**Student worksheet**

**Practical 6: The preparation of azo dyes**

**Equipment/materials**

* 5 cm3 phenylamine (toxic)
* 10 cm3 1.0 mol dm–3 hydrochloric acid solution (irritant)
* 5 cm3 concentrated hydrochloric acid (corrosive)
* Solid sodium nitrite (NaNO2) (toxic and oxidising)
* 4 cm3 0.5 mol dm–3 sodium hydroxide solution (corrosive)
* Phenol (toxic and corrosive)
* Naphthalen-2-ol (harmful)
* Six standard test tubes and stoppers
* Three boiling tubes and stoppers
* Two thermometers (-10 to 100 °C) × 1 °C
* Distilled/deionised water
* Dropping pipettes
* Glass rods
* Spatulas
* Ice
* Access to a balance
* Five 10 cm3 measuring cylinders

**Data/diagram**

**Analysis of results**

* Note the colours of the azo dyes

**Questions**

1. What is the type of mechanism for the reaction between phenol and benzenediazonium chloride?
2. Why does the reaction take place at the carbon–4 atom in the benzene ring of phenol?
3. What happens to benzenediazonium chloride on heating? Give an equation for the reaction.

**From the examiner**

* Make and record valid observations and organise your results suitably.
* Demonstrate safe practical techniques.
* Pay full regard to all essential safety precautions.

**Objective**

* Be able to prepare an azo dye.

Flammable

 Toxic Corrosive

 

Oxidising Harmful

**Safety**

* Perform the experiment in a well-ventilated room.
* Wear a lab coat and nitrile disposable gloves, tie long hair back.
* Wear safety goggles.

**Procedure**

1. To 3 cm3 of phenylamine and 10 cm3 of water in a boiling tube, add 8 cm3 of concentrated hydrochloric acid. Stopper and shake the tube until the amine has dissolved.
2. Cool the solution in an ice bath to about 5 °C.
3. Prepare a solution of sodium nitrite by dissolving 3 g of sodium nitrite in 8 cm3 of water in a boiling tube and cool this to 5 °C.
4. Combine the two solutions to make benzenediazonium chloride. Use some of the benzenediazonium chloride solution to find out what happens at temperatures above 10 °C. The rest can be used in coupling reactions to make azo dyes. Record your observations.
5. Warm 3 cm3 of the benzenediazonium chloride solution until it is nearly boiling. Carefully smell the product.
6. Dissolve a few crystals of phenol in 2 cm3 of sodium hydroxide solution. Cool the solution to 5 °C and then add 0.5 cm3 of the benzenediazonium chloride solution.
7. Repeat step 6 using naphthalen-2-ol instead of phenol.