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A Safe Way of Performing Some Dangerous Experiments. II. Construction of a Safety Dropper

Vladimir M. Petruševski* and Metodija Ž. Najdoski

Institute of Chemistry, Faculty of Natural Sciences and Mathematics,Sts. Cyril and Methodius University, Arhimedova 5, 1000 Skopje, Republic of Macedonia

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Abstract: Due to the high safety risks, chemistry instructors avoid demonstrating many remarkable experiments based on the addition of a liquid to a solid. Well-known examples of such demonstrations are various pyrotechnic mixtures of potassium chlorate and sugar (sucrose), which are usually activated with a drop of concentrated sulfuric acid. Other attractive demonstrations are the addition of water to freshly prepared magnesium phosphide and addition of water to burning magnesium. In all of these demonstrations the reaction that takes place immediately is very vigorous and can be hazardous for the instructor. Because chemistry teachers and instructors usually try to avoid performing experiments that include a hazard, a number of highly attractive experiments may remain unknown to the public. Using a simple homemade device called a safety dropper, one can perform all of these experiments with complete safety, both for the audience and the demonstrator. Details for performing some of these experiments as well as for the construction of the safety dropper are given in this paper. Video clips of demonstrations are included as an aid for inexperienced instructors.

Introduction

In the first paper of this series a simple homemade device, called a safety spoon, was described [1]. This remote-controlled device enables a reaction between a solid and a liquid (e.g. sodium or potassium and water; potassium and bromine, etc.) to be performed with complete safety, both for the audience and the instructor making the lesson more vivid and more instructive.

There are quite a number of situations where adding a liquid to a solid generates a spectacular result in a demonstration. Some known examples include different pyrotechnic mixtures of potassium chlorate and sugar, in which the reaction is usually triggered by a single drop of concentrated sulfuric acid [2–5]; addition of water to freshly prepared magnesium phosphide [6]; and addition of water to burning magnesium [7]. The first experiment is probably one of the oldest demonstrations of self-igniting mixtures. Sulfuric acid reacts with potassium chlorate giving ClO_2, an extremely strong oxidizing agent [8], which triggers the reaction. Once initiated, the sugar–chlorate mixture burns with a beautiful pale-violet flame [9]. The second demonstration is less known. Magnesium phosphide (previously prepared in a crucible by burning a mixture of homogenized magnesium powder and red phosphorus) can readily be hydrolyzed with water, generating phosphine [10] that ignites spontaneously in air. The third demonstration (definitely the most hazardous of the three mentioned) is based on addition of liquid water to burning magnesium. The result is a real fireworks display, with lots of sparks (i.e., small pieces of burning magnesium) being scattered in a wide area surrounding the initial magnesium pile [11]. This experiment is also important as a demonstration to show that water may not always be used for extinguishing a fire.

In all these examples the reaction that occurs upon addition of the liquid is instantaneous and very vigorous. As such, it includes a substantial risk for the instructor. It is somewhat surprising, therefore, that some authors simply ignore the hazard, recommending the above demonstrations be performed as lecture demonstrations in the classroom [2, 5]. The liquid should be added by a dropper [5], glass rod [2], or in some other way [4]. This operation definitely includes high risk, at least for the instructor [12].

Keeping in mind that (1) complete safety for both the audience and the instructor is "*conditio sine qua non*" when doing demonstrations and (2) that it is very important to perform chemical demonstrations (particularly demonstrations that are remarkable as these will always be remembered by the audience and will always serve as a link to the answers for particular questions/problems), we decided to construct devices that will enable the instructor to perform hazardous demonstrations with virtually no risk. In this paper, we describe the safety dropper and its use in several interesting experiments.

Construction of the Safety Dropper

Similarly, as in the case of the safety spoon [1], the safety dropper is a device that should be used in all cases where an experiment is either explosive in nature or a highly vigorous reaction (that can easily go out of control) occurs upon addition of a liquid to the reaction system. The design of one variant of the safety dropper is given in Figure 1 (other alternatives are also possible).

The dropper was built of five joined parts: a micro wash-bottle filled with the liquid that is to be used in the demonstration (a); a bent glass tube (b) ending with a narrow capillary opening (c); long rubber tubing (d), and a small rubber pump (e). The departments' glassblower made the micro wash-bottle, although such bottles are also available commercially. For best results we used a rubber pump from an

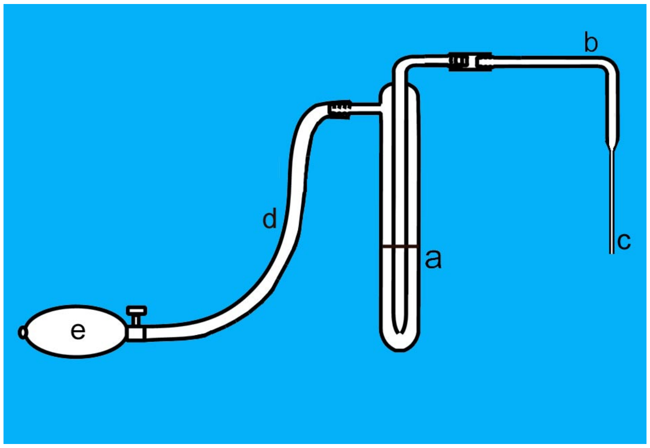

Figure 1. Construction of the safety dropper: micro wash bottle filled with liquid (a), bent glass tube (b), narrow capillary opening (c), long rubber tubing (d), small rubber pump (e).

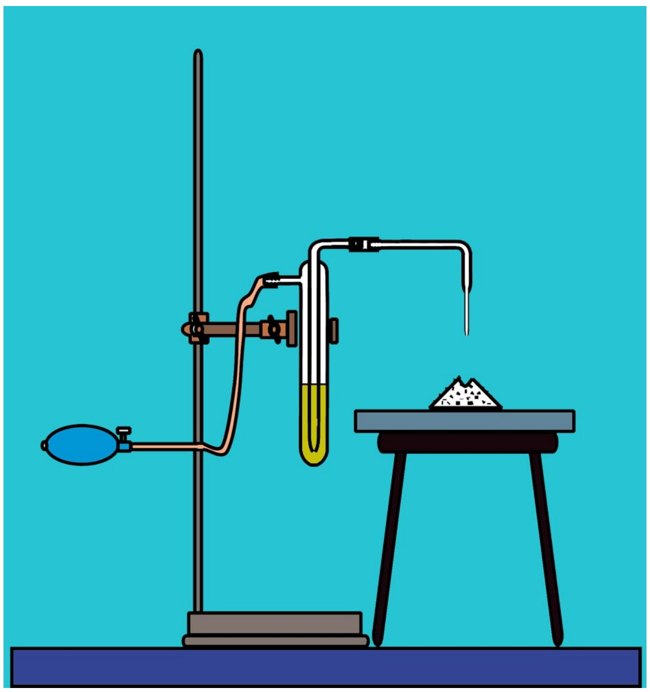

Figure 2. Setup for the experiment. The dropper contains concentrated H_2SO_4, and the heap is a mixture of potassium chlorate and sucrose.

apparatus for measuring blood pressure. After some practice it becomes possible to add only a few drops (by carefully operating the rubber pump and releasing the valve when necessary) to a few milliliters, as necessary for the intended demonstration.

Alternatively, the safety dropper may be constructed in one piece, so as to avoid the rubber connection between the micro wash bottle and the bent tube. We prefer the first version (Figure 1), because it is equally safe for the demonstrations, but is less likely to be broken when used.

Demonstrations

We shall briefly describe the three experiments listed above, which can be performed with complete safety using the safety dropper providing they are done exactly as described.

Pyrotechnic Mixture of Potassium Chlorate–Sucrose. Equal quantities (1–2 grams) of powdered sucrose (white table sugar) and powdered potassium chlorate are carefully mixed on a piece of paper, using a feather (see Safety Tips and Hazards about handling and pounding $KClO_3$). The mixture is transferred onto a brick (or an iron plate put over a tripod) in a pile (≈5 cm in diameter and ≈1.5–2 cm in height), with a shallow crater (≈1 cm) on top. The safety dropper (filled to one third with concentrated sulfuric acid) is mounted above the pile in such a way that the drops of acid will fall in the crater (see Figure 2).

When the instructor is ready to perform the demonstration, he or she should carefully squeeze the rubber pump until 1 to 2 drops of H_2SO_4 fall on the pile. An instantaneous reaction occurs, and the mixture burns with intense (although not very luminous) flame. The result can be seen on the enclosed video clip (Pyrotech.mpg, see the supporting material). Using Na, Sr, Ba, or Cu salts, the flame (that is otherwise pale violet) may be colored increasing the effect of the demonstration [9].

Synthesis of Magnesium Phosphide and Its Hydrolysis with Water. A quantity of 2 g of red phosphorus and 2.5 g of magnesium powder are homogenized and put in a crucible. The crucible is placed in a suitable vessel and is fixed with sand as a protection against fire. The mixture is ignited by a piece of magnesium ribbon (≈5 cm). The reaction is a very vigorous one, so this part of the experiment must be performed in a hood (see Mg3P2synth.mpg, available as supporting material).

After the reaction ceases and the crucible cools down to room temperature, the instructor may proceed with the demonstration. The safety dropper is filled with water and is placed above the vessel (exactly above the crucible mouth). Squeezing the rubber pump, the instructor adds about 0.5 mL of water into the crucible. A brisk evolution of gas occurs (due to hydrolysis of the phosphide) followed by a minor explosion due to instantaneous self-ignition of the PH_3–P_2H_4 mixture (see Mg3P2hydr.mpg available as supporting material). If traces of magnesium metal are still present a minor fireworks display may also be seen.

Addition of Water to Burning Magnesium. Several grams of powdered magnesium are placed on a brick to make a small pile. A piece of magnesium ribbon is used as a fuse, similar to the previous demonstration. The safety dropper filled with water is placed above the heap, and the ribbon is ignited. The powdered magnesium catches fire and soon a large part of the pile glows with a yellow-orange (or white, if larger pieces/aggregates of magnesium are present) glow. Upon pressing the pump, liquid water runs on the burning magnesium. Not only is the fire not extinguished, but a spectacular (usually unexpected) fireworks display results with small particles of burning magnesium scattered around the pile (see Mg+HOH.mpg available as supporting material). It would be very dangerous to add the water using an ordinary dropper, or even a graduated pipette.

By the end of the lecture or demonstration the instructor should discuss the need for use of the safety dropper as well as the chemistry involved in the demonstration(s) performed.

Important. As in our previous paper, we have performed the demonstrations described many times and they have always worked safely and successfully. We are trying to offer a safe way for the instructor to perform these demonstrations providing that he or she takes all safety precautions and performs the demonstration exactly as explained above. We are always willing to advise, discuss, or help in any way any reader who would like to try these demonstrations.

Conclusion

The demonstrations are impressive and educational (the first one is well-known), although they all include some safety hazard. Using the safety dropper the risk is substantially reduced. For those reluctant to perform such demonstrations, the available video clips are surely an absolutely safe alternative.

Safety Tips and Hazards

If any of the demonstrations described are not performed in a hood, place a large transparent safety shield (a polycarbonate one works well) between the demonstration desk and the audience.

Special care must be taken when handling potassium chlorate as it is a strong oxidizing agent. Always wear a face shield when preparing the mixture. Never pound mixtures of chlorate and reducing agents (sucrose, sulfur, phosphorus, etc.) because the mixture may explode. To prepare a fine powder of potassium chlorate, take small quantities of the salt (e.g., 1 g, repeating the procedure several times if larger quantities are needed) and use a clean mortar and pestle. Mixtures of potassium chlorate and sucrose should be blended on a piece of paper, using a feather. If chemicals are used for coloring the flame, particularly if barium compounds are used, the demonstration should be performed in a hood and the waste should be disposed of according to local regulations.

Phosphides and phosphines (both PH_3 and P_2H_4) are extremely poisonous. Always perform the demonstrations of synthesis and hydrolysis of Mg_3P_2 in a hood. These substances should be handled with extreme care. Dispose of the residue only after complete hydrolysis with water.

Never look directly at the flash of the burning magnesium unless you use special safety goggles that absorb UV radiation. This demonstration shows definitively that water should not be used to extinguish burning magnesium. In case of accident cover the burning magnesium with large quantities of sand.

Supporting Materials. A SONY camera (CCD-TR 620E PAL) coupled to ATI RAGE FURY PRO-vivo graphic card was used for recording and digitalization of the following supporting files. A WinZip file containing supporting files is available. (1) Pyrotech.mpg. An 18-s video clip showing the self-ignition of the chlorate–sucrose mixture with a drop of sulfuric acid. (2) Mg3P2synth.mpg. A 52-s video clip showing direct synthesis of magnesium phosphide. (3) Mg3P2hydr.mpg. A 29-s video clip showing hydrolysis of magnesium phosphide and self-ignition of the phosphine mixture. (4) Mg+HOH.mpg. A 42-s video clip showing the reaction of liquid water with burning magnesium. (http://dx.doi.org/10.1007/s00897020583b)

References and Notes

1. Petruševski, V. M.; Najdoski, M. Ž. *Chem. Educator* **2002,** *7,* 81–83; DOI: 10.1007/s00897020546a.
2. Fowles, G. *Lecture Experiments in Chemistry;* Bell & Sons Ltd.: London, 1959; p 23.
3. Eliason, R.; Lee, E. J.; Wakefield, D.; Bergren, A. *J. Chem. Educ.* **2000,** *12,* 1581–1583.
4. Gilbert, G. L.; Williams, L. G.; Shakhashiri, B. Z.; Dirreen, G. E.; Juergens, F. H. In *Chemical Demonstrations: A Handbook for Teachers of Chemistry*, Vol. 1; Shakhashiri, B. Z. Ed.; University of Wisconsin Press: Madison, 1983; pp 79–80.
5. Herak, J. *General and Inorganic Chemistry. Experiments;* Školska knjiga: Zagreb, 1968; p 281 (in Croatian).
6. Herak, J; *General and Inorganic Chemistry. Experiments;* Školska knjiga: Zagreb, 1968; p 333 (in Croatian).
7. Dragić, R. *Methods in Chemistry Education;* Svijetlost: Sarajevo, 1973; pp 64–65 (in Croatian).
8. The reaction is, actually, a very complex one. It includes several parallel channels, giving rise to ClO_2, $HClO_3$, $HClO_4$ and other strong oxidizers.
9. Flames of other colors may easily be obtained. $SrCl_2$ or $Sr(NO_3)_2$ produces a red color, $BaCl_2$ or $Ba(NO_3)_2$ produces green, a small amount of ordinary salt, NaCl, produces an intense yellow color, and $CuCl_2$ produces blue.
10. It probably also contains traces of diphopshine, P_2H_4. Diphosphine is a liquid that spontaneously catches fire when in contact with air.
11. When water is added to burning magnesium, magnesium is readily oxidized reducing water to hydrogen gas (magnesium oxide is the ‘waste product’). The temperature is high enough to ignite the hydrogen, so the gas also burns (usually with a pale pink or pale orange flame, which is less luminous when compared to the brilliance of the burning magnesium).
12. The authors of reference 4 say that “The reaction starts slow, evolving smoke after 1–2 seconds, and then the pile bursts into flame”[4]. This does not agree with our experience. We have found that the reaction is practically instantaneous and hence the demonstration, as described, is very hazardous (see the video clip Pyrotech.mpg).