Part 8:
Systematic Qualitative Analysis Experiments

CHAPTER-19

Experiment: 19

TO IDENTIFY THE GIVEN ORGANIC COMPOUND-1 (UREA) BY SYSTEMATIC QUALITATIVE ANALYSIS

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Chapter Id: ASU/NSP/EB/APHPC/2022/Ch-19

doi: https://doi.org/10.52458/9789391842611.nsp2022.eb.asu.ch19

Aim:

To identify the given organic compound-1 (Urea) by Systematic Qualitative Analysis

Requirements:

A. Glassware & Instruments:

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

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

ISBN: 978-93-91842-61-1

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

Steps for Systematic Qualitative Analysis:

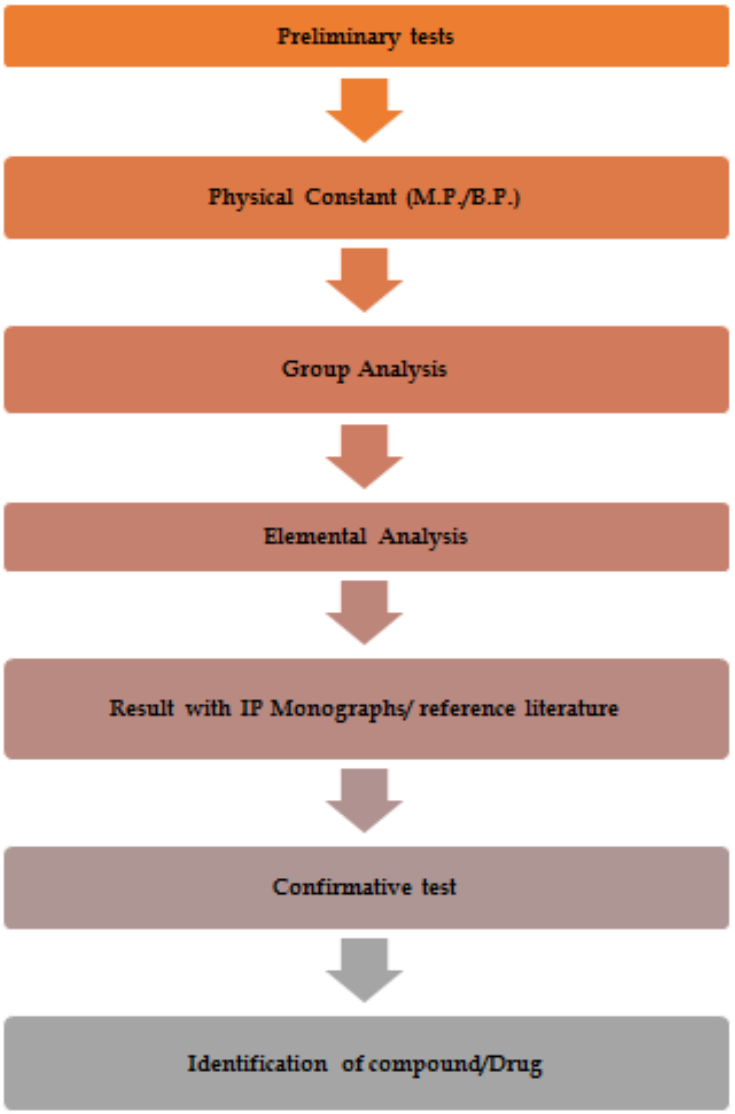

Figure 3. Steps for Systematic Qualitative Analysis

Observation Table and Conclusions:

A. Preliminary Tests:

S. No.	Test	Observation	Inference
1	**Colour**		
2	**Odour**		
3	**Solubility:** 0.2 ml or 4 drops of liquid /solid or 0.1 gm of solid +3 ml of the solvent. Shake thoroughly. If sample does not dissolve warm gently and cool to room temperature. a. Solubility in water b. Cold or hot solution, test with litmus. c. If acidic, add a substance to 10% sodium bi-carbonate solution d. If not soluble in water, then try in 2N NaOH e. If not soluble in 2 N NaOH, then try in dil. HCl		
4	**Action of reagents:** **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 **b. Action of hot con. $H2SO_4$:** 0.1g solid or 2 drops of liquid + 1ml con. H_2SO_4, warm. **c. Action of Na_2CO_3 solution:** 10 ml of 10% Na_2CO_3 solution +0.2 g of solid or 4 drops of liquid. **d. Action of $KMnO_4$ solution:** 0.2 g of solid		

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	or 4 drops of liquid + 10ml Na_2CO_3 soln + drop-by-drop $KMnO_4$ solution. **e. Action of bromine water:** Action of $FeCl_3$ soln.: Substance +water+ a drop or two of $FeCl_3$ soln. **f. Heating on oxidized copper gauze (Beilstain's test)** **g. Heating in a dry test tube** **h. Heating on a clean glass rod.** **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.)		
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 + HCI 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 laver.		

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Conclusion: The given organic drug/ compound found to contain……...elements.

D. Determination of Functional Group/Groups (For .....Elements)

S. No.	Test	Observation	Inference
1	**Reaction with NaOH solution:** Take 0.2 g of the substance in a boiling tube and add 2 ml of 10% NaOH solution. Boil the contents. Bring a glass rod dipped in conc. HCl near the mouth of the tube.		
2	**Hydroxamic Acid Test:** In a boiling tube, take 0.1 g of the substance with 2 ml of 1N hydroxylamine hydrochloride and 2 ml of 1N KOH solution. Boil the mixture for 5 minutes; cool and add 2-3 drops of ferric chloride.		
3	**Biuret test:** Heat 0.1 g of substance in a dry test tube, a sublimate of biuret with the evolution of NH_3 is formed on the sides of test tube. Dissolve this in 2 ml of distilled water; add a drop of very dilute $CuSO_4$ solution and 2 ml of NaOH solution.		
4	**Urea nitrate test:** To 0.5 g of urea, add 3-5 drops of conc. HNO_3 and heat. Cool.		

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, and ……..functional 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	**Biuret Reaction for aliphatic diamide:** Heat 0.1 g of substance in a dry test tube, a sublimate of biuret with the evolution of NH_3 is formed on the sides of test tube. Dissolve this in 2 ml of distilled water; add a drop of very dilute $CuSO_4$ solution (When aliphatic diamide is heated at a temperature above its melting point, ammonia is evolved and crystalline biuret is formed. This biuret in alkaline medium gives a violet colour with a drop of copper sulphate solution.)		

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.

ISBN: 978-93-91842-61-1