Microscale Gas Chemistry
Supplement: Other Microscale Gas Methods

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The in-syringe method first proposed by Hubert Alyea and extensively developed by our group is by no means the only microscale gas chemistry technique. A variety of other microscale gas chemistry methods are currently in use and we summarize here those of which we are aware. Other such methods will be added to this list as we become aware of them.

Viktor Obendrauf (Austria) has developed numerous methods of gas generation using 10-20 mL syringes, small test tubes, soft latex stoppers or septa and blunt needles similar to that proposed by Alyea. His refinements include the use of a reagent syringe (Figure 1). Much of his work with microscale gas chemistry (methods and numerous interesting experiments) has appeared in German-language journals such as *Chemie & Schule* (Salzburg) but a good overview of his methods can be found in his web-based article in *Chemical Education Journal* [Obendrauf, 2002]. Among many ingenious features, Obendrauf uses a syringe packed with charcoal to absorb unwanted gases as they continue to generate in the reaction test tube. Obendrauf's methods using medical apparatuses (septa, needles, etc.) for the generation of gases have led to various companies marketing the necessary equipment for European markets [Menzel, web].

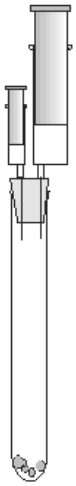

Figure 1. Viktor Obendrauf uses a small test tube containing a solid reagent with the second reagent in a small syringe; the large syringe is the gas receiving syringe [Obendrauf, 2002].

Alan Slater and Geoff Rayner-Canham generate gases in one cell of a 24-well plate and collect the gas in pipet bulbs (Figure 2) [Slater, 1994].

Figure 2. Slater and Rayner-Canham describe the generation of gases in a 24-well plate.

An ingeniously small-scale, inexpensive apparatus is described by Lise Kvittingen and Richard Verley (Norway) in which the reaction vessel is a centrifuge tube and the dropping funnel is a thin-stem pipet. The gas collection vessel is a wide-stem pipet and used the method of water displacement (Figure 3) [Kvittingen, 2004].

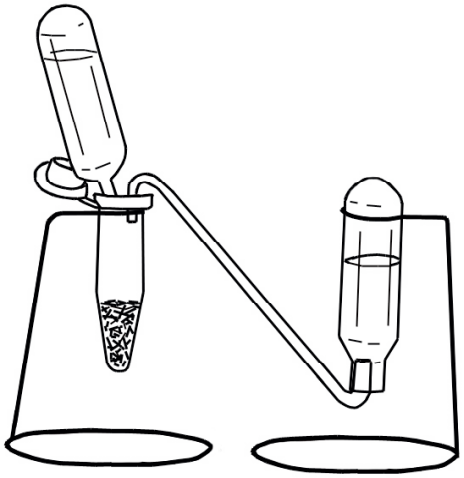

Figure 3. Kvittingen and Verley's apparatus is similar in principle to the traditional 19th Century equipment — only 100 times smaller.

Certain gases are readily produced by electrochemical methods and microscale methods for generating gases this way using pipet bulbs have also appeared. Per-Odd Eggen and Lise Kvittingen have described the electrolysis of water using one or two pipet bulbs as shown in Figure 4 [Eggen, 2004].

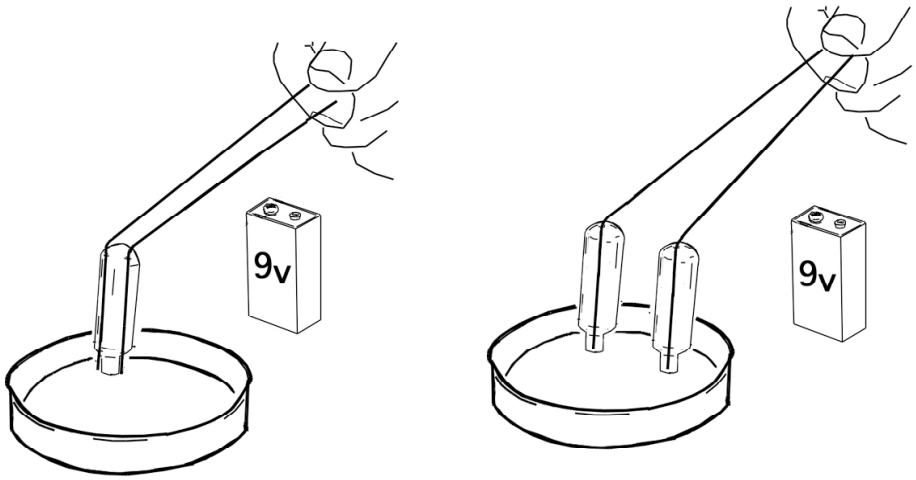

Figure 4. Electrochemical method for the generation of gases using a pipet bulb and a 9-volt battery.

Ozone is also conveniently produced by an electrochemical method as described by Jorge Ibáñez and Bruce Mattson and shown in Figure 5. This method is well-suited for use of gases *in situ* [Ibáñez, 2005a].

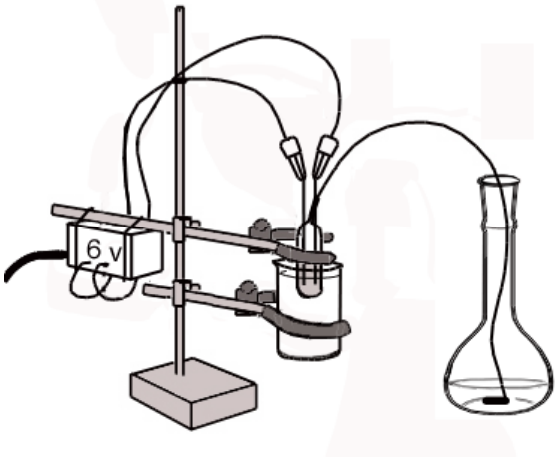

Figure 5. Electrochemical generation of ozone in a pipet bulb.

Generation of chlorine dioxide for *in situ* use has also been described by Ibáñez, Anderson and Mattson. The device which uses a Beral pipet as the generation chamber is shown in Figure 6. This method could be extended for use with a wide variety of gases. In this and the previous example, the stem of a Barel pipet has been stretched to 30 cm or more in length so that the gas can be conveniently delivered to the desired destination [Ibáñez, 2005b**].**

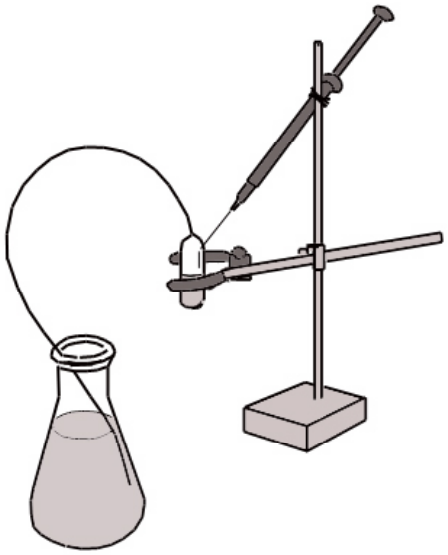

Figure 6. Use of a pipet bulb to generate gas in situ — especially useful for gas samples that cannot be stored because they are unstable.

Microscale gas chemistry need not involve expensive or expensive apparatus. Martin Choi describes making chlorine in a drop of solution in a Petri dish and allowing gas

diffusion to carry the chlorine to various reagents present in other drops in the dish (Figure 7) [Choi, 2002].

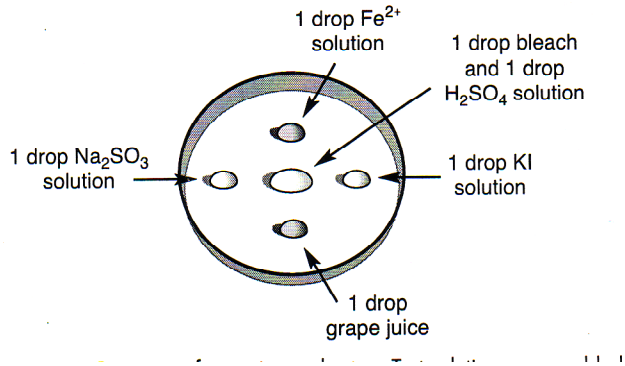

Figure 7. Gas chemistry by diffusion. [Choi, 2002] Used with permission from the Journal of Chemical Education, copyright 2002, Division of Chemical Education, Inc.

Using a test tube and Beral pipet with slits cut into the bulb, James Kilroy and Mary Virginia Orna describe the simple device pictured in Figure 8 in various attitudes. An especially nice feature is that one can stop gas generation at any time by lifting the reaction chamber out of the aqueous reagent [Kilroy, 1994].

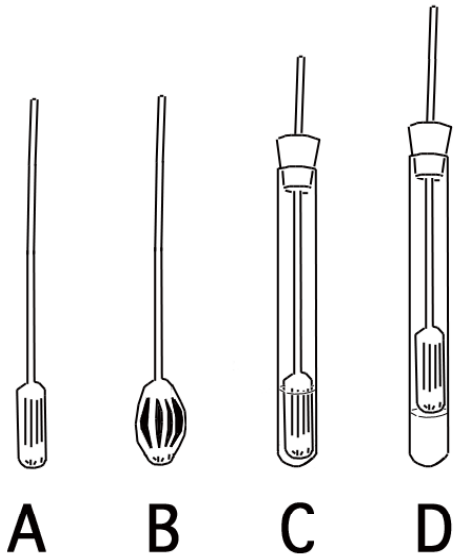

Figure 8. From left: A. Beral pipet with slits cut into the bulb; B. opening the slits to add solid reagent; C. pipet in contact with aqueous reagent and concomitant production of gas; D. pipet raised out of aqueous solution to stop gas generation

The Kipp generator, described in the Brief History supplement at this website has inspired similar small-scale devices. A 20-mL gas capacity one-piece clear plastic Kipp generator, pictured in Figure 9 was invented in the former Federal Republic of Germany and popularized by Andreas Kometz and others [Kometz, 2001].

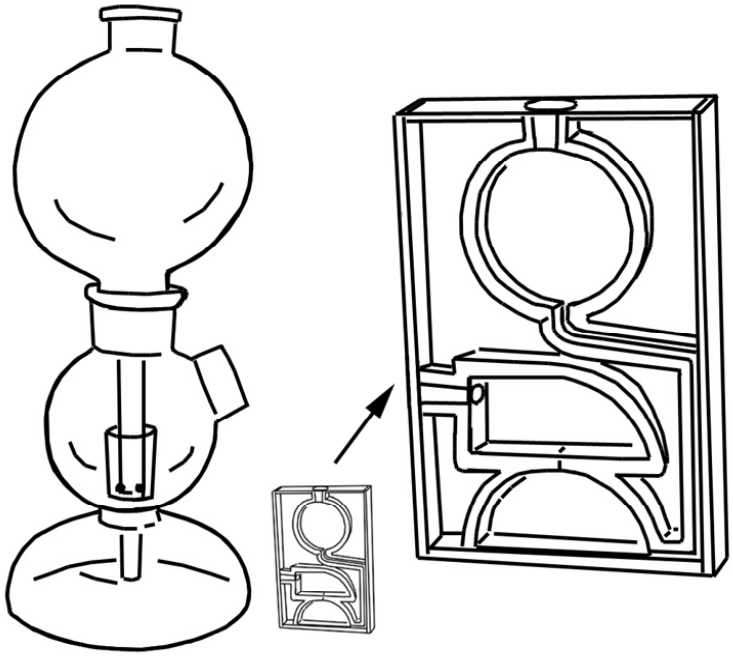

Figure 9. A 20 mL Kipp generator standing next to a traditional 2.5 L model from the 19th century

From its inception, microscale methods have developed along two lines in terms of equipment sophistication and expense. Glass manufacturers have created ingeniously small apparatuses that tend to be expensive, while other methods tend to be inexpensive and use "consumable" plasticware. Figure 10, pictures an example of the former, a micro-Kipp generator about the size of a 250 mL beaker and described by Jinhua Wang [Wang 2003]. The aqueous reagent is placed in the left reservoir and the solid reagent goes in the right reservoir where it rests on a sintered glass frit that separates it from the aqueous reagent.

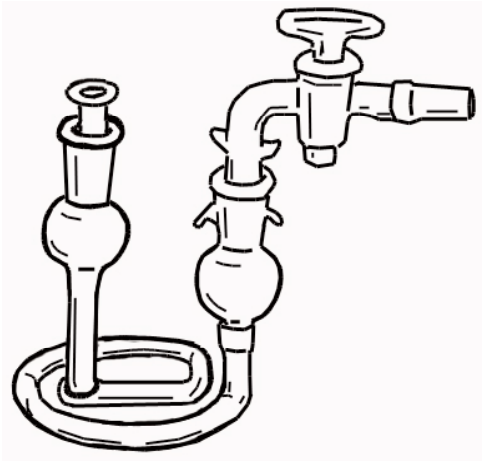

Figure 10. Another design of a microscale gas chemistry generator.

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Our Microscale Gas Chemistry Website.

Our gas book, numerous color photographs of procedures, experiments and demonstrations, a few QuickTime movies of techniques and experiments are available on the web at our microscale gas chemistry website. Equipment ordering information and historical information are also available at the site. Use of the site is free.

http://mattson.creighton.edu/Microscale_Gas_Chemistry.html