

Synthesis of Orange II

Materials:

Item	Amount per student	Amount for 24 students
Ethanol	10 mL	240 mL
Sulfanilic acid	2.4 g	60 g
2.5 % Na ₂ CO ₃ (aq) solution	25 mL	600 mL
Sodium Nitrite (NaNO ₂)	1 g	24 g
Ice	12 chunks	300 g? (a bucket full)
Conc. HCl	2.5 mL	60 mL
10% NaOH	10	240 mL
2-Naphthol	1.8 g	45 g
Sodium Chloride (NaCl) solid	5 g	120 g
Sodium Chloride (sat'd, aq)	10 mL	240 mL
Filter paper	2	48

Equipment:

Item	Amount per student	Amount for 24 students
Pasteur pipettes or droppers	1	24
Filter flasks	1	6
Buchner funnels	1	24
Rubber collars	1	6
Tubing	1 piece	6 pieces

Staff Notes: Please set up aspirators prior to class.

Safety Issues:

- The sulfanilic acid is an irritant to skin, eyes, and other mucous membranes. Flush any skin surface with copious amounts of water upon exposure. You may want to wear gloves during this procedure.
- Sodium hydroxide is caustic and corrosive to all tissues. Flush any skin surface with copious amounts of water upon exposure. The concentrated HCl is also caustic! Gloves are good....
- Sodium nitrite is used extensively as a preservative for smoked and cured meats, however in large quantities, it is toxic to humans, and irritating to our skin and mucous membranes. Again, wearing gloves would protect your hands.
- 2-Naphthol is highly toxic and can be readily absorbed through the skin. Contact with the skin can cause peeling and discoloration. Actually, gloves would be a really good idea here!

- Both acetone and ethanol are flammable organic solvents. Use care with these. The ethanol used for these labs is denatured making it unfit for human consumption; so don't consume it, OK?
- The product, Orange II, is not hazardous, but will stain your skin orange! Why not wear gloves so as not to become orange? ;-)

Procedure:

1. Diazotization of Sulfanilic Acid
 - a. Weigh 2.4 g of sulfanilic acid, and place it in a 125-mL Erlenmeyer flask. Add 25 mL of 2.5% aqueous Na_2CO_3 , and heat gently on a hot plate, just until the sulfanilic acid dissolves. Cool the flask under running tap water.
 - b. Weigh 1.0 g of sodium nitrite (NaNO_2) and add it to the Erlenmeyer flask from step 1a. Swirl to dissolve.
 - c. Prepare an acidic ice solution in a 250 mL beaker by putting ~10-12 chunks of ice and 2.5 mL of concentrated HCl into it. Add the sulfanilic acid/sodium nitrite solution from step 1b and watch for a white, powdery precipitate to form. If the precipitate does not form, add a few more drops of HCl until you see it forming. This is your "diazonium suspension".
2. Orange II Synthesis
 - a. Cool 10 mL of 10% NaOH in a 400 mL beaker. Add 1.8 g of 2-naphthol to this cold NaOH and stir with your glass rod to dissolve.
 - b. Pour the diazonium salt suspension (from step 1c) into this solution with stirring. Stir for 5-10 minutes, until thoroughly mixed and you cannot see any further solid coming out of solution.
 - c. Heat the beaker on the hot plate until the solid dissolves and then add 5 g of NaCl. Continue heating and stirring until all of the solid dissolves.
 - d. Allow the warm solution to cool slowly on the benchtop and then cool further in an ice bath. The orange solid that forms is the Orange II dye that we are seeking.
 - e. Collect the solid product by suction filtration, and "wash" it with a saturated NaCl solution while it is still in the suction apparatus and the suction is flowing. Allow the moist dye to dry in your drawer until next week.

CLEAN-UP:

After removal of the solid product, combine the filtrates, check the pH, and adjust as needed to neutralize. Dilute with lots of water and flush down the drain. The crystallization filtrate should go in the organic solvents waste bottle.

Reflections:

1. Write the chemical reactions for these two reaction steps.
2. Draw a chemical structure for Orange II and describe what structural features make it appear orange.