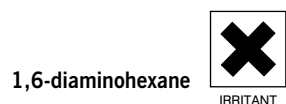
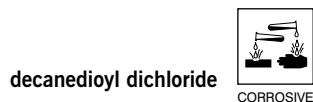



A nylon is made from its monomers by condensation polymerisation. You may already have done this or a similar experiment in an earlier course. If so, you can omit this activity and go on to Activity DP2.2.


Requirements

- 5 cm³ beaker
- decanedioyl dichloride, 5% solution in cyclohexane (1 cm³)
- 1,6-diaminohexane, 5% solution in 0.5 mol dm⁻³ sodium carbonate solution (1 cm³)
- glass rod or test-tube
- tweezers
- access to fume cupboard
- protective gloves



CARE Eye protection and gloves must be worn.

 WEAR EYE PROTECTION

 WEAR PROTECTIVE GLOVES

What you do

- 1 Pour about 1 cm³ of the 1,6-diaminohexane solution (**CARE** Irritant) into a 5 cm³ beaker.
- 2 Carefully add an equal volume of the decanedioyl dichloride solution to the beaker. (**CARE** Decanedioyl dichloride has an irritating vapour. The vapour is a powerful **lachrymator** (eye irritant) and this effect is often delayed. *Perform this part of the activity in a fume cupboard.*)
Two separate layers will form. Do not mix them.
- 3 Use a pair of tweezers to remove the nylon film which forms where the two layers are in contact. Do this slowly and hook the nylon thread which forms onto a glass rod or a test-tube.
Slowly wind the thread around the rod. As the nylon is removed more forms at the solution interface, so you should be able to keep on winding for some time.
- 4 Once you have made some nylon it needs to be washed thoroughly with tap water. *Take care not to touch the nylon* because it forms as a hollow tube, and there will still be some chemicals trapped in the middle.

QUESTIONS

- a Write out an equation to show the formation of nylon in this activity.
- b What is the name of the nylon you have made?

In this experiment you are going to convert some nylon-6,6 polymer back into its original di-acid and di-amine. The linkages in the nylon are broken down by hydrolysis using sulphuric acid. This activity will allow you to improve your skill in carrying out an organic reaction safely. You will learn how to purify an organic solid and how to measure its melting point.

Requirements

- nylon-6,6 granules (2 g)
- 100 cm³ boiling flask
- condenser
- sulphuric acid, 30% (35 cm³)
- 250 cm³ conical flasks (2)
- watch-glass
- thin-walled capillary tubes (or melting-point tubes)
- melting-point apparatus for use up to 150 °C
- apparatus for vacuum filtration
- saturated sodium hydrogencarbonate solution (20 cm³)
- sodium hydroxide solution, 2 mol dm⁻³ (5 cm³)
- 100 cm³ measuring cylinder
- ice
- electric heating mantle
- 250 cm³ beakers (2)
- 10 cm³ measuring cylinder
- Universal Indicator paper

sodium hydroxide solution



CORROSIVE

sulphuric acid



CORROSIVE

CARE Eye protection must be worn throughout.



WEAR EYE PROTECTION

CARE Sulphuric acid of this concentration is very corrosive.

Introduction

If you did **Activity DP2.1** you will have made a nylon from its monomers by condensation polymerisation. A reaction like this, in which a new substance is made from simpler substances, is called a **synthesis**.

The reverse process, in which a large molecule is broken down into simpler molecules, is called a **degradation**. This type of reaction is often used by chemists to find out about the composition of substances. If they can identify the degradation products, they may be able to work out the structure of the original compound.

The amide linkages in nylon can be **hydrolysed** (split by water) to give the parent di-acid and di-amine. The reaction with water is very slow indeed, but it can be speeded up by carrying out the hydrolysis in acid solution (**acid hydrolysis**). You will use moderately concentrated sulphuric acid (about 5.5 mol dm⁻³) for the hydrolysis.

What you do

Part 1: Breaking down the nylon

- 1 Place 2 g of nylon-6,6 granules into a 100 cm³ flask to which a reflux condenser can be attached.
- 2 Pour 35 cm³ of 30% sulphuric acid into the flask. (**CARE** Sulphuric acid of this concentration is very corrosive.) Fit the condenser to the flask (Figure 1).
- 3 Place the reaction flask and condenser in the heating mantle. Heat the reaction mixture under reflux for about 3 hours. (This technique is used when you want to heat reactants for some time, but not lose either the reactants or the products by evaporation.) Add a few boiling chips to help the mixture boil smoothly.

The nylon will all have dissolved after about 45 minutes, but you should carry on heating to complete the degradation.

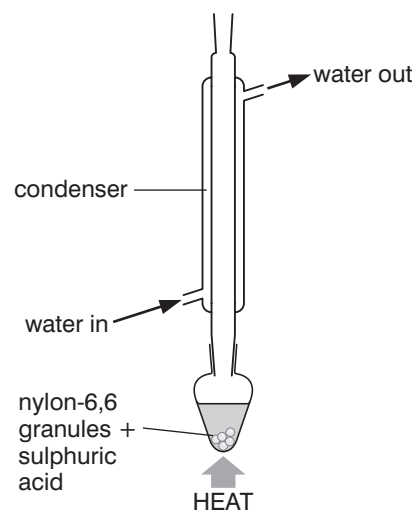


Figure 1 Breaking down the nylon

- 4 Allow the flask and contents to cool, then place them in an ice bath and leave them overnight for crystals to form.
- 5 Collect the crystals by vacuum filtration. Keep the filtrate for use later.

Part 2: Purifying the hexanedioic acid by recrystallisation

Your solid now needs to be purified by **recrystallisation**.

- 6 Place your hexanedioic acid crystals in a 250 cm³ conical flask. Add 10 cm³ of distilled water. Hold the neck of the flask with an insulating holder and gently heat the flask, swirling the contents at the same time. If some crystals remain when the water starts to boil, add a further 5 cm³ of water and reheat.
- 7 Carry on in this way until all the crystals have dissolved in the minimum quantity of water.
- 8 If your solution is clear, you can loosely cover the opening to the flask and leave the solution to cool overnight.
If the solution contains debris, this can be removed by carefully decanting most of the solution into a second flask, leaving the debris behind. You will need to reheat the solution to redissolve the crystals before covering it and leaving it to recrystallise.
- 9 Collect the crystals by vacuum filtration and leave them to dry on a watch-glass. To speed things up you can place the watch-glass in an oven or on a food-warming tray.

Part 3: Finding a melting point

Hexanedioic acid melts at 152 °C. Find the melting point of your crystals and compare it with this value.

This is what you do to determine a melting point.

- 10 Carefully heat one end of a small thin-walled capillary tube – a melting-point tube – so that the opening is just sealed. Leave the tube to cool.
- 11 Grind a small quantity of your dry crystals in one corner of the watch-glass until you have a fine powder.
Tap the open end of the melting-point tube into the fine powder so that a little powder packs into the tube. Invert the tube and tap it gently so that the powder falls to the closed end. Your teacher may show you an effective way of doing this. Do not try to put too much powder into the tube at once.
- 12 Repeat the procedure until you have about 1 cm depth of powder in the tube.
- 13 You may have an electrically-heated melting point apparatus which your teacher will show you how to use. Another way of determining melting points is described next.
- 14 Fix the tube into position in the melting-point apparatus as shown in Figure 2.

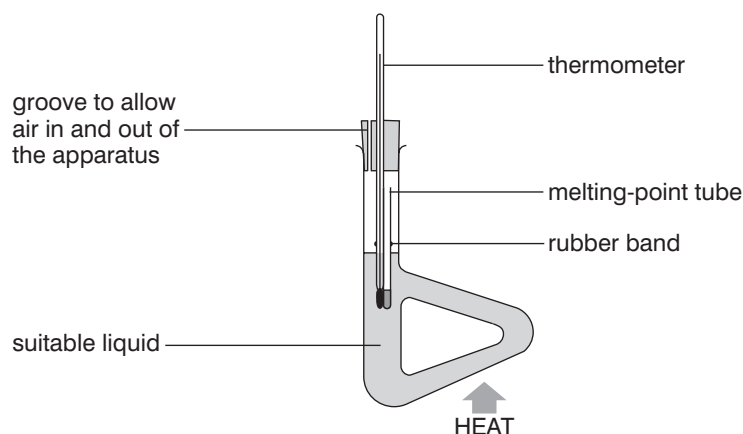


Figure 2 Melting-point apparatus

- 15 Slowly heat the side-arm of the apparatus with a very low Bunsen burner flame. The design of the apparatus should ensure a circulation of warm liquid around the sample and thermometer. Watch the sample carefully. When it melts, the powder will collapse into a sticky liquid. Record the temperature at which this happens.

- 16** This will be a rough value for the melting point because you were heating quite quickly. To determine an accurate melting point, allow the apparatus to cool down to about 10 °C below the value you have just recorded. Prepare another sample while this is happening. Then repeat the process with the fresh sample and a slower rate of heating.
- 17** Record the accurately determined melting point of your hexanedioic acid.

Part 4: Detecting the 1,6-diaminohexane produced

The diamine is still in solution in the filtrate obtained in Part 1 because it has formed a soluble salt by reacting with the sulphuric acid.

- 18** Take 5 cm³ of the filtrate and carefully pour it into 20 cm³ of saturated sodium hydrogencarbonate solution in a 250 cm³ beaker. (**CARE** Do not add the filtrate all in one go or the mixture will fizz dangerously.)
Use pH paper to make sure that the mixture is no longer acidic. If necessary add some more sodium hydrogencarbonate solution to achieve this.
- 19** Then add 5 cm³ of 2 mol dm⁻³ sodium hydroxide solution to make the solution alkaline. (**CARE** Sodium hydroxide solution is corrosive.)
Cautiously swirl the contents of the beaker and note the smell of the solution which contains 1,6-diaminohexane. (For comparison, the trivial names of 1,4-diaminobutane and 1,5-diaminopentane are putrescine and cadavarine respectively, both of which are associated with the putrefaction of proteins in flesh.)

QUESTIONS

- a** What property of hexanedioic acid is made use of in the recrystallisation process?
- b** Melting points are often used to identify compounds. They are also a good indication of the purity of a compound. Was your sample of hexanedioic acid pure? Explain your answer.
- c** Write an equation for the hydrolysis of a short section of nylon-6,6 to produce hexanedioic acid and 1,6-diaminohexane.
-

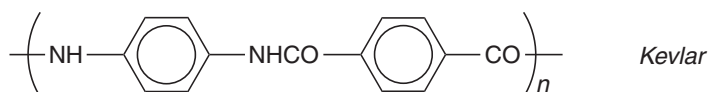
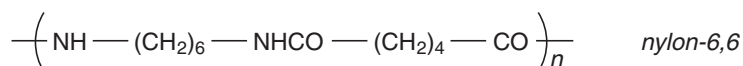
One thing you will achieve from studying this unit is an understanding of how the properties of polymers relate to their structures. In this activity you can examine this relationship for nylon and Kevlar.

Requirements

- molecular model kit

Building the model

The structures of the polymers nylon-6,6 and Kevlar are:



Use your model kit to make the structures that represent the repeating units for these polymers. Then join your nylon model together with those from other students' kits to make a long-chain model of the polymer. Do the same for the Kevlar structure. Use your models to help you work through the questions which follow.

QUESTIONS

- a How do the models of the polymer chains behave when stretched? Which polymer will be more elastic?
- b Use the models to explain the following data, which were obtained in experiments to test the strengths of the two polymers:

Elongation at fracture	
nylon-6,6	18%
Kevlar	3%

- c The bond lengths in Kevlar and nylon-6,6 can be estimated from comparable functional groups in other organic molecules:

	Bond length/nm	
	nylon-6,6	Kevlar
N-C (in CONH group)	0.132	0.132
N-C (attached to a hydrocarbon group)	0.147	0.135
C-C (in a hydrocarbon group)	0.154	0.139
C-C (attached to CONH group)	0.150	0.148

There is usually a good correlation in chemistry between bond length and bond strength: shorter bonds are stronger, longer bonds are weaker.

- Draw the structures of nylon-6,6 and Kevlar, and mark on each of them the bond lengths from the table.
- Use the bond lengths to explain why Kevlar is more stable than nylon-6,6 when heated.

The polymer in bubble gum can be elastic or glassy, depending on temperature. You can study both forms with the help of a domestic freezer.

Requirements

- bubble gum (eg Hubba Bubba)
- freezer

CARE Do this experiment at home, not at school.

CARE Do not handle other people's bubble gum.

What you do

- 1 Chew a piece of bubble gum until all the taste has gone.
- 2 Gently pull the gum and then release it.
 - a Does it show any elastic properties?
- 3 Pull it so that it stretches to about eight times its original length.
 - b Does it still show elastic properties? Does it completely return to its original length? What term is used to describe this irreversible change?
- 4 Now shape your piece of bubble gum so it's about the same size as a 5p coin. Wrap it in some plastic film and place it in the coldest part of your freezer for about 15 minutes.

When you remove the gum from the freezer, quickly bend it.

 - c What happens? Can you explain the result? What happens as it returns to room temperature?

This activity helps you get your notes in order at the end of this unit.

Use this list as the basis of a summary of the unit by collecting together the related points and arranging them in groups. You may also wish to check how this unit has further developed ideas first met in **The Polymer Revolution**. You may want to link the summaries of parts of **The Polymer Revolution** to the summaries you prepare for this unit.

Most of the points are covered in the **Chemical Ideas**, with supporting information in the **Storyline** or **Activities**. However, if the *main* source of information is the Storyline or an Activity, this is indicated.

- The historical development of condensation polymers (polyamides and polyesters) (**Storyline DP2** and **DP3**).
- How to distinguish between addition and condensation polymerisation.
- Predicting the structural formula of the condensation polymer formed from given monomer(s) and vice versa.
- The characteristic properties of carboxylic acids, including: acidic nature and ester formation with alcohols and phenols.
- The hydrolysis of esters.
- Be able to recognise members of the following homologous series: acyl chlorides, amines and amides.
- The use of systematic nomenclature to name primary amines.
- The characteristic properties of the amino group, including: basic nature and acylation.
- The reaction between a primary amine and an acyl chloride to produce an amide.
- The hydrolysis of an amide (**Activity DP2.2**).
- The procedure for purifying an organic solid product (**Activity DP2.2**).
- The properties of condensation polymers in terms of intermolecular attractions.
- The effect of temperature changes on polymers.
- The relationship between the properties of polymers and their molecular structure.
- The ways that chemists can modify the properties of a polymer by physical and chemical means (including the use of co-polymers), to meet particular needs.
- The use of given data to design a polymer for a particular purpose.
- The disposal and recycling of polymers (**Storyline DP7**).