

Experiment 9

Chemiluminescence: Glow stick in a beaker

For the Instructor

All CH_2Cl_2 and oxalyl chloride solutions should be stored in a hood. The MSDS sheets for these chemicals should be reviewed before the experiment.

In part A, the carbonate buffer solution with the luminol and copper should have a pH of approximately 9 if made correctly. The fluorescein and rhodamine B solutions should be made by adding 0.05 g of the dye to a dropping bottle and then adding 20 mL water.

We have found starting with a purchased solution of oxalyl chloride much easier than starting with the pure reagent. The smell of oxalyl chloride necessitates working with the 2 M solution in a hood. All of part B is best performed in the hood. Waste containing dichloromethane should be placed into its own waste container. To make the oxalyl chloride solution for the students to use, dilute 25 mL of the 2 M oxalyl chloride solution in 230 mL CH_2Cl_2 ; then dilute this solution 1 part in 5. Need 2.4 L of final dilution for class of 24 students. This solution should be stable for prolonged periods of time if kept in a sealed container in the dark.

The volumes of water used can be reduced by up to a half to reduce the amount of waste.

A truly excellent discussion of chemiluminescence can be found in Shakhshiri, *Chemical Demonstrations*, Vol. 1 (1).

Part A

Preparation Information - 24 students

Sodium carbonate	48.0 g
Luminol (3-aminophthalhydrazine)	2.4 g
Sodium bicarbonate	48.0 g
Ammonium carbonate monohydrate	6.0 g
Copper sulfate pentahydrate	4.8 g
30% hydrogen peroxide	60 mL
Fluorescein	0.05 g
Rhodamine B	0.05 g

Necessary Equipment - 24 students

500 mL Erlenmeyer flask	2 per student or group
200 mL beaker	3 per student or group
100 mL graduated cylinder	1 per student or group
10 mL graduated cylinder	1 per student or group
Glass stirring rod	1 per student or group

Dropping bottle 2
Paper towels
Disposable gloves
Weigh boats or weighing paper

Part B

Preparation Information - 24 students

30% Hydrogen peroxide	1.5 mL per student
Dichloromethane	≤3 L per class
9,10-Bis(phenylethynyl)anthracene	0.005 per student or group
9,10-Diphenylanthracene	0.005 per student or group
2 M Oxalyl chloride in dichloromethane (or oxalyl chloride)	≤50 mL per class

Necessary Equipment – 24 students

Glass stirring rod	2 per student or group
20 mL beaker	1 per student or group
100 mL beaker	2 per student or group
10 mL graduated cylinder	1 per student or group
25 mL graduated cylinder	1 per student or group
100 mL graduated cylinder	1 per student or group
Disposable gloves	
Weigh boats or weighing paper	
Paper towels	

Literature Cited

(1) Shakhashiri, B. Z. *Chemical Demonstrations: A Handbook for Teachers of Chemistry*; The University of Wisconsin Press: Madison, WI, 1983; Vol. 1; pp 125-204.

Answers to Pre-Laboratory Questions

1. Search the Internet to find the structure of fluorescein and rhodamine B. Draw the structures below. Do they have any similarities? What is the role of these molecules in the experiment?

Structures can be found by SciFinder search if institution as subscription; good chance to teach students how to use SciFinder. Also can be found using Wikipedia.

They have a identical C₁₉O cores.

They are sensitizers.

2. Find the MSDS on the Internet for 30% hydrogen peroxide, dichloromethane, and oxalyl chloride. What hazards do they represent?

Search name of chemical and "MSDS".

Oxalyl chloride (as CHCl_2 solution) – lachrymator, corrosive, poison by ingestion, and possible cancer hazard.

30% H_2O_2 - Strong oxidizer. Contact with other material may cause a fire. Harmful if inhaled. Corrosive. Causes eye and skin burns. May cause severe respiratory tract irritation with possible burns. May cause severe digestive tract irritation with possible burns.

Dichloromethane - Harmful if swallowed or inhaled. May be harmful by skin contact. Eye and skin irritant. Readily absorbed through the skin. Asphyxiant. Causes CNS depression. Possibly carcinogenic in humans. Possible mutagen. Experimental reproductive effects.

3. What parts of the experiment require the use of a hood?

All parts that utilize oxalyl chloride.

