Purpose Designed Reaction Chambers as a Tool for Improving the Efficiency of Demonstration Experiments

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Abstract: Chemical demonstrations are very often performed in closed vessels of limited volume. This, of course, affects the duration of the demonstration. Flow chambers may be used instead, allowing for a significant increase in the time of the demonstration. Several attractive experiments (ranging from very simple to more complex) are described in this paper and are offered as either new or improved lecture demonstrations for both first-year students of chemistry and chemistry majors.

Introduction

For many decades, a number of experiments have been performed as lecture demonstrations, but the viewing of these demonstrations has been limited by the short time period in which the reactions occur. For example, the burning of various materials in pure oxygen is a simple and effective demonstration that is usually performed in a large test tube with a deflagration spoon that passes through a cork [1] using an oxygen-filled glass jar or beaker [2, 3]. The duration of the demonstration in a corked test tube is usually only a few seconds, which is a bit short for a demonstration. If some properties of the reaction product are to be illustrated, further complications arise when using either a corked test tube or a glass jar, as the products are not visible long enough to permit discussion. Other demonstrations, for example, the oxidation of ammonia by oxygen in air in the presence of a platinum wire or copper foil, have also been performed [4–6], but suffer from similar shortcomings. For these reasons, we decided to seek alternative methods of performing these classic demonstrations. We wanted an alternative that would make the demonstrations more vivid and would also make the equipment more compact and the demonstration more automated.

The solution we developed is a flow chamber. It is designed specifically to produce long-lasting (more than just few seconds) and effective demonstrations. Several demonstrations are presented that use two variants of the flow chamber.

Construction of the Chambers

Flow chambers are devices that can be effectively used for chemical demonstrations. They are ideal for demonstrating the burning of different materials in various gasses and are useful for many other demonstrations as well. Because of their construction (gas flows through the chamber), there is no depletion of the gaseous reactant (inevitable in closed vessels)

The volume of the flow chambers we use varies from 100 to 500 cm^3. All the chambers have the same basic construction; two variants are shown in Figure 1.

Because quite often these flow chambers are used for the burning of various materials in oxygen (or chlorine, nitrous oxide, nitrogen dioxide, etc.), it is vital that the best quality of

borosilicate glass be used. The glass should be of the type that survives rather abrupt temperature changes. A glassblower can easily build these chambers. The first chamber (Figure 1a, left) is made from a 20-cm glass tube with ϕ approximately 5 cm. The sidearms are 3 cm in length and are set about 1 cm from the bottom (lower, left) and about 4 cm from the top (higher, right) of the glass tube. The second chamber (Figure 1a, right) is similar to the first chamber, but ϕ is approximately 3.5 cm and the lower-left (inlet) tube is not just attached to the sidewall of the chamber, but is narrowed and ends in the chambers' interior to enable effective bubbling of the gas (see Figure 1b). The inner diameters of the inlet and outlet tubes for both chambers are 4 to 5 mm.

Demonstrations

It is very easy to perform demonstrations in these chambers. We describe three simple, well-known demonstrations using the chambers and discuss the effectiveness of the chambers for the demonstrations.

(1) Burning of Sulfur, Phosphorus, or Graphite in Oxygen. The setup for the demonstration is shown in Figure 2a–b. This demonstration is useful for showing the vigorousness of oxidation in pure oxygen and the nature of the products. We used the larger chamber in this demonstration (there were about 100 students in the lecture room that could easily follow everything that happened on the demonstration table).

Chemicals. Sulfur, phosphorus, graphite, magnesium, oxygen, diluted solution of sodium hydroxide ($c \approx 0.1$ mol dm^{-3}), diluted solutions of various indicators.

Procedure. The rubber balloon is filled with pure oxygen from a container [7]. The micro wash bottles (see Figure 2a for the construction) all contain diluted solutions of different indicators (methyl red, bromophenol blue, and thymol blue were used, see Figure 2b, but other indicators might also be appropriate). The use of three different indicators is justified by their different pK values, that is, by the different pH range in which the color change occurs. Methyl red changes color in the pH interval of 4.8 to 6.0 (red to yellow), bromophenol blue from 3.0 to 4.6 (yellow to blue), and thymol blue (two

Figure 1. (left) Flow chambers for demonstration experiments (two variants). (right) Enlarged photograph of the inlet tube used for bubbling a gas through a liquid (see Figure 1a, right chamber).
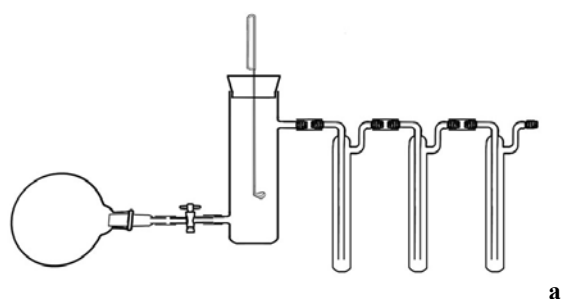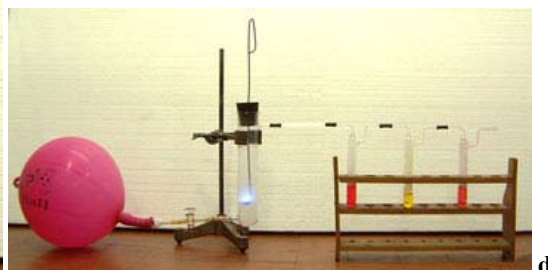

Figure 2. (a) Schematic for the demonstration of oxidation in a flow chamber. (b) Setup for the demonstration of oxidation of sulfur in a flow chamber. The micro wash bottles contain, left to right, alkalized water solutions of methyl red (yellow liquid), bromophenol blue (intense-blue liquid), and thymol blue (deep-blue liquid). (c) Intense blue flame of sulfur burning in pure oxygen. (d) Oxidation of sulfur. The reaction product (SO_2, an acidic oxide) induces the color changes of all indicator solutions.

transitions) from 1.2 to 2.8 (red to yellow) and 8.0 to 9.6 (yellow to blue).

It is helpful to make all the above indicator solutions slightly alkaline (adding, say, one drop of 0.1 mol dm^{-3} NaOH solution). A few milliliters of each solution are poured into three test tubes and are used as a blank. The wash bottles are all connected in a series with rubber or plastic tubing.

Some sulfur (0.5–0.8 g) is put in a deflagration spoon (the spoon passes through a suitable rubber stopper). The sulfur is ignited with a burner and the spoon is put into the chamber. A flow of oxygen (typically 3–5 mL s^{-1}) is established by carefully opening the stopcock until the desired flow rate is reached [8]. The sulfur burns with an intense blue flame. In less than 30 seconds the color changes in all the test tubes. An explanation is given for the very vigorous oxidation in pure oxygen (see Figure 2c) and for the color change of the indicators (Figure 2d).

The same experiment is performed with phosphorus ($\approx$0.7 g) and with graphite rod (a piece of used graphite electrode works well). The instructor points out that the graphite burns although no flame is seen (it only glows brightly). This time the color change in the micro wash bottle containing thymol blue is different (from blue to brownish-green, which is a transition color between blue and yellow). Using these results, the relative strengths of the corresponding acids can be discussed and it can be concluded that the water solution of CO_2 is a much weaker acid than the corresponding solution of SO_2. The solutions formed are sometimes (probably incorrectly) designated as $H_2CO_3(aq)$ and $H_2SO_3(aq)$, respectively, although there is no clear evidence that such molecules really exist.

Other related demonstrations are also possible. It is possible to burn magnesium ($\approx$0.5 g) in a deflagration spoon and to show that the white smoke exhibits basic properties. In this case, however, it is important to put some sand in the bottom of the reaction chamber as a safety precaution. Note that it is not necessary to use pure oxygen for the last demonstration (in fact, if this demonstration is to be performed, we recommend using a balloon filled with air only).

(2) Catalytic Oxidation of Ammonia. The setup for this demonstration is shown in Figure 3a. Unlike demonstrations performed in a closed vessel, this demonstration can last up to 10 minutes. Furthermore, the products of the oxidation of ammonia (nitric oxide, nitrogen dioxide) including the products formed in the side reactions (ammonium nitrite and ammonium nitrate) may readily be recognized and their presence detected.

Chemicals. Concentrated aqueous ammonia, 1% solution of diphenylamine in sulfuric acid (2:1 acid diluted with water works well), Pt foil (or Pt wire).

Procedure. The rubber balloon is filled with air. The U-tube contains a solution of diphenylamine in sulfuric acid. The platinum foil is fastened to a suitable holder, which passes through a rubber stopper that tightly closes the chamber. The chamber is filled by concentrated aqueous ammonia so that the liquid just covers the inlet glass tube (see Figures 1b and 3a).

The demonstration starts by opening the stopcock on the balloon. Air bubbles through the ammonia solution (1–2 mL s^{-1}; for best results, experiment to find the optimum flow rate and the exact volume of ammonia solution) and through the U-tube. Nothing happens (apart from the formation of some white coating on the surface of the liquid in the U-tube, which may easily be explained as due to formation of ammonium hydrogen sulfate). No oxidation reaction occurs at room temperature (at least, not one that leads to the formation of a measurable quantity of products).
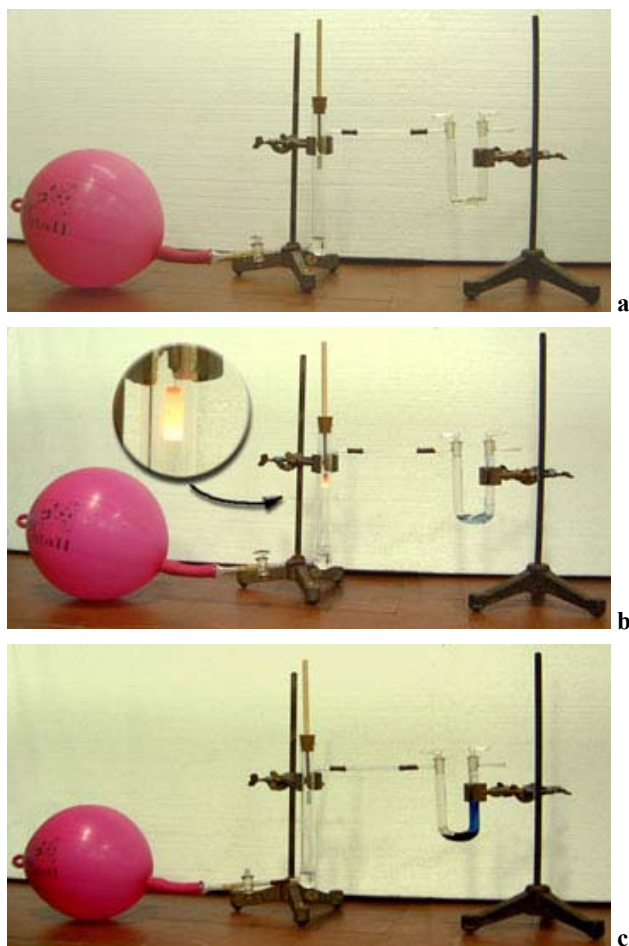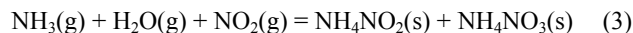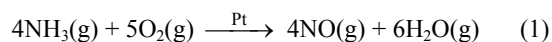
Figure 3. (a) Setup for the demonstration of the catalytic oxidation of ammonia by air in a flow chamber. The U-tube contains a solution of diphenylamine in sulfuric acid. (b) Initiation of the catalytic oxidation of ammonia by air. Note the glowing Pt foil (enlarged photo). The solution in the U-tube has just started to change color. (c) The end of the reaction for the catalytic oxidation of ammonia by air. The deep-blue color is due to the reaction product (ammonium phenylbenzidine hydrogensulfate) of a strong oxidizer (NO₂, nitrites, nitrates) with the diphenylamine in sulfuric acid.

Now, the platinum foil (catalyst) is heated to red hot on a Bunsen burner and placed into position in the chamber. The red-hot appearance of the foil (see Figure 3b) persists for a long time (it fades only after the solution in the chamber is ammonia depleted). White smoke is soon evident, both in the chamber and in the U-tube. After a few seconds, the diphenylamine solution turns deep blue (Figure 3c). This is evidence of the presence of an oxidizing agent. Because neither ammonia nor air gives this reaction, it must be due to some product of the catalyzed reaction of ammonia oxidation. Air is used for the oxidation reaction and as carrier gas; thus, the upper part of the chamber contains a gaseous mixture of air and ammonia as well as water vapor.

The chemical changes that take place in the chamber are rather complex and the composition of the products depends highly on the initial composition of the gas mixture. The following chemical equations might be considered:

$$4NH_3(g) + 5O_2(g) \xrightarrow{Pt} 4NO(g) + 6H_2O(g) \quad (1)$$

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

$$NH_3(g) + H_2O(g) + NO_2(g) = NH_4NO_2(s) + NH_4NO_3(s) \quad (3)$$

The white smoke is, actually, an aerosol of ammonium nitrate and ammonium nitrite. Depending on the composition of the gaseous mixture that is in contact with the catalyst, the product may contain more ammonium salts (if the mixture is oxygen depleted) or more nitrogen oxides (if the mixture is ammonia depleted). The approximate composition of the mixture may be controlled by the level of the solution in the reaction chamber. The higher the concentration, the more ammonia that is contained in the gaseous mixture; hence, more ammonium salts and less nitrogen oxides will be produced. No matter what the true composition of the products, they all oxidize the diphenylamine solution giving a dark blue product (ammonium phenylbenzidine hydrogensulfate).

(3) Catalytic Oxidation of Methanol. The setup for the demonstration is similar to that in Figure 3a, but instead of a U-tube, a glass tube, bent at a right angle, is attached to the chamber, and this tube ends in a test tube (see Figure 4a). This demonstration may also last a long time (the duration is limited by the capacity of the balloon). The product of the oxidation (formaldehyde) is detected using Tollens reagent (giving the silver mirror reaction).

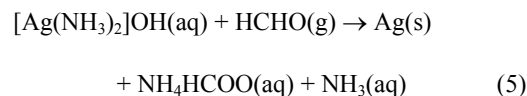*Chemicals.* Methanol, Tollens solution, Pt foil (or Pt wire).

Procedure. The rubber balloon is filled with air. The test tube contains a few milliliters of freshly prepared Tollens solution [9]. The platinum foil is fastened to a suitable holder, which passes through a rubber stopper that tightly closes the chamber.

Again, the demonstration starts by opening the stopcock on the balloon. The stream of air, in this case, **must** be slow—up to 1 mL s⁻¹. Air bubbles through the methanol but no reaction occurs. When the platinum foil is only slightly heated (well before it is red-hot) and is placed in the chamber, it starts to glow. After a short time, the colorless liquid in the test tube darkens until (in about one minute) it is black (see Figure 4b). Silver mirror (albeit not of highest quality) is seen on the wall of the test tube after several minutes (see Figure 4c).

The reaction in the chamber is a catalytic oxidation of methanol:

$$CH_3OH(g) + \tfrac{1}{2}O_2(g) \xrightarrow{Pt} HCHO(g) + H_2O(g) \quad (4)$$
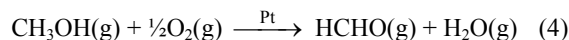
In the test tube, the aldehyde reduces the Tollens solution:

$$[Ag(NH_3)_2]OH(aq) + HCHO(g) \rightarrow Ag(s)$$

$$+ NH_4HCOO(aq) + NH_3(aq) \quad (5)$$

Fehling's reagent could be used instead of Tollens reagent but in that case heating of the test tube is inevitable, which slightly complicates the demonstration.

For the latter two experiments we used the smaller chamber. It is appropriate for lecture rooms with up to 50 students.

Safety Tips and Disposal. Safety goggles should be worn at all times while performing these demonstrations.

To the best of our knowledge, there are no commercially available chambers of this type. The help of a glassblower seems to be inevitable. There is a possibility that the chamber
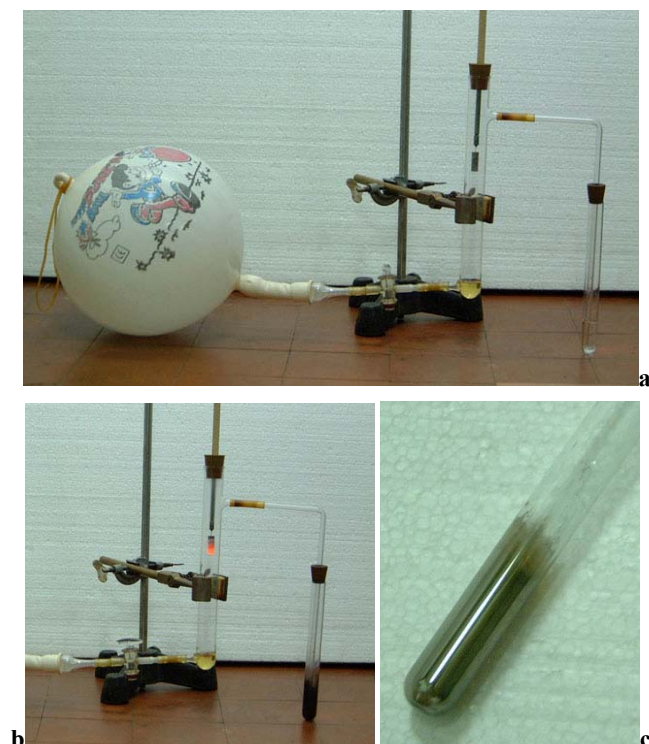
Figure 4. (a) Setup of the demonstration of the oxidation of methanol with oxygen in air in a reaction chamber. (b) Oxidation of methanol with oxygen in air in a reaction chamber (the product of oxidation is identified with Tollens reagent in the test tube, the black color being due to elemental silver). (c) After a few minutes, the formation of a silver mirror is evident.

might crack (most probably at the joints) if not properly constructed resulting in a serious risk of injury!

Concentrated sulfuric acid is very corrosive and highly irritating. Gloves must be worn and a face shield is highly recommended. Aqueous ammonia is corrosive and an irritant. Gloves are recommended.

Pure oxygen is strong oxidizer. When burning various materials in pure oxygen, there is always a fire hazard.

Use only the minimum amount of any substance for the demonstration (this is particularly important when using magnesium). Prepare a vessel with dry sand to extinguish any fire. Never use water to extinguish a fire if alkali or alkaline earth metals are involved.

All products may be diluted in large quantity of water and disposed of in the drain. If Tollens reagent has been used do so immediately. It has been reported that after period of time, highly explosive silver fulminate may be formed. For this reason, never prepare larger quantities of Tollens reagent than required for the demonstration, and never keep prepared solutions for more than one day.

Oxidation of methanol can safely be demonstrated only if air is used as the oxidizer. Never use pure oxygen in this demonstration; an explosion with a very serious risk of injury will occur!

Conclusion

The demonstrations described above are much more vivid when performed using the flow chambers. The experiments last long enough to illustrate all of the important concepts and products and are also attractive to the students. Although they can be performed in a closed vessel, this limits their ability to demonstrate some important properties of the products (especially for demonstrations 2 and 3). The authors encourage instructors to make a few of these flow chambers and try these demonstrations and many others as well.

References and Notes

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7. Alternatively, oxygen may be generated by pyrolysis of $KClO_3$ with about 5% MnO_2; both chemicals must be of reagent-grade purity to prevent explosion! In the latter case, perhaps the easiest way is to fill a gas reservoir with oxygen and then use it instead of the rubber balloon.

8. For a gas-flow control, the bubbles could be counted (thus determining the bubbling rate) in the micro wash bottles. Prior to this, the volume of a typical bubble of gas should be determined (probably the easiest way to do this is to collect the gas escaping from the right-most micro wash bottle into a graduated cylinder under water, and measure the volume of 50 or 100 bubbles).

9. Tollens reagent is prepared by the reaction of KOH(aq) and $AgNO_3$(aq), adding just enough NH_3(aq) to dissolve the dark-brown precipitate. The reagent is used the same day (preferably within few hours).