

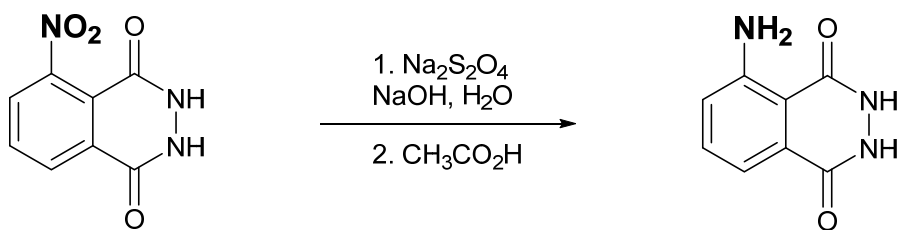
Luminol.

Chemiluminescence. Energy changes in chemical reactions are most often accompanied by the emission or absorption of heat. In chemiluminescent reactions the energy change is instead accompanied by the emission of light. When the reaction is of biological origin, the phenomenon is known as bioluminescence. Most of us have observed the display of the firefly but bioluminescence is produced in a great number of other organisms as well. These include algae, fungi, and many marine organisms.

The oxidation of luminol is one example of a chemiluminescent reaction. During the reaction, an intermediate chemical species is produced in an electronically excited state. When the excited state intermediate returns to the normal ground state, energy is given off in the form of light.

In this experiment, luminol is synthesized by the reduction of 3-nitrophthalhydrazide with sodium hydrosulfite and oxidized using a mixture of potassium ferricyanide and hydrogen peroxide.

(1.) Synthesis of Luminol. To a reaction tube, add 140 mg of 3-nitrophthalhydrazide and 1.0 mL of 3 M sodium hydroxide solution. Stir with a rod, and to the resulting deep brown-red solution add 0.6 g of sodium hydrosulfite dihydrate ($\text{Na}_2\text{S}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$, MW 210.2). Wash down the sides of the tube with a small amount of water. Heat to a gentle boil and keep the tube hot for 5 minutes. During this time some product may begin to crystallize. Add 0.4 mL of acetic acid, cool the tube in cold water, and stir. Collect the light-yellow product by suction filtration.



5-nitro-2,3-dihydrophthalazine-1,4-dione
AKA . . . 3-nitrophthalhydrazide

5-amino-2,3-dihydrophthalazine-1,4-dione
AKA . . . 3-aminophthalhydrazide (**Luminol**)

(2.) The Light-Producing Reaction. For the most dramatic effect, the light-producing reaction should be done in as dark a location as possible. In this lab, the best way to accomplish this is to do the light-producing reaction in your fume hood with the fume hood light turned off (coordinate with your fume hood neighbor) and the overhead room lights turned down as much as possible. Dissolve the moist luminol in 2 mL of 3 M NaOH and 38 mL of water. Call this Solution A. Prepare Solution B by mixing 4 mL of 3% aqueous potassium ferricyanide ($\text{K}_3[\text{Fe}(\text{CN})_6]$, MW 329.2), 4 mL of 3% hydrogen peroxide, and 32 mL of water. In the darkened area, pour solutions A and B simultaneously into a funnel which is resting in a 125 mL Erlenmeyer flask.

WASTE: Place all liquid waste and acetone rinses into the Organic Liquid Waste bottle.

At Experiment Completion: turn off the sand bath and vacuum and air valves, clean up your bench, fume hood and close the fume hood sash completely. Check out of lab with the check-in/out sheet you signed at the beginning of the semester.

The write-up for this last exp will be submitted on the same day that the experiment is done (ultra-short write-up). ALL written work for the semester must be submitted at your last meeting, unless you have a valid reason for submitting something late and have made arrangements with your instructor. No work will be accepted after the deadline given in the “Schedule of Experiments”.

CHECK-OUT

IMPORTANT: NOT CHECKING OUT OF LAB PROPERLY WILL COST YOU POINTS. If you fail to check out properly, you will lose the credit equivalent to one non-formal report (50 pts). Check-out will be done on the last scheduled lab period, not before. Come to your regularly scheduled lab period so your TA can credit you with checking out. If you miss your regular lab period and must check out during another period, contact Raina Kittilstved so that you will be credited with checking out. **BRING YOUR LOCKER KEY ON CHECK-OUT DAY AND BE SURE TO LEAVE IT IN THE LOCKER.** Carefully follow the check-out procedure given on the back of your equipment list. If for some reason you do not return your key during the check-out period, be sure to return it within the next week or you will not be credited with check-out.

Check-out Procedure

- Wash all equipment and glassware. Leave it on the benchtop.
- While you wait for your TA, dispose of all products from the semester in the proper waste containers. Clean out the drawer with paper towels and a little water.
- You will put the equipment back into your drawer, piece by piece, only when your TA is there checking it off and making sure it is clean for the student next semester.
- Place all equipment that is not on the list and all extra equipment into the plastic bin in the central area of the lab.
- Replace missing and broken equipment. You may obtain equipment from the usual places in the lab. You may also use surplus equipment from the side benches. Be sure that all equipment on the list, including the very small pieces, are present.

When and only when you have completed all of the above steps,

- Ask your TA to check your equipment. Be patient. Your TA will place the equipment back into the drawer as soon as he or she checks it.
- Place your key into a small beaker at the front of the drawer so that it can be easily noticed, close the drawer, and submit the equipment list to your TA, making sure that your TA signs it. Checkout

will not be final until you submit your key. If you do not have your key at checkout time, you may return it sometime within the following week.