Practical Manual

Pharmaceutical Organic Chemistry - I



Prepared By

Chinmaya Chidananda Behera (M. Pharm. Pharmaceutical Chemistry) Asst. Prof., School of Pharmacy and Life

Sciences, CUTM, Bhubaneswar

Object:

To perform the Preliminary test: Color, odour, aliphatic/aromatic compounds, saturation and unsaturation of Organic compounds.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Chemicals/ Reagents: Supplied Organic compound, Nichrome Wire, Bunsen Burner, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Before outlining the general scheme, one or two points of practical importance should be noted.

(a) Quantities of substance for tests. For most tests about 0.1 g solid or 0.1 - 0.2 mL (2 - 3 drops) of liquid material (NOT MORE) should be used.

(b) Reagents likely to be met within organic analysis are on the reagent shelves. Students are advised to develop a general knowledge of the physical characteristics of common organic compounds. If in doubt about the expected result of a test between a certain compound and a reagent, carry out a trial test with a known compound and compare with the unknown.

(c) Quantities of substance derivatives. Students have wasted much time and material in the past by taking too large a quantity of substance for preparation of a derivative. In general, 0.5 - 1 g (or 0.5 - 1 mL) of substance gives the most satisfactory results.

Date: ----- Practical Manual, Pharmaceutical Organic Chemistry - I If a practical book instructs one to use larger quantities (3 - 4 g or more), the quantities should be scaled down to 1 g or 1 mL of the unknown substance and corresponding quantities of reagents should be used.

Procedure:

Preliminary Tests

(a) Note physical characteristics - solid, liquid, colour and odour.

(b) Perform an ignition test (heat small amount on metal spatula) to determine whether the compound is aliphatic or aromatic (i.e. luminous flame - aliphatic; sooty flame - aromatic).

Test, observation and inference should be given.

Physical property:

Color	
Odour	
Physical State	
Saturation/Unsaturation	

Result:

Product	Theoretical yield	Practical Yield	Melting point	% Yield

Use:

Signature of In-charge

Object:

To perform the Detection of elements like Nitrogen by Lassaigne's test.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Fusion Tube, Sodium Metal, Distilled Water, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Qualitative Analysis for Elements

In organic compounds the elements commonly occurring along with carbon and hydrogen, are oxygen, nitrogen, sulphur, chlorine, bromine and iodine. The detection of these elements depends upon converting them to water-soluble ionic compounds and the application of specific tests.

Lassaigne's Sodium Fusion Test

C, H, O, N, S, X NaX NaCN Na₂S NaCNS

Procedure:

Place a piece of clean sodium metal, about the size of a pea into a fusion tube. Add a little of the compound (50 mg or 2 - 3 drops).* Heat the tube gently at first, allowing any distillate formed to drop back onto the molten sodium. When charring begins, heat the bottom of the tube to dull redness for about three minutes and finally plunge

Date:Practical Manual, Pharmaceutical Organic Chemistry - Ithe tube, while still hot, into a clean dish containing cold distilled water (6 mL) and

cover immediately with a clean wire gauze.**

*For liquids it is better to first melt the sodium add the liquid drop by drop.

**CAUTION: The tube shatters, and any residual sodium metal reacts with water. Stir the mixture, boil for 1 - 2 minutes, on a tripod and filter hot through a fluted paper.

The 'fusion' filtrate which should be clear and colourless, is used for the specific tests described below:

1. NITROGEN (NITRILE) To a portion (2 mL) of the 'fusion' filtrate add 0.2 g of powdered ferrous sulphate crystals. Boil the mixture for a half a minute, cool and acidify by adding dilute sulphuric acid dropwise. Formation of a bluish-green precipitate (Prussian blue) or a blue solution indicates that the original substance contains nitrogen. If no precipitate appears, allow to stand for 15 minutes, filter and inspect filter paper.

2. SULPHUR (SULPHIDE) To the cold 'fusion' filtrate (1 mL) add a few drops of cold, freshly prepared, dilute solution of sodium nitroprusside. The latter may be prepared by adding a small crystal of the solid to 2 mL of water. Production of a rich purple colour indicates that the original substance contains sulphur. This test is very sensitive. Only strong positive results are significant.

3. HALOGENS (HALIDES) Acidify a portion (1 mL) of the 'fusion' filtrate with 2N nitric acid, and if nitrogen and/or sulphur are present, boil for 1 - 2 minutes.* Cool and add aqueous silver nitrate (1 mL), compare with a blank. Formation of a heavy, white or yellow precipitate of silver halide indicates halogen. If a positive result is obtained: acidify the remaining portion of the 'fusion' filtrate with dilute sulphuric acid, boil and cool. Add carbon tetrachloride (1 mL) and a few drops of freshly prepared chlorine water. Shake the mixture.

(a) If the carbon tetrachloride layer remains colourless - indicates chlorine.

(b) If the carbon tetrachloride layer is brown - indicates bromine.

(c) If the carbon tetrachloride layer is violet - indicates iodine.

Date: ----- Practical Manual, Pharmaceutical Organic Chemistry - I *If nitrogen and/or sulphur are also present, the addition of silver nitrate to the acidified 'fusion' solution will precipitate silver cyanide and/or silver sulphide in addition to the silver halides. The removal of hydrogen cyanide and/or hydrogen sulphide is effected by boiling the 'fusion' solution.

Result:

Sample Name	Inference	Conclusion

Signature of In-charge

Object:

To perform the Detection of elements like Sulphur by Lassaigne's test.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Fusion Tube, Sodium Metal, Distilled Water, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Qualitative Analysis for Elements

In organic compounds the elements commonly occurring along with carbon and hydrogen, are oxygen, nitrogen, sulphur, chlorine, bromine and iodine. The detection of these elements depends upon converting them to water-soluble ionic compounds and the application of specific tests.

Lassaigne's Sodium Fusion Test

C, H, O, N, S, X NaX NaCN Na₂S NaCNS

Procedure:

Place a piece of clean sodium metal, about the size of a pea into a fusion tube. Add a little of the compound (50 mg or 2 - 3 drops).* Heat the tube gently at first, allowing any distillate formed to drop back onto the molten sodium. When charring begins, heat the bottom of the tube to dull redness for about three minutes and finally plunge

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cover immediately with a clean wire gauze.**

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**CAUTION: The tube shatters, and any residual sodium metal reacts with water. Stir the mixture, boil for 1 - 2 minutes, on a tripod and filter hot through a fluted paper.

The 'fusion' filtrate which should be clear and colourless, is used for the specific tests described below:

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2. SULPHUR (SULPHIDE) To the cold 'fusion' filtrate (1 mL) add a few drops of cold, freshly prepared, dilute solution of sodium nitroprusside. The latter may be prepared by adding a small crystal of the solid to 2 mL of water. Production of a rich purple colour indicates that the original substance contains sulphur. This test is very sensitive. Only strong positive results are significant.

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(a) If the carbon tetrachloride layer remains colourless - indicates chlorine.

(b) If the carbon tetrachloride layer is brown - indicates bromine.

(c) If the carbon tetrachloride layer is violet - indicates iodine.

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Result:

Sample Name	Inference	Conclusion

Signature of In-charge

Object:

To perform the Detection of elements like Halogens by Lassaigne's test.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Fusion Tube, Sodium Metal, Distilled Water, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Qualitative Analysis for Elements

In organic compounds the elements commonly occurring along with carbon and hydrogen, are oxygen, nitrogen, sulphur, chlorine, bromine and iodine. The detection of these elements depends upon converting them to water-soluble ionic compounds and the application of specific tests.

Lassaigne's Sodium Fusion Test

C, H, O, N, S, X NaX NaCN Na₂S NaCNS

Procedure:

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2. SULPHUR (SULPHIDE) To the cold 'fusion' filtrate (1 mL) add a few drops of cold, freshly prepared, dilute solution of sodium nitroprusside. The latter may be prepared by adding a small crystal of the solid to 2 mL of water. Production of a rich purple colour indicates that the original substance contains sulphur. This test is very sensitive. Only strong positive results are significant.

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(c) If the carbon tetrachloride layer is violet - indicates iodine.

Date: ----- Practical Manual, Pharmaceutical Organic Chemistry - I *If nitrogen and/or sulphur are also present, the addition of silver nitrate to the acidified 'fusion' solution will precipitate silver cyanide and/or silver sulphide in addition to the silver halides. The removal of hydrogen cyanide and/or hydrogen sulphide is effected by boiling the 'fusion' solution.

Result:

Sample Name	Observation	Inference

Signature of In-charge

Object:

To perform the Solubility Test for the supplied Organic compound.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Different solvent in the order of polarities, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Solubility tests

The solubility of the unknown in the following reagents provides very useful information. In general, about 3 mL of the solvent is used with 0.1 g or 0.2 mL (2 - 3 drops) of the substance. The class of compound may be indicated from the following table:

REAGENT AND TEST	CLASS	GROUP OF COMPOUNDS	
Soluble in cold or hot	Neutral, acidic or	Lower members of series.	
water. (If the unknown is	basic. (Test with	Neutral, e.g. alcohols; Acidic,	
soluble do NOT perform	litmus or universal	e.g. acids, phenols; Basic, e.g.	
solubility tests below)	indicator paper)	amines	
Soluble in dil. HCl	Basic	Most amines (except III amines	
		with only aromatic groups	
Soluble in dil. NaOH	Acidic	Most acids, most phenols.	
Soluble in NaHCO ₃	Strongly acidic	Most carboxylic acids.	
Insoluble in water, acid and	Neutral	Hydrocarbons, nitrohydro-	
alkali		carbons, alkyl or aryl halides,	
		esters and ethers. Higher	

Date:	Practical Manual, Pharmaceutical Organic Chemistry - I			
		molecular	weight	alcohols,
		aldehydes a	nd ketones	5

Procedure:

Place a small amount of supplied organic compound in the 15 ml test-tube and add about 5 ml of the solvents as listed in the table and shake manually for about Two minutes. If required heat gently the mixture in boiling water bath to ensure solubility. Report the solubility of the organic compound.

Result:

Sample Name	Solubility	Inference

Signature of In-charge

Object:

To perform the Functional group test of Phenols, Amides/ Urea.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Different reagents, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Practical Notes

Before outlining the general scheme, one or two points of practical importance should be noted.

(a) Quantities of substance for tests. For most tests about 0.1 g solid or 0.1 - 0.2 mL (2 - 3 drops) of liquid material (NOT MORE) should be used.

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(c) Quantities of substance derivatives. Students have wasted much time and material in the past by taking too large a quantity of substance for preparation of a derivative. In general, 0.5 - 1 g (or 0.5 - 1 mL) of substance gives the most satisfactory results.

Date: ----- Practical Manual, Pharmaceutical Organic Chemistry - I If a practical book instructs one to use larger quantities (3 - 4 g or more), the quantities should be scaled down to 1 g or 1 mL of the unknown substance and corresponding quantities of reagents should be used.

Procedure:

I. UNSATURATED COMPOUNDS

Two common types of unsaturated compounds are alkenes and alkynes characterised by the carbon-carbon double and triple bond, respectively, as the functional group. The two common qualitative tests for unsaturation are the reactions of the compounds with (a) bromine in carbon tetrachloride and (b) potassium permanganate.

(a) 2% Bromine in carbon tetrachloride: Dissolve 0.2 g (or 0.2 mL) of the compound in 2 mL of carbon tetrachloride or another suitable solvent and add the solution dropwise to 2 ml of 2% bromine solution in carbon tetrachloride and shake. e.g.



Rapid disappearance of the bromine colour to give a colourless solution is a positive test for unsaturation.

NOTE: The reagent is potentially dangerous. Keep it off your skin and clothes; protect your eyes and nose. (b) 2% Aqueous potassium permanganate Dissolve 0.2 g (or 0.2 mL) of the substance in 2 mL of water (acetone may also be used as solvent). Add the potassium permanganate solution dropwise and observe the result. e.g.



Date:Practical Manual, Pharmaceutical Organic Chemistry - IFor a blank determination, count the number of drops added to 2 mL of acetonebefore the colour persists. A significant difference in the number of drops required inthe two cases is a positive test for unsaturation.

II. COMPOUNDS CONTAINING NITROGEN

1 Amides R-CO-NH2:

Simple primary amides can be decomposed by boiling with alkali and thereby evolving ammonia.e.g.

CH3-CO-NH2 + NaOH ----> CH3-CO2- Na+ + NH3

Boil 0.5 g of the compound with 5 mL of 10% sodium hydroxide solution and observe whether ammonia is evolved.

III. BIURET REACTION FOR ALIPHATIC DIAMIDE (UREA):

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.



Result:

The Supplied Organic compound was found to contain _____

_____ functional groups.

Signature of In-charge

Object:

To perform the Functional group test of Carbohydrates, Amines.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Different reagents, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Practical Notes

Before outlining the general scheme, one or two points of practical importance should be noted.

(a) Quantities of substance for tests. For most tests about 0.1 g solid or 0.1 - 0.2 mL (2 - 3 drops) of liquid material (NOT MORE) should be used.

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(c) Quantities of substance derivatives. Students have wasted much time and material in the past by taking too large a quantity of substance for preparation of a derivative. In general, 0.5 - 1 g (or 0.5 - 1 mL) of substance gives the most satisfactory results.

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Procedure:

I. SUGARS, CARBOHYDRATES

a. Molisch's Test: This is a general test for carbohydrates. Dissolve 20 - 30 mg of the compound in 2 mL water and add 0.5 mL of the reagent (a 20% solution of 2-naphthol in ethanol). Pour 2 mL of concentrated sulphuric acid from a dropper carefully down the side of the tube so that the acid forms a layer beneath the aqueous solution without mixing with it. A red colouration, changing to dark purple forms at the interface. Carry out a second test on a blank solution.

II. ALDEHYDES AND KETONES (For Aldose and Ketoses)

(i) Semicarbazones: Dissolve semicarbazide hydrochloride (1 g) and sodium acetate (1.5 g) in water (8 - 10 mL), add the aldehyde or ketone (0.3 mL) and shake. Shake the mixture for a few minutes and then cool in ice-water. Filter off the crystals, wash with a little cold water and recrystallise from methanol or ethanol.

(ii) 2,4-Dinitrophenylhydrazones: Suspend 0.25 g of 2,4-dinitrophenylhydrazine in 5 mL of methanol and add 0.5 mL of concentrated sulphuric acid cautiously. Filter the warm solution and add a solution of 0.2 g of the carbonyl compound in 1 mL of methanol. Recrystallise the derivative from methanol, ethanol or ethyl acetate.

(*iii*) Oximes: Hydroxylamine hydrochloride (0.5 g) is dissolved in water (2 mL). 10% sodium hydroxide (2 mL) and the carbonyl compound (0.2 - 0.3 g) dissolved in alcohol (1 - 2 mL) are added, the mixture warmed on a steam bath for 10 min and then cooled in ice. Crystallisation is induced by scratching the sides of the test tube with a glass rod. The oximes may be crystallised from alcohol.

III. COMPOUNDS CONTAINING NITROGEN

1. Amines

Date: -----Practical Manual, Pharmaceutical Organic Chemistry - I (a) Reaction with nitrous acid: Dissolve the amine (0.5 mL) in concentrated acid (2.0 mL) and water (3 mL) and cool the solution to 0 - 5 in an ice-bath for 5 minutes. Add a cold solution (ice-bath) of sodium nitrite (0.5 g) in water (2.0 mL) from a dropper, with swirling of the test tube, still keeping the mixture in the ice-bath.

AMINE REACTION

I aliphatic:	N2 evolved.
	$RNH2 + HNO2 \rightarrow ROH + N2 + H2O$
I aromatic:	Diazonium salt is formed.
	ArNH2 + HNO2 -> ArN=N+
	Add the cold diazonium solution and with swirling to a cold solution of 2-naphthol (0.2 g) in 5% NaOH solution (2 mL). An orange-red azo dye is formed.
II aliphatic:	Yellow oily nitrosamines are generally formed.

II aromatic $R2NH + HNO2 \rightarrow R2N-NO$

- III aliphatic No visible reaction.
- **III** aromatic Dialkylanilines yield green solid p-nitroso compounds (if p-position unsubstituted).



(b) Reaction with benzenesulphonyl chloride: Benzenesulphonyl chloride reacts with primary and secondary but not with tertiary amines to yield substituted sulphonamides. e.g.

(a) C6H5SO2Cl + H-NHR + NaOH -> C6H5SO2NHR + NaCl + H2O

(b) C6H5SO2Cl + H-NR2 + NaOH -> C6H5SO2NR2 + NaCl + H2O

Date: -----

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The substituted sulphonamide formed from a primary amine dissolves in the alkali medium whilst that produced from a secondary amine is insoluble in alkali.

Place 0.5 mL (or 0.5 g) of the compound, 15 - 10 mL of 5% NaOH and 1 mL of benzenesulphonyl chloride in a test tube, stopper the tube and shake until the odour of the sulphonyl chloride has disappeared. The solution must be kept alkaline (if no reaction has occurred, the substance is probably a tertiary amine).

If a precipitate appears in the alkaline solution, dilute with about 10 mL of water and shake; if the precipitate does not dissolve, a secondary amine is indicated.

If there is no precipitate, acidify it cautiously to congo red with concentrated hydrochloric acid (added dropwise): a precipitate is indicative of a primary amine.

Result:

The Supplied Organic compound was found to contain

functional groups.

Signature of In-charge

Object:

To perform the Functional group test of Carboxylic acids, Aldehydes and Ketones.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Different reagents, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Practical Notes

Before outlining the general scheme, one or two points of practical importance should be noted.

(a) Quantities of substance for tests. For most tests about 0.1 g solid or 0.1 - 0.2 mL (2 - 3 drops) of liquid material (NOT MORE) should be used.

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(c) Quantities of substance derivatives. Students have wasted much time and material in the past by taking too large a quantity of substance for preparation of a derivative. In general, 0.5 - 1 g (or 0.5 - 1 mL) of substance gives the most satisfactory results.

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Procedure:

I. CARBOXYLIC ACIDS:

(a) Test with 5% aq. NaHCO₃:

 $R-CO2H + NaHCO3 - R-CO^2 Na^+ + CO2 + H2O$

Sodium hydrogen carbonate reacts with carboxylic acids to give the sodium salt of the acid and liberates carbon dioxide. If the acid is insoluble in water and the reaction is sluggish dissolve the acid in methanol and add carefully to a saturated sodium hydrogen carbonate solution, when a vigorous effervescence will be observed.

(b) Amides, anilides and p-toluidides Formation:

The acid (0.5 g) is refluxed with thionyl chloride (2 - 3 mL) in a fume cupboard for about 30 mins.* It is advisable to place a plug of cotton wool in the top of the reflux condenser to exclude moisture. The condenser is removed and the excess of thionyl chloride is distilled off (b.p. 78). The acid chloride thus produced is treated with concentrated ammonia solution (5 mL) or aniline (0.5 - 1 mL) or p-toluidine (0.5 - 1 g), when the solid derivative separates out. It is collected and recrystallised from alcohol adding decolourising charcoal if found necessary. Alternately use PCl_5 to form the acid chloride.

II. ALDEHYDES AND KETONES (For Aldose and Ketoses)

(i) Semicarbazones: Dissolve semicarbazide hydrochloride (1 g) and sodium acetate (1.5 g) in water (8 - 10 mL), add the aldehyde or ketone (0.3 mL) and shake. Shake the mixture for a few minutes and then cool in ice-water. Filter off the crystals, wash with a little cold water and recrystallise from methanol or ethanol.

(ii) 2,4-Dinitrophenylhydrazones: Suspend 0.25 g of 2,4-dinitrophenylhydrazine in 5 mL of methanol and add 0.5 mL of concentrated sulphuric acid cautiously. Filter the

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Result:

The Supplied Organic compound was found to contain _____

____ functional groups.

Signature of In-charge

Object:

To perform the Functional group test of Alcohols, Esters, Aromatic compounds.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Different reagents, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Practical Notes

Before outlining the general scheme, one or two points of practical importance should be noted.

(a) Quantities of substance for tests. For most tests about 0.1 g solid or 0.1 - 0.2 mL (2 - 3 drops) of liquid material (NOT MORE) should be used.

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Procedure:

I. ALCOHOLS

(*i*) 3,5-Dinitrobenzoates: 3,5-Dinitrobenzoyl chloride is usually partially hydrolysed and should be prepared in the pure state by heating gently a mixture of 3,5dinitrobenzoic acid (1 g) and phosphorus pentachloride (1.5 g) in a dry test tube, until it liquifies (5 min).* The liquid is poured on a dry watch glass and allowed to solidify. The phosphoryl chlorides are removed by pressing the solid with a spatula on a wad of filter paper. The residual acid chloride is suitable for immediate use in the preparation of the derivatives.

*Work under fume hood. Fumes are irritating to the eyes and nose.

The 3,5-dinitrobenzoyl chloride is mixed with the alcohol (0.5 - 1 mL) in a loosely corked dry test tube and heated on a steam bath for about 10 min. Secondary and tertiary alcohols require up to 30 min. On cooling add 10 mL sodium hydrogen carbonate solution, stir until the ester crystallises out, and filter at the pump. Wash with a little carbonate solution, water and suck dry. Recrystallise from the minimum hot ethanol or light petroleum. Cool slowly to avoid the formation of oily droplets of your ester.

II. ESTERS

(a) Hydroxamic acid test:

R-CO-OR' + H2N-OH -> R-CO-NH-OH + R'-OH

Esters react with hydroxylamine in the presence of sodium hydroxide to form the sodium salt of the corresponding hydroxamic acid. On acidification and addition of ferric chloride the magenta-coloured iron (III) complex of the hydroxamic acid is formed.

Date: -----

It is always advisable to ensure that an unknown compound does not give a colour with iron (III) chloride before carrying out the hydroxamic acid test.

Procedure for hydroxamic acid test:

(a) Ferric chloride test: Dissolve a drop or a few small crystals of the compound in 1 mL of 95% ethanol (rectified spirit) and add 1 mL of M hydrochloric acid. Note the colour produced when 1 drop of 5% iron (III) chloride is added to the solution. If a pronounced violet, blue, red or orange colour is produced, the hydroxamic acid test described below is NOT APPLICABLE.

(b) Hydroxamic acid test: Mix 1 drop or several small crystals (ca 0.05 g) of the compound with 1 mL of 0.5 M hydroxylamine hydrochloride in 95% ethanol and add 0.2 mL of 6 M aqueous sodium hydroxide. Heat the mixture to boiling and after the solution has cooled slightly add 2 mL of M hydrochloric acid. If the solution is cloudy, add 2 mL of 95% ethanol. Observe the colour produced when 1 drop of 5% iron (III) chloride solution is added. If the resulting colour does not persist, continue to add the reagent dropwise until the observed colour pervades the entire solution. Usually only 1 drop of the iron (III) chloride solution is necessary. Compare the colour with that produced in test (a). A positive test will be a distinct burgundy or magenta colour as compared with the yellow colour observed when the original compound is tested with iron (III) chloride solution in the presence of acid. It is often advisable to conduct in parallel the test with, say, ethyl acetate, to ensure that the conditions for this test are correct.

III. AROMATIC COMPOUNDS (test for Unsaturation)

Two common types of unsaturated compounds are alkenes and alkynes characterised by the carbon-carbon double and triple bond, respectively, as the functional group. The two common qualitative tests for unsaturation are the reactions of the compounds with (a) bromine in carbon tetrachloride and (b) potassium permanganate.

(a) 2% Bromine in carbon tetrachloride: Dissolve 0.2 g (or 0.2 mL) of the compound in 2 mL of carbon tetrachloride or another suitable solvent and add the solution dropwise to 2 ml of 2% bromine solution in carbon tetrachloride and shake. e.g.



Rapid disappearance of the bromine colour to give a colourless solution is a positive test for unsaturation.

Result:

The Supplied Organic compound was found to contain

_ functional groups.

Signature of In-charge

Object:

To perform the functional group test for Halogenated Hydrocarbons, Nitro compounds and Anilides.

Reference:

Sharma A. Practical in Pharmaceutical Chemistry-III (Organic Chemistry-I), 1st ed. Meerut: Shri Sai Prakashan, 2009: 54-55.

Requirement:

Supplied Organic Compound, Different reagents, etc.

Equipments/ Glassware: Beaker, glass rod, Round Bottom Flask, Funnel, filter paper, Magnetic Stirrer.

Theory:

Practical Notes

Before outlining the general scheme, one or two points of practical importance should be noted.

(a) Quantities of substance for tests. For most tests about 0.1 g solid or 0.1 - 0.2 mL (2 - 3 drops) of liquid material (NOT MORE) should be used.

(b) Reagents likely to be met within organic analysis are on the reagent shelves. Students are advised to develop a general knowledge of the physical characteristics of common organic compounds. If in doubt about the expected result of a test between a certain compound and a reagent, carry out a trial test with a known compound and compare with the unknown.

(c) Quantities of substance derivatives. Students have wasted much time and material in the past by taking too large a quantity of substance for preparation of a derivative. In general, 0.5 - 1 g (or 0.5 - 1 mL) of substance gives the most satisfactory results.

Date: ----- Practical Manual, Pharmaceutical Organic Chemistry - I If a practical book instructs one to use larger quantities (3 - 4 g or more), the quantities should be scaled down to 1 g or 1 mL of the unknown substance and corresponding quantities of reagents should be used.

Procedure:

I. HALOGENATED HYDROCARBONS

Halogenated hydrocarbons are usually unreactive and are therefore widely used as solvents in organic chemistry.

a) Reaction with silver nitrate: At room temperature Add a drop (= 0.03 mL) (or 30 mg) of the compound to 0.5 mL of a saturated solution of AgNO3 in ethanol. If the test is positive, a precipitate forms within two minutes. Compounds that react positively are alkyl iodides and bromides, tertiary alkyl chlorides, alicyclic iodides and bromides, allylic halides and 1,2-dibromoalkanes.

b) Reaction with silver nitrate under boiling: If no precipitate forms in the previous test after two minutes, the sample is boiled for half a minute. A positive reaction (the formation of a precipitate) now occurs in case the sample contains a primary or secondary alkyl chloride. Aryl and vinyl halides as well as polyhalogenated compounds (eg.chloroform) give a negative result.

c) Hydrolysis: Mix 100 mg of the compound to be examined with 5 mL of a 5 % solution of KOH in alcohol and boil the mixture for 5 min. Add 10 mL of water to the cool mixture, acidify with HNO3 and filter if necessary until the solution is clear. Add two drops of a 5 % silver nitrate solution. A positive reaction (precipitate) is obtained with all alkyl and cycloalkyl iodides, bromides and chlorides. Fluorides and aryl halides give a negative result (with the latter a cloudy solution might appear).

II. NITRO COMPOUNDS

(a) Iron (II) Hydroxide Test for Nitro Groups:

$BNO_2 + 6 Fe(OH)_2 + 4 H_2O \longrightarrow$ $BNH_2 + 6 Fe(OH)_3$

Add about 10 mg of the compound to 1 mL of the ferrous ammonium sulfate reagent in a test tube, and then add 0.7 mL of the 2*N* alcoholic potassium hydroxide reagent. Stopper the tube, and shake. Note the color of the precipitate after 1 minute. *Ferrous Ammonium Sulfate Reagent:* To 50 mL of recently boiled, distilled water add 2.5 g of ferrous ammonium sulfate crystals and 0.2 mL of concentrated sulfuric acid. The Alcoholic Potassium Hydroxide Reagent will already be prepared for you. A positive test is the formation of the red-brown precipitate of iron(III) hydroxide.

II. ANILIDES (AMIDES)

Date: -----

Amides are decomposed by NaOH to evolve ammonia. The gas can be tested by a moist red litmus paper which is then turned blue.

Result:

The Supplied Organic compound was found to contain

functional groups.

Signature of In-charge

Object:

To determine and report the melting point of the given sample.

Reference:

 Furniss BS, Hannaford AJ, Smitt PWG, Tatchell AR. Vogel's Text book of practical organic chemistry. 5th ed. Singapore: Longman Singapore Publishers Pte. Ltd., 2004: 236.

Requirements:

Chemical/ Reagents: Supplied Sample of solid.

General/Glassware: Thiele's Tube, Burner, Thermometer, Capillary Tubes.

Principle: Melting point serves as a criterion of purity of an organic compound. Ions or molecules in a crystalline solid are arranged in a regular geometric fashion. Melting point occurs when a temperature is reached at which the thermal energy of the particles is great enough to overcome the inter-crystalline forces that hold them together. A pure organic compound usually has a sharp and definite melting point.

Theory:

The melting point of a compound is the temperature at which the solid phase is in equilibrium with the liquid phase. A solid compound changes to a liquid when the molecules acquire enough energy to overcome the forces holding them together in an orderly arranged crystalline lattice. For most organic compounds, these intermolecular forces are relatively weak. The melting point range is defined as the span of temperature from the point at which the crystals first begin to liquefy to the point at which the entire sample is liquid. Most pure organic compounds melt over a narrow temperature range of 1-2 $^{\circ}$ C.

Melting points are used for three reasons.

1. To help characterize (identity) an unknown sample.

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- 2. Recorded for new compounds for future identity by others.
- 3. To determine the purity of a compound. (A solid is considered pure, if its melting point does not rise after re-crystallization.)

Factors that determine melting point.

1. Intermolecular forces.

Covalent compounds – most melt < 300°C

London forces

Dipole-dipole attraction

H-Bonding

Ionic compounds – have much higher melting points, e. g. NaCl-800°C

Ionic forces

2. Larger molecules have higher melting points

Methane	Ethane	Propane	Butane
CH ₄	CH ₃ CH ₃	CH ₃ CH ₂ CH ₃	CH ₃ CH ₂ CH ₂ CH ₃
-184°C	-172°C	-190	-135°C

3. More symmetrical compounds will have higher melting points. (Solid arrangement more orderly)

Pentane	iso-Pentane	neo-Pentane
CH ₃ CH ₂ CH ₂ CH ₂ CH ₃	CH ₃ CH ₂ CH(CH ₃) ₂	CH ₃ C(CH ₃) ₃
m.p.	m.p.	m.p.
-129.72°C	-159.9°C	-16.55°C
lowest		highest
However, branching low	ers boiling points	

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36.1°C	27.85°C	9.5°C	
highest		lowest	

Procedure:

- 1. Seal the capillary tube at one end by keeping 2-3 mm of capillary tube in Bunsen flame contact and continuously rotating it till the end is completely sealed by forming a bulb at the end.
- 2. Now fill the capillary with compound the melting point of which is to be determined, about 0.05 g of compound is placed on the plate and open end is then gently tapped.
- 3. Tie the capillary tube with thermometer and place the whole in Thiele's tube filled with liquid paraffin.
- 4. Now put Bunsen flame under the tube constantly and properly.
- 5. Note the temperature at which the compound starts melting and then note the temperature at which it completely melts (both the temp. should not vary more than the range of 2°C. Take the average of the two temperatures.



Fig: Thiele's tube assembled

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Laboratory Safety Precaution

- 1. Wear safety goggles and lab coat at all times while working in the laboratory.
- 2. Many chemicals are potentially harmful. Prevent contact with your eyes, skin, and clothing. Avoid ingesting any of the reagents.
- 3. Unknowns may be flammable, toxic, and irritating.
- 4. Capillary tubes are fragile and easily broken.
- 5. Wash your hands thoroughly with soap or detergent before leaving the laboratory.
- 6. Take care to avoid burning yourself when using paraffin oil, glycerol and electric hotplates.

Melting Point of given substances

Crude Melting Pt Determination:

Accurate Melting Pt Det.:

Literature Melting Point:

Result:

Signature of In-charge

Object:

To determine and report the boiling point of the given sample.

Reference:

 Furniss BS, Hannaford AJ, Smitt PWG, Tatchell AR. Vogel's Text book of practical organic chemistry. 5th ed. Singapore: Longman Singapore Publishers Pte. Ltd., 2004: 241.

Requirements:

Chemical/ Reagents: Supplied Sample of Liquid.

General/Glassware: Thiele's Tube, Burner, Thermometer, Capillary Tubes, Fusion tubes.

Principle:

In a liquid, the particles are not arranged in a regular manner and are thus free to move. A liquid boils when a temperature is reached at which the thermal energy of the particles is great enough to overcome the cohesive forces that hold them together. This is the point at which the vapor pressure of the liquid becomes equal to the atmospheric pressure. Boiling point of the liquid is significant for a particular compound.

Theory:

Boiling points are also useful physical properties for indicating the purity of an organic compound. Boiling point is the temperature at which the vapor pressure of a liquid equals atmospheric pressure or some other applied pressure. A boiling point is commonly measured during a distillation, in which a liquid is heated to form vapor, and then the vapor is condensed and collected in another container. The boiling temperature is measured as distillation vapor covers the bulb of a thermometer suspended above the boiling liquid. Typically, the most accurate boiling point measurement is the relatively constant temperature achieved during a distillation.

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The phenomenon of vapor pressure is interpreted in terms of molecules of liquid escaping into the empty space above the liquid. In order for the molecules to escape from the liquid phase into the vapor phase, the intermolecular forces (in order of increasing strength: Van-Der-Waals, dipole-dipole, hydrogen bonding) have to be overcome which requires energy. Since the nature of the intermolecular forces is determined by the molecular structure, then the amount of energy required to vaporize the sample also depends on the molecular structure. As the number of molecules in the vapor above the liquid becomes larger, the rate of return of the molecules from the vapour to the liquid increases until the rate of return is equals the constant rate of escape. This is the equilibrium condition and the corresponding concentration of molecules in the vapor space gives rise to the equilibrium vapor pressure. At higher temperatures, the greater average kinetic energy of the molecules in the liquid results in a greater constant rate of escape. Equilibrium is established at higher temperatures, and so larger numbers of molecules are present in the vapor phase and the pressure is higher. The vapor pressure-temperature curve for water is shown on the following page.

When the vapor pressure of a liquid is equal to the atmospheric (or applied) pressure then boiling occurs. The temperature, at which this occurs, for a given pressure, is the boiling point. It should be noted, therefore, that the boiling point of a liquid decreases as the atmospheric (or applied) pressure decreases.

Procedure:

Date: -----

- 1. Take the ignition tube.
- 2. Now fill the tube with liquid sample to $1/4^{th}$ of the tube
- 3. Tie the ignition tube with thermometer and place the whole in Thiele's tube filled with liquid paraffin (Take care that ignition tube should be out of liquid paraffin).
- 4. Now put Bunsen flame under the tube constantly and properly.
- 5. Note the temperature at which the compound starts boiling.



Test Tube Assembly 1

Observations:

Boiling Point, 1st Trial:

Boiling Point, 2nd Trial:

Boling Point, 3rd Trial:

Avg. Boiling Point:

Normal Boiling Point:

Literature Boiling Point:

Result:

Signature of In-charge

Date: -----

Object:

To synthesize and characterize Iodoform from ethyl alcohol (Haloform Test).

References:

 Furniss BS, Hannaford AJ, Smitt PWG, Tatchell AR. Vogel's Text book of practical organic chemistry. 5th ed. Singapore: Longman Singapore Publishers Pte. Ltd., 2004: 1220.

Requirements:

Chemical/ Reagents: Ethyl alcohol, sodium carbonate, iodine, NaOH.

General/Glassware: Round bottom Flask, Beaker, Funnel, and thermometer.

Theory:

Iodoform is the organo-iodine compound with the formula CHI₃. A pale yellow, crystalline, volatile substance, it has a penetrating odor (in older chemistry texts, the smell is sometimes referred to as the smell of hospitals) and, analogous to chloroform, sweetish taste. It is occasionally used as a disinfectant. It is also known as tri-Iodomethane, and sometimes also referred to as Carbon tri-iodide (which is not strictly correct, as this compound also contains hydrogen) or Methyl tri-iodide (which is somewhat ambiguous as that name could also refer to the Methylated tri-iodide ion, CH₃I₃).

Chemical Reaction:

From ethanol:

CH ₃ CH ₂ OH	+	I_2	>	CH ₃ CHO	+	2HI
				Acetaldehyd	e	
CH ₃ CHO	+	$3I_2$	>	Cl ₃ CHO	+	3 HI
				Iodol		
Cl ₃ CHO	+	KOH	>	CHI ₃	+	HCOOK
Tri-iodoaceta	ldehyde	e		Iodoform		Pot. formate

Procedure:

Date: ------

- 1. Take 10 ml ethyl alcohol, 20ml of 5% Sodium hydroxide solution 500ml round bottom flask.
- 2. Heat the flask to about $70-80^{\circ}$ C on a water bath, to the warm solution.
- 3. Add small amount of Potassium iodide Iodine reagent at a time with constant shaking.
- 4. Add more Potassium iodide Iodine reagent so that the reaction product should have a pale yellow color.
- 5. Add drop wise a dil. Solution of NaOH, if any brown colour of iodine persists.
- 6. Filter the crude Iodoform, wash with cold water and recrystalise from hot ethanol.

* Potassium iodide-Iodine reagent is the mixture of 20g Potassium iodide and 10g Iodine in 100ml water.

Physical Property:

Formula	CHI
1 offitidid	emy
Molecular weight	$393.73 \text{ g mol}^{-1}$
D1 1 0	D 1 1 1 1 1
Physical State	Pale, light yellow, opaque crystals
Malting Daint	121 °C
Menning Form	121 C

Result:

Product	Theoretical yield	Practical Yield	Melting point	% Yield

Uses:

Date: --

- 1. The compound finds small scale use as a disinfectant.
- 2. Around the beginning of the 20th century it was used in medicine as a healing and antiseptic dressing for wounds and sores, although this use is now superseded by superior antiseptics.
- 3. Adolf Hitler's mother, Klara Hitler, died of Iodoform poisoning brought on by her treatment for breast cancer.
- 4. It is the active ingredient in many ear powders for dogs and cats, to prevent infection and facilitate removal of ear hair, along with zinc oxide and propanoic acid.
- 5. As an antiseptic, due to iodine it liberates. However due to its unpleasant smells it has been replaced by better antiseptics.
- 6. The reaction is used in Iodoform test.

Signature of In-charge

Object:

To synthesize and characterize Thiourea.

Reference:

- Mann FG, Saunders BC. Practical Organic Chemistry, 4th ed. New Delhi (India): Pearson Education, 2009:124.
- Takao B, Agimoto Y. The Transformation of Ammonium Thiocyanate into Thiourea under High Pressures. The Review of Physical Chemistry of Japan, 1954: 24.

Requirement:

Chemicals/ Reagents: Ammonium Thiocyanate (16.5g)

General/Glassware: Beaker, glass rod, funnel, tripod stand, thermometer, etc.

Theory:

Thiourea is a planar molecule available in two tautomeric forms.



It is also called Thiocarbamide or Sulfourea. It is the diamide of Thiocarbonic acid that resembles urea but contains sulfur instead of oxygen. It can be prepared from Ammonium Thiocyanates. At 135°C Ammonium Thiocyanates decomposes in to thiourea.



Procedure:

Date: -----

- 1. Placed 16.5g of Ammonium thiocyanate in a round bottom flask and heated over oil bath until all the content just melted down. The temperature was kept constant at which the content just remained liquid, i. e. 170°C for 1hr.
- 2. Then the content was cooled and the solid was ground with 20ml cold water, which dissolved unchanged ammonium thiocyanate, but little of thiourea also.
- 3. The crude product was recrystallized from hot water as color less silky needles.
- 4. The % yield and melting point were calculated.

Physical property:

	· · · · · · · · · · · · · · · · · · ·
Formula	CH_4N_2S
Molecular weight	76.12g/mol
	$D^{-1} W^{1} (1)$
Physical State	Brownish white crystals
Malting Doint	170 17400
Mennig Folin	1/0-1/4 C

Result:

Product	Theoretical yield	Practical Yield	Melting point	% Yield

Use:

- 1. Thiourea is used directly in ore filtering, metal refinery and cleaning, isomerization catalyst.
- 2. Used as additive in fertilizers, drilling auxiliaries, light sensitive photocopy papers and explosives.

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- 3. It is used as a liquefying agent in animal hide, as an insecticide, as textile treating agent and as intermediate to other compounds.
- 4. Thiourea derivatives are versatile medicinal compounds.

Signature of In-charge

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