

# The microscale synthesis of indigo dye

## Topic

Colour chemistry, dyes. Organic chemistry – dyes, synthesis.

## Timing

10 min.

## Description

In this experiment students prepare the dye indigo by the condensation of 2-nitrobenzaldehyde with propanone in the presence of sodium hydroxide. The experiment is very easy to do.

## Apparatus (per group)

- One 10 cm<sup>3</sup> beaker or a test-tube
- Small filter funnel and filter paper.

## Chemicals (per group)

Solutions contained in plastic pipettes, see 'Apparatus and techniques for microscale chemistry' handout.

- Sodium hydroxide 0.5 mol dm<sup>-3</sup>
- Deionised water
- Propanone
- 2-Nitrobenzaldehyde.

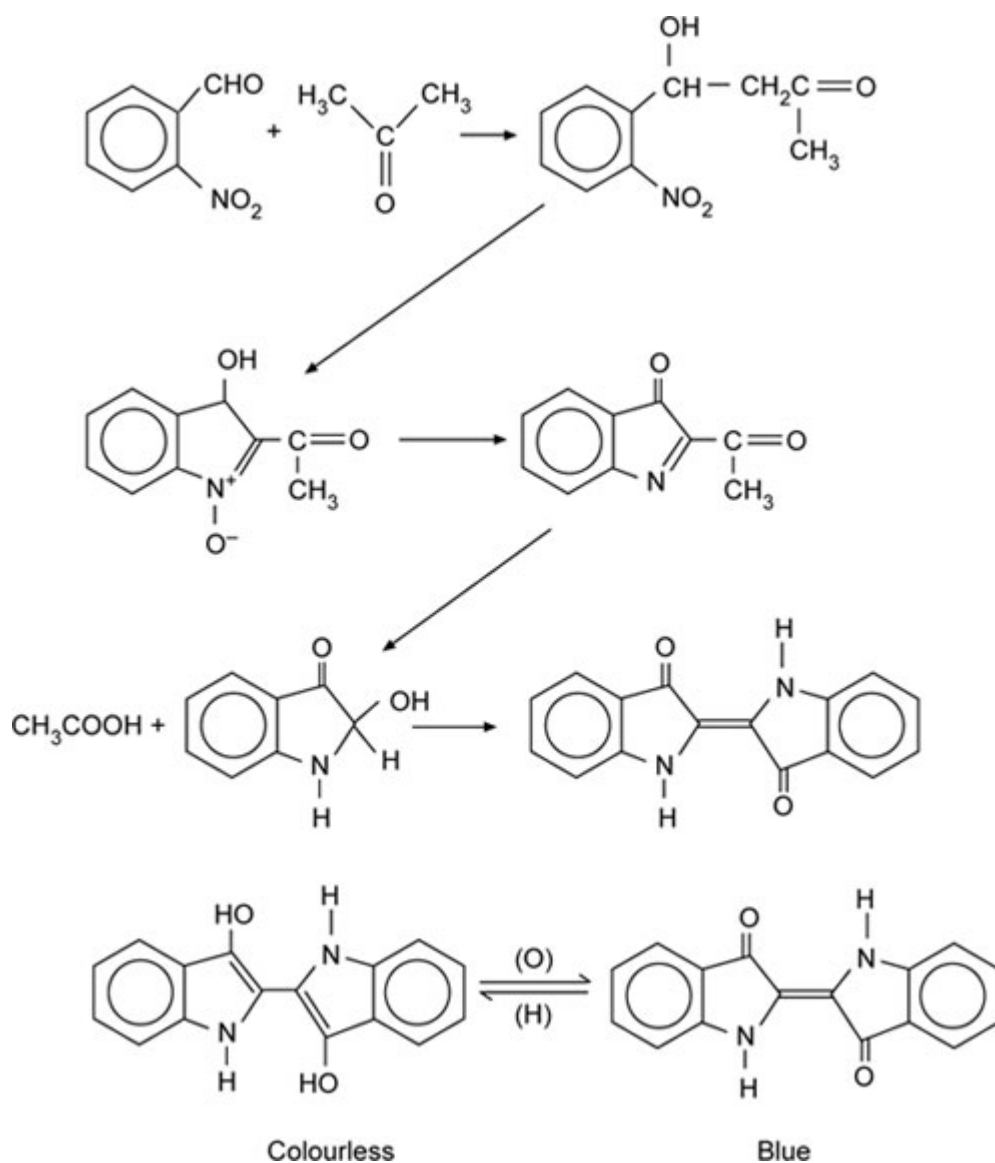
## Observations

2-Nitrobenzaldehyde dissolves in propanone to form a pale yellow solution. When sodium hydroxide solution is added the solution darkens after a few seconds and a purple precipitate of indigo forms. The synthesis is very simple and quick to perform yet it is mechanistically complex involving a series of condensations, disproportionations and oxidations. The sequence of reactions is given in the *Journal of Chemical Education* and this, together with the structures of the two forms of indigo, is shown overleaf. There is one unusual step which produces ethanoic acid and what appears to be a hydroxy-derivative of indoxyl prior to the final oxidation to form indigo (indigotin).

It is worthwhile pointing out to students that it took Baeyer 14 years (from 1865 to 1879) to find a route for the original synthesis of indigo and even when he did it took him another four years to deduce the correct formula (in 1883)!

The indigo can be filtered off and then dissolved in an alkaline solution of sodium dithionite to form the colourless, soluble form (leucoindigo). A piece of cotton cloth dipped into this solution and then exposed to air produces the indigo-dyed fabric – this is the procedure used in vat dyeing.





The preparation of indigo from 2-nitrobenzaldehyde and propanone

## References

1. *J.Chem.Ed.*, 68, 10, 1991.
2. S.W. Breuer, *Microscale practical organic chemistry*. Lancaster: Lancaster University, 1991.

## Health & Safety

Students must wear suitable protection (Splash resistant goggles to BS EN166 3).

Sodium hydroxide solution,  $0.5 \text{ mol dm}^{-3} \text{ NaOH (aq)}$  is corrosive.

Propanone is highly flammable, and an eye eye/respiratory irritant

2-Nitrobenzaldehyde is Harmful if swallowed and a skin/eye and respiratory irritant. See ChemSpider-10630.

## Credits

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*Health & safety checked May 2018*

Page last updated August 2018

