

Synthesis of Indigo

Materials:

Item	Amount per student	Amount for 24 students
2-nitrobenzaldehyde	0.5 g	12 g
Acetone	5 mL	120 mL
10% Aqueous sodium hydroxide (NaOH)	1 mL	24 mL
Ethanol	10 mL	240 mL
Ice	12 g	300 g (a bucket full)
HCl, dilute (to neutralize mother liquor)	10 mL	240 mL

Equipment:

Item	Amount per student	Amount for 24 students
Disposable gloves	1 pair	24 pairs
Pasteur pipettes or droppers	1	24
Filter paper	2	48
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 2-nitrobenzaldehyde is harmful as an irritant to skin eyes and other mucous membranes. It is a possible mutagen. Please use gloves and be careful with this material! Flush any skin surface for at least 5 minutes with copious amounts of water upon exposure.
- Sodium Hydroxide is caustic and corrosive to all tissues. Flush any skin surface with copious amounts of water upon exposure. Treat the dilute HCl with similar care.
- Both acetone and ethanol are flammable organic solvents. Use care with these. Note that the ethanol used in these labs is denatured and not safe for consumption; please avoid doing so.
- The product, indigo, will stain your skin blue!

Procedure:

1. Dissolve 0.5 g of 2-nitrobenzaldehyde in 5 mL of acetone in a 6-inch test tube. BE CAREFUL and WEAR GLOVES!

2. Mix one mL of 10% aqueous NaOH with 1.5 mL of distilled water. Add this mixture dropwise to the test tube prepared in step 1. Record what you see, and what you feel on the outside of the test tube, in your LNJ. Let the mixture return to room temperature and remain at room temperature for at least 5 minutes.
3. Cool the test tube in an ice bath for about 5 minutes while you set up your suction filtration apparatus. Collect the solid indigo by suction filtration. The indigo particles are very small, so it looks somewhat like the dark blue color in the liquid is simply removed! If the solution is still very dark blue, you may want to pour it back into the test tube to filter it again with the piece of filter paper....
4. With the suction still on and the indigo still on the filter paper, "wash" the indigo by pouring 10 mL of distilled water over it and then 10 mL of ethanol over it. Continue to let the suction flow for 5 or 10 minutes, if you have time.
5. Allow the indigo to dry in your drawer by placing the filter paper on top of several paper towels.

CLEAN UP: Neutralize the liquid in the filtration flask (the "mother liquor"), with dilute HCl. When the pH is between 6 and 8, you can flush it down the drain with lots of water.

Reflections:

1. Write the chemical equation for the synthesis in your LNJ.