the microscale laboratory

A Microscale Synthesis of Indigo: Vat Dyeing

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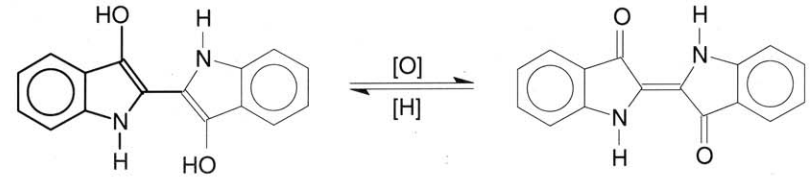
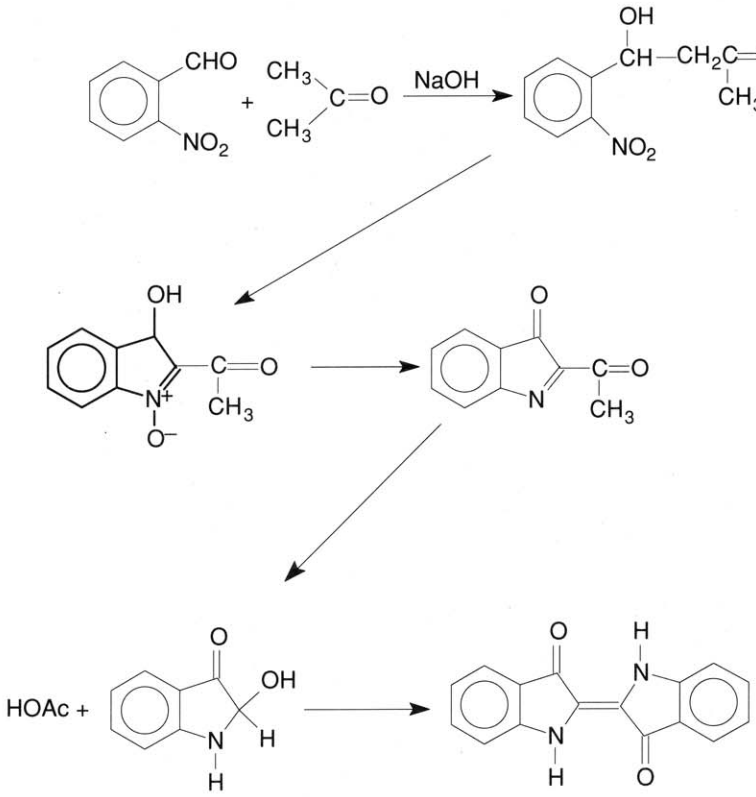
Indigo was first synthesized by Baeyer[2] who prepared it from isatin in 1870. The commercial manufacture of this material, which followed shortly, was based on a method devised by Heumann. In this process anthranilic acid is reacted with chloroacetic acid to produce phenylglycine-*o*-carboxylic acid. When heated with strong base this material produces indoxyl which can be converted to indigo by air oxidation.[3]

The insolubility of indigo in water led to the development of a special procedure known as vat dyeing (eq 1). In this process a variety of mild reducing agents may be used to convert the insoluble indigo to the alkali-soluble, colorless, leucoindigo (indigo white). Then the material to be dyed is placed in a tank or vat containing the leucoindigo. Oxidation deposits indigo in the matrix of the cloth fibers. The dyed material is then washed to remove excess pigment and dried.[4]



(1)

Indigo in the following experiment is produced quite differently from the traditional method described above. In the experiment, *o*-nitrobenzaldehyde is dissolved in acetone. When aqueous sodium hydroxide is added, a series of condensations, disproportionations, and oxidations takes place within a few seconds to produce indigo in good yield (eq 2). After purification this material is reduced by an aqueous solution of sodium dithionite to produce leucoindigo. Dipping a piece of cotton cloth in this solution and exposing it to the air produces indigo-dyed fabric in the traditional manner. Although the procedure outlined here is similar to more complex methods described in laboratory textbooks,[5] it greatly reduces the time required for the reaction, and it can be carried out with simple equipment (a test tube).



(2)

Experimental

The Synthesis of Indigo

To synthesize indigo in microscale, we use the Baeyer–Drewson Reaction.[6] *o*-Nitrobenzaldehyde (0.5 g) is dissolved in 5 mL of acetone in a 6-in. test tube. Five milliliters of water is added and the suspension is stirred. Sodium hydroxide (2.5 mL of a 1.0 N solution) is added dropwise. As the base is added, the blue color of indigo can be seen almost at once, and the solution may boil.

After the exothermic reaction has subsided, the reaction is allowed to stand for 5 min. The precipitated indigo is collected by suction filtration. The filtrate is washed successively with 10 mL of water and 10 mL of ethyl alcohol. Since indigo melts above 360 °C, the melting point determination should not be attempted.

Caution: Normal precautions[7] should be observed when handling any chemical. Acetone is **flammable**. Acetone is

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[2]Baeyer, A. *Ber.* **1870**, *3*, 514.
[3]Heumann, K. *Ber.* **1890**, *23*, 3043.
[4]Karrer P. *Organic Chemistry*; Nordemann: New York, 1938; p 527.
[5]Wilcox, C., Jr. *Experimental Organic Chemistry—A Small Scale Approach*; Macmillan: New York, 1988; pp 406–408.
[6]Fitton, A; Smalley, R. *Practical Heterocyclic Chemistry*; Academic Press: New York, 1968; p 13.
[7]Lenga, R. E. *The Sigma–Aldrich Library of Chemical Safety Data*, 2nd ed.; Sigma–Aldrich Corporation, Milwaukee, 1988.

a skin **irritant** and is moderately **toxic** by ingestion. *o*-Nitrobenzaldehyde is a **mutagen,** and gloves should be worn when using it. Sodium hydroxide is a **caustic agent,** so goggles should be worn. Ethyl alcohol is **flammable**. Indigo is a mild **irritant**. Indigo will be absorbed by the skin, and it causes a blue coloration. Gloves should be worn when handling this compound.

Vat Dyeing with Indigo

The indigo produced above is placed in a 6-in. test tube with 10 mL of water, 3 pellets of sodium hydroxide, and a boiling chip. The solution is heated to boiling using a microburner, and 2 mL of 10% aqueous sodium dithionite are added. Additional dithionite is then added dropwise until the indigo dissolves and a clear, yellowish leucoindigo solution forms.

The leucoindigo solution is then poured into a 250-mL beaker containing 100 mL of cool water. A piece of cotton cloth (1 in. × 3 in.) is worked into the dye solution with a heavy glass rod. It is removed when completely saturated and allowed to air dry. As the cloth dries, the blue color of indigo will appear. After the cloth is completely dry, it is rinsed with cold water to remove any residue of indigo which is not incorporated into the fibers of the cloth. When the washings are completely clear, the dyed cloth should be allowed to dry.

A Mercury-Free Alternative for Temperature Measurement in Aluminum Blocks

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As heat transfer devices, aluminum blocks offer advantages over sand baths because the blocks warm much more rapidly and uniformly. These differences have been detailed previously.[1]

However, measuring the temperature of an aluminum block with standard thermometers presents practical difficulties. Thermometers inserted into either a vertical or horizontal hole in the block are easily broken off by an accidental sideways blow. The resulting spillage of mercury onto a heated surface can represent a significant health risk. Furthermore, mercury can form an amalgam with aluminum, complicating the decontamination of the block. In addition, thermometers inserted horizontally tend to unbalance the block and require additional support.

We have identified a simple, durable, and mercury-free alternative for these temperature measurements: a dial thermometer, similar to the metal candy thermometers available in supermarkets. They can be inserted into aluminum blocks via an appropriately sized hole, then an-