# Practical 5.16 Polymerisation reactions

## Purpose

To describe:

(i) condensation polymerisation for the formation of polyesters such as terylene and polyamides such as nylon and Kevlar

(ii) addition polymerisation including poly(propenamide) and poly(ethenol) [5.4.2f]

## Safety

**Wear eye protection. Phenylethene (styrene) is harmful. Di(dodecanoyl) peroxide (lauroyl peroxide) is oxidising. This experiment should be carried out in a well-ventilated laboratory, or preferably in a fume cupboard. Decanedioyl dichloride (sebacoyl dichloride) is corrosive. 1,6-diaminohexane is corrosive. Wear gloves if handling the polymer product because it is in the form of a fine tube which may contain unreacted materials. Benzene-1,2-dicarboxylic anhydride (phthalic anhydride) is harmful. Avoid skin contact and wear gloves.**

Each group of students will need:
Eye protection
Gloves (nitrile)

**1 Poly(phenylethene)**
Beaker, 250 $cm^3$
Test tube
Cotton wool
Phenylethene
Di(dodecanoyl) peroxide 0.2 g OXIDISING
Wooden splints
Fume cupboard

**2 The 'nylon rope trick'**
Beakers, 100 $cm^3$ × 2
Measuring cylinders, 25 $cm^3$ × 2
Crucible tongs or tweezers
Glass rods × 2
Decanedioyl dichloride (0.5 $cm^3$) in petroleum spirit or cyclohexane (15 $cm^3$) HIGHLY FLAMMABLE, CORROSIVE
1,6-diaminohexane (0.7 g) CORROSIVE
Sodium carbonate-10-water (2 g) in water (15 $cm^3$) solid IRRITANT
50% aqueous ethanol, 20 $cm^3$

It may save a lot of time if the technician makes up the two solutions needed.

**3 Polyester resin**
Test tube
Thermometer, 0–360 °C
Oil bath
Dropping pipette
Benzene-1,2-dicarboxylic anhydride (phthalic anhydride), 3 g HARMFUL, avoid contact with the skin
Propane-1,2,3-triol (glycerol), 2 $cm^3$
Gloves (nitrile)

Access to:
Fume cupboard

**4 Cross-linked polymer**
Disposable container (plastic drinking cup)
Measuring cylinder, 50 $cm^3$
Measuring cylinder, 10 $cm^3$
Glass rod
8% aqueous poly(ethenol), 25 $cm^3$
4% aqueous sodium borate, 5 $cm^3$

## Method

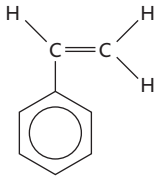
You should be able to carry out at least one of the reactions described, but look at them all to identify the common features. Wear gloves to do these experiments and do the work in a fume cupboard if possible.

# **Practical 5.16 (cont.)** Polymerisation reactions

## 1 Preparation of poly(phenylethene)

Mix thoroughly 5 $cm^3$ of phenylethene and 0.2 g of di(dodecanoyl) peroxide in a test tube. Plug the test tube with some cotton wool and warm in a beaker of boiling water in a fume cupboard for about 30 minutes.

Test the viscosity of the mixture by stirring with a wooden splint from time to time. Allow it to cool in the water bath when the polymerisation nears completion.




**a** What type of polymerisation is involved here?

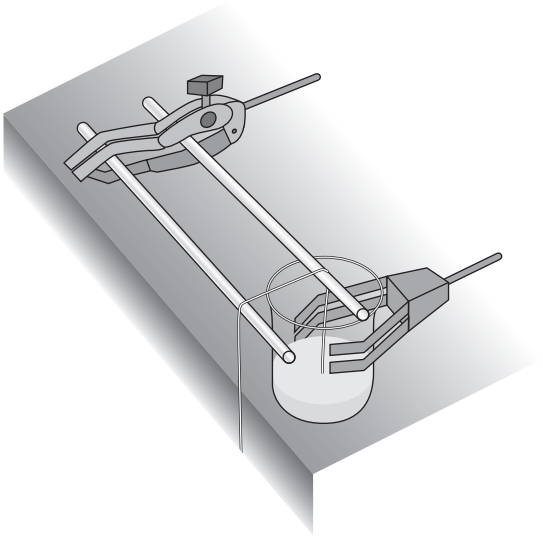
**b** Write an equation for the polymerisation reaction using structural formulae.

## 2 The 'nylon rope trick'

Use the prepared solutions of:

- 0.5 $cm^3$ of decanedioyl dichloride (take care) in 15 $cm^3$ of hydrocarbon solvent in a 100 $cm^3$ beaker
- 0.7 g of 1,6-diaminohexane (take care) and 2 g of sodium carbonate in 15 $cm^3$ of water in a 100 $cm^3$ beaker.

Clamp the beaker holding the aqueous solution, and alongside it clamp a pair of glass rods as shown in the diagram. If possible allow a drop of about 1 metre from the rod to the receiver.



Edexcel practical materials adapted from Nuffield Advanced Chemistry materials

# Practical 5.16 (cont.) Polymerisation reactions

Now pour the hydrocarbon solution carefully onto the aqueous solution in the 100 $cm^3$ beaker and, using crucible tongs or forceps, pull the interfacial film out, over the rods, and down towards the receiver. When a long enough rope has formed, the process should go on of its own accord until the reagents are used up, but the rope may need to be pulled out gently using the tongs. Take care not to get either solvent or reagent on your fingers.

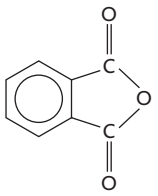
To obtain a dry specimen of nylon polymer, wash it thoroughly in 50% aqueous ethanol, and then in water until litmus is not turned blue by the washings. Note that the nylon 'rope' is likely to be a hollow tube containing solvent and possibly reagent. You should therefore take care when handling it, even after washing in this way.

**c** Did the reaction involve addition or condensation polymerisation?

**d** Write an equation for this polymerisation reaction.

## 3 The preparation of a polyester resin

Wearing gloves, mix 3 g of benzene-1,2-dicarboxylic anhydride with 2 $cm^3$ of propane-1,2,3-triol in a test tube. Measure out the propane-1,2,3-triol with a dropping pipette and allow the pipette plenty of time to drain.




benzene-1, 2-dicarboxylic anhydride

In an oil bath, heat to 160 °C, and then more slowly to 250 °C in a fume cupboard. When the mixture ceases to bubble, allow it to cool. Test the viscosity of your product.

**e** Did the reaction involve addition or condensation polymerisation?

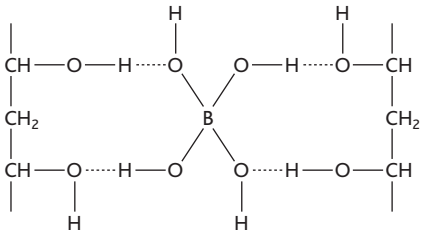
**f** Write an equation for a possible polymerisation reaction – use benzene-1,4-dicarboxylic acid and ethane-1,2-diol to make it more straightforward.

# **Practical 5.16 (cont.)** Polymerisation reactions

## 4 The preparation of a cross-linked polymer

Measure about 25 $cm^3$ of a 4% aqueous solution of poly(ethenol) into a disposable container. Rinse out your measuring cylinder thoroughly. Add about 5 $cm^3$ of 4% sodium borate solution and stir well immediately. Continue stirring as the mixture thickens. Put on gloves and examine the properties of the 'slime' you have produced. Take care as 'slime' sticks to clothing and removes paint.

In 'slime' the poly(ethenol) chains have been cross-linked by hydrogen bonds to borate ions, so the links are not permanent.




hydrogen bonding

**g** What monomer is needed to make poly(ethenol)?

**h** The molar mass of the poly(ethenol) used in this experiment is about $10^5$ g $mol^{-1}$. How many monomer units does this involve?