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Microscale Determination of Oxygen in Air by Reaction with Nitric Oxide

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Abstract: The composition of the air we breathe is fundamental for the preservation of human life. Since the time when it was determined for the first time by the British chemist Henry Cavendish, it has been a subject for frequent quantitative analysis. In this paper we present an experiment to determine the oxygen content in air. It can be performed in a normal laboratory setting and is based on the reaction between nitric oxide (NO) and oxygen. Nitric oxide is generated by the reaction between $NaNO_2$ and $FeSO_4$ in HCl in a Beral pipet. The solution of $NaNO_2$ and the air sample are introduced in the pipet bulb with syringes. This is an easy, ecological, low cost, fast, highly educational, and safe experiment performed with a simple chemistry kit.

Introduction

Many chemistry demonstrations have been proposed for the determination of oxygen content in air. Forster [1] was perhaps the first to propose an educational experiment that involved the oxidation of pyrogallol in an alkaline solution. Different modifications have been developed since then [2, 3]. Another variation involves the rusting of clean steel wool that has been previously immersed in acetic acid and left to rust in an air-filled vessel; the attendant oxygen consumption is then measured by difference.

The well known candle-and-cylinder experiment, long practiced [4–7], turned out to involve a misconception [8]. Behind this experiment which fortunately gives a favorable result there lie a series of extremely important questions. For example, what is the new composition of air when the candle stops burning? What volume of carbon dioxide does the burning of paraffin generate? Consequently, could one dissolve this volume in the available quantity of water, knowing the solubility of the gas at a certain temperature, and then draw conclusions as to whether it is significant or not?

We developed an alternative method involving the reaction between nitric oxide and oxygen [9] and downscaled it to the microscale level [10]. We present here a novel adaptation of this method. To the best of our knowledge, a microscale experiment for determining oxygen content in the air - with all of its microscale advantages, for example, economy of materials, reduced cost, time, space, energy, and amount of waste requiring disposal [10] - has not yet been reported.

Nowadays, besides all new analytical methods and sophisticated instruments [11, 12] we believe that the proposed microscale method will be welcome in the chemistry education arena.

Experimental

Have the contents of the kit shown in Figure 1 ready in advance. Prepare the Fe(II) solution by dissolving 0.75 g of $FeSO_4 \cdot 7H_2O$ in 15 mL of 15-20 % HCl. Mark with a permanent marker the pipet stem at ca. 75 mm from its bulb to be cut at a later stage (see below); this will provide stability to the pipet when placed inside the test tube, as well as capillary contact with its contents at the test tube bottom thereby facilitating the inward and outward transport of the solution from the pipet.

Insert the test tube in the hole of the stand. Draw into a Beral pipet enough $FeSO_4$ solution so as to fill it completely, including its stem. Invert the filled pipet (avoid pressing its bulb) and cut the stem at the mark (ca. 75 mm) with the scissors; then insert it in the test tube bottom up (Figure 2).

Draw 0.05 mL of a saturated $NaNO_2$ solution into one of the insulin syringes (hereafter called *syringe A*). Fill the second insulin syringe (called *syringe B*) with 1 mL of air and place it on the lab bench for later use. Then, insert the needle of syringe *A* into the pipet bulb. Slowly add all the 0.05 mL of the $NaNO_2$ solution by pressing the plunger. This will produce a colorless gas (NO) that will occupy roughly 1/3 of the bulb (see Figure 3) and the solution will change color.

(All the chemicals that we used were Pro-analysis grade, Merck and used as received with no further purification.) **Warning: NO is a toxic radical, avoid breathing its vapors. The entire experiment should be performed under a fume hood. We recommend that the MSDS (Material Safety Data Sheet) for all chemicals be consulted prior to the experimental work.**

Holding the two syringes, shake the pipet to facilitate the reaction between NO and O_2 and thus the dissolution of the NO_2 generated. Then, withdraw enough gas from the bulb with syringe *B* so as to bring the liquid level back to the initial mark. Read and record the new volume of gas in syringe *B*. Assuming that O_2 is the limiting reagent, the gas in the bulb is now essentially composed of air components that do not react with NO and of unreacted NO. This excess NO should be eliminated from the bulb by drawing it into a 5-mL syringe (syringe *C*) containing 1 mL of H_2O and 2-mL of air so as to produce soluble NO_2; allow the NO_2 to dissolve and neutralize with a base. Discard according to local regulations. (A suggested disposal procedure and further safety tips are presented in the Supporting Materials). **Warning: NO_2 is toxic, avoid breathing its vapors.**

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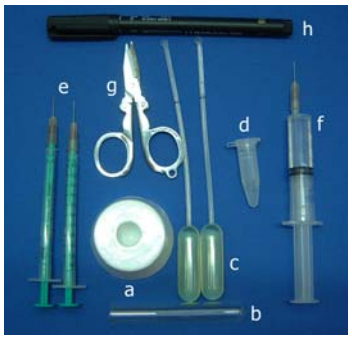

Figure 1. Materials and equipment: a) Beral pipet stand (e.g., made of gypsum or by drilling a hole in the center of a large rubber stopper), b) 12 × 75 mm test tube, c) Beral pipets, d) Eppendorf plastic vial containing a saturated solution of $NaNO_2$, e) 1-mL insulin syringes with 0.01 mL divisions, f) 5-mL syringe with needle, g) permanent marker, and h) folding scissors.

Figure 2. Experimental setup.

Figure 3. Insertion of the $NaNO_2$ solution.

Figure 4. Insertion of 1 mL air in the pipet bulb.

Mark the new liquid level in the bulb with the permanent marker. Next, insert the needle of syringe *B* into the top of the bulb (see Figure 4) and slowly push 1 mL of air into it.

Results and Discussion

The reaction between Fe^{2+} and NO_2^- produces a given volume of NO(g), $V_{NO,i}$ according to equation 1:

$$Fe^{2+} + 2H^+ + NO_2^- \longrightarrow NO(g) + Fe^{3+} + H_2O(l) \quad (1)$$

Note that the spectator ions are not shown here. Protons are used instead of hydronium ions for simplicity. *All species are aqueous, unless otherwise specified*.

Then, adding the oxygen contained in 1 mL of air makes it react quantitatively with NO in a 1:2 molar ratio, as shown in equation 2:

$$2NO(g) + O_2(g) \longrightarrow 2NO_2(g) \quad (2)$$

The $NO_2(g)$ thus generated is highly soluble in water and will no longer be present in the gas phase. This dissolution is not a physical process, but rather a chemical one (i.e., a nitrogen disproportionation):

$$2NO_2(g) + H_2O(l) \longrightarrow HNO_3 + HNO_2 \quad (3)$$

After this reaction is over, only excess NO (volume is $V_{NO,f}$) and N_2 plus trace gases from the air (this volume is hereafter called $V_{air\text{-}O_2}$) are left as gases in the system [9]. Prior to the reaction between NO and O_2 the total volume of NO(g), $V_{NO,i}$ plus that of air, V_{air} is given by V_i in equation 4:

$$V_i = V_{NO,i} + V_{air} \quad (4)$$

where V_{air} = 1 mL. Although $V_{NO,i}$ is not known, its value is "fixed" by the mark previously made on the bulb. Following the reaction between NO and O_2, the liquid level is brought back to the mark by removing from the bulb the necessary amount of gas with syringe *B*. The final total volume of gas, V_f in the bulb and in syringe *B* is given by equation 5.

$$V_f = V_{NO,i} + V_{syr\ B} \quad (5)$$

V_f is actually the volume of NO left unreacted plus the remaining components of air. In other words,

$$V_f = V_{NO,f} + V_{air\text{-}O_2} \quad (6)$$

Equations (5) and (6) can be equated to obtain:

$$V_f = V_{NO,i} + V_{syr\ B} = V_{NO,f} + V_{air\text{-}O_2} \quad (7)$$

The volume change, ΔV is due to the volume decrease predicted by equations 2 and 3, and is given by the following difference:

$$\Delta V = V_i - V_f = (V_{NO,i} + V_{air}) - (V_{NO,i} + V_{syr\ B}) = V_{air} - V_{syr\ B} \quad (8)$$

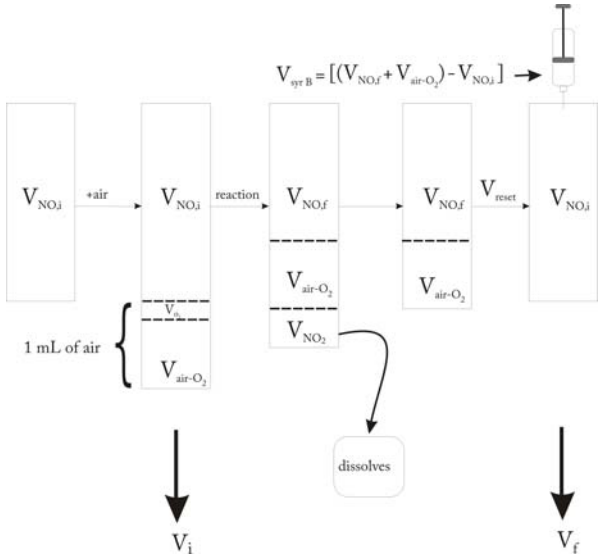

Figure 5. Process scheme.

Because V_{air} = 1 mL, and $V_{syr\ B}$ can be easily read in the syringe, ΔV can be obtained. Since the difference between the initial and final volumes of NO gives the volume that reacted, $V_{NO,reacted}$ and this is twice the initial volume of O_2 (see reaction 2), then:

$$V_{NO,reacted} = V_{NO,i} - V_{NO,f} = 2V_{O_2} \quad (9)$$

Knowing that

$$V_{air} = V_{O_2} + V_{air\text{-}O_2} \quad (10)$$

one obtains from equations (7–10):

$$\Delta V = V_i - V_f = \left(V_{NO,i} + V_{O_2} + V_{air\text{-}O_2}\right) - \left(V_{NO,f} + V_{air\text{-}O_2}\right) = V_{O_2} + \left(V_{NO,i} + V_{NO,f}\right) = V_{O_2} + V_{NO,reacted} = V_{O_2} + 2V_{O_2} = 3V_{O_2} \quad (11)$$

and

$$V_{O_2} = \Delta V/3 \quad (12)$$

The entire sequence is schematized in Figure 5.

Lastly, one can calculate the percentage of oxygen in air:

$$\%\ \text{of oxygen in air} = \left[V_{O_2}/\left(V_{O_2} + V_{air\text{-}O_2}\right)\right] \times 100 = \left[(\Delta V/3)/1\ \text{mL}\right] \times 100 = \left\{\left[\left(1\ \text{mL} - V_{syr\ B}\right)/3\right]/1\text{mL}\right\} \times 100 = \left[\left(1 - V_{syr\ B}\right)/3\right] \times 100 \quad (13)$$

where $V_{syr\ B}$ is given in mL.

We consistently obtain values for $V_{syr\ B}$ between 0.35 mL and 0.45 mL, which translates into an oxygen content in air between 18% and 22% (i.e., reasonably close to the true value of ca. 21%).

Additional projects can be performed with similar procedures and materials. These may involve, for example, determining the oxygen content in the air exhaled by living creatures as a function of the time they keep that air in their lungs. Monitoring photosynthetic processes can be another project; in this case, one can determine the oxygen content in a closed system containing a given plant, with appropriate light irradiation during different periods in a day.

Conclusions

Oxygen determination can be performed with a simple procedure, equipment and solutions in a short time and at the microscale level with the associated advantages.

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Supporting Materials. Suggested disposal procedure and further safety tips are presented in the Supporting Material (http://dx.doi.org/10.1333/s00897102281a).

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