

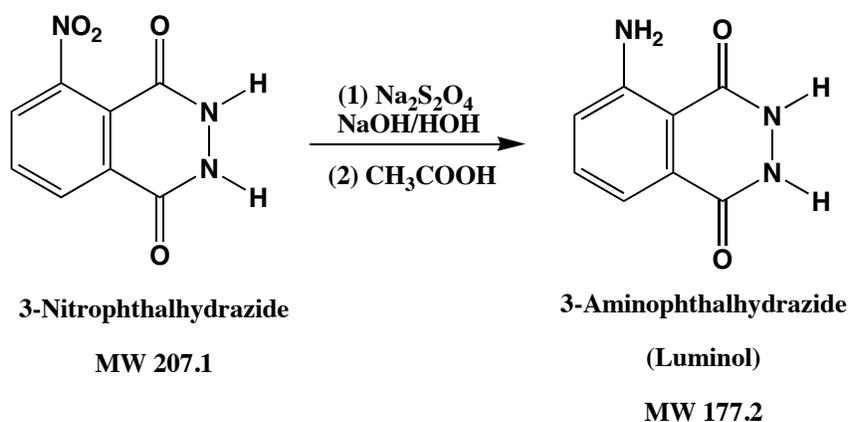
## Luminol.

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.



(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.

**BEFORE YOU LEAVE THE LAB:** turn off the sand bath and vacuum and air valves, clean up your work areas, close the fume hood sash completely and complete the check-out procedure given below. If you see caps off of bottles, replace the caps. If you see spilled chemicals, clean them up or report it to your TA.

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 experiment (10 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 Don Taylor 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.

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### Check-out Procedure

- Place all of your equipment onto your benchtop in the order in which it appears on the apparatus list and leave it there until your TA has checked it.
- 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.
- Clean the glassware. Dispose of all products in the proper waste containers. Clean out the drawer.

### **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.