

# The microscale synthesis of azo dyes

## Topic

Organic chemistry – azo dyes.

## Timing

20 min.

## Description

In this experiment students prepare an azo dye and use it to dye a piece of cotton. The synthesis is unusual in that whereas most organic syntheses require ambient or elevated temperature, this synthesis requires low temperatures.

## Apparatus (per group)

- Three 10 cm<sup>3</sup> beakers
- Thermometer
- Tweezers.

## Chemicals (per group)

- Ice
- Aminobenzene (aniline)
- Hydrochloric acid (concentrated?)
- Sodium nitrite
- 2-Naphthol (also called  $\beta$ -Naphthol; naphthalene-2-ol)
- Sodium hydroxide solution 2 mol dm<sup>-3</sup>
- Ethanol
- Urea.

## Observations

The orange-red azo dye forms in the fibres of the cotton, dyeing the cloth. The melting point of 1-phenylazo-2-naphthol is 133 °C.

## Note

The urea decomposes excess HNO<sub>2</sub> formed and prevents many side reactions from occurring. A better 'red' dye is usually produced.

## Reference

S. W. Breuer, *Microscale practical organic chemistry*. Lancaster: Lancaster University, 1991.

## Health & Safety

Students must wear suitable eye protection (splash resistant goggles to BS EN166 3). This experiment should be done in a fume cupboard.



Aminobenzene (aniline) is TOXIC by all routes, a carcinogen and mutagen, a skin sensitiser, causes eye damage and is toxic to aquatic life.

Hydrochloric acid, HCl (aq), is CORROSIVE and a respiratory irritant.

Sodium Nitrite, NaNO<sub>2</sub> is an OXIDISER, toxic if swallowed and hazardous to aquatic life.

2-Naphthol is HARMFUL if swallowed or in contact with skin and toxic to aquatic life.

Sodium hydroxide solution, 2 mol dm<sup>-3</sup> NaOH (aq), is CORROSIVE.

Ethanol is highly FLAMMABLE.

Urea is of low hazard.

## Credits

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*Health & safety checked May 2018*

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