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tested demonstrations

A Multicolored Luminescence Demonstration

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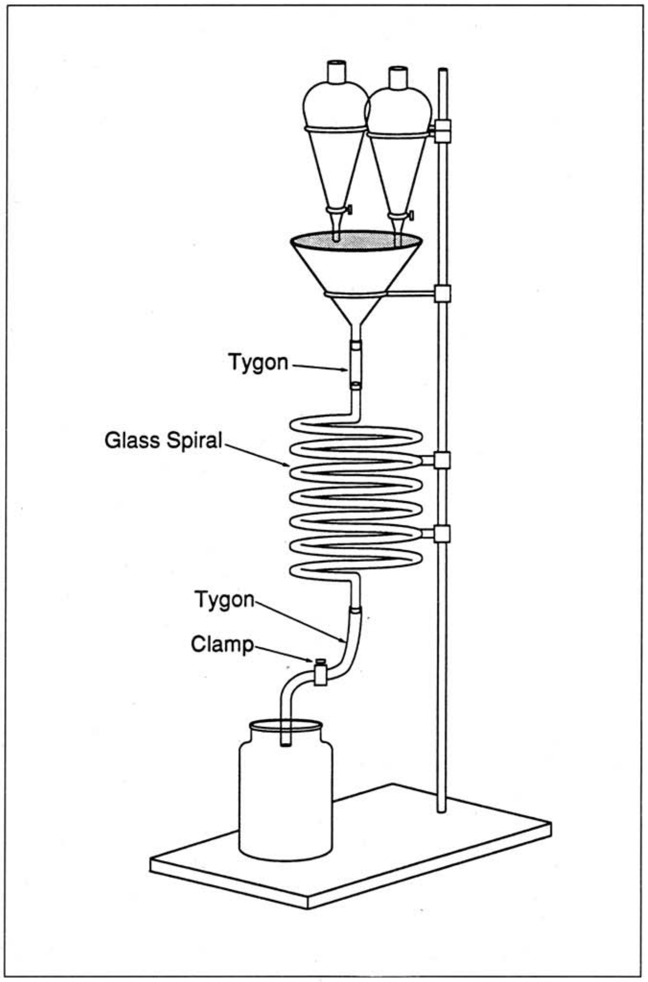
Chemiluminescence demonstrations are so fascinating to both students and their instructors that it is tempting to describe them as "exocharmic" (*1, 2*). One of the most popular demonstrations in this category involves oxidation of luminol (3-aminophthalhydrazide) with $Fe(CN)_6^{3-}$ in basic solution (*3, 4*). The products of this reaction are N_2 and an aminophthalate ion in an electronically excited state, which decays to the ground state by emission of a photon with λ_{max} = 425 nm. Because of the low quantum yield and short emission time of luminol, many people have switched to the H_2O_2 oxidation of lucigenin (bis-*N*-methylacridinium nitrate) (*5*). In their discussion of chemiluminescence demonstrations, Shakhashiri, et al., described the use of a glass-spiral assembly that significantly enhances this demonstration.

While studying possible variations on the lucigenin demonstration (*6*), we encountered a method of producing a demonstration that gradually turns color from one end of the visible spectrum to the other. By using a clamp to slow down the rate at which liquid flows through the spiral, it is possible to generate almost the entire visible spectrum within the spiral at the same time. Once this has been accomplished, the clamp can be closed and the glow will last for as long as 15 minutes. (Best results are obtained when the demonstration is done in a darkened room.)

Materials

The first step in assembling the apparatus used in this demonstration is to build a glass-spiral assembly,[1] such as the one described by Shakhashiri, et al. (*4, 5*). Attach the glass spiral to a ring stand. Support a glass funnel in an iron ring at the top of the ring stand and connect the stem of the funnel to the top of the glass spiral with Tygon tubing as shown in the figure. Attach two iron rings to the ring stand above the glass funnel. Then place two 1-L separatory funnels in the iron rings and position their spouts against the inner wall of the glass funnel. Attach a second piece of Tygon tubing to the bottom of the glass spiral and use a pinch-clamp to collapse this tubing partially. Insert the exit tube of the spiral into a 2-L Erlenmeyer flask, which is used to collect the liquid that flows through the apparatus.

[1] This demonstration also can be done by replacing the glass spiral with an apparatus constructed by coiling Tygon tubing around a Plexiglas tube roughly 6 in. in diameter.



Set-up for the luminescence demonstration (see cover for color photo of results).

Prepare solutions A, B, C, and D as described below. Gloves and safety goggles must be worn during both the preparation and handling of these solutions.

Solution A

Dissolve 8.0 g of sodium hydroxide in 650-mL of distilled water. Add 300 mL of ethanol and 50 mL of 30% hydrogen peroxide. Stir until thoroughly mixed. (This solution must be prepared on the day the demonstration is done.)

Solution B

Dissolve 0.2 g of lucigenin [Aldrich: B4,920-3] in 1 L of distilled water. (This solution will keep for several months.)

Solution C

Dissolve 0.5 g of rhodamine B [Aldrich: R95-3] in 1 L of ethanol. Note that rhodamine B is a mutagen and a cancer-suspect agent.

Solution D

Dissolve 1 g of fluorescein [Aldrich: 16,630-8] in 1 L of ethanol

Procedure

In their description of the H_2O_2 oxidation of lucigenin demonstration (*5*), Shakhashiri, et al., proposed two alternate procedures. The first involved mixing solutions A and B in the glass funnel of the glass-spiral apparatus described above. The second involved adding a fluorescent dye solution, such as solution C or D, to a beaker containing solution B, and then adding solution A to this beaker.

We propose a third alternative. Pour 1 L of solutions A and B into the separatory funnels in the figure. Simultaneously open the stopcocks on the two separatory funnels, allowing solutions A and B to pour into the funnel at the top of the glass-spiral apparatus until the funnel is approximately one-third full. Optimum results are obtained by adjusting the stopcocks so that the funnel remains about one-third full. The rate at which liquid flows through the spiral also can be controlled by opening or closing the pinch-clamp on the exit tube.

If you find that this approach generates air bubbles within the glass spiral, it is possible to start by filling the spiral with ethanol, and then allowing this solvent to flow through the exit tube as the pale-blue luminescent solution flows out of the glass funnel into the top of the spiral.

While solutions A and B are flowing into the glass funnel, add a small (5-mL) aliquot of solution C. The rhodamine B dye in this solution will gradually change the color of the luminescence from pale-blue to red. Once this process has begun, add a small aliquot of solution D. The fluorescein dye will now shift the color toward the green end of the spectrum. To shift the color back toward the red end of the spectrum, add a second aliquot of solution C. This process can be repeated until the whole spiral is a brilliant blend of colors, at which point you can clamp off the exit tube and freeze the colors for as long as 15 min. (*6*).

Caution: If you fill the funnel too full with solutions A and B, you will reduce the concentration of the dye solution and not achieve good color separation in the spiral.

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Rediscovering the Wheel

The Flame Test Revisited

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With this paper we hope to establish awareness of a forgotten but useful flame test demonstration that can be easily viewed by all students in a large or small lecture hall. In one of the typical techniques used today, a platinum wire is dipped in a test solution and then exposed to the flame of a Fisher burner or the solid chemical is put directly on the platinum wire. This produces a small flame of short duration. Spraying a concentrated chemical solution into a Fisher burner's flame is more successful. (The burner is clamped at 90° to its normal position over a sink to minimize cleanup.) However, with this approach the flame emission is brief. Also, the spray bottles are difficult to aim in the dark, and the cleanup is time-consuming.

Recently, a device for another flame test demonstration was discussed in which a 2-L plastic bottle is placed over a Tirrill burner. An atomizer is used to introduce the chemical salt into the air volume of the 2-L bottle (*1*).

A Revival of an Elegant and Colorful Demonstration

The flame test demonstration presented in this paper is much quicker and more convenient than those discussed above. In fact, a dozen flame tests can be demonstrated all at the same time. Our search for a superiorly bright test was rewarded when we came across an old test with a simple arrangement that gives an intense flame for several minutes. The height of the flame is usually 10–15 cm.

This demonstration was reported in the literature by Professor Paul Arthur in 1939 (*2*). How this simple and convenient flame test was lost from the chemical educator's repertoire is a mystery. Perhaps it never reached a large audience because it was given in a speciality book.

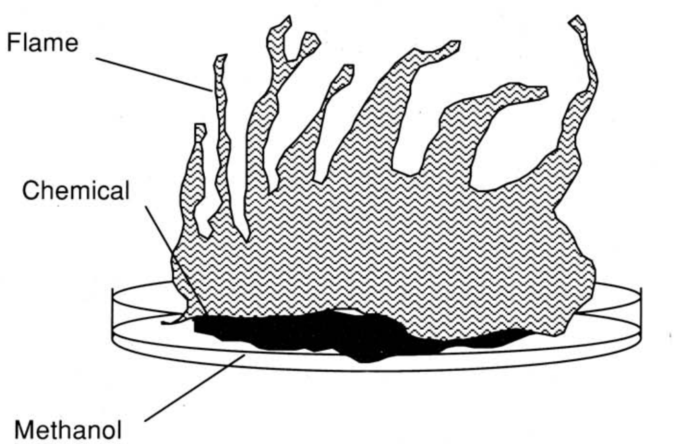
Setup

To maximize the effect of the demonstration, the room should be darkened as much as possible (even though several of the flames can be seen in a classroom with normal lighting). The apparatus for the flame test is a large Petri dish, a watch glass to cover it (or a matching pair of Petri dishes), and a heat-resistant pad.

Procedure

- ***Caution***: Adequate ventilation must be used.
- ***Caution***: Methanol must be handled with care. It is labeled as a health hazard. Its flammability is considered "severe". Methanol's reactivity is "slight", and its contact hazard is "moderate." Methanol may be fatal if swallowed, inhaled, or endured in prolonged contact. It may cause blindness or irritation. Methanol's flashpoint is 11 °C. Methanol should be kept enclosed in a bottle.
- ***Caution***: People standing close to the demonstration should not stare too intently at the flames.

Place about a teaspoon of the desired chemical in the Petri dish. Add about 5–10 mL of methanol, and ignite (see figure). After several seconds, the full intensity of the color is seen. The brilliant flames are clearly visible to over 350 students.



Demonstration setup for flame tests in a Petri dish.