

CIE Chemistry A-Level
Practicals for Papers 3 and 5
Organic Analysis Practical



Chromate VI and Dichromate VI

Note:

- Chromate VI is CrO_4^{2-} . Dichromate VI is $\text{Cr}_2\text{O}_7^{2-}$.
- K_2CrO_4 is **yellow**. $\text{K}_2\text{Cr}_2\text{O}_7$ is **orange**.

Test for an alcohol: using potassium dichromate (or chromate)

1. To distinguish between different types of alcohols, add **acidified** potassium dichromate to the solution.
2. Primary and secondary alcohols will be reduced from orange dichromate (VI) ions to **green** chromium (III) ions. (Chromate (VI) also goes green.)
3. It will remain orange if a **tertiary** alcohol is present.

Reaction tips

- If no colour change occurs, try **gently** heating the mixture. However, don't heat excessively as it may evaporate the alcohol or other organic liquid. Heating should cause a rapid colour change.
- Also, use a **small** mass of chromate/dichromate because if used in excess, the colour will remain yellow/orange from the remaining unoxidised chromate/dichromate will mask any green colour from the alcohol present. A small amount ensures **all** of the chromate/dichromate will react if the alcohol is present.

Test for an alcohol: using metal

1. Add a small piece of metallic **sodium** to alcohol.
2. Produces **hydrogen** gas which is shown by 'squeaky pop' test.
3. Dispose any excess sodium **safely** using the beaker of ethanol provided. (Sodium will completely react with the excess ethanol in order to be safely washed away because if any water comes into contact with the sodium there is a **serious fire risk**).

Test for an aldehyde: Fehling's solution

1. In a clean test tube mix together equal volumes of **Fehling's solution A** (blue aqueous solution of copper (II) sulfate pentahydrate crystals) and Fehling's solution B (clear solution of aqueous potassium sodium tartrate and a strong alkali). The resultant Fehling's test reagent should be a clear **dark blue solution**.
2. Add 5 drops of this test reagent to a test tube along with a few **anti-bumping granules**, and then add the aldehyde.
3. **Warm gently** for around two minutes in a beaker of hot water, gradually bring the beaker of water to boiling and maintain this temperature for a few minutes.
4. Carefully lift the test tube out of the boiling water and allow its contents to stand for several minutes.



5. A **brick red precipitate** formed if an aldehyde is present.

Test for an alkene: bromine water

1. Add about 1 cm³ of bromine water to 2 drops alkene.
2. Shake the contents of the tube vigorously from side to side.
3. Bromine water **decolourised from orange** if an alkene is present.

Test for a carboxylic acid: sodium carbonate

1. Place spatula of solid **sodium carbonate** in a test tube and add about 2 cm³ of dilute ethanoic acid using a pipette.
2. Collect the gas produced and bubble through limewater (calcium hydroxide). It will turn cloudy if a carboxylic acid is present as **CO₂** is produced. (Phenols are also acidic but are not sufficiently acidic to react with carbonates.)

Test for a halogenoalkane: sodium hydroxide and silver nitrate

1. Using a test pipette, add 5 drops of 1-bromobutane to about 1cm³ of **sodium hydroxide** solution in a test tube. (OH⁻ ions replace the Br by **nucleophilic substitution**).
2. **Warm** the contents of the test tube for a few minutes in a beaker filled with hot water at approximately 60°C.
3. Acidify the contents of the test tube by adding 2cm³ of **dilute nitric acid** and then add about 1cm³ of silver nitrate solution. (Nitric acid removes carbonate and hydroxide impurities which would form **precipitates**. Silver Bromide precipitate then forms).

Thin layer chromatography

Method	Accuracy	Explanation
1) Wearing gloves carefully use a pencil to draw a faint line 1 cm above the bottom of a TLC plate and mark five spots, equally spaced, along this line.	<ul style="list-style-type: none"> • Gloves • Pencil line 	<ul style="list-style-type: none"> • Gloves prevent contamination from the hands to the plate • Pencil line –will not dissolve in the solvent
2) Use a capillary tube to apply a tiny drop of each solution to a different origin spot and allow the plate to air dry . If required repeat this process to achieve small but concentrated spots	Tiny drop	Too big a drop will cause different spots to merge



3) Add approximately 10 cm ³ of solvent to a development chamber (or suitable container with a lid)	Depth of solvent	If the solvent is too deep it will dissolve the sample spots from the plate.
4) Place the TLC plate into the development chamber , making sure that the level of the solvent is below the spotting line. Replace the lid and make sure it is a tight seal.	Lid	To prevent evaporation of toxic solvent and so that the inside of the tank is saturated with the solvent vapour.
5) When the level of the solvent reaches about 1 cm from the top of the plate, remove the plate and mark the solvent front with a pencil. Allow the plate to dry in the fume cupboard.	<ul style="list-style-type: none"> Allow solvent line to rise near to the top of the plate Dry in fume cupboard 	<ul style="list-style-type: none"> Will get more accurate results if the solvent is allowed to rise to near the top of the plate but the R_f value can be calculated if the solvent front does not reach the top of the plate Dry in a fume cupboard as the solvent is toxic
6) Place the plate under a UV lamp in order to visualise the spots. Draw around them lightly in pencil.	UV lamp	UV lamp used if the spots are colourless and not visible.
7) Calculate the R _f values of the observed spots.	Use mm ruler	Higher resolution - more precise R_f value calculated

N.B

- If you use less solvent and have a high baseline you will get large spots
- If your sample is too concentrated then your spots overlap

Melting Points

Melting point of synthesised product:

- If the sample is very pure then the melting point will be **sharp** (same value as quoted in data books).
- If impurities are present (and this can include solvent from the recrystallization process) the melting point will be **lowered** and the sample will **melt over a range** of several degrees.
- Can be measured in an electronic melting point machine or by using a practical set up where the capillary tube is strapped to a thermometer immersed in some heating oil. In both cases a small amount of the salt is put into a capillary tube. The tube is heated up and is **heated slowly when near the melting point**.
- Compare** experimentally determined melting point value with one quoted in a data source to determine purity.
- Error may occur if the temperature on the thermometer is not the same as the temperature in the actual sample tube.



Measuring boiling point of synthesised liquid:

- This can be done in a distillation set up or by simply boiling a tube of the sample in a heating oil bath.
- **Pressure** should be noted- changing pressure can change the boiling point of a liquid.
- Not the most accurate method of identifying a substance as several substances may have the same boiling point.
- To get a correct measure of boiling point the thermometer should be **above** the level of the surface of the boiling liquid and be **measuring the temperature of the saturated vapour**.

