On the Oxygen Content of Air: An Alternative Determination Using Digital Manometer

Vladimir M. Petruševski* and Nataša Stavrevska

Institute of Chemistry, Faculty of Natural Sciences and Mathematics, Ss Cyril and Methodius University, Arhimedova 5, PO Box 162, 1000 Skopje, Republic of Macedonia, vladop@pmf.ukim.mk

Abstract: In order to determine the oxygen content in the air, an excess of alkaline solution of pyrogallol was placed in a reaction chamber connected to a digital manometer. The reaction is completed in less than 24 hours, the determined oxygen content is in perfect agreement with the widely adopted value of 21 % by volume. Also the process follows, almost ideally, a first order kinetics.

Introduction

Experiments aimed to determine the content of oxygen in the air are quite numerous in the literature [1–15]. All of the above experiments are basically of one type: measuring the volume change of the gaseous mixture after completion of a chemical reaction that is supposed to eliminate the oxygen from the system.

In the early works [1, 2] the authors used an alkaline solution of pyrogallol for elimination of the oxygen from the air. There is a disagreement, however, whether the demonstration can be completed in about 10 minutes, as claimed by Forster [1], or a significantly longer time period as noticed by Munro [2]. A modification of this method (using 'hypodermic' syringes) was developed more than 3 decades later [5] and may perhaps be considered to be at the origin of the nowadays very popular microscale equipment [15]. Interestingly, the chemistry behind the reaction of the alkaline solution of pyrogallol and oxygen still seems to be obscured [16]. One could also object that pyrogallol is toxic, and the solution of NaOH (or KOH) used is very corrosive, thus making this experiment undesired.

A eudiometer filled with a mixture of air and hydrogen gas was used by Cooke [3]. Although this equipment seems to be important from both methodical and historical points of view, it seems to be out of use for many years (meaning, it cannot be found any more in most of the general chemistry and chemistry education labs). Furthermore, the eudiometer if not used wisely may be a source to serious hazard [17].

Lincoln and Klug [4] used the so-called Hempel buret and Hempel pipet (these pieces of equipment too are practically out of use in the general chemistry laboratories), and a mixture of elemental copper and aqueous solution of ammonia and ammonium chloride, that reacts with the oxygen enabling its removal to be 'rapid and complete'.

A series of publications [6–8, 10] devoted to variants of the well-known 'candle and cylinder experiment' are actually based on the misconception that all of the oxygen in the cylinder will be exhausted at the end of the experiment. This assumption, however, is wrong as has been pointed to by Birk and Lawson [13]. In a way related (although claimed as giving perfect results) is the attempt of Fang [12], where burning of ethanol is recommended. We have personally tried both the 'candle & cylinder' and the 'ethanol burning' and cannot report any satisfactory results.

Steel wool rusting [9, 11, 13] definitely gives good results, although not quite in 20 minutes time, as reported [9]. The reason for the above discrepancy might lie in some minor differences under which the experiments were performed. Anyway, this method is acceptably fast, relatively safe and reliable/creditable.

Another (even faster) method was offered by Najdoski and Petruševski [14]. This one is based on oxidation of nitric oxide by oxygen. The method is not only fast, but is also a very reliable one. Its microscale variant was published quite recently [15]. One disadvantage of the method, however, is the use of the noxious gas (NO).

In the present work we introduce equipment at the heart of which is a digital manometer, for accurate and elegant determination of the oxygen content in air, thus directly measuring the pressure in a purposely designed reaction chamber. Alkaline solution of pyrogallol was used, for it is very efficient in removing the oxygen from air. To the best of our knowledge, the only demonstration bearing any similarity with our approach is the work by Gordon and Chancey [18], where they pay attention to the kinetics of the rusting of steel wool (they use gas sensors to measure the partial pressure of oxygen). The experiment we propose lasts several hours, so it is one more example of marathon experiments [19–24]. This one, however, may be considered as a short marathon experiment. A potential educational advantage of the proposed method is that it offers an alternative: to measure the decrease of pressure (under isochoric conditions) during the oxygen consumption, instead of the decrease of volume (under isobaric conditions).

Experimental

We used a digital manometer Lutron VC–9200 that could be coupled to a PC through one of its USB ports (the price of the instrument together with the software is less than 300 euro, which seems more than acceptable). The manometer could operate in units of Torr, bar, hPa, psi etc. We used the Torr scale. The instrument resolution was 1 Torr, meaning the maximum error of a measurement should be ±0.5 Torr, providing the factory calibration of the instrument is perfect. No possibilities existed for the user to recalibrate it, which is a real disadvantage. Nevertheless, we noticed that the measured values

Figure 1. The digital manometer. 1. body of the manometer; 2. connection cable; 3. the pressure gauge; 4. screw covered with teflon tape; 5. nut; 6. brass inlet/otlet tube; 7. glass tube with a stopcock.

Figure 2. The reaction chamber used for the determination of the oxygen content of air.
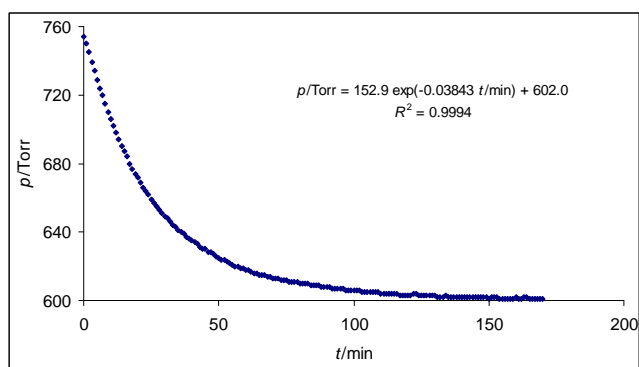

Figure 3. Variation of the pressure in the reaction chamber with time and the equation of the best-fit line.

of the atmospheric pressure were in a very good agreement with the reported daily values so the factory calibration should be considered as more than satisfactory. We were conservative, however, and considered the error of the measurement to be equal to the resolution of the instrument (±1 Torr).

The digital manometer we used is shown in Figure 1.

A medium problem was to mount a glass tube with a stopcock on the brass screw connected to the pressure gauge (cf. Figure 1, parts 7 and 4). We managed to find a suitable nut (together with a

corresponding brass inlet/outlet tube, parts 5 and 6), but it did not fit the screw hermetically until we used several layers of a teflon tape between the two. Hermeticity was checked by partial evacuation of the air out of the gauge (down to ~ 300 Torr). It was then left overnight. The result was quite satisfactory, as after 24 hours the pressure (measured at the same temperature of the room) did not vary by more than 1 Torr.

Basically, the reaction chamber (Figure 2) that was used in the present experiment was constructed earlier, by a modification of a standard wash-bottle (all details are given elsewhere [24]). The top part of the inlet glass tube was sealed, and part of its glass tube that enters the wash-bottle was cut off. The outlet tube was joined (by means of a piece of plastic tube 3–4 cm in length) to the pressure gauge, via the glass tube with a stopcock (Figure 1 part 7). Of course, other vessels from standard laboratory equipment could be used as well.

Performing the experiment. A mixture of 15 mL concentrated aqueous solution of KOH ($w = 0.4$) and 1.5–2 g of solid pyrogallol was shaken in a stoppered test tube, till the solid was completely dissolved (**wear a face shield and protection gloves during this operation!**). The solution was quickly transferred to the reaction chamber. The chamber was covered with the ground glass stopper connected to the pressure gauge and data acquisition starts immediately, at a rate of 1 reading per minute. (One of the reviewers suggested to put the vessel on an electromagnetic stirrer. On the positive side, this should decrease the duration of the experiment; however, the equipment becomes much more complex in this way.)

A word on the above quantities: as the chemistry of the process is not known in detail, in order to be on the safe side, we assumed that one molecule of pyrogallol will absorb one atom of oxygen. Knowing that the volume of the reaction chamber is about 300 mL (roughly 1/5 of which is oxygen) the above mass of pyrogallol (1.5–2 g) is an order of magnitude larger than the assumed stoichiometric mass. Therefore, the concentration of the pyrogallol will basically remain constant during the reaction.

Results and Discussion

The data acquisition lasted for about 3 hours, a period in which the pressure dropped from the initial value in the chamber of 754 Torr to 601 Torr. The instrument was then set off and was left overnight but the equipment **was not** dismounted. This was done to save battery life, on one hand (weakened batteries induce higher pressure values than they are in reality), and to prevent readings of the pressure under non-isothermal conditions (as a consequence of central heating reductions in the evening hours, the temperature variation may be as large as 5 °C). The next day the instrument reads the value of the final pressure as 596 Torr (the temperature being the same, i.e. 22 °C as in the beginning of the experiment).

Keeping in mind that we adopted $\Delta p = 1$ Torr as the error of the measurement, one easily calculates the oxygen content in the air as

$$\varphi(\mathrm{O_2}) = \frac{p_{\mathrm{initial}} - p_{\mathrm{final}} \pm \Delta p}{p_{\mathrm{initial}}} = \frac{158\ \mathrm{Torr} \pm 1\ \mathrm{Torr}}{754\ \mathrm{Torr}} = \quad (1)$$
$$(20.95 \pm 0.13)\%$$

The variation of the gas pressure in the reaction chamber with time is presented in Fig. 3. The equation of the best-fit line (obtained using non-linear fit of the Kevin Rainer's WinCurveFit free software package [26]) together with the regression coefficient are

$$p/\text{Torr} = 152.9 \exp(-0.03843\ t/\text{min}) + 602.0$$
$$R^2 = 0.9994 \qquad (2)$$

The variation reveals almost perfect exponential behavior of the measured pressure with time. The first term on the right-hand side (the exponential one) should ideally correspond to the variation of the partial pressure of oxygen in the chamber with time. Similarly, the second term should ideally correspond to the total pressure of all other gases but oxygen. This would be the case, if the entire set of values (scanned through, say, 24 hours) was available. Due to the mentioned potential errors of the weakened battery and the non-isothermal conditions in the laboratory it was not obtained. This being the case, one must notice that the first numerical value (the 152.9 of the exponential term) is underestimated (would be close to 158), and the constant term of 602.0 is overestimated (would be close to the numerical value of the pressure measured the other day, i.e. 596).

Equation 2 could be rewritten to account for the partial pressure of oxygen as

$$p(\text{O}_2)/\text{Torr} = 152.9 \exp(-0.03843\ t/\text{min}) \qquad (3)$$

Knowing that for gases (ideal behavior is assumed) $p = kc$, where c is the concentration and k is a proportionality constant, one sees that Eq. 3 is equivalent to

$$c(\text{O}_2) = c_0(\text{O}_2) \exp(-0.03843\ t/\text{min}) \qquad (4)$$

and it is clear that the reaction follows a 1st order kinetics. This was enabled by making the concentration of the alkaline solution of pyrogallol an order of magnitude higher than necessary.

Conclusion

The reaction of the alkaline solution of pyrogallol with air oxygen in closed vessel follows a 1st order kinetics. The determined oxygen content (20.95 ± 0.13 %) perfectly agrees with the widely adopted value [25].

Safety Tips and Disposal

The alkaline solution of pyrogallol is highly corrosive and causes serious burns to skin! Always wear a face shield and protection gloves during manipulations with it. After the experiment is over, the solution might be disposed under the drain with large quantities of water (or following local safety regulations).

References and Notes

1.　Forster, A. C. *J. Chem. Educ.* **1927, 4**, 638–639.

2.　Munro, L. A. *J. Chem. Educ.* **1928,** *5*, 741–742.

3.　Cooke, G. B. *J. Chem. Educ.* **1929,** *6*, 105–106.

4.　Lincoln, A. T.; Klug, H. P. *J. Chem. Educ.* **1935,** *12*, 375–376.

5.　Davenport, D. A.; Saba, A. N. *J. Chem. Educ.* **1962,** *39*, 617–620.

6.　Cooper, E. K. *Discovering Chemistry*; Butterworth: London, 1963; pp 118–119.

7.　Farrah, K. H. *Middle School Chemistry*; G. Bell: London, 1965; pp 22–23.

8.　Mullin, V. L. *Chemistry Experiments for Children*; Dover; New York, 1968; pp 30–31.

9.　Birk, J. P.; McGrath, L; Gunter, S. K. *J. Chem. Educ.* **1981,** *58*, 804–805.

10.　Cobb, V. *Chemically Active*; Lippincott: New York, 1985; p 45.

11.　Martins, G. F. *J. Chem. Educ.* **1987,** *64*, 809–810.

12.　Fang, C. H. *J. Chem. Educ.* **1998,** *75*, 58–59.

13.　Birk, J. P.; Lawson, A. E. *J. Chem. Educ.* **1999,** *76*, 914–916.

14.　Najdoski, M.; Petruševski, V. M. *J. Chem. Educ.* **2000,** *77*, 1447–1448.

15.　Najdoski, M.; Ibanez, J. G. *Chem. Educator*, **2010,** *15,* 215–217; DOI: 10.1007/s00897102281a.

16.　http://www.tutorvista.com/bow/alkaline-pyrogallol　　　(accessed December 2010).

17.　Herak, J. *General and Inorganic Chemistry. Experiments*; Školska knjiga: Zagreb, 1968; pp. 114–117 (in Croatian).

18.　Gordon, J.; Chancey, K. M. *J. Chem. Educ.* **2005,** *82*, 1065–1067.

19.　Najdoski, M.; Aleksovska, R.; Petruševski, V. M. *Chem. Educator*, **2001,** *6*, 319–320; DOI 10.1333/s00897010519a.

20.　Petruševski, V. M.; Najdoski, M. Z. *Chem. Educator*, **2001,** *6*, 91.94; DOI 10.1333/s00897010465a.

21.　Petruševski, V. M.; Monković, M.; Najdoski, M. *Chem. Educator*, **2004,** *9*, 39–41; DOI 10.1333/s00897040752a.

22.　Petruševski, V. M.; Monković, M.; Najdoski, M. *Aust. J. Educ. Chem.*, **2006,** *66*, 24–26.

23.　Monković, M.; Petruševski, V. M.; Najdoski, M. *Chem. Educator*, **2006,** *11*, 259–261; DOI 10.1333/s00897061051a.

24.　Stavrevska N.; Petruševski, V. M.; Ivanovski, V. *Chem. Educator*, **2010,** *15,* 321–324; DOI 10.1333/s00897102294a.

25.　http://www.physlink.com/reference/aircomposition.cfm　　　(accessed December 2010).

26.　http://www.simtel.net/free-download/Education-Science-related-Programs/WinCurveFit/52878.html (accessed January 2011).