Apparatus and techniques for microscale chemistry

Teacher's Information Sheet

This section provides information about some of the apparatus and techniques that are needed in the microscale chemistry experiments described in this series. CLEAPSS, SSERC and microchemuk have up-to-date resources for microscale chemistry.

Apparatus

1. Transparent plastic sheet (OHP sheet)

These sheets are widely used in schools and colleges and are used to overlie the student worksheets in several experiments. Students add drops of solutions onto the sheets to do the reactions. The sheets are reasonably resilient and may be wiped clean with household tissues and re-used many times. However, they could be attacked by strong acids and they are stained by iodine solution if left in contact for more than a few minutes.

Alternatively, clear plastic wallets (A4 size) can be used or the student worksheets could be laminated. Again the wallets or laminated sheets can be wiped clean after use.

Whichever type is used, aqueous solutions form nicely-defined drops on the surface which enable chemical reactions to be conveniently carried out. A discussion of the shape of the drops can provide students with interesting insights into the effects of hydrogen bonding on surface tension.

2. Plastic pipettes

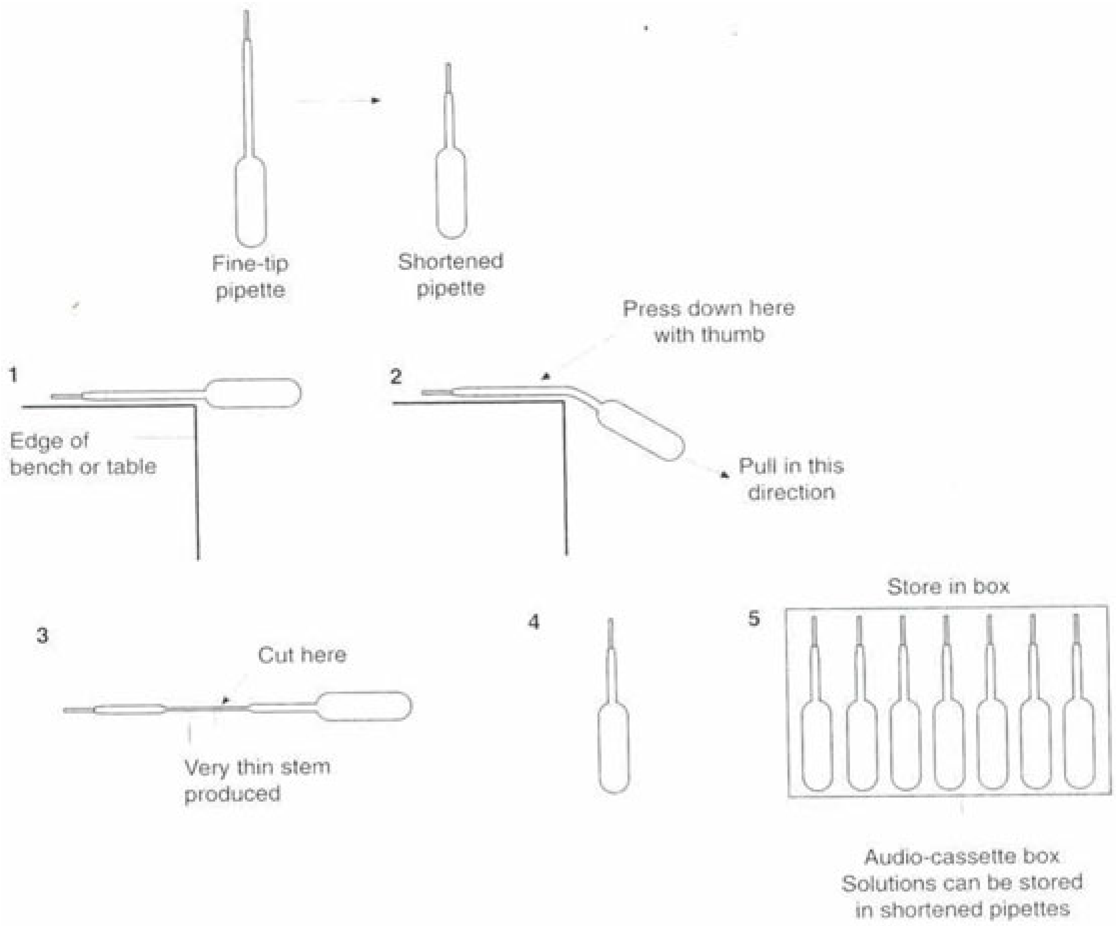
These very versatile pieces of plastic apparatus may be used for storing solutions and dispensing drops of solution during experiments. By cutting and re-shaping them it is possible to make scoops and spatulas, filter funnels, mini reaction vessels, and build electrolysis apparatus. The two most useful forms of pipette are:

Fine-tip form, *eg* Aldrich Z13,503-8

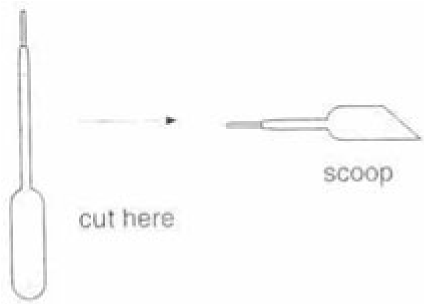
Standard-tip form, *eg* Aldrich Z13,500-3

These plastic pipettes have many uses. Two of the most useful are preparing a shortened pipette for storing solutions and making a scoop.

Preparing a shortened pipette



Making a scoop

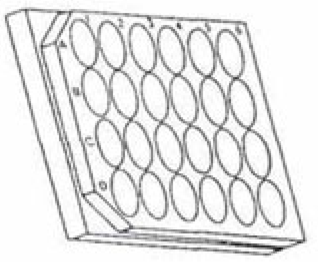


Plastic petri dishes

These are used as containers in which test gases can be generated. Examples are the 4.5 cm and 9 cm diameter sizes supplied by Philip Harris (ref: B8H64838).

Well-plates

Clear plastic well-plates are sometimes listed in catalogues under culture apparatus. The most useful version is the 24-well plate which consists of an arrangement of 6 × 4 cylindrical wells each well having a capacity of *ca* 3 cm^3.



These well-plates are used for organic chemistry experiments, equilibria, rates of reaction and colorimetry experiments. They are compatible with many organic liquids.

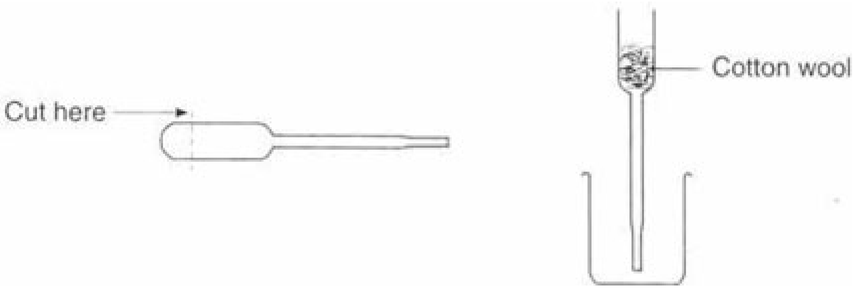
Other apparatus

Other useful pieces of equipment are audio-cassette boxes for storing shortened plastic pipettes, plastic boxes for 35 mm film, plastic display boxes for pens, temperature strips and screw-top sample bottles.

Techniques

1. Microscale filtration

This is a simple but effective method for constructing a filter funnel from a plastic pipette.



Microscale filtration

Your filter funnel is now ready to use. The efficiency of the filter funnel depends mainly on how compact the cotton wool is in the funnel. For coarse particles the cotton wool need not be packed very tightly. However, if very fine particles are to be separated from the liquid tight packing is essential for effective separation.

NB In microscale filtration, transfer of liquids is always by pipette never by pouring.

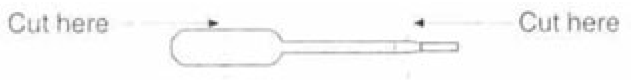
2. Sampling a bottle of hydroxybenzene (phenol)

Hydroxybenzene is a hazardous substance and sampling a bottle of hydroxybenzene using a spatula is usually difficult. The need to ensure that crystals of hydroxybenzene do not come into contact with the skin, the wearing of gloves and the fact that hydroxybenzene is hygroscopic, causing the crystals to stick together, all add to the difficulties. The following procedure reduces safety hazards and allows students to gain in confidence and practical skills. Students must still wear eye protection and gloves.

This technique illustrates the use of two plastic pipettes for obtaining small samples of hydroxybenzene crystals suitable for use in microscale chemistry experiments.

Procedure

1. Take a standard form plastic pipette and cut off the ends as indicated.



2. Cut the tip off the end of a fine-tip pipette as indicated.

3. Taking the modified standard pipette, press it gently down into the crystals in the hydroxybenzene bottle and withdraw. A small column of solid hydroxybenzene should be held on the inside of the bevelled end

4. Place the pipette over the petri dish and insert the fine-tipped pipette to press out a small quantity of hydroxybenzene crystals. (Repeat if necessary at other locations in the petri dish for example if you are doing the 'Reactions of Hydroxybenzene' experiment).

5. Place the ends of both pipettes into a 100 cm^3 beaker about half full with 1 mol dm^{-3} sodium hydroxide solution. This dissolves any solid hydroxybenzene remaining on the pipettes.

6. The pipettes may then be washed with deionised water, dried on tissue paper and stored ready for re-use.

Sampling a bottle of hydroxybenzene

Microscale titrations

Teacher's guide

Titrations are widely used in post-16 chemistry courses. The traditional apparatus comprises 50 cm^3 burettes and 25 cm^3 pipettes. For a whole class, experiments with this size of apparatus consume large quantities of solutions and students often take a long time to do the titrations. They are frequently very messy with spillages from pouring solutions and leaking taps on burettes!

Microscale titrations have several advantages compared with conventional titrations. Details of how to construct the apparatus are given in the student worksheet.

Description

In these microscale titrations the 50 cm^3 burette is replaced by a 2 cm^3 graduated pipette and the 25 cm^3 pipette by a 1 cm^3 pipette. A diagram of the apparatus is shown in the student worksheet overleaf.

To do the titration the plunger is gently pressed down. The volume of the drops produced is *ca* 0.02 cm^3 – without the fine tip the drop volume is *ca* 0.04 cm^3. The apparatus can be read to 0.01 cm^3 (compared with 0.05 cm^3 with a conventional burette) although the volume of solution delivered (less than 2 cm^3) is far less.

The main advantages of this technique are:

- the greatly reduced volumes of solutions required (with the associated reductions in cost);
- the removal of the need for pouring solutions;
- increased speed of titration; and
- the smaller quantities of solutions to dispose of at the end of the experiment.

The apparatus can be used for the following experiments:

- Acid-base neutralisation
- Measuring an equilibrium constant
- Finding out how much salt there is in seawater
- Measuring the amount of vitamin C in fruit drinks

Apparatus (per pair or group)

- One clamp stand with two bosses and clamps
- One 2 cm^3 graduated pipette
- One plastic pipette (fine-tip form)
- One 10 cm^3 plastic syringe
- Rubber tubing (for connectors)
- Adaptor (made from a 1 cm^3 plastic syringe)
- One 10 cm^3 beaker.

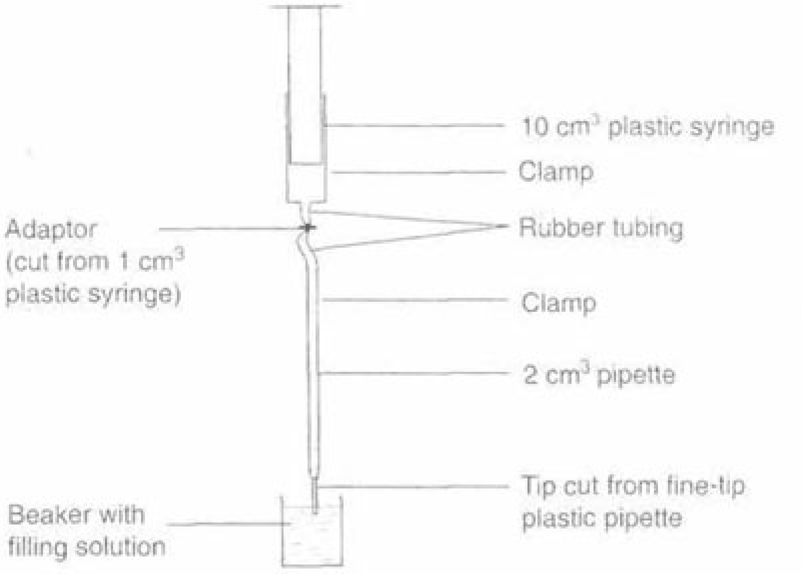
Acknowledgement

I thank Dr Mono M. Singh, Director, National Microscale Chemistry Centre, North Andover, Massachusetts, US for help in preparing this technique.

Microscale titrations

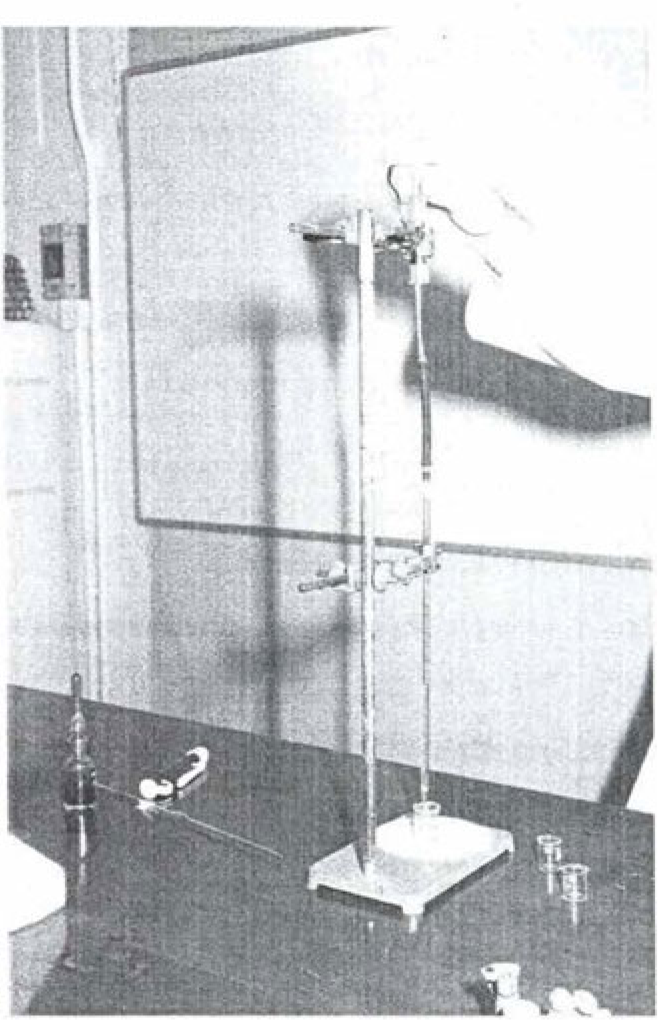
Student worksheet

In this technique you will be constructing a microscale titration apparatus and then using it to do titrations.



Instructions

1. Place the 2 cm^3 pipette in a clamp. This is your 'burette' during the titration.
2. Cut the end off a fine-tip plastic pipette and push it carefully onto the end of the 'burette'.
3. Attach the 10 cm^3 plastic syringe to a clamp above the 'burette'.
4. Cut the end off a 1 cm^3 plastic syringe to make an adaptor.
5. Cut two short pieces of rubber tubing and use them to attach the syringe to the top of the 'burette' via the adaptor.



Filling a microscale titration apparatus.

Filling the 'burette'

1. Take a 10 cm^3 beaker and using a plastic pipette half-fill it with the solution for the 'burette'.
2. Place the tip of the 'burette' in the solution and slowly raise the plunger. The solution is drawn up into the 'burette'. (If air bubbles are drawn up raise and lower the plunger slowly a few times to expel them). When the desired level is reached release the plunger and the liquid level in the 'burette' should remain stationary. If the level falls, adjust the connections on the apparatus to ensure that the system is air-tight and repeat the filling process.

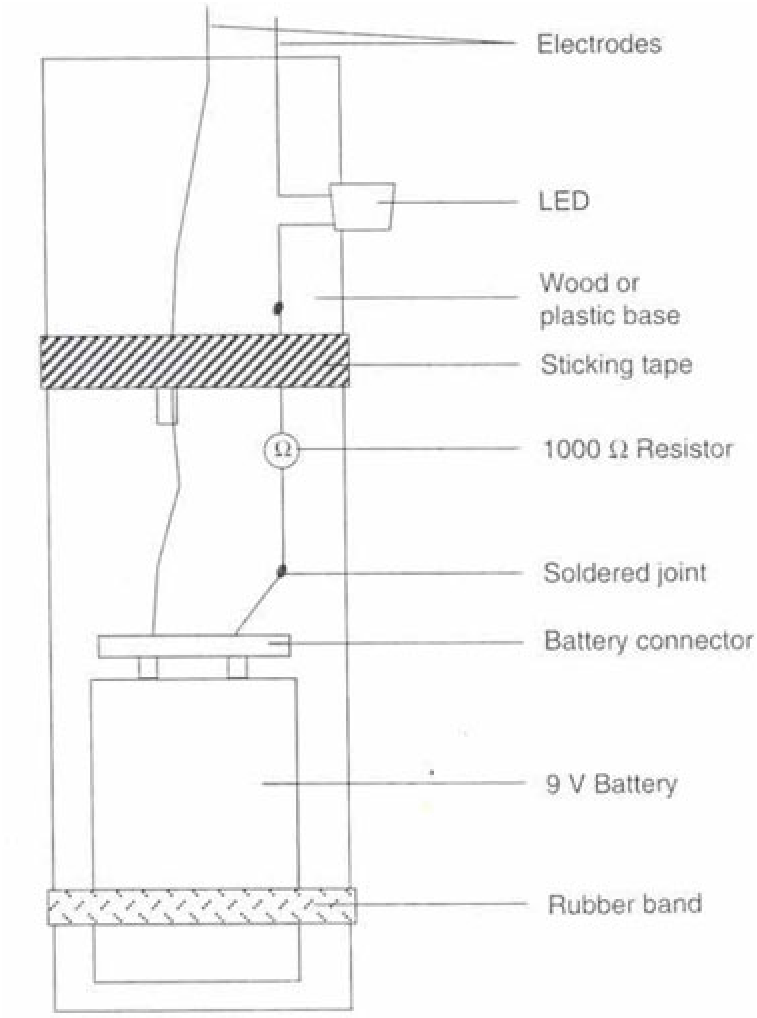
Constructing a conductivity meter

Teacher's guide

This procedure gives instructions for constructing a conductivity meter that can be used for testing conductivity of solutions or solids. It is possible for students to make the apparatus themselves. Alternatively students could be given the instruments ready-made.

Apparatus (per group)

- One light-emitting diode (LED)
- One 1000 Ω resistor
- One 9 V battery
- One battery connector
- One piece of thin wood or plastic - *ca* 150 × 25 mm
- Solder wire or tape
- Soldering iron
- Sticking tape
- One thick rubber band.


Electrodes
LED
Wood or
plastic base
Sticking tape
1000 Ω Resistor
Soldered joint
Battery connector
9 V Battery
Rubber band

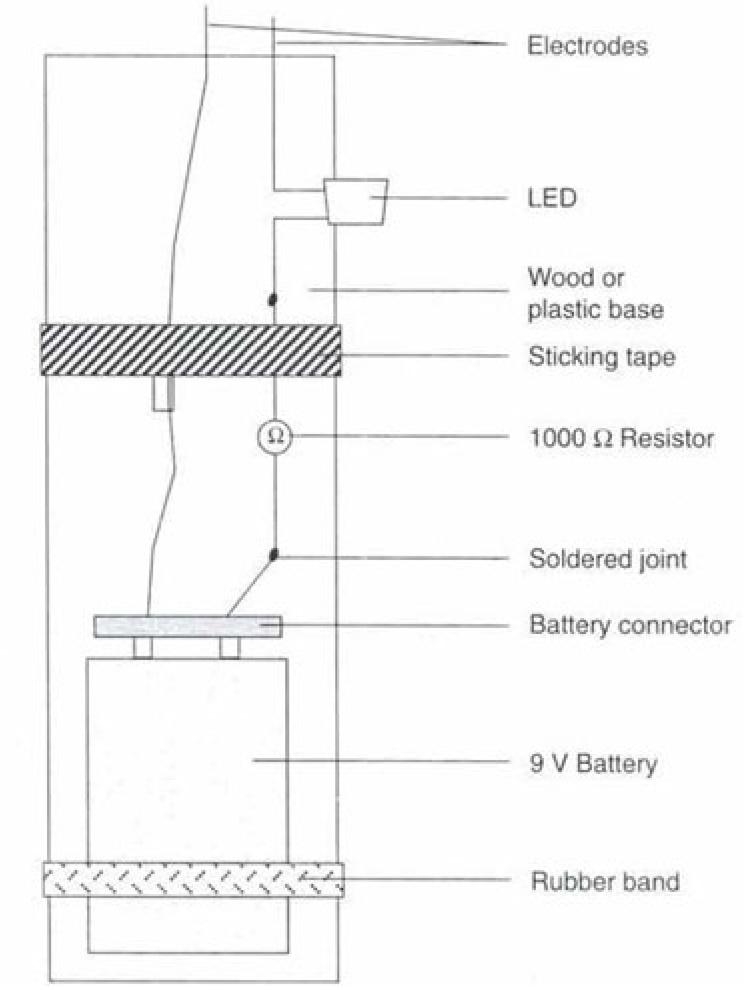
Constructing a conductivity meter

Student worksheet

In this experiment you will be making a conductivity meter which you can use to test the conductivity of solutions and solids such as metals.

Instructions

A diagram of the meter is shown here. Your teacher will give you guidance on how to build the conductivity meter.



Microscale Hoffman apparatus

Teacher's guide

Topic

Electrolysis.

Level

Pre-16 and post-1 6,

Timing

20 min.

Description

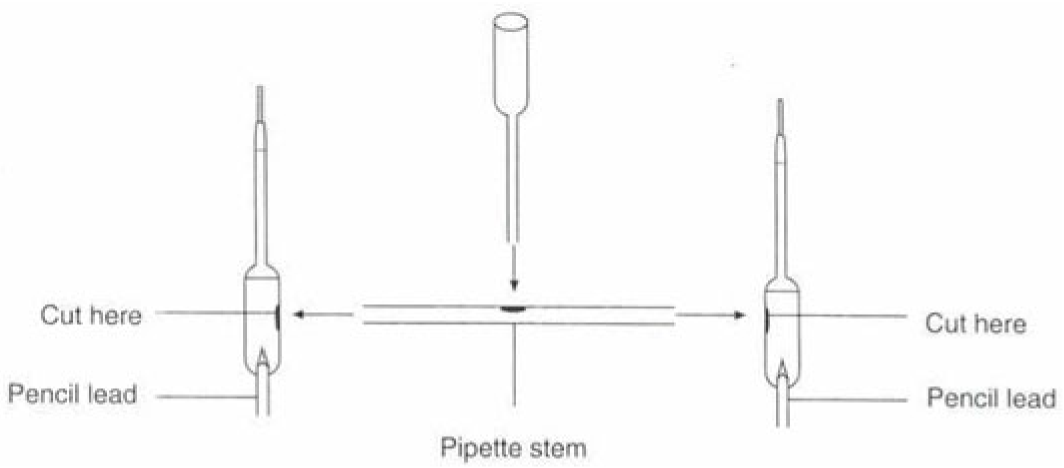
In this experiment students make a microscale Hoffman apparatus from plastic pipettes and use it to investigate aspects of electrolysis. Instructions are given here for the construction of the apparatus. Experiment 38 describes an electrolysis using the apparatus.

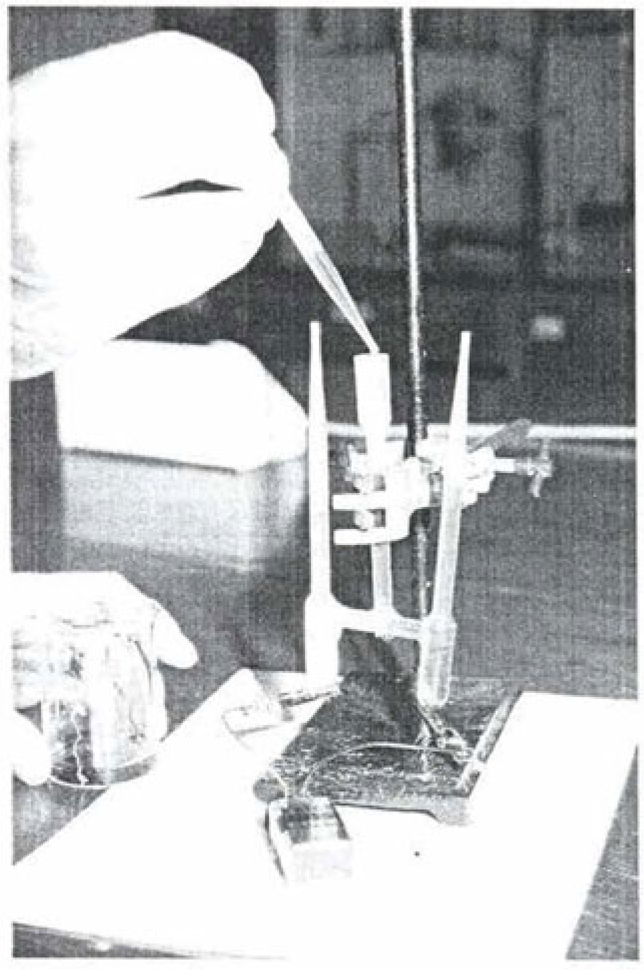
Apparatus

- Four plastic pipettes (standard form, *eg* Aldrich ref' Z13,500-3)
- Scissors
- A pin
- Pencil leads (HB 0.9 mm)
- Adhesive, *eg* polystyrene glue.

Construction

1. Using the scissors cut two holes in two of the pipettes as indicated in the diagram.
2. Cut off the stem from a third pipette and insert it in the first two pipettes to join them.
3. Cut off the tip and end of the bulb of the fourth pipette and insert the tip end into a hole in the middle of the stem joining the first two pipettes.
4. With the pin make a hole in the bottom of the two bulbs and carefully insert a pencil lead into each.
5. Apply adhesive to each joint and leave to dry.





A set-up showing a microscale Hoffman apparatus.

Reference

Chemunity News, 1994, American Chemical Society.

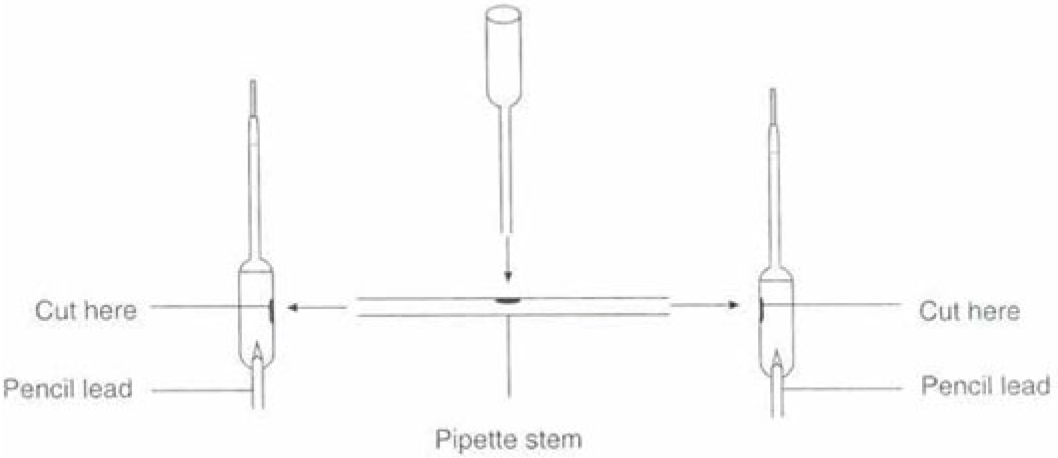
Microscale Hoffman apparatus

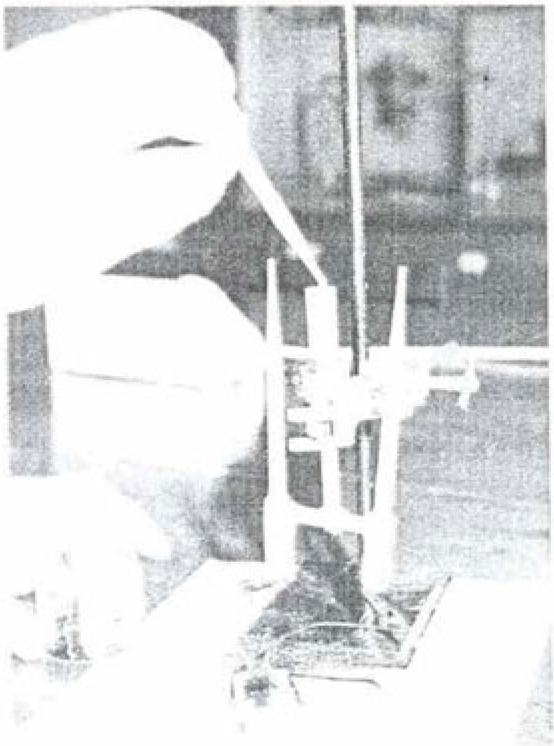
Student worksheet

Follow these instructions to make a microscale Hoffman electrolysis apparatus out of plastic pipettes.

Construction

1. Using the scissors cut two holes in two of the pipettes as indicated in the diagram.
2. Cut off the stem from a third pipette and insert it in the first two pipettes to join them.
3. Cut off the tip and end of the bulb of the fourth pipette and insert the lip end into a hole in the middle of the stem joining the first two pipettes.
4. With the pin make a hole in the bottom of the two bulbs and carefully insert a pencil lead into each.
5. Apply adhesive to each joint and leave to dry.





Element solutions

Barium

Barium nitrate solution (0.2 mol dm^{-3}) – dissolve 5.2 g $Ba(NO_3)_2$ in 100 cm^3 of deionised water.

Barium nitrate is an oxidizing agent and is harmful if swallowed or inhaled.

A 0.2 mol dm^{-3} solution is of low hazard

Calcium

Calcium nitrate solution (0.5 mol dm^{-3}) – dissolve 11.8 g of $Ca(NO_3)_2.4H_2O$ in 100 cm^3 of deionised water.

Calcium nitrate solution (0.2 mol dm^{-3}) – dissolve 4.7 g of Ca $(NO_3)_2.4H_2O$ in 100 cm^3 of deionised water.

Calcium nitrate is an oxidizing agent, is harmful if swallowed and is a skin/eye irritant.

A 0.5 mol dm^{-3} solution is a skin/eye irritant and a 0.2 mol dm^{-3} solution is of low hazard.

Chromium

Potassium chromate solution (0.2 mol dm^{-3}) – dissolve 3.9 g of K_2CrO_4 in 100 cm^3 of deionised water.

Potassium chromate is a carcinogen, mutagen and skin sensitiser as well as a skin/eye/respiratory irritant

A 0.2 mol dm^{-3} solution is a carcinogen, mutagen and skin sensitiser.

Cobalt

Cobalt nitrate solution (0.5 mol dm^{-3}) – dissolve 14.6 g of $Co(NO_3)_2.6H_2O$) in 100 cm^3 of deionised water.

Both the solid and a 0.5 mol dm^{-3} solution is a carcinogen, mutagen, reproductive toxin, skin and respiratory sensitiser and toxic to aquatic life.

Copper

Copper sulphate solution (0.5 mol dm^{-3}) – dissolve 12.5 g of $CuSO_4.5H_2O$ in 100cm^3 of deionised water.

Copper sulphate solution (0.2 mol dm^{-3}) – dissolve 5.0 g of $CuSO_4.5H_2O$ in 100cm^3 of deionised water.

Copper sulphate causes serious eye damage, is harmful if swallowed and is toxic to aquatic life.

Both 0.5 and 0.2 mol dm^{-3} solutions cause serious eye damage and are toxic to aquatic life.

Iron

Iron(lll) nitrate solution (0.2 mol dm^{-3}) – dissolve 8.1 g of $Fe(NO_3)_3.9H_2O$ in 100 cm^3 of deionised water.

Iron III nitrate is an oxidiser and a skin/eye/respiratory irritant.

A 0.3 mol dm^{-3} solution is of low hazard

Iron(ll) sulphate solution (0.2 mol dm^{-3}) – dissolve 5.6 g of $FeSO_4.7H_2O$ in 100 cm^3 of deionised water. Add sulphuric acid (1 mol dm^{-3}) to make up to 200 cm^3. (The presence of the acid minimises the hydrolysis of iron(ll).)

Iron II sulphate is harmful if swallowed and a skin/eye irritant.

A 0.2 mol dm^{-3} solution made as above will be of low hazard.

Lead

Lead nitrate solution (0.5 mol dm^{-3}) – dissolve 16.6 g of $Pb(NO_3)_2$ in 100 cm^3 of deionised water.

Lead nitrate is an oxidiser, harmful if swallowed or inhaled, is corrosive to skin and eyes, is a reproductive toxin and is very toxic to aquatic life.

A 0.5 mol dm^{-3} solution is corrosive to skin and eyes, a reproductive toxin and very toxic to aquatic life.

Lithium

Lithium bromide solution (1 mol dm^{-3}) – dissolve 4.3 g of lithium bromide in 50 cm^3 of deionised water.

Lithium bromide is harmful if swallowed and a skin/eye irritant.

A 1 mol dm^{-3} solution is of low hazard.

Magnesium

Magnesium nitrate solution (0.5 mol dm^{-3}) – dissolve 7.4 g of $Mg(NO_3)_2$ in 100 cm^3 of deionised water.

Magnesium nitrate is an oxidiser and a skin/eye/respiratory irritant.

A 0.5 mol dm^{-3} solution is of low hazard.

Manganese

Potassium manganate(VII) solution (0.01 mol dm^{-3}) – dissolve 0.16 g of $KMnO_4$ in 100 cm^3 of deionised water.

Potassium manganate VII is an oxidiser, is harmful if swallowed and is toxic to aquatic life.

A 0.01 mol dm^{-3} solution is of low hazard.

Molybdenum

Ammonium molybdate solution (0.05 mol dm^{-3}) – dissolve 6.2 g of $(NH_4)_6Mo_7O_{24}.4H_2O$ in 100 cm^3 of water.

Ammonium molybdate Is harmful if swallowed and a skin/eye/respiratory irritant.

A 0.05 mol dm^{-3} solution is of low hazard.

Nickel

Nickel nitrate solution (0.5 mol dm^{-3}) – dissolve 14.5 g of $Ni(NO_3)_2.6H_2O$ in 100 cm^3 of deionised water.

Nickel nitrate is an oxidiser, Is harmful if swallowed ir inhaled, is a skin irritant, causes serious eye damage, is a skin and respiratory sensitiser, is a carcinogen (by inhalation), a mutagen, a reproductive toxin, causes damage to organs and is very toxic to aquatic life.

A 0.5 mol dm^{-3} solution is a skin irritant, causes serious eye damage, is a skin and respiratory sensitiser, is a carcinogen (by inhalation), a mutagen, a reproductive toxin, causes damage to organs and is very toxic to aquatic life

Potassium

Potassium bromide solution (0.2 mol dm^{-3}) – dissolve 2.4 g KBr in 100 cm^3 of deionised water.
Potassium iodide (0.2 mol dm^{-3}) – dissolve 3.3 g KI in 100 cm^3 of deionised water.

Potassium bromide and iodide are both eye irritants.

0.2 mol dm^{-3} solutions of both salts are of low hazard.

Silver

Silver nitrate solution (0.1 mol dm^{-3}) – dissolve 1.7 g of $AgNO_3$ in 100 cm^3 of deionised water. Store in a dark place.

Silver nitrate is an oxidiser, is corrosive to skin and eyes and is very toxic to aquatic life.

A 0.1 mol dm^{-3} solution is a skin/eye irritant.

Sodium

Sodium fluoride solution (0.5 mol dm^{-3}) – dissolve 1.1 g of NaF in 50 cm^3 of deionised water.

Sodium fluoride is toxic if swallowed and a skin/eye irritant.

A 0.5 mol dm^{-3} solution is of low hazard.

Sodium carbonate solution (0.5 mol dm^{-3}) – dissolve 5.3 g of Na_2CO_3 in 100 cm^3 of deionised water.

Sodium carbonate is an eye irritant.

A 0.5 mol dm^{-3} solution is of low hazard.

Sodium chloride solution (0.5 mol dm^{-3}) – dissolve 2.9 g of NaCl in 100 cm^3 of deionised water.

Sodium sulphate (0.5 mol dm^{-3}) – dissolve 7.1 g of Na_2SO_4 in 100 cm^3 of deionised water.

Sodium chloride and sodium sulphate are of low hazard.

Strontium

Strontium nitrate solution (0.5 mol dm^{-3}) – dissolve 10.6 g of $Sr(NO_3)_2$ in 100 cm^3 of deionised water.

Strontium nitrate is an oxidiser and causes serious eye damage.

A 0.5 mol dm^{-3} solution causes serious eye damage.

Tungsten

Sodium tungstate solution (0.2 mol dm^{-3}) – dissolve 6.6 g of $Na_2WO_4.2H_2O$ in 100 cm^3 of deionised water.

Sodium tungstate is harmful if swallowed, causes serious eye damage and is toxic to aquatic life.

A 0.2 mol dm^{-3} solution causes serious eye damage and is toxic to aquatic life.

Vanadium

Ammonium vanadate solution (0.2 mol dm^{-3}) – dissolve 2.3 g NH_4VO_3 in 100 cm^3 of deionised water.

Ammonium vanadate is harmful if swallowed, inhaled or in contact with the skin.

A 0.2 mol dm^{-3} solution is of low hazard.

Zinc

Zinc sulphate solution (0.2 mol dm^{-3}) – dissolve 5.8 g of $ZnSO_4.7H_2O$ in 100 cm^3 of deionised water.

Zinc sulphate is harmful if swallowed, causes serious eye damage and is toxic to aquatic life.

A 0.2 mol dm^{-3} solution causes serious eye damage and is toxic to aquatic life.

Credits

Health & safety checked May 2018

Page last updated August 2018