Synthesis of Luminol

Discussion: The oxidation of 3-aminophthalhydrazide, commonly known as luminol, occurs with a striking emission of blue-green light. Most exothermic chemical reactions produce energy in the form of heat, but a few produce light and release little or no heat. This phenomenon, chemiluminescence, is usually an oxidation reaction.

NOTE: hydrazine is a carcinogen. Wear gloves and use caution.

Microscale Procedure:

a. Heat a tube with 3 mL water on steam bath.

b. Then heat a mixture of 200 mg of 3-nitrophthalic acid and 0.4 mL of 8% aqueous solution of hydrazine in a 10 x 100 mm reaction tube over a hot sand bath.

c. Wait until the solid has dissolved.

d. Add 0.6 mL of triethylene glycol, and clamp the tube vertically. Insert a boiling chip, a thermometer and an aspirator tube connected to an aspirator. (see figure in reference text)

e. Boil the solution to distill the excess water.

f. There will be a period during which the solution will boil at 110°C and then over a 3- or 4-minute period it will rise to 215°C.

g. At this temperature (215°C) remove the tube from the sand bath, BUT make sure that you keep the temperature at 215°C by placing the tube back on the sand bath when a temperature drop is noticed.

h. Maintain this temperature for about 2 min.

i. Then cool to 100°C and add 3 mL of hot water.

j. Cool the tube in cold water.

k. Collect the yellow granular nitro compound using vacuum filtration.

l. Dry weight of the product should be about 140 mg.

m. The nitro compound does not need to be dried and can be reduced immediately.

n. Place the nitro compound back to the unclean tube that it was made in, and add 1.0 mL of 3 M sodium hydroxide solution, stir.

o. A brown red solution should form. To this solution you should add 0.6 g of fresh sodium hydrosulfite dihydrate (Na₂S₂O₄ · 2 H₂O).

p. Wash the solid down the walls of the tube using a little water.

q. Heat the solution to its boiling point, stir and keep the mixture hot for 5 min.
r. Add 0.4 mL acetic acid and cool the tube in a beaker of cold water.
s. Stir and collect the resulting precipitate of yellow luminol by vacuum filtration

Clean up: Dilute filtrate with water and neutralize with sodium carbonate. Add 3 mL of bleach and heat the mixture to 50°C for 1hr. only then dilute the mixture with water and flush down the sink.

**Light Producing Reaction**

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\[ \begin{align*}
\text{NH}_3^+ & \quad \text{O}^- \\
\text{N} & \quad \text{NH} \\
\text{O} & \quad 2\text{OH}^- \\
\text{N} & \quad \text{N} \\
\text{O}^- & \quad \text{O}^- \\
\text{NH}_2 & \quad \text{O} \\
\end{align*} \]
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**Microscale Procedure:**

a. Dissolve the moist luminol in 2 mL of 3 M sodium hydroxide solution and 18 mL of water. This is stock solution A.
b. Prepare stock solution B, by mixing 4 mL 3% aqueous potassium ferricyanide, 4 mL of 3% hydrogen peroxide and 35 mL of water.
c. Dilute 5 mL of solution A with 35 mL of water and in a dark place pour this solution and solution B into an Erlenmeyer flask.
d. Swirl the flask and if you want to increase the brilliance add further small quantities of alkali and ferricyanide crystals.

Clean up: Add 2 mL of 3 M hydrochloric acid, dilute the solution with water and wash down the drain.