National Initiative on Undergraduate Science - Teacher Development Workshop
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Homi Bhabha Centre for Science Education
Tata Institute of Fundamental Research

In collaboration with

Gogate Joglekar College, Ratnagiri

Organic Chemistry Task IA

This workshop is organized by Centre for Excellence in Science and Mathematics Education (CESME-HBCSE) and set up under the Pandit Madan Mohan Malaviya National Mission for Teachers and Teaching Programme

This experiment has been adapted from 48th International Chemistry Olympiad held in Tbilisi, Georgia in 2016 which is available in the public domain and is accessed from: http://www.icho2016.chemistry.ge/icho48_problems.php

Functional group/s in aroma related molecules: Can we use qualitative tests to identify these molecules?

List of glassware

Items	Quantity
Test tubes	16
Burner	1
Test tube holder	1
Pasteur pipettes or 2 mL insulin syringes	10
Test tube stand	1

List of Chemicals

Reagent	Quantity	Labelled	Safety
Organic unknowns	Liquid (0.5 mL) Solid (0.5 g)	**1** to **8**	
Potassium permanganate 0.1% (w/v)	10 mL solution	$KMnO_4$	
2,4-dnitrophenylhydrazine	10 mL solution	2,4-DNP	
Ammonium Cerium (IV) nitrate reagent prepared in 2.0 M HNO_3	10 mL solution	Ce (IV) reagent	
0.5 mol/L ethanolic hydroxylamine hydrochloride solution	10 mL solution	$NH_2OH.HCl$	
2.5 % (w/v) Fe(III) chloride solution	10 mL solution	$FeCl_3$	
Acetonitrile	10 mL	Acetonitrile	

On the table for the common use
Ethanol, Liquid waste container, Solid waste container, Distilled Water

Hazard Symbols

Corrosive 

Flammable 

Irritant 

Carcinogenic 

Toxic 

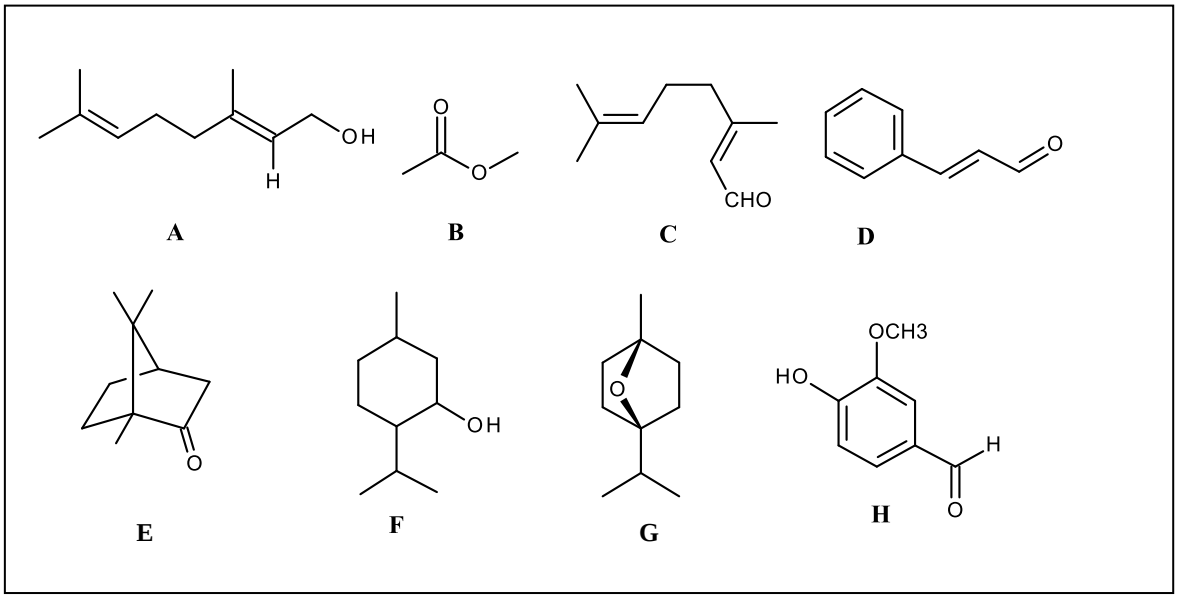
Hazard to environment 

Functional group/s in aroma related molecules: Can we use qualitative tests to identify these molecules?

Introduction

Flavours and fragrances are significant constituents of food and beverage though they may be needed in minute quantities. These compounds are characterized by smell or odour and flavours additionally affect taste. Flavours are largely naturally occurring and can be used to achieve a desired taste and flavour perception whereas fragrances are mostly synthetic and are generally not ingested. The impact of these compounds is experienced in all spheres of daily life since aromas/flavouring compounds can be found in food, spices, perfumes and essential oils. Essential oils are distilled from herbs and spices (like rose, cinnamon, rosemary, mint, eucalyptus, etc.) and the fragrant compounds are derivatives of petrochemicals or natural sources or are often synthesized in laboratory.

In the current experiment, you are given 8 compounds **(labelled 1 to 8),** that are popular 'aroma' compounds. The samples provided to you are pure and their possible structures are as follows (**A to H**). You will be performing qualitative tests to determine the functional groups present in the compounds and identify them.



To help you map the structures **A-H** to the vials labelled as **1 to 8**, you first need to understand the structural features and functional groups present in **A-H**.

Few facts about solubilities of the given samples

All the samples are soluble in common solvents like alcohol and ether and normally insoluble in dilute aqueous NaOH and HCl. However, one of the compounds is soluble in water as well as NaOH.

Brief descriptors for the qualitative tests to be used are presented below.

Qualitative Tests

Perform the following tests with all the compounds and note your observations carefully. For each test take adequate quantities**, a small pinch (if solid) or 2-3 drops (if liquid)** of the compound**.** Indicate first whether the test is positive **(+)** / negative **(–)** for a compound followed by selecting the most appropriate descriptor/s given to you from (**I to V**).

Note: for the same compound (from **A to H**) there may be **one or more** descriptors applicable.

1. $KMnO_4$ test (Baeyer test)

Dissolve a small quantity of the compound in about 1 mL of ethanol in a test tube. Add 1 drop of the given $KMnO_4$ solution and shake the mixture. Observe what happens to the permanganate colour after shaking.

2. 2,4-dintrophenylhydrazine (2,4-DNPH) test

Dissolve a small quantity of the compound in about 1 mL of ethanol in a test tube and add 1 mL of DNPH reagent to the prepared solution. Shake the mixture and let it stand for 1-2 minutes and record your observation.

3. Ferric hydroxamate test

Mix 1 mL of 0.50 M ethanolic hydroxylamine hydrochloride solution with 5 drops of 6 M NaOH solution in a glass test tube. Add a small quantity of the compound (dissolved in alcohol) and heat the mixture to boiling while gently swirling the test tube. Allow it to cool down slightly and add 2 mL of 1M HCl solution. To this add 1 drop of 2.5% Fe (III) chloride solution.

4. Cerium (IV) nitrate test

To 2 drops of the Ce (IV) reagent in a test tube, add 2 drops of acetonitrile and then a small quantity of the compound. Shake the mixture in the test tube thoroughly and observe the colour change.

The descriptors of the qualitative tests that you will use to mark are given below.

- Baeyer's test: involves decolourization of the purple colour (I)
- 2,4-dinitrophenylhydrazine test: a deep yellow / orange red precipitation (II)
- Ferric hydroxamate test- a deep red colouration (III)
- Ce (IV) nitrate test- an orange red colouration (IV)
- If there is no visible change or none of the above-mentioned descriptors are applicable then the relevant description would be (**V**)

Observations

You are required to summarize the observations of the various tests in the table below as indicated in the columns. Ensure that every cell is filled with **+ / -** / at least one Roman numeral from I to V.

Sample No.	1	2	3	4	5	6	7	8
Baeyer test results**: (+/-)**								
Descriptor used for Baeyer test observations								
2,4-DNPH test result **(+/-)**								
Descriptor used for 2,4-DNPH test observations								
Fe (III) hydroxamate test result **(+/-)**								
Descriptor used for Fe (III) hydroxamate test observations								
Ce (IV) test results (+/-)								
Descriptor used for Ce (IV) test observations								

Questions

List the appropriate compounds from **A to H**, which give positive results for

a) Baeyer test

b) 2,4-DNPH test

c) Ferric hydroxamate test

d) Ce (IV) test

e) Keeping the structures of the compounds in mind and based on your observations, what could be the function of $KMnO_4$? Use equations to represent your answer.

f) Two of the compounds are structural isomers of each other. However, they do not have the same observations for the tests performed. Examine the structures **A to H**, identify the two compounds and identify the relationship.

Structures: Relationship:

g) Two of the given compounds are α, β – unsaturated carbonyl compounds. Is there any test physical/chemical that could help you identify between these two compounds? Explain.

Based on your results for different qualitative tests, allocate the aroma samples to the proper alphabet codes.

Sample No.	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Alphabet codes of the identified compound								