

CHAPTER-20



Experiment: 20

**TO IDENTIFY THE GIVEN ORGANIC COMPOUND-2
(ETHANOL) BY SYSTEMATIC QUALITATIVE ANALYSIS**

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Chapter Id: ASU/NSP/EB/APHPC/2022/Ch-20

doi: <https://doi.org/10.52458/9789391842611.nsp2022.eb.asu.ch20>

Aim:

To identify the given organic compound-2 (Ethanol) by Systematic Qualitative Analysis

Requirements:

A. Glassware & Instruments:

1. Test tube
2. Beaker
3. Graduated pipettes
4. Buchner's flask
5. Water bath
6. Measuring cylinder
7. Thiel's tube
8. Capillary
9. Thermometer

B. Chemicals & Reagents:

1. Sodium metal
2. Sodium nitroprusside
3. Potassium antimonate solution
4. 15% w/v solution of K_2CO_3
5. Magnesium uranyl acetate solution
6. Alcohol
7. Distilled water (DW)
8. Dilute acetic acid
9. HCl
10. Ferric chloride
11. Silver nitrate solution

12. Ferrous sulphate
13. 1N hydroxylamine hydrochloride, etc.

Steps for Systematic Qualitative Analysis:

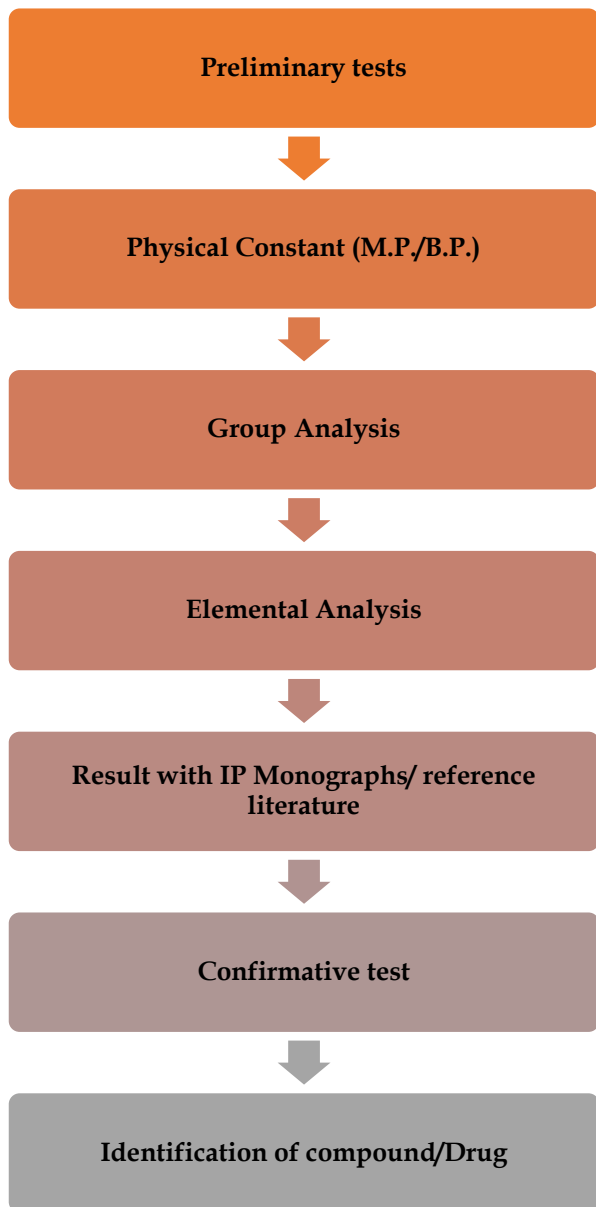


Figure 4. Steps for Systematic Qualitative Analysis

Observation Table and Conclusions:**A. Preliminary Tests:**

S. No.	Test	Observation	Inference
1	Colour		
2	Odour		
3	<p>Solubility: 0.2 ml or 4 drops of liquid /solid or 0.1 gm of solid +3 ml of the solvent.</p> <p>Shake thoroughly. If sample does not dissolve warm gently and cool to room temperature.</p> <p>a. Solubility in water</p> <p>b. Cold or hot solution, test with litmus.</p> <p>c. If acidic, add a substance to 10% sodium bi-carbonate solution</p> <p>d. If not soluble in water, then try in 2N NaOH</p> <p>e. If not soluble in 2 N NaOH, then try in dil. HCl</p>		
4	<p>Action of reagents:</p> <p>a. Action of cold NaOH: About 0.2 g/3 drops of compound + 2 ml of water + 2ml 10% NaOH and the mixture is shaken well</p> <p>b. Action of hot con. H₂SO₄: 0.1g solid or 2 drops of liquid + 1ml con. H₂SO₄, warm.</p> <p>c. Action of Na₂CO₃ solution: 10 ml of 10% Na₂CO₃ solution +0.2 g of solid or 4 drops of liquid.</p> <p>d. Action of KMnO₄ solution: 0.2 g of</p>		

	<p>solid or 4 drops of liquid + 10ml Na₂CO₃ soln + drop-by-drop KMnO₄ solution.</p> <p>e. Action of bromine water: Action of FeCl₃ soln.: Substance +water+ a drop or two of FeCl₃ soln.</p> <p>f. Heating on oxidized copper gauze (Beilstein's test)</p> <p>g. Heating in a dry test tube</p> <p>h. Heating on a clean glass rod.</p> <p>i. Heating with soda lime: Take in hard glass test tube, 0.5 g of substance + 2g of finely powdered soda lime + 1 g of coarse soda lime. (If the substance is liquid, add 5 drops of it to a fine layer of soda lime. Close the tube by a cork with a bent delivery tube, heat from top of test tube downwards gently at first and than strongly, and collect the product.)</p>		
5	Nature: Dip a wet litmus paper in the sample		

Conclusion: On the basis of the tests performed above and with reference to handbook the given organic drug/compound is

1. Aromatic/Aliphatic
2. Saturated /Unsaturated
3. Acid/Base/ /Neutral/ Phenol Halide
4.
5.

B. Determination of Physical Constant:

Conclusion: The melting point/ boiling point of the given organic compound was found to be.....°C.

C. Determination of Elements (Lassaigne's Test):

S. No.	Test	Observation	Inference
1	Test for Nitrogen: 3-4 ml filtrate + solid FeSO_4 till saturation, heat to boil for few minutes, then acidify with con. H_2SO_4		
2	Test for Sulphur: i. 2 ml filtrate + 1 drop of dilute sodium hydroxide solution + four drops of freshly prepared and very dilute solution of sodium nitroprusside. ii. 2 ml filtrate + acetic acid to acidify + few drops of lead acetate solution. iii. 2 ml filtrate + HCl to neutralize the soln+ few drops of FeCl_3 solution.		
3	Test for Halogens: i) Acidify 5 ml the filtrate with dilute H_2SO_4 boil well to reduce the volume to one third to expel H_2S if S and /or HCN if N, already found to be present. Then add few drops of dil HNO_3 (to acidify) and AgNO_3 solution. ii) Chloroform layer test Filtrate, acidify with mineral acid+ 1 ml of chloroform + few drops of fresh chlorine water, shake well and observe the colour of		

	the chloroform layer.		
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Conclusion: The given organic drug/ compound found to contain.....elements.

D. Determination of Functional Group/Groups (ForElements)

S. No.	Test	Observation	Inference
1	Cerric-ammonium nitrate Test: To 1 ml of the substance, add a few drops of ceric ammonium nitrate reagent.		
2	Sodium test: To 2 ml of the unknown, add a small piece of sodium metal		
3	Ester Formation: Heat about 0.5 ml of the sample with 1 ml of the acetic acid and a few drops of conc. H ₂ SO ₄ in a dry test tube for about a minute. Cool and pour the contents in a beaker containing water; observe the odour.		
4	Reaction with Acetyl chloride: To 1 ml of the substance add 2-3 drops of acetyl chloride. Bring a rod dipped in NH ₄ OH in contact with the vapours evolved.		
5	Lucas' test: To 1 ml of alcohol in a small test tube, add quickly 6 ml of Lucas' reagent at 26-27 °C, close the tube with cork, shake and allow it to stand. Observe the mixture.		
6	Benzoylation: Take about 500 mg of powdered 3,5 dinitrobenzoyl chloride in a dry test tube and add 2 ml of the sample. Warm the mixture in a beaker hot water until		

	a clear solution is obtained. Cool the tube.		
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Conclusion: The given organic drug/compound was found to contain.....functional group/groups.

Result:

The given organic drug/ compound was having.....m.p/b.p.....element, andfunctional group/groups, therefore with reference to literature, the organic drug/compound may be.....

E. Confirmative Test: (Specific Colour Reaction or Derivative)

S. No.	Test	Observation	Inference
1	Iodoform test: Take 5 ml of ethyl alcohol and 2 ml of 10% solution of sodium hydroxide. Add iodine solution drop by drop until slight iodine colour persists. Keep the tube in warm water bath for 15 minutes. Take out and cool		

Structural formula and Category (Student shall write it from Indian Pharmacopoeia):

Viva Questions:

- Include all organoleptic tests for organic compounds in the list.
- Which preliminary tests can be performed to determine whether the organic compound is unsaturated?
- Give one significant characteristic of aromatic and aliphatic compounds, respectively.
- Why do all aromatic compounds burn with a sooty flame?
- Specify the concentration of sulphuric acid used in the laboratory.