

29. Preparation of poly(pyrrole) (a conducting polymer)

Student sheet

In this exercise, you are going to make a **conducting** polymer. You will then, using a circuit of your own devising, test its conductivity.

Intended lesson outcomes

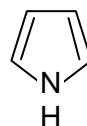
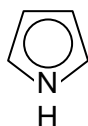
By the end of this exercise you should be able to:

- handle hazardous materials safely
- work with delicacy and precision
- measure the conductivity of a polymer
- understand the polymerisation process and the concept of a conducting polymer

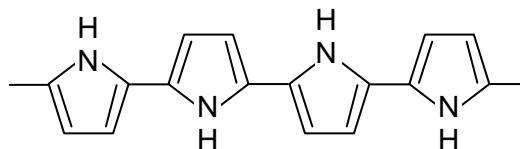
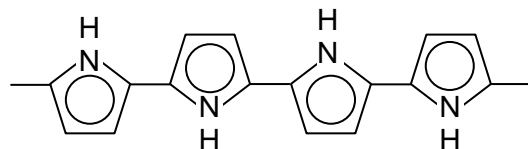
Background information

Electrical conductors work by allowing a flow of electrons through them. This requires that delocalised (mobile) electrons are present in the conductor. Metals conduct because they easily lose their outer shell electrons to form what may be described as a 'sea of delocalised electrons'. Graphite conducts owing to its bonding/structure; the carbon atoms form an extended system of π bonds which are delocalised across the whole plane of a graphite sheet. Consider the bonding in benzene. It may be thought of as having alternate double (π bonds) and single bonds (σ bonds). However, the benzene ring does not behave like an alkene. The reason is that an electron from each carbon atom is delocalised. The bonding in the ring is, therefore, described as being **delocalised**, and the mobile electrons are called **delocalised electrons**. Molecules with alternating double and single bonds are described as having a **conjugated system** of bonding. In a conjugated system, some electrons are delocalised and so can move freely throughout the conjugated system.

The structure of pyrrole, C_4H_5N , is shown below. You will notice that the two double bonds are conjugated. Also, there is a lone pair of electrons on the nitrogen atom that can also join in the conjugation so that together they provide six delocalised electrons, similar to those in benzene; see the diagrams below.



Under appropriate conditions, pyrrole polymerises to form poly(pyrrole), the structure of which is shown below.







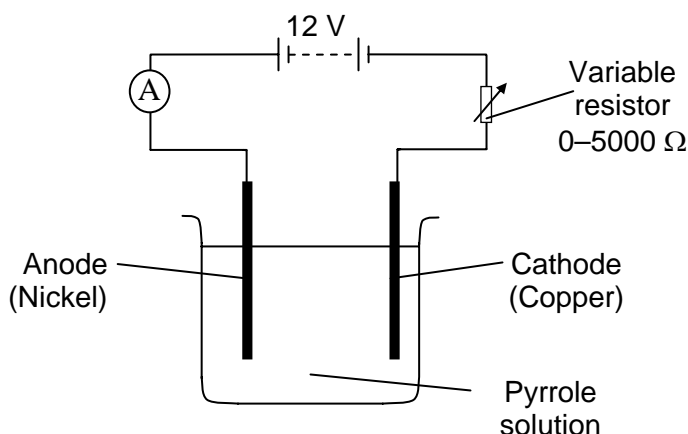
In both poly(pyrrole) and poly(ethyne), $-(CH=CH)_n-$, the delocalisation extends along the whole length of the polymer chain. However, in order to make such polymer chains conductors, electrons have to be added (reduction) or removed (oxidation) from the conjugated system. Electrons can then move along the polymer chain, through the conjugated system just as they do through graphite or a metal.

In this exercise, you will make some poly(pyrrole) by **growing** it on a nickel anode as a thin film. You will then remove this film by carefully peeling it off the anode, and test its conducting properties.

Safety

There are potentially hazardous substances involved in this exercise. You **must** follow all health and safety instructions given to you by your teacher. Materials data safety sheets should be consulted so that the correct action can be taken in event of a spillage and/or accident.

	You must wear eye protection throughout this experiment
	Pyrrole is toxic
	Sodium 4-methylbenzenesulphonate is irritant and harmful
	Propanone is highly flammable

**Procedure**

1. **Copper electrode:** Clean the piece of copper supplied with wire wool or emery paper and then rinse it with distilled water. Loop it over the side of the 250 cm³ beaker so that it reaches the bottom.
2. **Nickel electrode:** Clean the flat end of a nickel spatula with a non-abrasive metal cleaner such as 'Brasso' (you will only be able to peel the polymer off if the nickel electrode is very **clean** and **smooth**). Rinse thoroughly with distilled water, and then with propanone. Allow the spatula to dry in the air.
3. **Do not touch the flat part of the spatula with your fingers once you have washed it.**
4. Work in a fume cupboard. Using a teat pipette, drip about 0.4 g of pyrrole into a 250 cm³ conical flask (**CARE: irritant**). If you **spill** any pyrrole on your hands, **wash** it off with **lots** of **water**.
5. Add 100 cm³ of sodium 4-methylbenzenesulphonate solution (concentration
6. 0.100 mol dm⁻³) to the pyrrole. Swirl the flask thoroughly until the pyrrole has dissolved.
7. Pour the pyrrole solution into the 250 cm³ beaker and set up the circuit as shown in the diagram.
8. Start with a low current and gradually increase it to 30 mA.

Appendix 2

9. The nickel electrode should turn black within seconds and bubbles of hydrogen will form at the copper cathode.
10. Continue passing a current for about 45 minutes.
11. Remove the nickel electrode and wash it with water. Carefully peel off the poly(pyrrole) film in **one piece** using a scalpel or razor blade.

Testing the conductivity of poly(pyrrole)

1. Place your film on a glass slide. Fold the film in half and half again (four thicknesses) to reduce the possibility of it burning out when a current is passed through it.
2. Using crocodile clips, connect the film into a simple series circuit containing a 1.5 V bulb and a variable 12 V d.c. supply. Initially, place the crocodile clips about 5 cm apart and start with a p.d. of 2 V. Gradually increase the p.d. and then move the crocodile clips closer together. You should find that when the p.d. is 12 V and the crocodile clips are about 1 cm apart, the bulb will light.

Question

Suggest, with explanation, how the conductivity of poly(pyrrole) compares with that of copper, and with polymers such as poly(ethene) or nylon.

29. The preparation of poly(pyrrole) (a conducting polymer)

Teachers' Notes

The exercise provides a novel way of preparing a polymer which, once formed, has unusual properties. To work properly, care and precision are required.

Intended learning outcomes

Please see the Student Sheet

A suggested approach

This is a relatively simple practical exercise but it does require great care and patience if it is to work properly. The chemicals used must be handled with care but the exercise is not beyond the capabilities of A level students.

The polymer is formed at the anode of an electrolytic cell, with hydrogen gas being evolved at the cathode. You may wish to revise the theory of electrolysis with your students prior to starting the exercise.

More able students may well deduce that H^+ ions are being produced at the anode, along with the polymer, and that hydrogen atoms are being removed from the 2- and 4- positions on the pyrrole ring. Putting these two ideas together could well result in an interesting discussion of the polymerisation process.

The polymer film is quite fragile and will need to be handled carefully. Students should aim to remove it in one piece and then transfer it to a microscope slide for support. The crocodile clips used to connect the film into the circuit will also need to be carefully positioned if the film is not to be damaged.

It is normal for the teacher to trial experiments before using them with a class of students. The wise teacher will certainly trial this exercise!

Answers to question

The conductivity of the polymer is:

- lower than that of copper (copper doesn't need 12 V to light a 1.5 V bulb, so must have a much higher concentration of delocalised electrons)
- greater than Terylene and nylon (they are non-conductors due to the absence of delocalised electrons)

Technical information

Requirements per student/group

Apparatus

- copper foil (1 cm wide and long enough to be folded over the edge of a beaker and still reach to the bottom)
- nickel spatula
- one 250 cm³ beaker
- one 250 cm³ conical flask
- 0–5000 Ω variable resistor
- 12 V d.c. supply (2 V steps)
- ammeter range 0–30 mA
- 1.5 V light bulb and holder
- teat pipette
- razor blades or equivalent

Appendix 2

- microscope slides
- access to fume cupboard
- crocodile clips
- wires

Materials

- pyrrole (0.4 g)
- 100 cm³ of sodium 4-methylbenzenesulphonate (*p*-toluenesulphonic acid sodium salt) solution of concentration 0.10 mol dm⁻³
- propanone (20 cm³)
- wire wool or emery paper
- 'Brasso' metal polish
- small pieces of cloth
- paper towels

Safety

The main points are included on the Student Sheet. However:

- It is essential that a risk assessment is carried out before a decision is taken to go ahead with this exercise. MSDS sheets should be consulted so that the correct action can be taken in event of a spillage and/or accident.
- It must be **made clear** to students that potentially hazardous materials are in use and that precautions are needed to minimise the risk to themselves and to others.
- Your **MUST** be prepared to **intervene** if a student seems to be unsure of a procedure, or is performing an unsafe operation.