**PHOTOChemistry: SYNTHESIS OF LUMINOL**

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Luminol (4) is a chemiluminescent substance that is made by reduction of nitrophthalhydrazide (3), formed upon thermal dehydration of 3-nitrophthalic acid (1) with hydrazine (2). Most exothermic reactions release energy in the form of heat, but chemiluminescent reactions release energy in the form of light. When luminol is allowed to react with hydrogen peroxide and potassium ferricyanide in a basic solution, the emission of a blue-green light is observed.

Luminol is used by crime scene investigators to locate traces of blood, even if it has been cleaned or removed. The investigator prepares a solution of luminol and the activator and sprays it throughout the area under investigation. The iron present in any blood in the area catalyzes the chemical reaction that leads to the luminescence revealing the location of the blood. The amount of catalyst necessary for the reaction to occur is very small relative to the amount of luminol, allowing the detection of even trace amounts of blood. The glow lasts for about 30 seconds and is blue. Detecting the glow requires a fairly dark room. Any glow detected may be documented by a long exposure photograph.

**Procedure**

A. Synthesis of luminol

Heat a test tube containing 3 mL of distilled water in a warm (~ 80° C) water bath.

In a second test tube (10 x 100 mm), heat a mixture of 200 mg of 3-nitrophthalic acid (3-nitrophthalic anhydride can also be used) and 0.4 mL of 8% aqueous hydrazine on a hot oil bath until the solid dissolves.
Clamp the test tube so that it stands vertically in the oil bath. Once the solid has dissolved, add 0.6 mL of triethylene glycol to the test tube along with a boiling chip, a thermometer, and an aspirator tube connected to an aspirator. Boil solution vigorously to distill the excess water. The solution should boil at 110° C and then over a 3-4-minute time period it will rise to 215° C. Maintain the temperature of the solution between 215-220° C for 2 min by adding and removing heat as necessary.

Remove the tube from the oil bath and allow the solution to cool to ~ 100° C. Then add the 3 mL of hot water to the solution. Cool the solution in cool water and collect the light yellow solid precipitate (nitrophthalhydrazide) by vacuum filtration.

Transfer the nitrophthalhydrazide compound back into the uncleaned test tube in which it was prepared and add 1.0 mL of 10 % sodium hydroxide solution with stirring. To the resulting deep brown-red solution add 0.6 g of sodium hydrosulphite dihydrate. Wash down the walls of the test tube with a little water and heat to boiling with stirring. Keep the mixture hot for 5 min and then add 0.4 mL of acetic acid. Cool the test tube in a beaker of cold water while stirring. Collect the resulting precipitate of light-yellow luminol by vacuum filtration. Weigh the crude product.

B. Chemiluminescent reaction

Dissolve the crude luminol product in 2 mL of 10% sodium hydroxide solution and 38 mL of water – this is “Solution A”. Prepare a second solution, B, by mixing 4 mL of 3 % aqueous potassium ferricyanide, 4 mL of 3 % hydrogen peroxide and 32 mL of water.

In a dark place (with the lights out), pour solution A and solution B simultaneously into a funnel connected to tubing that drains into a large beaker.

As an alternative experiment, the chemiluminescent reaction can be produced using blood. If there are any volunteers, mix solutions A and B, omitting the ferricyanide from solution B, and then add a few drops of blood to the mixture and swirl.

References


Chemiluminescence figure can be access at: http://en.wikipedia.org/wiki/File:Luminol2006.jpg