Identification of Inks in Felt Tip Pens Using Paper Chromatography

Felt tip pens have a 'rod' inside them. It's made of an absorbent material and is impregnated with a mixture of different coloured dyes. The colour you get from a pen depends on the mixture of inks used. Ink dyes can be separated using paper chromatography.

There are two types of pens – permanent and non-permanent. The difference is how easily they can be washed from what they have been used to mark (whether deliberately or accidently).

1. Scope

The procedure described below may be used to identify the ink dyes used in non-permanent and permanent felt tip pens.

2. Principle

Substances are separated in chromatography because of the difference in their attraction for the mobile phase (the solvent) and the stationary phase. The greater the attraction for the stationary phase relative to the mobile phase, the slower a substance moves through it.

3. Reagents

- Water
- Propan-2-ol (**HIGHLY FLAMMABLE**; **IRRITANT**).

4. Equipment

- Boiling tubes
- Scissors
- Ruler
- Test-tube racks
- Chromatography paper.

5. Health and safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.

6. Procedure

- Cut pieces of chromatography paper (one for each pen to be tested) slightly longer than the boiling tubes and slightly narrower than the diameter of the tubes.
- Place boiling tubes (one for each piece of chromatography paper) in a test-tube rack.
- Draw a pencil line about 2 cm from one end. At the centre of the line use a felt tip to make a dot. Take care that it doesn't spread out too much. Ideally it should be a dark dot no more than 2 mm across.
- Fold the chromatography paper at the opposite end to the dot so that it can rest on a small wooden stick (such as a cocktail stick).
- Place about 1 cm depth of water in each tube.
- Carefully insert the chromatography paper into the test-tube, dotted end down. The dot must be above the water, and the sides of the chromatography paper cannot touch the sides of the test-tube.
- Leave the test-tube until good separation is obtained or until the solvent front reaches 1-2 cm from the top of the chromatography paper.
- Remove the chromatogram and hang it to dry.
- Mark the solvent front with a pencil and put a small cross at the centre of each spot.
- Repeat the experiment using a 1:1 (volume:volume) mixture of propan-2-ol and water mixture as the solvent.

7. Calculations

Calculate the retention factors (R_f) for spots found on the developed chromatogram using the formula

$R_f = X/Y$

where X = distance moved by 'spot' (measured to the centre of the spot), and Y = distance moved by the solvent

8. Expression of results

Give the colour and R_f values of each spot that appears on the chromatograms.

Identification of Amino Acids in Fruit Juice Using Paper Chromatography

'Fruit and vegetable juices. Determination of free amino acids content. Liquid chromatographic method' (BS EN 12742:1999) ***is the British Standard method for determining the free amino acids content of fruit and vegetables. This method is used to determine the quantities present (in other words, for quantitative analysis). In this experiment non-quantitative paper chromatography is used. However, it is simpler and enables the amino acids present to be identified.***

1. Scope

The procedure may be used to identify some of the amino acids present in freshly squeezed fruit juice.

2. Principle

Substances in solution 'travel' up chromatography paper at different rates depending on their relative solubility in the mobile and stationary phases. By measuring the retention factors or using standard reference materials, the components in a mixture can be identified.

3. Reagents

- Ethanol (**HIGHLY FLAMMABLE**)
- Developing solvent:
  24.0 cm^3 butan-1-ol (**HARMFUL; FLAMMABLE**)
  5.0 cm^3 glacial ethanoic acid (**CORROSIVE**)
  10.0 cm^3 distilled water
- Ninhydrin spray (0.5% solution of ninhydrin in butan-1-ol) (**HARMFUL; FLAMMABLE**)
- Standard reference solutions:
  0.01 mol dm^{-3} solutions of amino acids in propan-2-ol (eg *Amino acids reference substances for chromatography. Collection A* from VWR International)
  Note: propan-2-ol (**HIGHLY FLAMMABLE**; **IRRITANT**).

4. Apparatus

- Test-tube with stopper/bung
- Filter funnel and cotton wool
- 400 cm^3 beaker, with Petri dish as lid
- Glass rod
- Melting point tubes (or equivalent) for spotting the chromatogram
- Spray for the locating agent.
- Access to fume cupboard

5. Health and Safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.

6. Procedure

- Cut the piece of fruit and squeeze to obtain a small quantity of juice.
- Put 1 cm^3 of the freshly squeezed fruit juice into a test-tube and add 3 cm^3 of ethanol. Put a stopper or bung on the test-tube. Shake well and filter through a small cotton wool plug in a filter funnel.
- Keep the filtrate in a labelled sample tube.
- Before handling the chromatography paper, put on gloves to avoid contamination by the amino acids from the sweat on the skin surface.
- Place a glass rod across the top of a 400 cm^3 beaker. Cut a piece of chromatography paper 5 cm longer than necessary to reach to the bottom of the jar, and fold it over the rod. The bottom of the paper should be 0.5 cm above the bottom of the beaker.
- Remove the paper, and pour developing solvent into the beaker to a depth of about 1 cm. Cover the jar with a Petri dish.
- Rule a pencil line 2 cm from the bottom of the chromatography paper. Mark a small cross for each substance that you are going to run, and label them (also in pencil).
- Spot the filtrate (containing the amino acids in the fruit juice) on the chromatogram. Use the smallest quantity possible and allow the spot to dry. Repeat several times to build up a small, concentrated spot. Now spot the standard reference solutions of amino acids.

- Carefully hang the chromatogram over the glass rod. Make sure it does not touch the sides and that the spots do not dip into the developing solvent. Replace the lid. Let it run until the solvent has travelled nearly to the top of the paper.
- Remove the chromatogram from the tank and mark the level of the solvent front with a pencil.
- Allow to dry, then spray with ninhydrin solution in a fume cupboard. Allow to dry again and mark the centre of each spot.

7. Calculations

Calculate the retention factors (R_f) for spots found on the developed chromatogram using the formula

$R_f = X / Y$

where X = distance moved by 'spot' (measured to the centre of the spot), and Y = distance moved by the solvent

8. Expression of results

State which amino acids are found in the sample analysed.

Identification of Lipsticks using Thin Layer Chromatography

Forensic scientists use chromatography to analyse some types of evidence left at the scene of crime. The techniques are usually very sophisticated (gas-liquid chromatography and high performance liquid chromatography), but sometimes the relatively simple technique of thin layer chromatography is used. Forensic scientists use this most often for the identification of fabric dyes and drugs.

1. Scope

This procedure may be used to identify lipstick smears found on surfaces such as paper tissues, handkerchiefs and clothing.

2. Principle

Lipsticks consist of fats, waxes, oils, flavourings, perfumes, and dyes (mainly aluminium, calcium, or barium dyes). Identifying the lipstick responsible for leaving a smear can be done by straightforward colour matching. This will leave a small number, all of which give a reasonable colour match. The colourings in these lipsticks may be separated by thin layer chromatography. This colour analysis may be used to identify the lipstick used to make the smear.

3. Reagents

- Methanol (**HIGHLY FLAMMABLE**; **TOXIC**)
- Developing solvent:
  50 cm^3 3-methylbutan-1-ol (**HARMFUL; FLAMMABLE**)
  50 cm^3 propanone (**FLAMMABLE**; **HARMFUL**)
  25 cm^3 distilled water
  5 cm^3 drops of 6 mol dm^{-3} ammonia (**CORROSIVE**).
- Access to fume cupboard

4. Apparatus

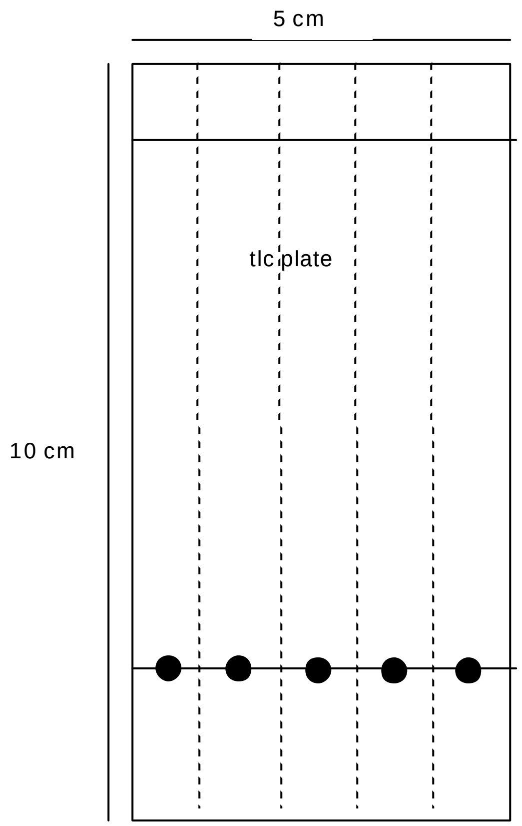
- 400 cm^3 beaker
- Large watch glass
- Filter paper
- Silica gel tlc plate
- Sharp knife
- 5 small sample tubes with adhesive labels
- 5 small glass rods
- Dropping pipette
- Capillary tubes.

5. Health and Safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.

6. Procedure

- Cut a piece of tlc plate (5 x 10 cm). Mark it with a pencil as shown by the solid lines in the diagram. Use a knife to score lines on the plate (in other words, remove the coating from the tlc plate) as shown by the dashed lines in the diagram. These will help to prevent the ‘lanes’ from running into each other as the tlc plate is developed.



- Pour the developing solvent into a 400 cm^3 beaker to a depth of 1 cm. This should be done in a fume cupboard. Place two pieces of filter paper in the beaker so that they are leaning against the inside of the beaker. Cover with the large watchglass. Leave so that the atmosphere becomes saturated with the solvent.

- Five paper tissues are provided. Four have been smeared with known lipsticks. The fifth has been smeared with an 'unknown' lipstick.
- Cut a 1 x 2 cm section of the lipstick sample on the tissue and put in a sample tube. Label with the lipstick name.
- Repeat this for each tissue.
- In a fume cupboard, add 5-10 drops of methanol to each sample. Use a glass rod to 'pound' the tissue so that as much of the lipstick dye goes into solution. Pour these solutions onto seperate watchglasses and leave until most of the methanol has evaporated.
- Use capillary tubes to put one very small drop of each of the coloured methanol solutions on the tlc plate (see diagram).
- Place the tlc plate in the beaker containing the developing solvent and replace the cover. Leave it until the solvent reaches the solvent front line on the plate (see diagram).
- Remove the plate from the solvent and leave it to dry in the fume cupboard.
- Once the plate is dry, draw and label each of the chromatograms.

7. Results

- Compare the spots from the unknown lipstick sample with the four known colours lipsticks.
- Calculate the retention factors (R_f) for spots found on the developed chromatogram using the formula

  $R_f = X / Y$

  where X = distance moved by 'spot' (measured to the centre of the spot), and Y = distance moved by the solvent
- State which lipstick the 'unknown' appears to be.

Identification of Active Ingredients in OTC Pain Relievers Using Thin Layer Chromatography

Chemical compounds that are going to be used in medicines must meet strict purity standards. These standards, and how they are determined are given in pharmacopoeias such as the British Pharmacopoeia (BP) and the European Pharmacopoeia (PhEur). Analyses fall into two categories:* Identification *(qualitative analysis to identify impurities) and* Assay *(quantitative analysis to determine percentage purity). Thin layer chromatography is one of the techniques used for* Identification*.

Two of the most common over-the-counter (OTC) pain-relievers, in other words those you can get without a doctor's prescription, are aspirin and paracetamol.

1. Scope

This procedure may be used to identify aspirin and/or paracetamol in an OTC pain reliever.

2. Principle

Chromatography is a separation technique that may be used to separate components of a mixture.

3. Reagents

- Aspirin
- Paracetamol
- Ethanol (**FLAMMABLE**)
- Developing solvent:
  95 cm^3 ethyl ethanoate (**HIGHLY FLAMMABLE; IRRITANT**)
  5 cm^3 glacial ethanoic acid (**CORROSIVE**)
- Iodine crystals (**HARMFUL**)

4. Apparatus

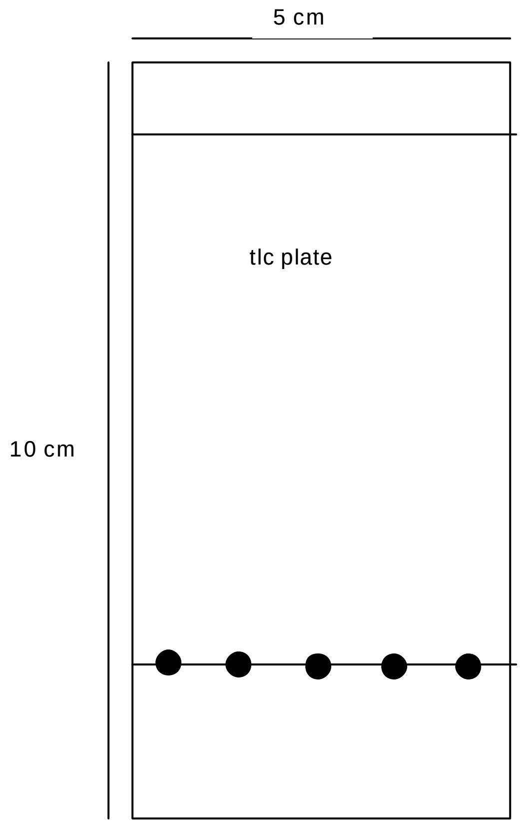
- Tlc plate (coated in silica gel and containing a fluorescing agent)
- Screw-capped jar or tank large enough to take the tlc plates
- Finely drawn glass tubes (for spotting the plates) **CARE** these are fragile and break easily
- Test-tube rack
- Four test-tubes
- UV lamp. **CARE** do not look directly at UV light.

5. Health and Safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.

6. Procedure

- Carefully mark a tlc plate as shown in the diagram.



- Into each of the four test-tubes put about 2 cm^3 ethanol. Label the test-tubes 1 to 4 and add small amounts of the following to the suitably labelled tube:
  – Tube 1: aspirin

- Tube 2: paracetamol
- Tube 3: aspirin and paracetamol
- Tube 4: a powdered sample of the unknown tablet

- Spot samples from test-tubes 1, 2, 3 and 4 on to the tlc plate. Allow a few minutes for the spots to dry.
- Develop the plate in the screw-capped jar containing about 1 cm depth of the developing solvent (make sure it does not cover the spots themselves). Leave until the solvent has risen to about 1 cm from the top of the plate.
- Remove the plate from the jar and lightly mark in pencil (not pen) the position of the solvent front. Leave it in a fume-cupboard to dry.
- Examine the plate under the UV lamp, taking care not to look at directly at the UV light, or stand it in the third screw-capped jar containing 2-3 iodine crystals.

7. Results

By comparing the number and position of spots on the chromatogram, state which OTC pain relievers are in the tablets given.

Determination of Water Hardness

Hard water causes 'furring' in kettles, 'limescale' in sinks and baths and a 'scum' when you wash with soap. There are two types of hardness: temporary and permanent. Temporary hardness causes furring, limescale and scum. It can be removed by boiling. Permanent hardness doesn't cause furring or limescale, but it does form a scum when washing with soap. It can't be removed by boiling.

1. Scope

All samples of water may be tested in this way. The method only gives a measure of relative hardness, in other words, how hard one water sample is compared with another.

2. Principle

The volume of soap solution needed to make a permanent froth is a measure of the hardness. The more soap solution required to make a permanent froth, the harder the water.

3. Reagents

- Soap solution.

4. Apparatus

- 50 cm^3 burette, with stand, clamp and white tile
- 100 cm^3 measuring cylinder
- 250 cm^3 conical flask with stopper
- Tripod and gauze
- Bunsen burner
- Water sample
- Wanklyn's soap solution (**HIGHLY FLAMMABLE**).

5. Health and Safety

- You must always wear protective clothing and eye protection in the laboratory.
- A risk assessment must be carried out before you start work.

6. Procedure

Total hardness (permanent + temporary)

- Measure 100 cm^3 of the water being tested into a 250 cm^3 conical flask.
- Titrate with soap solution, adding 1 cm^3 of soap solution at a time. Stopper the flask and shake it vigorously after each addition.
- When the froth takes longer to disappear, add the soap solution 0.5 cm^3 at a time.
- Continue adding the soap solution until a permanent froth forms.

Permanent hardness

- Measure 100 cm^3 of the water being tested into a 250 cm^3 conical flask.
- Put the conical flask on the tripod and gauze and bring the water to the boil. Simmer for about 5 minutes. **CARE** because of the flammability of soap solution, keep well away from Bunsen flame.
- Allow to cool and add distilled water to make the volume about 100 cm^3 again (some water will have been lost due to evaporation).
- Titrate with soap solution in the same way as for total hardness.

'Blank' titration

- Carry out the same titration as for total hardness except use distilled water instead of the water being tested.

7. Calculations

Sample being tested	Volume of soap solution needed to make a permanent froth/cm^3
100 cm^3 of unboiled water	(a)
100 cm^3 of boiled water	(b)
100 cm^3 of distilled water ('blank' titration)	(c)

Unboiled water has both temporary and permanent hardness. Calculate the volume of soap solution needed to react with the salts causing the hardness using these formulae:

- volume of soap solution needed to react with salts causing total hardness in 100 cm^3 of water = $a - c$
- volume of soap solution needed to react with salts causing permanent hardness in 100 cm^3 of water = $b - c$

- volume of soap solution needed to react with salts causing temporary hardness in 100 cm^3 of water = *a* - *b*

where

a = volume of soap solution needed to react with 100 cm^3 of unboiled water

b = volume of soap solution needed to react with 100 cm^3 of boiled water

c = volume of soap solution needed to react with 100 cm^3 distilled water

8. Expression of results

Give the hardness of water as number of cm^3 required to give a permanent froth with 100 cm^3 of the water being tested.

Analysis of Sodium Carbonate Solution

The miniaturisation of electronic equipment owes much to the introduction of printed circuit boards. The connecting 'wires' for an electrical circuit are etched onto a copper-clad plastic board. The design is marked by a chemically resistant material (called the mask). Unwanted copper is dissolved away using an etching solution. Sodium carbonate is sometimes used. This standard procedure is based upon product data sheets supplied by manufacturers of sodium carbonate solutions used to develop printed circuit boards. The data sheets specify the product and give analytical methods to check concentrations.

1. Scope

This procedure may be used for sodium carbonate solutions with a concentration in the range 0.3 – 0.7 grams of sodium carbonate per 100 cm^3 of solution.

2. Principle

Sodium carbonate reacts with hydrochloric acid to give sodium chloride, carbon dioxide and water. This reaction can be used to determine the concentration of sodium carbonate in solution. The end-point is seen using methyl orange (an acid-base indicator).

3. Reagents

- 0.100 mol dm^{-3} hydrochloric acid
- Methyl orange indicator solution (0.1%).

4. Apparatus

- 25 cm^3 pipette with safety filler
- Pipette filler
- 250 cm^3 conical flask
- 100 cm^3 measuring cylinder
- 50 cm^3 burette with stand, clamp and white tile.

5. Health and Safety

- Wear protective clothing and eye protection.
- A risk assessment must be carried out before starting work.
- Sodium carbonate is a HARMFUL substance. Solutions of sodium carbonate are caustic (pH about 11).

6. Procedure

- Pipette 25 cm^3 of the sodium carbonate solution into 250 cm^3 conical flask.
- Add 100 cm^3 distilled water and then 1 cm^3 methyl orange indicator solution.
- Titrate with 0.100 mol dm^{-3} hydrochloric acid to a pink-orange end-point.
- Repeat the titration.

7. Calculations

Calculate the mass of sodium carbonate in 100 cm^3 solution using the formula:

mass of sodium carbonate = 0.053 x t g

where t is the average volume (cm^3) of 0.100 mol dm^{-3} hydrochloric acid used in the titrations

8. Expression of results

Give the concentration of sodium carbonate in grams per 100 cm^3 (g 100 cm^{-3}).

Determination of Citric Acid in *Hubba Bubba* Bubble Gum

Citric acid (as its monohydrate) is added to food products to give them a sharp, acidic taste. It's used, for example, to flavour Awesome Orange ("It's an orange attack! Let your mouth go wild with this awesome flavour")* Hubba Bubba *bubble gum.

Hubba Bubba *bubble gum is made by* Wrigley *in Plymouth and can be bought in most parts of Europe. The method described here is based on an analytical procedure used by the* Wrigley *company in their Plymouth laboratories.*

1. Scope

This procedure may be used to determine the citric acid content of *Hubba Bubba* bubble gum. It may be adapted to analyse citric acid found in a range of food products. The sampling and extraction procedures may require modification, as may the concentration of sodium hydroxide solution used.

2. Principle

The determination is based on an acid/base reaction between the citric acid in the bubble gum and standard sodium hydroxide. The percentage citric acid in the bubble gum can be calculated from titration results.

3. Reagents

- 0.100 mol dm^{-3} sodium hydroxide solution **(IRRITANT)**
- Phenolphthalein indicator solution (0.2% in ethanol) (**HIGHLY FLAMMABLE**).

4. Apparatus

- Kitchen rolling pin
- 250 cm^3 conical flask
- 250 cm^3 graduated flask
- 100 cm^3 graduated flask
- Magnetic stirrer and follower
- 10 cm^3 burette reading to nearest 0.02 cm^3 with clamp, stand and white tile
- Top pan analytical balance reading to two decimal places.

5. Health and Safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.
- Remember: do not taste, eat or chew in the laboratory

6. Procedure

- Take one piece of orange flavoured Hubba Bubba bubble gum, unwrap it and place on a wood block.
- With a kitchen rolling pin, roll the gum into a very thin strip approx. 160 x 30 x 0.5 mm. Cut the thin strip into small pieces about the size of long grain rice.
- Weigh out 1.00 g of gum bits into a 250cm^3 conical flask.
- Pour 100 cm^3 of distilled water into the flask. Add a magnetic follower and stopper.
- Stir vigorously for 30 minutes making sure the bubble gum bits do not stick together.
- Add 0.5 cm^3 of phenolphthalein indicator solution and titrate with 0.1 mol dm^{-3} sodium hydroxide contained in a 10 cm^3 burette. The end-point is when a pink colour appears and remains after 15 seconds. Record the titre.
- Repeat twice more and average all three results.

7. Calculations

- Calculate the average titration (t cm^3) for the three samples analysed.
- Use the following formula to calculate the mass of citric acid monohydrate, in milligrams, in 100 g of the Hubba Bubba bubble gum:

  mass of citric acid monohydrate in 100 g of gum = $t \times 0.71$ g

8. Expression of results

Give the citric acid content as a percentage by mass (w/w; grams per 100 grams).

Analysis of Washing Soda Crystals

Washing soda is mainly sodium carbonate-10-water, $Na_2CO_3.10H_2O$. However, $Na_2CO_3.10H_2O$ loses water to the atmosphere (it effloresces). A white powder forms on the surface of the crystals. It's better to represent washing soda crystals as $Na_2CO_3.xH_2O$, where x is less than 10. The composition of washing soda crystals can be expressed as a percentage by mass of anhydrous sodium carbonate, Na_2CO_3. In sodium carbonate-10-water there is 37.1% w/w Na_2CO_3.

1. Scope

This procedure may be used to determine the composition of washing soda crystals, both when they are bought and after the package has been opened and closed a number of times.

2. Principle

The sodium carbonate, Na_2CO_3, content of washing soda crystals may be determined by titrating a solution made from the washing soda with standard hydrochloric acid.

Na_2CO_3 (aq) + 2HCl (aq) → 2NaCl (aq)+ H_2O (l) + CO_2 (g)

Screened methyl orange is a suitable indicator.

3. Reagents

- 0.100 mol dm^{-3} hydrochloric acid
- Screened methyl orange indicator solution.

4. Apparatus

- Balance that can measure mass to the nearest 0.01 g or better
- 100 cm^3 beaker
- 250 cm^3 volumetric flask
- 250 cm^3 conical flask
- 50 cm^3 burette with clamp, stand and white tile
- 25 cm^3 pipette with safety filler
- Dropping pipette.

5. Health and Safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.

6. Procedure

- Weigh accurately about 3 g of washing soda crystals into a 100 cm^3 beaker. Stir well until they dissolve completely.
- Transfer the solution quantitatively to a 250 cm^3 graduated flask and make up to the mark with distilled water. Mix well.
- Pipette 25.0 cm^3 of the washing soda solution into a 250 cm^3 conical flask.
- Add 3-4 drops of screened methyl orange indicator.
- Titrate with 0.100 mol dm^{-3} hydrochloric acid until the solution just turns mauve
- Repeat with further 25.0 cm^3 until consistent titrations are obtained.

7. Calculations

- Calculate the average titration volume.
- Calculate the mass of anhydrous sodium carbonate, Na_2CO_3, in 100 g of washing soda crystals using the formula

  $$\%\ w/w\ Na_2CO_3 = \frac{0.53 \times V}{m}$$

  where,
  m is the mass of washing soda crystals in grams.

8. Expression of results

Give the composition of the washing soda crystals as a % w/w, in other words the mass of anhydrous sodium carbonate in 100 g of washing soda crystals.

Analysis of Calcium Carbonate Tablets

Indigestion is caused by excess acid in the stomach. Antacids are substances that neutralise acids by chemical reaction and help to relieve the symptoms. Active ingredients in antacid medicines include magnesium hydroxide, aluminium hydroxide, magnesium carbonate, calcium carbonate and sodium hydrogencarbonate.

1. Scope

This procedure may be used for any antacid tablets where the only active ingredient is calcium carbonate. Tablets usually contain 0.5 g calcium carbonate.

2. Principle

Carbonates react with acids to give a salt, water and carbon dioxide, for example

$MgCO_3(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2O(l) + CO_2(g)$

Soluble carbonates (e.g. sodium carbonate) may be dissolved in distilled water and titrated against a standard acid solution using a suitable indicator. Insoluble carbonates (e.g. magnesium carbonate, calcium carbonate) are not so straightforward. One method is to dissolve the insoluble carbonate in an excess of a suitable acid and determine the amount of acid remaining by titration with a suitable alkali. This is called a back-titration.

3. Reagents

- 1.00 mol dm^{-3} hydrochloric acid
- 0.50 mol dm^{-3} sodium hydroxide solution (**IRRITANT**)
- phenolphthalein indicator solution (0.2% in ethanol) (**HIGHLY FLAMMABLE**).

4. Apparatus

- Balance that can measure mass to the nearest 0.01 g or better
- 3 x 250 cm^3 conical flasks
- Tripod, gauze and Bunsen burner
- glass funnel
- 50 cm^3 burette with clamp, stand and white tile
- 25 cm^3 pipette with safety filler
- Dropping pipette.

5. Health and Safety

- Wear protective clothing and eye protection
- A risk assessment must be carried out before starting work.

6. Procedure

- Weigh one tablet and put it in a 250 cm^3 conical flask.
- Pipette 25.0 cm^3 of 1.00 mol dm^{-3} hydrochloric acid into the flask.
- Use a measuring cylinder to add 25 cm^3 of distilled water. Swirl the contents of the flask until the tablet has completely dissolved.
- Place the conical flask on a wire gauze supported by a tripod. Put a glass filter funnel in the neck of the flask.
- Heat with a Bunsen burner so that the solution boils gently for about 5 minutes. The volume of the solution should be kept roughly constant by adding distilled water.
- Allow the solution to cool. Add 2-3 drops of phenolphthalein and titrate against 0.50 mol dm^{-3} sodium hydroxide until a permanent pink tinge to the solution is seen.
- Repeat the analysis for at least one more tablet.

7. Calculations

For each tablet calculate the mass of calcium carbonate using the formula:

$$\text{mass of calcium carbonate} = \frac{(25 - 0.5V)}{20}\ \text{g}$$

where V is the average volume of 0.500 mol dm^{-3} sodium hydroxide solution required for a tablet

8. Expression of results

Report the analysis of the antacid tablets as grams of calcium carbonate per tablet.

Determination of Chloride Ions in River Water

Sodium chloride, common salt, is a common ingredient in our diets. It is barely affected during sewage treatment and its concentration in river waters indicates the amount of treated sewage in a river. In America, for example, the New York State Department of Environmental Conservation sets a limit of 250 ppm for chloride ions in river water. At this concentration the water actually tastes salty. The method here is based on one given in Standard Methods for the Examination of Water and Wastewater, 16th Edition, APHA, AWWA, WPCF.

1. Scope

Sodium chloride concentrations in the range 1 - 3 g dm^{-3} may be determined.

2. Principle

The concentration of a sodium chloride solution can be determined by a precipitation titration using silver nitrate solution and potassium chromate(VI) as the indicator. This is called the Mohr method.

Sodium chloride solution reacts with silver nitrate solution to give a precipitate of silver chloride.

$AgNO_3$ (aq) + NaCl (aq) $\rightarrow$ AgCl (aq) + $NaNO_3$ (aq)
When all the sodium chloride has reacted, further silver nitrate reacts with potassium chromate(VI) to give a red precipitate of silver chromate(VI). The end point is shown by the first permanent appearance of a pinkish yellow colour.

3. Reagents

- sodium chloride
- 0.01 mol dm^{-3} silver nitrate solution
- 5% potassium chromate(VI) indicator (**TOXIC**).

4. Apparatus

- balance that can measure mass to the nearest 0.01 g or better
- several 250 cm^3 conical flasks
- 100 cm^3 measuring cylinders
- 100 cm^3 pipette with safety filler
- 50 cm^3 burette with clamp, stand and white tile
- dropping pipette

5. Health and Safety

- You must always wear protective clothing and eye protection in the laboratory.
- A risk assessment must be carried out before you start work.

6. Procedure

Standardisation of silver nitrate solution

- Weigh accurately about 0.2 g of sodium chloride.
- Transfer it quantitatively and dissolve in approximately 100 cm^3 of distilled water in a 250 cm^3 conical flask.
- Measure the pH of this solution using a pH meter. Adjust the pH to be between 7 and 10 by adding 1 mol dm^{-3} sulfuric acid (**CORROSIVE**) or 1 mol dm^{-3} sodium hydroxide (**IRRITANT**) if necessary.
- Add 1.0 cm^3 of potassium chromate(VI) indicator.
- Titrate this solution with silver nitrate solution (approx. 0.01 mol dm^{-3}) until a permanent orange pink colour forms.
- Repeat with further 0.2 g samples of sodium chloride until consistent results are obtained.

Analysis of river water

- If the river water is cloudy filter it through a sintered glass crucible under reduced pressure.
- Pipette 100 cm^3 of river water into a 250 cm^3 conical flask and adjust its pH to between 7 and 10 (see standardisation of silver nitrate solution).
- Add 1.0 cm^3 of potassium chromate(VI) indicator.
- Titrate this solution with the standardised silver nitrate solution until a permanent orange pink colour forms.
- Repeat with further 100 cm^3 samples of river water until consistent results are obtained.

7. Calculations

Standardisation of silver nitrate solution

- Calculate the concentration of silver nitrate solution using the formula

  concentration of silver nitrate solution (mol dm^{-3}) = $\frac{1000m}{58.5t}$

  where m = mass of sodium chloride (in grams) and t = volume of silver nitrate used in the titration

  Repeat the calculation for each sample of sodium chloride used and average the results

Analysis of river water

- Calculate the average titration for 100 cm^3 of river water with the standardised silver nitrate solution.
- Calculate the concentration of sodium chloride in 1 dm^3 of river water sample using the formula

  concentration of sodium chloride (mol dm^{-3})
  = average titration (cm^3) x 0.585

8. Expression of results

Give the concentration of sodium chloride in river water in g dm^{-3}.

Determination of Hydrogen Peroxide Concentration by Manganate(VII) Titration

This Standard Procedure is based on a product datasheet produced by US Peroxide, a company that markets solutions of hydrogen peroxide.

1. Scope

This method is suitable for determining the concentration of hydrogen peroxide in aqueous solutions, ranging from 0.25 to 10 grams per 100 cm^3 of solution.

2. Principle

Potassium manganate(VII) ($KMnO_4$) is reduced by hydrogen peroxide in sulfuric acid. The reaction mixture effervesces as oxygen gas is given off. The characteristic pink/purple colour of potassium manganate(VII) disappears and a colourless solution forms.

Using a potassium manganate(VII) solution of known concentration, this reaction is used for the determine the concentration of hydrogen peroxide in solution.

potassium manganate(VII) + sulfuric acid + hydrogen peroxide → potassium sulfate + manganese(II) sulfate + water + oxygen

3. Reagents

- 1 mol dm^{-3} sulfuric acid (**IRRITANT**)
- 0.0200 mol dm^{-3} potassium manganate(VII) solution (**LOW HAZARD**)
- hydrogen peroxide solution
  Solutions greater than about 20% w/v are **CORROSIVE**. Solutions between 5% and 20% w/v are less corrosive, but they must still be handled with care. They are an **IRRITANT**.

4. Apparatus

- Balance that measures mass to the nearest 0.01 g or better
- Weighing bottle and lid
- 250 cm^3 volumetric flask
- 25 cm^3 pipette and safety filler
- 250 cm^3 conical flask
- 50 cm^3 burette with stand, clamp and white tile.

5. Health and Safety

- Wear protective clothing and eye protection.
- A risk assessment must be carried out before starting work.

6. Procedure

- Using the table below, decide roughly what mass of hydrogen peroxide solution to take.

Expected hydrogen peroxide concentration (grams per 100 cm^3 of solution)	Suggested sample amount (grams)
0.25-1.0	30-80
1-3	10-30
3-10	3-10

- Weigh a weighing bottle with a loose-fitting aluminium cap as its lid (do not use a tight fitting stopper).
- Put the approximate chosen quantity of hydrogen peroxide into the weighing bottle. Replace the aluminium cap and weigh again.
- Carefully wash the weighed sample into a 250 cm^3 volumetric flask with distilled water, dilute to the mark and mix thoroughly.
- Pipette 25 cm^3 of the diluted solution of hydrogen peroxide into a 250 cm^3 conical flask containing 100 cm^3 of 1 mol dm^{-3} sulfuric acid.
- Titrate against 0.0200 mol dm^{-3} potassium manganate(VII) solution until the pink colour persists for 15-20 seconds.
- Repeat with further 25 cm^3 portions of the diluted hydrogen peroxide solution.

7. Calculations

- Calculate the value of the average titration.
- Calculate the mass of hydrogen peroxide in the original sample (before it was diluted) using the formula

  percentage by mass of hydrogen peroxide in 100 cm^3 of solution = $\frac{0.0171 \times t}{m} \times 100$

  where,

  t = the average titration of 25 cm^3 of the diluted hydrogen peroxide solution against 0.0200 mol dm^{-3} potassium manganate(VII) solution

  m = mass of original hydrogen peroxide solution taken for analysis

8. Expression of results

Give the hydrogen peroxide concentration in grams per 100 cm^3 of solution.