

A General Chemistry Experiment for the Determination of the Oxygen Content of Air

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An experiment to determine the percentage by volume of oxygen in air appears in many general chemistry laboratory manuals. All the methods are based on a chemical absorption of O_2 resulting in the rise of a liquid level. None of the published methods is consistent with all our criteria for a good experiment: it should be safe, rapid, simple, accurate, and reproducible.

The methods in common use involve alkaline pyrogallol, white phosphorus, copper in ammoniacal ammonium chloride, or steel wool as the oxygen-absorption reagent. The pyrogallol method¹ is satisfactory in all respects except for safety. Pyrogallol is highly toxic when introduced internally or by percutaneous absorption, and the concentrated sodium hydroxide solution commonly used with it is very corrosive to tissues. The white phosphorus method suffers with respect to both safety and speed. White phosphorus is poisonous, with a 50-mg fatal dose. In addition, it may cause skin burns and may flame spontaneously. The absorption of O_2 generally is slow under experimental conditions, with most procedures suggesting a 24-hr waiting period. Copper in ammoniacal ammonium chloride can give rapid, accurate, reproducible results.² However, a complex apparatus consisting of a Hempel buret connected through a three-way stopcock to a Hempel pipet was used. We attempted to adapt this method to a simpler apparatus but were unsuccessful.

The steel wool method is satisfactory except for speed. As commonly described, the experiment requires 4–6 hr to remove the oxygen completely from a 25-ml air sample. Because this method satisfied our other criteria, we sought conditions such that the rusting of steel wool would be complete in less than one 2-hr laboratory period. The general procedure is as follows:

(1) Soak a piece of fine steel wool (size 00 is best) in acetic acid solution for about 1 min to clean the surface.

(2) Shake off the excess acid. Rinse the steel wool once by dipping the steel wool into 200 ml of distilled water in a beaker, swishing it around 4–5 times, then shaking off the excess water. (Some experiments involved no rinsing or repeating the rinsing procedure two or more times.) This is an important departure from published variations of this method, which wash the acid off completely.

(3) Quickly insert the steel wool into a 25-ml graduated cylinder and invert into a beaker of water. The initial volume of air is then assumed to be the total capacity of the cylinder.

(4) Periodically measure the height of water in the cylinder after equalizing the internal and external levels.

(5) When the water level stops changing, calculate the volume % O_2 from the % volume change.

One obvious factor to be investigated is the amount of steel wool used. In one series of experiments, various amounts of steel wool were soaked in glacial acetic acid and rinsed once in distilled water. The optimum experiment is shown in Figure 1 and complete results are listed in Table 1. These experiments suggest that at least 0.5 g, but no more than about 1.0 g Fe should be used. More steel wool than 1.0 g gives no time advantage, and the volume occupied by the wet steel wool

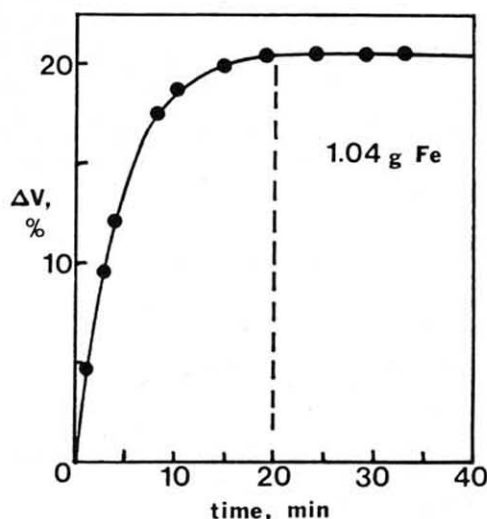


Figure 1. Plot of volume change expressed as percent of the initial air volume versus time, for 1.04 g steel wool soaked in glacial acetic acid and rinsed once in distilled water.

Table 1. Effect of Weight of Steel Wool^a

Weight Fe (g)	Half-life (min)	Time for Complete O_2 Removal (min)	% O_2 ^b
1.47	2.5	20	19.6
1.04	3.2	20	20.5
0.72	11.5	40	20.6
0.51	14.5	60	20.3
0.29	~100	270	20.2

^a The steel wool was soaked in glacial acetic acid and rinsed once in distilled water.

^b Uncorrected for the volume occupied by the wet steel wool or for vapor pressure of water.

begins to become significant. With less than 1.0 g Fe, the volume occupied is generally less than 1% of the initial air volume. Other experiments using 6 M acetic acid instead of glacial acetic acid gave similar results.

A second factor, which was discovered late in our investigation but which makes interpretation of other results easier, is the rinsing process. One series of experiments, shown in Figure 2, involved soaking about 1.0 g of steel wool in glacial acetic acid and rinsing in fresh 200-ml portions of distilled water various numbers of times. For 0, 1, 2, and many rinses (i.e. in running water) the half-lives were 2.6, 7.7, 25, and >100 min, respectively, while the corresponding times for completion were 10, 25, 60, and >>100 min. These experiments suggest that some residual acid is essential to a rapid rusting process and that a single rinse provides optimum conditions. While the reaction is faster if the acid is not rinsed off, the acidity is then high enough to cause H_2 evolution, giving poor results.

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¹ See for example Davenport, D. A. and Saba, A. N., J. CHEM. EDUC., 39, 617 (1962).

² Lincoln, A. T. and Klug, H. P., J. CHEM. EDUC., 12, 375 (1935).

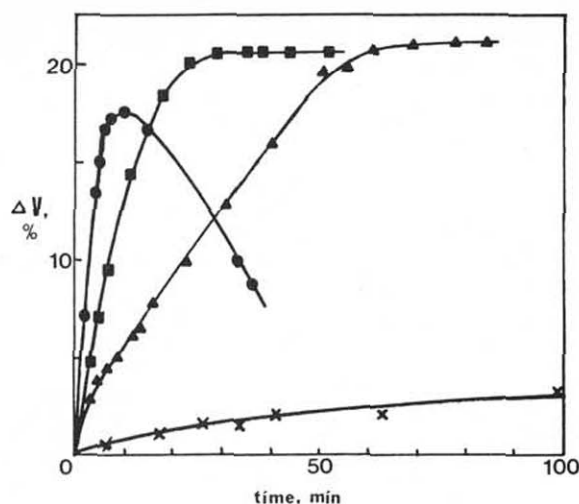


Figure 2. Effect of rinsing on the rusting of 1.0 g steel wool soaked in glacial acetic acid. Number of rinses: ● none, ■ one, ▲ two, × many (complete removal of $\text{CH}_3\text{CO}_2\text{H}$).

The effect of acetic acid was investigated further in a series of experiments in which 0.7 g of steel wool was soaked in acetic acid of various concentrations then rinsed twice in distilled water. Results for one series of experiments are presented in Figure 3. For 17.5 M, 6 M, 2 M, and 0 M acetic acid, the half-lives were about 5, 11, 22, and >100 min, respectively, while the times for completion were about 30, 45, 80, and >>100 min. These experiments again suggest that the amount of residual acetic acid influences the time required for complete reaction.

In a series of experiments to determine the optimum residual acetic acid concentration, about 1.0 g of steel wool was soaked in acetic acid solution of various concentrations but was not subsequently rinsed in distilled water. As shown in Table 2, with acetic acid concentrations in excess of 1.0 M, the water level rose to a maximum value corresponding to less than 20% O_2 , then dropped, due to H_2 evolution. Long-term stability in the final volume was observed for 0.5 M or lower concentrations of acetic acid. With >0.5 M acid solutions, a single distilled-water rinse is thus essential to obtain good results. Residual acetic acid concentrations of 0.2–0.5 M appear to be optimal for rapid reaction and accurate % O_2 determination.

Reproducibility of reaction times is demonstrated by a series of experiments in which ≈ 1.0 g of steel wool was soaked in glacial acetic acid and rinsed once in distilled water. Times for complete reaction in five experiments were 25, 20, 25, 20, and 30 min. In 30 experiments under a variety of conditions, excluding only those experiments in which H_2 was evolved, excellent results were obtained. The average % O_2 in air was $20.6 \pm 0.4\%$ (uncorrected for steel wool volume or vapor pressure of water). The range of values was 19.6–21.7 %.

We recommend the use of about 1.0 g of fine steel wool, soaked in 6 M acetic acid and rinsed once in 200 ml distilled water. Alternatively, students could soak 1.0 g steel wool in about 0.25 M acetic acid with no distilled water rinse. **Glacial acetic acid is not recommended for student use**, because it is toxic and can cause skin burns.

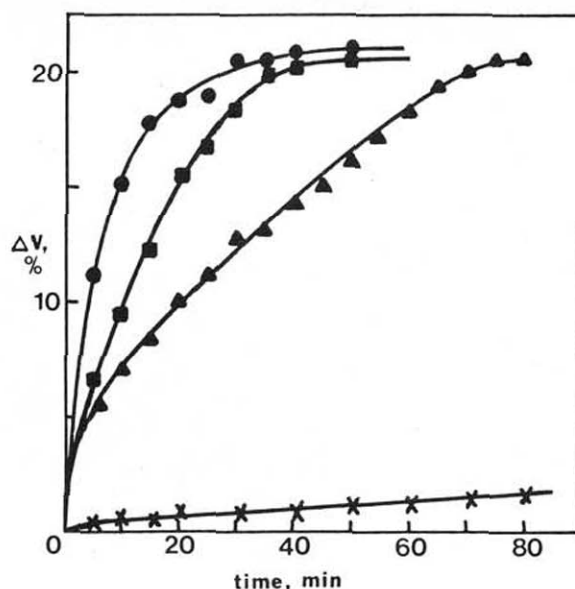


Figure 3. Effect of soaking 0.7 g steel wool in acetic acid solution, followed by two distilled-water rinses. Acetic acid concentration: ● 17.5 M (glacial), ■ 6 M, ▲ 2 M, × 0 M (distilled water).

Table 2. Effect of Residual Acetic Acid^a

$[\text{CH}_3\text{CO}_2\text{H}]$ (M)	Time for Maximum Volume Change (min)	% O_2
17.5	10 ^b	15.1
6.0	12 ^b	16.7
2.0	15 ^b	19.1
1.0	17 ^b	19.5
0.50	18	19.9
0.25	25	20.6
0.10	40	20.4

^a About 1.0 g steel wool was soaked in acetic acid solution, with no subsequent distilled water rinse.

^b The water level rose to a maximum value, then decreased due to hydrogen evolution.

We carried out a number of unsuccessful variations on these procedures. Reaction was slow when 6 M HCl ($t_{1/2} = 40$ min) or 6 M H_3PO_4 ($t_{1/2} > 2$ hr) was substituted for $\text{CH}_3\text{CO}_2\text{H}$ in the initial cleaning process. Soaking in glacial acetic acid, rinsing thoroughly with distilled water, then rinsing once in 1.0 M NaCl or 1.0 M NaCH_3CO_2 also produced slow reactions— $t_{1/2} = 70$ min for NaCl and $t_{1/2} > 2$ hr for NaCH_3CO_2 . Finally, amalgamation of the steel wool had no effect on the rate of reaction.

This system could be adapted also for a longer-term student project. Experiments to determine the conditions for optimal rusting rates would provide an effective introduction to both qualitative and quantitative aspects of kinetics. The concepts of relative rates and half-life could be introduced easily. In many of our experiments (though not all), the volume change followed excellent first-order kinetics, so that actual rate constants could be calculated.