The glow stick reaction

Education in Chemistry
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Technician notes

Preparation of the chemiluminescent compound, bis(2,4,6-trichlorophenyl) oxalate (TCPO)

Kit

- 4.7 g 2,4,6-trichlorophenol (TCP) (irritant, harmful if swallowed, dangerous for the environment, suspected carcinogen)
- 40 cm^3 acetone (flammable, irritant)
- 3.5 cm^3 triethylamine (flammable, harmful if swallowed, toxic by skin contact)
- 1.5 cm^3 oxalyl chloride (corrosive, respiratory irritant, contact with water releases flammable gases)
- 100 cm^3 round-bottomed flask
- drying tube
- large beaker and approx. 200 g ice for an ice bath
- suction filtration apparatus
- magnetic stirrer and follower

Preparation

Work in a fume cupboard. Wear gloves and splash-proof goggles. Place a beaker to act as an ice bath on a magnetic stirrer and clamp a 100 cm^3 round bottomed flask within it. Add 4.7 g of TCP followed by 40 cm^3 acetone. Start stirring the mixture and add 3.5 cm^3 of triethylamine. Cool the mixture by adding ice and water to the beaker. Slowly add 1.5 cm^3 of oxalyl chloride with time for cooling on each addition. Add one drop of triethylamine, place a drying tube on the flask and leave stirring for 20 minutes. The product will crystallise out and can be filtered, air-dried under suction and will remain stable in a sample vial for several months.

The glow stick reaction

Kit

- glass 20 cm^3 sample vial with lid
- 5 cm^3 ethyl acetate (flammable, irritant)
- 5 cm^3 ethanol (flammable, harmful if swallowed)
- 2 cm^3 10 vol hydrogen peroxide in ethanol (flammable)
- 0.1 g TCPO (irritant)
- 0.05 g sodium acetate
- 0.05 g rhodamine B (harmful if swallowed, causes serious eye damage, dangerous for the aquatic environment)
- spatulas and wooden splints/microspatulas

In front of the class

For the best effect work in a darkened room. Wear eye protection and gloves. Transfer approx. ½ a spatula of TCPO to the sample vial (approx. 0.1 g) and the tip of a splint/microspautula of the other two solids (approx. 0.05 g). Add the ethyl acetate and ethanol then shake with the lid on to dissolve the dye and suspend the base. Finally add the hydrogen peroxide and shake the mixture one more time to initiate the reaction. The red glow will persist for several minutes.

Disposal

The mixture can be left to evaporate in a beaker at the back of a fume cupboard to leave a crystalline residue of 2,4,6-trichlorophenol and dye. The bulk of the solid material can be scraped into a container for collection by a registered waste contractor and any remaining material can be diluted and poured down a foul water drain.