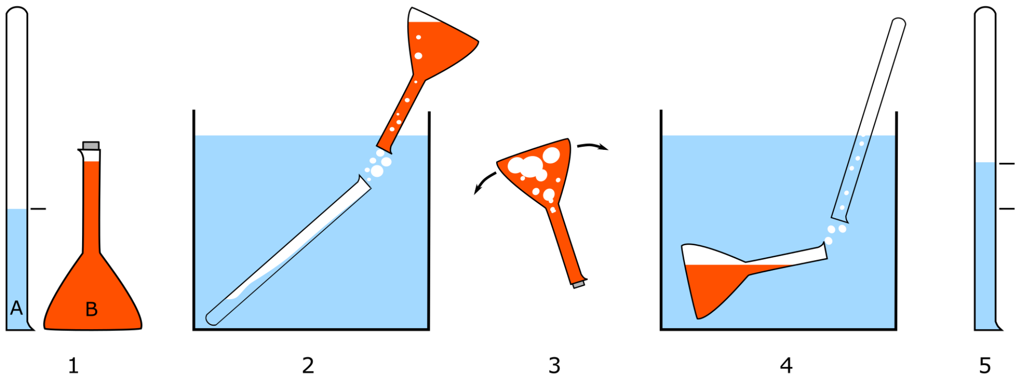

Figure 1. Steps of the experiment. A and B indicate the vessels as referred to in the text.

4 **Mix the 100 ml of atmospheric air with the polysulfide solution** 10m

Introduce at least 100 ml of the polysulfide solution into a volumetric flask (B). An excess is recommended. Any flask can be used. We recommend a volumetric flask with a sealable narrow opening of a diameter slightly larger than the diameter of the tube A used in the previous step.

With one hand, introduce the volumetric flask B with the polysulfide solution upside down in a water bath. This step needs to proceed fast, in order to avoid an excessive diffusion of polysulfide solution into the water. A narrow opening will help (Figure 1.2)

With the other hand, introduce the volumetric tube A upside down into the water bath and bubble its contents into the volumetric flask B containing the polysulfide solution.

5 **Shake** 10m

Close the volumetric flask and shake it vigorously (Figure 1.3). The reaction between the polysulfide solution and oxygen occurs in the interface between the air bubbles and the liquid. Therefore, the shaking needs to produce as many bubbles as possible. Stirring or shaking that results in a smooth movement of the liquid will unlikely be sufficient.

It is possible to achieve a complete reaction in 5 minutes of shaking. The reaction time depends on the concentration of polysulfides. If some water has diffused into flask B in step 4, the reaction time will be longer.

6 **Measure the new volume of air**

Submerge vessels A and B into the water bath (Figure 1.. Ensure that vessel A is completely filled with water. With care, let the air contained into flask B scape, bubbling it into vessel A. If any air is lost during this process, the measurement will not be valid. A funnel may be of assitsance.

The volume of air in the tube A should be smaller than the initial volume of air collected. If the experiment has been conducted correctly, there should be 79 ml of air (in fact, mostly N_2) in the tube.

You have determined the volumetric concentration of Oxygen in the atmosphere, which is 100 - 79 = **21 % v/v.**

Tips and precautions

7 In the XVIII century, eudiometrists took samples of air in many different locations, in the hope of finding significant differences. They did not find any. To demonstrate the homogeneity of the volume of Oxygen in the atmosphere, the experiment can be repeated with samples from very different locations.

CO_2, even in crowded spaces, is in concentrations that are too low to alter the measurements obtained with this procedure. Argon, however, can reach 1%. If it does not react with polysulfides, and does not dissolve in water, it should be contained within the 79 ml of remaining air.

A trained experimenter can obtaina very high repeatibility with this experiment, with an standard error smaller than 1%.