

Chem 268H. Luminol and check-out. (revised 5/07)

**Finish the Unknowns lab if necessary. If time allows, do Luminol. Check out according to the procedure given below.**

(1.) Synthesis of Luminol. To a reaction tube, add 140 mg of 3-nitrophenylhydrazide (compound 13 in Chapt 61) 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. 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 product by suction filtration.

(2.) The Light-Producing Reaction. The light-producing reaction should be done in as dark a spot as possible. In this lab, the best way to accomplish this is to do the light-producing reaction in a fume hood at the opposite end of the lab with the hood lights off and if possible the room lights off. 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, 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: Use the separate waste bottles provided for "Reaction Filtrate Waste", "Light Reaction Waste", and Acetone Rinse Waste (if acetone is used to clean equipment for check-out).

DUE DATES. The write-up for Luminol will be turned in on the same day that it is done (ultra-short, nearly nonexistent write-up, although be sure to have a prelab).

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, see Peter Samal or 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 below and on the back of your equipment list. If for some reason you do not return your key during the check-out period, please return it within the next week.

### 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 onto one of the benches at the sides 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 you carefully check the glassware kit and replace missing or broken items.)
- Clean the glassware. Dispose of 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. Place the equipment back into the drawer as soon as it is checked.
- 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.