WJEC (Eduqas) Chemistry A-level

SP OA4d - Planning a Series of Tests to Identify Organic Compounds

Methods and images taken from the WJEC practical handbook

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Aim

Plan and carry out a **sequence of tests** to **identify** organic compounds from a given list.

Apparatus and Chemicals

- Deionised water
- Access to disposable gloves (or suitable long-term use gloves such as nitriles)
- Test tubes with stoppers
- Boiling tubes
- Small beakers
- Test tube racks
- Red and blue litmus or similar indicator papers
- 2.0 mol dm^{-3} NaOH solution
- 1.0 mol dm^{-3} H_2SO_4 solution
- $NaHCO_3$
- 2,4-DNPH
- Materials for preparing Tollens' reagent
- Materials for preparing the reagent for the iodoform test
- $NaNO_2$

Safety Considerations

★ 2.0 mol dm^{-3} NaOH solution - irritant
★ 1.0 mol dm^{-3} H_2SO_4 solution - irritant
★ 0.1 mol dm^{-3} NH_3 solution - flammable, toxic
★ 0.1 mol dm^{-3} $AgNO_3$ solution - irritant
★ $NaNO_2$ - harmful
★ 2,4-DNPH - oxidising, toxic, dangerous to the environment
★ $CH_3CH_2CH_2CH_2NH_2$ - flammable, harmful
★ C_6H_5CHO - flammable, harmful
★ $CH_3COCH_2CH_3$ - flammable, irritant
★ $C_6H_5COC_6H_5$ - irritant
★ $C_6H_5COOC_2H_5$ - irritant
★ CH_3CONH_2 - carcinogenic
★ C_6H_5CN - harmful, irritant

Planning

1. Complete a **written method** detailing how you will **identify** these chemicals in the **fewest possible steps**. This method should include a diagram showing the sequence of tests and the outcomes for each sequence.
2. Complete a full **risk assessment** for this investigation.

General method

1. You are provided with a sample of **6 unknown organic chemicals**. These chemicals may be any six from the following eight chemicals:

    - 1-aminobutane ($CH_3CH_2CH_2CH_2NH_2$)
    - Benzenecarbaldehyde (C_6H_5CHO)
    - Benzenecarboxylic acid (C_6H_5COOH)
    - Diphenylmethanone ($C_6H_5COC_6H_5$)
    - Butanone ($CH_3COCH_2CH_3$)
    - Ethanamide (CH_3CONH_2)
    - Ethylbenzenecarboxylate ($C_6H_5COOC_2H_5$)
    - Benzenecarbonitrile (C_6H_5CN)

2. You are also provided with the **apparatus** and **chemicals** listed on the previous page.

Method

1) Test for aldehydes and ketones
   Add **2,4-DNPH** (Brady's reagent) to the 6 unknown chemicals. If a bright **orange precipitate** forms then it is a positive result (reacts with the C=O of an aldehyde or ketone).

2) Test for aldehydes
   To identify if any of the previous positive results are an aldehyde (**benzenecarbaldehyde),** add **Tollens reagent**. Tollens' reagent is a solution of silver nitrate ($AgNO_3$) and ammonia (NH_3). A **silver mirror** will form in a positive result.

3) Test for methyl Ketones
The samples that were positive for 2,4-DNPH but negative for aldehyde will be tested for a methyl ketone which has the formula R-CO-CH_3 (**butanone)**.
Warm the sample with **iodine** and **sodium hydroxide**. A positive result is indicated by a **yellow precipitate** and an **antiseptic smell** (triiodomethane, iodoform). A negative result will be a ketone that is not a methyl ketone (**diphenylmethanone).**

4) Test for acids
When **blue litmus** paper is exposed to an acid, it will turn **red**. Test all the samples that gave negative results in Test 1 with blue litmus paper. A positive result will indicate an acid (**benzenecarboxylic acid).**

5) Test for carboxylic acids
When a **carbonate** is added to a carboxylic acid, **effervescence** occurs due to the production of CO_2. Use this to confirm the results of Test 4 by adding $NaHCO_3$ to the sample which turned blue litmus paper red. If a positive result occurs (effervescence) then Test 4 is correct.

6) Test for bases
Red litmus paper will turn **blue** when exposed to a base. Test all the unknown samples remaining with red litmus paper. A positive result will be basic like an amine (**1-aminobutane)**.

7) Test for amides
Carry out **alkaline hydrolysis**. Heat the samples gently with sodium hydroxide. If the sample is an amide, it will produce **ammonia gas**. Test the vapour produced using damp **red litmus** paper. If ammonia is present, the litmus paper will turn blue - identifying the sample as an amide (**ethanamide)**.

8) Test for esters
Use **alkaline hydrolysis**. Heat the samples with sodium hydroxide. If the sample is an ester, it will produce a **sodium carboxylate salt**. Add dilute sulfuric acid to produce a carboxylic acid. Test this with blue litmus paper - the acid will cause it to turn red. This result will confirm that the original sample was an ester **(ethylbenzenecarboxylate)**. If it does not have this positive result the final sample is **benzenecarbonitrile.** You can also confirm if a carboxylic acid is present by using the same method as Test 5.

All of the 6 samples should now be identified.