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Diploma in Pharmacy 1st Year
Pharmaceutical Chemistry
Experiment

To determine special elements in the given sample

Aim:

To determine special elements in the given sample.

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 88 - 90

Theory

Lassaingne's Test

A small piece of metallic sodium is cut and dried between filter paper before being place in a dry ignition tube. The tube is gently heated until the sodium metal solution forms a gleaming ball. The tube is then cooled, and a pinch of the specified substance (3-4 drops) is greatly heated before being dipped into chloride free 10ml distilled water to filter it. The filtrate is called as sodium fusion extract.

Table 8: Qualitative Analysis Test

S. No.	Experiments	Observations	Inferences
1)	**Test for Nitrogen:** 2mL sodium fusion extract is treated with freshly prepared saturated ferrous sulphate solution. It is then boiled and cooled. Then add dil. H_2SO_4 to this solution.	Formation of Prussian blue colour. $Na+C+N \rightarrow NaCN$ (sodium cyanide) $FeSO_4 + 2NaOH \rightarrow Fe(OH)_2 + Na_2SO_4$ $6NaCN + Fe(OH)_2 \rightarrow Na_4[Fe(CN)_6]$ (Sodium ferrocyanide) $+ 2NaOH$ $3Na_4[Fe(CN)_6] + 2Fe_2 \rightarrow (SO_4)[Fe(CN)_6]_3 + 6Na_2SO_4$ Ferric ferrocyanide (Prussian blue)	Presence of Nitrogen.
2)	**Test for Sulphur** i) To 1mL sodium fusion extract, add 2-3ml of freshly prepared sodium nitroprusside solution.	Formation of violet or purple colour (this colour may disappear gradually)	Presence of sulphur.
	ii) 2ml of extract are acidified with 2ml of acetic acid. A solution of lead acetate is added to this.	Formation of black colour ppt. $2Na + S \rightarrow Na_2S$ (Sodium sulphide)	Presence of sulphur.
		$Na_2 + Pb(CH_3COO)_2\ PbS \rightarrow 2\ CH_3COONa$ (Lead sulphide) $Na_2S + Na_2Fe(CN)_5NO \rightarrow Na_2Fe\ NOS$ (Sodiumthionitroprusside)	
3)	**Test for Halogen** **Silver Nitrate Test:** 1ml of conc. HNO_3 is added to 2-3ml of sodium fusion extract, which is then heated and cooled. Add $AgNO_3$ solution to this solution.	Formation of white ppt. which is soluble in ammonium hydroxide solution.	Presence of chlorides.
		$Na + X \rightarrow NaX$ [X = Cl, Br or I]	

Table 9: Preliminary Test

S. No.	Experiments	Observations	Inferences
1)	**Soda Lime Test:** 100mg of the substance is treated with soda lime and heated it strongly.	Ammonia gas is produced, resulting in white dense fumes with a glass rod dipped in conc. HCl.	May be an amide
3)	**Sodium Bicarbonate Test:** The small amount of the substance is treated with freshly prepared saturated sodium bicarbonate solution.	With the evolution of CO_2, a rapid effervescence occurs, turning lime water milky.	May be carboxylic acid.
4)	**Neutral Ferric Chloride Test** Substance is treated with neutral ferric chloride solution.	The solution turns violet, blue, or green in colour.	May be phenol.
		The solution turns into a buff-coloured ppt.	May be mono carboxylic acid.
5)	**Schiff's Reagent Test:** Substance is treated with Schiff's reagent.	Formation of magenta colour.	May be aldehyde.
6)	**Ceric Ammonium Nitrate Test:** Substance is treated with ceric ammonium nitrate solution.	Formation of blood red colour.	May be alcohol.
7)	**Molisch's Reagent:** Substance is dissolved in a molisch's reagent. (Or alcoholic alpha naphthol solution). And add Conc. H_2So_4 on sides of the test tube.	Formation of violet ring between the junctions of two layers.	May be carbohydrate.
8)	**Conc. H_2So_4 Test:** In a test tube, 50mg of the drug is mixed with 5ml of conc. H_2So_4.	The sample burnt into a black mass with a sugar smell.	May be carbohydrates.
		The sample is blackening and forming effervescence.	May be an aliphatic acid.
9)	**Sodium Nitro Pruside Test :** The substance is dissolved in water or alcohol then a few drops of sodium nitroprusside and 6 drops of NaOH are added.	An orange ppt. is formed.	May be ketone.

Table 10: Confirmation Test

S. No.	Experiments	Observations	Inferences
1)	**Test for Alcohol:**		
	i) **Esterification Test:** 2ml glacial acetic acid and 1ml conc. H_2SO_4 are mixed in a clean test tube and boil in a beaker containing hot water for a few minutes. The content is poured into a beaker containing cold water and then stir the solution and smell it.	A fruity odour detected.	Presence of alcohol.
	ii) **Ceric Ammonium Nitrate Test:** Substance is treated with ceric ammonium nitrate.	Formation of blood red colour.	Presence of alcohol.
2)	**Test for Amides:**		
	i) **Soda Lime Test:** The substance is dissolved with soda lime.	Formation of ammonia gas.	May be an aide.
	ii) **Biutet Test:** Add 100mg of substance in a dry test tube and is heated until it melts and ammonia gas is formed. The melted substance is then cooled till it gets solidified. It is dissolved in 10% NaOH solution and one drop of very dilute solution $CuSO_4$ is added.	Formation of purple or violet colour.	Presence of diamide.
	iii) **Urea Nitrate Test:** Few drops of conc. HNO_3 is treated with the conc. Solution of substance in water.	Formation of white crystalline ppt.	Presence of urea.

)	**Test For Phenol:**		
	i) **Ferric Chloride Test:** A small quantity of substance is treated with ferric chloride.	Solution becomes blue (or) violet and green.	Presence of phenol.
	ii) **Phthalic Fusion Test:** In a dry test tube, take 100mg of the substance and equal amount of phthalic anhydride is added. Add 2 to 3 drops of conc. H_2SO_4 to this solution. The tube is gently heated for about 1 min. It is then cooled and 10% NaOH solution is added to this.	i) Formation of red colour. ii) Formation of green fluorescence.	Presence of phenol Presence of resorcinol (or) di hydric phenol.
	iii) **Dye Test for Phenol:** 2 drops of Aniline is dissolved in slight excess of conc. HCl and then cool in ice water and then add $NaNO_2$ solution to it. Add 2gm of sample in NaOH solution in another test tube and mix two solutions.	Formation of orange red dye.	Presence of phenol like, napthol, resorcinol.
	iv) **Libermann Test:** In dry test tube, take a small amount of substance and add few drops of sodium nitrite to this. Add conc. HCl and water. The layer of water is removed and boiled. Conc. H_2SO_4 and dil NaOH is added to this solution.	Formation of deep blue or violet solution.	Presence of phenol.
4)	**Test for Nitro Compounds**		
	i) **Baker And Mullein's Test:** 50ml of substance is dissolved in 2ml of ethanol and add 1ml of zinc dust to this solution. It is heated on water bath about 10 minute and then filtered. Add Tollen's reagent to the filtrate and heat on water bath.	Formation of silver mirror.	Presence of nitro compounds.

8)	**Test For Amines:**		
	i) **Dye Test:** 100mg of substance is dissolved in 1ml of conc.HCl and the solution are diluted with 3ml of water. It is cooled in ice to 5-10°C. Cool 2ml 10% solution of $NaNO_2$ in another test tube. It is then added to the first test tube and mixed well. Add 2ml of cool alkaline beta-naphthol solution to this solution.	Formation of orange or orange red dye. Formation of oily liquid.	Presence of primary amine. Presence of secondary amine.
	ii) **Mayer's Test:** Add twice the volume of Mayer's reagent to 2gm of substance. It is then heated and allowed to stand.	Formation of yellow ppt.	Presence of tertiary amine.
9)	**Test For Aldehyde:**		
	i) **Schiff's Reagent Test:** Substance is treated with Schiff's reagent.	Formation of magenta colour.	Presence of aldehyde.
	ii) **Fehling's Test:** Equal volume of Fehling's solution is mixed and then 50mg of the substance is added and then heated on water bath.	Formation of red colour ppt.	Presence of aldehyde.
	iii) **Benedict's Test:** Substance is added with Benedict's solution and heated on a water bath.	Formation of red colour ppt.	Presence of aldehyde.
	iv) **Tollen's Test:** Sample is treated with Tollen's reagent and heated for 30min in a water bath.	Formation of silver mirror along the sides of test tube.	Presence of aldehyde.

10)	**Picrate Test:** The material is dissolved in methyl alcohol and ether, and then mixed well in a watch bath with a few drops of cold conc. picric acid solution.	Formation of yellow needle shaped crystals.	May be hydrocarbon.
11)	**Saponification Test:** The substance is heated in a water bath with 5 drops of NaOH solution and 2 drops of phenolphthalein.	The pink colour will discharge over time. Also, the odour disappears.	May be an ester.

Result: The special elements in the given sample was determined.

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