**Name: ..... ( ) CT Group: ..... Date: .....**

**Raffles Institution**
**Year 6 H2 Chemistry 2014**
**Experiment 15: Organic Chemistry QA**

**Points to take note for Organic QA**

- You may be given three to five organic compounds and tasked to plan an experiment involving a sequence of simple chemical tests to identify these compounds.

- The unknown organic compounds given are either liquids or solids at room temperature.

| State at room temperature | Possible classes of organic compounds | **Note:** For simplicity, always write the following statement before writing the procedure for each test:<br><br>*Dissolve 0.5 g of any given unknown solid organic compound (e.g. amide and ammonium carboxylate) in 10 $cm^3$ of distilled water in a beaker. Use the resultant solution to carry out chemical tests.* |
|---|---|---|
| liquid | alkenes, halogenoalkanes, alcohols, aldehydes, ketones, carboxylic acids, acyl chlorides, esters, amines | |
| solid | phenols, aromatic carboxylic acids, amides, ammonium carboxylates, amino acids | |

- For simplicity,
  - if an unknown **liquid** organic compound is given, **1 $cm^3$** of it is to be used for each test.
  - if an unknown **solid** organic compound is given, **0.5 g** of it is to be dissolved in 10 $cm^3$ of distilled water in a beaker and **1 $cm^3$ of the resultant solution** is to be used for each test.

- Frequently, the **volume of reagent** to be added to the unknown sample is **2 $cm^3$**. However, for tests in which a colour change is expected to be observed when a reagent is added to the unknown, only **2 to 3 drops (and not excess)** should be used. Such reagents include $Br_2$(aq), acidified $KMnO_4$(aq), and neutral $FeCl_3$(aq).

- Substances that require hydrolysis (e.g. halogenoalkanes, esters and amides) should be heated in a water bath (i.e. **a beaker of hot water placed on a hot plate**) for at least 5 minutes. These tests should preferably be done last. No naked flames should be used as most organic compounds are flammable.

- Present the procedure in a tabulated form with three headings as shown below.

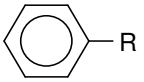
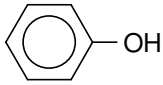
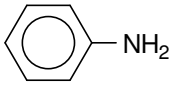
| **Procedure for each test** | **Expected observations** | **Compound identified** |
|---|---|---|
| **Test (1)**<br>Describe clearly the procedure for each test (i.e. how each test is to be carried out) by specifying the following:<br><br>1. the appropriate apparatus (e.g. test-tube, dropper, etc.)<br>2. the quantity of materials (e.g. 1 $cm^3$, 2 drops, etc.)<br>3. the reagents (e.g. $Br_2(aq)$, NaOH(aq), etc.)<br>the conditions (e.g. warm gently, heat strongly, etc.) | State the positive observation and the organic compound which gives this positive observation. | State the compound identified. |
| **Test (2)** | - - - | |
| **Test (3)** | - - - | |
| **Test (4)** | | |

**Safety considerations**

| **Safety precautions** | **Reasons** |
|---|---|
| **1** Use a water bath (e.g. a 250 $cm^3$ beaker half-filled with water placed on a hot plate) whenever heating of a reaction mixture is required. Avoid the use of any naked flame for heating. | **1** This is to prevent any fire from occurring since many organic compounds are volatile and highly flammable. |
| **2** Stopper all bottles containing the organic compounds and reagents when they are not in use and clear up any accidental spillage of chemicals immediately. | **2** This to prevent any poisonous fumes from volatile organic compounds escaping into the air and bringing harm to anyone who inhales the fumes. |
| **3** Ensure that the lab is well ventilated by opening the windows and turning on the fans. | **3** This is to allow rapid dispersal of any fumes from volatile organic compounds. |
| **4** Wear a lab coat and wear suitable safety gloves. Use droppers to transfer chemicals to test tubes. | **4** This is to avoid direct contact with chemicals as they may be toxic or corrosive. |
| **5** Wear safety goggles at all times in the lab when carrying out the tests. | **5** This is to protect the eyes from chemicals or fumes. |

## Common Chemical Tests

- Suggested strategy for the planning task:
    - Use simple specific distinguishing tests first, if possible, e.g. use 2,4-dinitrophenylhydrazine to identify any given aldehyde or ketone.
    - Distinguishing tests for **halogenoalkanes, amides** and **esters** which require hydrolysis and strong heating should preferably be done last.

| | **Functional Group / Class of Organic Compounds** | ***Formula/Part formula*** | **Suggested chemical test** |
|---|---|---|---|
| **1** | alkene | C=C | $Br_2$(aq) |
| **2** | alkylbenzene |  | hot, acidified $KMnO_4$(aq) |
| **3** | halogenoalkane | R–X | "The 4-step test" |
| **4** | alcohol ($1^o$ or $2^o$ or $3^o$) | R–OH | sodium |
| | alcohol ($1^o$ or $2^o$) | R–OH | hot, acidified $KMnO_4$(aq)<br>or hot, acidified $K_2Cr_2O_7$(aq) |
| | alcohol of the type $CH_3CH(OH)R$<br>(and no ketone of the type $CH_3COR$)<br>where R is H, alkyl group or aryl group | $CH_3CH(OH)R$ | alkaline aqueous iodine, warm |
| **5** | phenol |  | neutral $FeCl_3$(aq) |
| **6** | aldehyde (and no ketone is given) | RCHO | 2,4-DNPH |
| | aldehyde (and ketone is also given) | RCHO | Tollens' reagent, warm |
| | aliphatic aldehyde (and ketone is also given) | RCHO | Fehling's solution, warm |
| **7** | ketone (and no aldehyde is given) | RCOR' | 2,4-DNPH |
| | ketone of the type $CH_3COR$<br>(and no alcohol of the type $CH_3CH(OH)R$)<br>where R is H, alkyl group or aryl group | $CH_3COR$ | alkaline aqueous iodine, warm |
| **8** | carboxylic acid | RCOOH | $Na_2CO_3$(aq) |
| **9** | acyl halide | RCOX | $AgNO_3$(aq) |
| **10** | ester | $RCO_2R'$ | hot, acidified $KMnO_4$(aq) |
| **11** | phenylamine |  | $Br_2$(aq) |
| **12** | primary amide | $RCONH_2$ | NaOH(aq), heat strongly |
| **13** | ammonium carboxylate | $RCOO^-NH_4^+$ | NaOH(aq), warm gently |
| **14** | etc. | | |

**Part 1: Distinguishing Tests**

You are given four organic compounds: ethanol, propanal, benzaldehyde and propanone. Answer the following questions in the spaces provided.

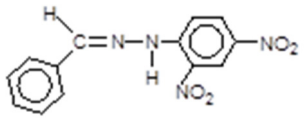
**(A) Oxidation Reactions**

| Test | Questions |
|---|---|
| **(a)** Add 1 $cm^3$ of aq $KMnO_4$ to 2 $cm^3$ of dil $H_2SO_4$.<br><br>Add one drop acidified $KMnO_4$ to 1 $cm^3$ of the organic compound in a test-tube. Shake well.<br><br>If no reaction occurs, carefully heat the mixture by placing the test-tube in a beaker of hot water.<br><br>[Perform this test with ethanol.] | • State the positive observation of the test.<br><br>Purple aq $KMnO_4$ decolourises<br><br>• Which of the four organic compounds would give a positive result? *You may select more than one compound*.<br><br>$CH_3CH_2OH$, $CH_3CHO$, $C_6H_5CHO$<br><br>• Using [O] to represent oxygen atom, write a balanced equation for the oxidation of ethanol in this test.<br><br>$CH_3CH_2OH + [O] \rightarrow CH_3CHO + H_2O$<br>OR<br>$CH_3CH_2OH + 2[O] \rightarrow CH_3COOH + H_2O$ |

| | |
|---|---|
| **(b)** Place 3 drops of the organic compound in a test-tube. Add 3 $cm^3$ of $I_2$, then add NaOH carefully until the colour of iodine has almost discharged. Warm in a water bath.<br><br>[Perform this test with propanone.] | • State the positive observation of the test.<br><br>Yellow ppt<br><br>• State the species responsible for the positive observation of the test.<br><br>$CHI_3$<br><br>• What structural unit must be present in the above sample to obtain a positive result?<br><br>$-CH(OH)CH_3$ OR $-COCH_3$<br><br>• Which of the four organic compounds would give a positive result? *You may select more than one compound*.<br><br>$CH_3CHO$, $CH_3COCH_3$ and $CH_3CH_2OH$ |
| **(c)** **<u>Preparation of Tollens' reagent</u>**<br>Add a few drops of NaOH to 1 $cm^3$ of $AgNO_3(aq)$. Then add aq $NH_3$ until the brown precipitate just dissolves.<br><br>Add 1 $cm^3$ of the organic compound to 2 $cm^3$ of Tollens' reagent in a clean test-tube (the test-tube should preferably be cleaned with hot nitric acid). If no reaction occurs, carefully heat the mixture by placing the test-tube in a beaker of hot water.<br><br>**Caution:** After the test, pour the contents of the test-tube into the waste bottle and wash the test-tube with dilute nitric acid. Any fulminating silver (or $Ag_3N$), which is highly explosive when dry, will thus be destroyed.<br><br>[Perform this test with benzaldehyde.]<br>Note: Benzaldehyde is found in the fumehood. | • State the positive observation of the test.<br><br>Ag mirror formed<br><br>• What functional group would give a positive result?<br><br>Aldehyde group<br><br>• Which of the four organic compounds would give a positive result? You may select more than one compound.<br><br>$CH_3CHO$, $C_6H_5CHO$ |

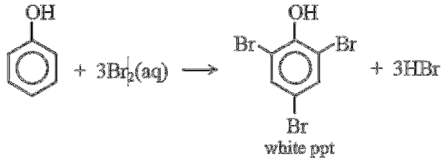
| | |
|---|---|
| **(d)** Add 1 $cm^3$ of Fehling's solution to 1 $cm^3$ of the organic compound in a test-tube. Shake well.<br><br>If no reaction occurs, carefully heat the mixture by placing the test-tube in a beaker of hot water.<br><br>[Perform this test with propanal.] | • State the positive observation of the test.<br><br>Reddish-brown ppt<br><br>• State the species responsible for the positive observation of the test.<br><br>$Cu_2O$<br><br>• What functional group would give a positive result?<br><br>Aliphatic aldehyde group<br><br>• Which of the four organic compounds give a positive result?<br><br>$CH_3CHO$ |

**(B) Condensation Reaction**

| Test | Questions |
|---|---|
| **(e)** Add 1 $cm^3$ of 2,4-dinitrophenylhydrazine (Brady's reagent) to 3 drops of the organic compound in a test-tube.<br><br>[Perform this test with benzaldehyde.] | • Write the structural formula of the product formed between benzaldehyde and 2,4-dinitrophenyl hydrazine.<br><br>  |

Use the phenol provided for the tests in part (C).

**(C) Distinguishing Tests for Phenol (Note: Teacher demonstration only)**

| Test | Questions |
|---|---|
| **(f)** Add neutral iron(III) chloride solution dropwise to 1 $cm^3$ of the organic compound in a test-tube. Shake well.<br><br>[Perform this test with phenol.] | • State the positive observation of the test.<br><br>Purple colouration observed |
| **(g)** Add bromine water drop by drop to 1 $cm^3$ of the organic compound in a test-tube. Shake well.<br><br>[Perform this test with phenol.] | • Write a balanced equation for the reaction between phenol and bromine water.<br><br>  <br>white ppt |

**Part 2: Qualitative Analysis**

You are provided with samples **FA 4**, **FA 5**, **FA 6** and **FA 7**, each of which is an aqueous solution containing a different one of the following:

an alcohol, an aldehyde, a ketone group and a carboxylic acid.

Each compound contains a maximum of 4 carbon atoms. Two of the four compounds also contain the alkene functional group.

Perform the test-tube experiments described below and record your observations in the table. Test for any gases evolved. **Use a fresh sample of each solution in each test.**

| | Test | Observations with **FA 4** | Observations with **FA 5** | Observations with **FA 6** | Observations with **FA 7** |
|---|---|---|---|---|---|
| **(a)(i)** | Place about 1 cm depth of **FA 4** in a test-tube.<br><br>To this test-tube, add about 1 cm depth of 2,4-dinitrophenylhydrazine.<br><br>**Repeat using FA 5, FA 6 and FA 7.** | No orange ppt. | Orange ppt. | No orange ppt. | Orange ppt. |
| **(a)(ii)** | Place about 1 cm depth of **FA 4** in a test-tube.<br><br>To this test-tube, add a small amount of magnesium powder (from teacher's bench).<br><br>**Repeat using FA 5, FA 6 and FA 7.** | No change. No effervescence. | No change. No effervescence. | Effervescence of $H_2$ gas. Lighted splint extinguished with a "pop" sound | No change. No effervescence. |
| **(a)(iii)** | Place about 1 cm depth of **FA 4** in a test-tube.<br><br>To this test-tube, add 6 drops of sodium hydroxide solution, then add iodine solution, drop by drop, until a permanent brown colour is present.<br><br>Warm the mixture in the water-bath for two minutes.<br><br>Add sodium hydroxide solution dropwise until the colour of the solution | Yellow ppt. of $CHI_3$. | No yellow ppt. | No yellow ppt. | Yellow ppt. of $CHI_3$. |

| | | | | | |
|---|---|---|---|---|---|
| | fades.<br><br>**Repeat using FA 5, FA 6 and FA 7.** | | | | |
| **(a)(iv)** | Place about 1 cm depth of **FA 4** in a test-tube.<br><br>To this test-tube, add $Br_2$(aq) dropwise.<br><br>**Repeat using FA 5, FA 6 and FA 7.** | No decolourisation of brown $Br_2$(aq). | No decolourisation of brown $Br_2$(aq). | Immediate decolourisation of brown $Br_2$(aq). | Immediate decolourisation of brown $Br_2$(aq). |

| | Test | Observations with **FA 4** | Observations with **FA 5** | Observations with **FA 6** | Observations with **FA 7** |
|---|---|---|---|---|---|
| Place about 2 cm depth of aqueous silver nitrate in a boiling tube and add to it a few drops of aqueous sodium hydroxide. Then, carefully add aqueous ammonia until the brown precipitate just dissolves. Use this solution in part **(a)(v).** | | | | | |
| **(a)(v)** | Place about 1 cm depth of this solution in a test-tube.<br><br>To this test-tube, add about 1 cm depth of **FA 4**.<br><br>Shake and warm the test-tube in a water-bath.<br><br>**Repeat using FA 5, FA 6 and FA 7.** | No silver mirror.<br>No grey ppt. | Grey ppt. /Silver mirror formed. | No silver mirror.<br>No grey ppt. | No grey ppt. |

**(b) (i)** From your observations, identify, and give evidence for, the type of compound present in **FA 4**, **FA 5**, **FA 6** and **FA 7**.

| | Compound type | Evidence |
|---|---|---|
| **FA 4** | alcohol | **FA4** gives a **negative carbonyl test in (a)(i)** and a **positive iodoform test in (a)(iii)**. Hence **FA4** contains the **$CH_3CH(OH)$– structure** and is an alcohol. |
| **FA 5** | aldehyde | **FA5** gives a **positive carbonyl test in (a)(i)** and gives a **positive Tollens' test in (a)(v).** |
| **FA 6** | carboxylic acid and alkene | **FA6** gives a **positive acid-metal test in (a)(ii) with effervescence of $H_2$ gas**.<br><br>**FA6** also contains C=C bond since it gives a positive aqueous bromine test in **(a)(iv).** |
| **FA 7** | ketone and alkene | **FA7** gives a **positive carbonyl test in (a)(i)** but a **negative Tollens' test in (a)(v).**<br>**FA7** also contains C=C bond since it gives a positive aqueous bromine test in **(a)(iv).** |

**(ii)** From your observations and the fact that **FA 4** can be oxidised to a carboxylic acid, deduce and explain the identity of **FA 4.**

**FA 4** ethanol

Explanation
**FA4** is a primary alcohol since it can be oxidised to a carboxylic acid
**FA4** gives a positive iodoform test, indicating that it contains the $CH_3CH(OH)–$ structure.

Since the **only primary alcohol that gives a positive iodoform test** is ethanol, **FA4** is ethanol.

**(iii)** From your observations, suggest a suitable identity each for **FA 5**, **FA 6** and **FA 7**.

| FA 5 | FA 6 | FA 7 |
|---|---|---|
| $CH_3CHO$ or $CH_3CH_2CHO$ or $CH_3CH_2CH_2CHO$ | $CH_2=CHCOOH$ or $CH_3CH=CHCOOH$ or others | $CH_2=CHCOCH_3$ |