

PHARMACEUTICAL ORGANIC CHEMISTRY- II

III SEMESTER (2nd YEAR B.PHARM)

PRACTICAL LAB MANUAL

Organic Chemistry Laboratory manual
(III rd. Semester)

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Expt. No.1**CRYSTALLIZATION AND RECRYSTALLIZATION**

The purpose of crystallization and recrystallization is to get pure compound. Crystallization may be defined as the process in which a solid compound precipitates from a saturated solution in the form of crystals. Saturation is usually effected through cooling or evaporation. In certain cases, recrystallization may be used for the separation of a solid mixture. When the impure solid is dissolved in a minimum volume of a suitable hot solvent and the resulting solution is gradually cooled, saturation and eventual crystallization of the pure compound occurs. Impurities in a solid are of two kinds: soluble and insoluble and recrystallization involves the removal of both to purify a solid. Insoluble impurities are first removed by gravity filtration of the hot solution while the soluble impurities remain dissolved in the cold saturated solution (mother liquor) after precipitation of the desired compound. The pure crystals are separated from the supernatant liquid by suction filtration. After drying, the purity is checked by a melting point determination.

DISTILLATION

In practice, separation of a liquid mixture into its components by a single distillation (simple distillation) is possible only when the boiling points of the components are 80 degrees or more apart. For mixtures of liquids having boiling points much less than 80 degrees apart, separation can be achieved only by fractional distillation. Such a distillation is equivalent to several repeated simple distillations. It uses a fractionating column which provides a large surface area for continuous heat exchange between the hot ascending vapour and the cooler descending liquid, thus resulting in a series of evaporations and condensations leading to separation of the two components. Vacuum distillation is a technique for the distillation of high boiling liquids, and for compounds that decompose at atmospheric pressure. At the low pressures employed, those compounds distil at much lower temperatures.

Steam-distillation

Many water-insoluble compounds, both solid and liquid, may be readily purified by distillation in a current of steam, provided that the compound is volatile, and the impurities non-volatile, under these conditions.

Apparatus

The apparatus consists of a round-bottomed flask having a glass inlet tube which reaches almost to the bottom of the flask and which is connected to a steam generator the flask has also a glass outlet tube, connected in turn to the watercondenser. The flask is clamped at an angle as shown to prevent the solution in round bottom flask from being splashed into the entrance of the outlet tube and thus being blown over mechanically into the condenser: this purpose is achieved more completely if in addition a still-head is used in place of the outlet tube, since spray will be trapped in the still-head, and returned as a steady stream of liquid to the flask. The material to be steam-distilled (mixed with some water if a solid compound, but not otherwise) is placed in flask, and a vigorous current of steam blown in from steam generator. The mixture in flask is thus rapidly heated, and the vapour of the organic compound mixed with steam passes over and is condensed in condenser. For distillations on a small scale it is not necessary to heat the flask if, however, the flask contains a large volume of material or material which requires prolonged distillation, it should be heated by a Bunsen burner, otherwise the steady condensation of steam in flask will produce too great a volume of liquid.

Expt. No.-2**Synthesis of benzil from benzoin**

Aim: To prepare benzil from benzoin.

REQUIREMENTS

Chemicals: Benzoin, conc. HNO_3 , Ethanol etc.

Apparatus: Round bottom flask, Beaker, Pipette, Glass rod, Buchner funnels etc.

PROCEDURE

20 g of benzoin and 100 ml of concentrated nitric acid are taken in a round bottomed flask and heated on a boiling water bath for about 1.5 hour with occasional shaking until the evolution of oxides of nitrogen ceases. The content is poured into 300 – 400 ml ice cold water with shaking. Then the product is filtered with suction and washed with cold water. It is recrystallized from ethanol, taking 2.5 ml per gram; m.p. 94-96 C.

Calculation:

Here limiting reagent is benzoin; hence yield should be calculated from its amount.

Molecular formula of benzoin = $\text{C}_{14}\text{H}_{12}\text{O}_2$

Molecular formula of benzil = $\text{C}_{14}\text{H}_{10}\text{O}_2$

Molecular weight of benzoin = 212 g/mole

Molecular weight of benzilic acid = 210 g/mole

Theoretical yield: 212 g benzoin forms 210 g benzil

Therefore, 20 g benzoin will form $(210/212) \times 20$ g benzil

Hence, Theoretical yield = 19.8 g

Then, Percentage Practical yield = $(\text{Practical yield} / \text{Theoretical yield}) \times 100$.

CONCLUSION

The percentage yield of the benzil is about __%

Expt. No.3

Synthesis of Benzoic Acid from Benzyl Chloride

Aim: To prepare benzoic acid from benzyl chloride.

REQUIREMENTS

Chemicals:

Anhydrous sodium carbonate – 5 g

Potassium permanganate – 10 g

Benzyl chloride – 5 ml

Sodium sulphite – 20g

Conc. HCl – 50 ml

Apparatus: Round-bottomed flask, Reflux condenser set, Beaker, Measuring cylinder, Buchner funnels etc.

PROCEDURE

200 ml of water is taken in a 500 ml flask 5 g of anhydrous sodium carbonate, 10 g of potassium permanganate, and 5 ml (5.5 g) of benzyl chloride are added one by one. The flask is fitted with a reflux water-condenser, and boiled gently for 1 to 1.5 hours to complete the reaction. During this boiling, the permanganate is slowly reduced, and manganese dioxide separates as a dark brown precipitate. Then the flask is cooled, and about 50 ml concentrated hydrochloric acid is added cautiously until the mixture is strongly acidic, and all the benzoic acid is precipitated. Then about 100 ml of 20% aqueous solution of sodium sulphite is added slowly with shaking until the manganese dioxide is completely dissolved and only the white precipitate of benzoic acid remains. The product is filtered at the pump when cold, and washed with water. It is recrystallised from boiling water. Benzoic acid is obtained as colour-less needles, m.p. 121°.

Calculation of yield:

140.57 g of benzyl chloride yield Benzoic Acid = 122.12 g

5.5 g of benzyl chloride shall yield Benzoic Acid = $(122.12 / 140.57) \times 5.5 = 4.78$ g

Hence, Theoretical yield = 04.78 g

Then, Percentage Practical yield = $(\text{Practical yield} / \text{Theoretical yield}) \times 100$.

CONCLUSION

The percentage yield of the benzoic acid is about __% having m.p. 121°C.

Expt. No.4**Synthesis of Phenyl Benzoate from Phenol****Chemicals required:**

Phenol (1 gm.), benzoyl chloride (2 ml)

Procedure: -

Dissolve 1 gm of phenol in 15 ml of 10% sodium hydroxide solution contained in a strong wide-mouthed flask of about 50 ml capacity. Then add 2 ml of benzoyl chloride, the flask securely closed and shake the mixture vigorously for 15 minutes. At the end of this time the reaction should be complete, and a solid product obtained, although a faint smell of unchanged benzoyl chloride may possibly still persist. Filter off the solid ester at the Buchner funnel, breaking up any lumps on the filter with a spatula, and then wash thoroughly with water and drain the filtrate.

Recrystallized the crude ester by methanol

Phenyl benzoate is thus obtained as colourless crystals, m.p. 69°C.

Expt. No.5**Synthesis of Benzanilide from Aniline**

Aim: To prepare Benzanilide from Aniline

REQUIREMENTS

Chemicals: Aniline – 2 ml, 10% NaOH -30 ml, Benzoyl chloride – 3 ml, Cold water, Hot alcohol

Apparatus: Conical flask – 250 ml, Buchner funnel, Measuring cylinder, Filter paper

PROCEDURE

Place 2 ml (2.08 g) of aniline 30 ml of 10% NaOH solution in 250 ml conical flask, then add 3 ml (3.4 g) of benzoyl chloride slowly with vigorous shaking. Cork the flask and shake for further 15-20 min or till the odour of benzoyl chloride can no longer be detected. Dilute the reaction mixture with cold water, filter the crude benzanilide with suction on a Buchner funnel, wash with cold water and crystallize from hot alcohol. Dry the product and calculate the percentage yield.

Calculation:

Here limiting reagent is aniline; hence yield should be calculated from its amount taken.

Molecular formula of aniline = C_6H_7N

Molecular formula of benzanilide = $C_{13}H_{11}ON$

Molecular weight of aniline = 93 g/mole

Molecular weight of benzanilide = 197 g/mole

Theoretical yield:

93 g aniline forms 197 g benzanilide

Therefore, 2.08 g (2 ml) aniline will form? (X) g benzanilide

$X = (197 \times 2.08) / 93 = 4.4 \text{ gm.}$

Theoretical yield = 4.4 gm

Practical yield = _____ gm

% Yield = (Practical Yield) / (Theoretical Yield) \times 100

Expt. No.6**Preparation of 1-Phenylazo-2-Naphthol from Aniline**

Aim - To Prepare and Submit 1-Phenylazo-2-Naphthol from Aniline

REQUIREMENTS

Chemicals: Aniline – 4.0 g conc.

Hydrochloric acid – 12.8 ml

β -Naphthol -6.24 g

10% (w/v) Sodium hydroxide solution – 40 ml

Sodium nitrite – 3.2 g

PROCEDURE

Dissolve 4.0 g (3.92 ml) aniline in 12.8 ml conc. HCl in a 250 ml beaker and dilute it with 12.8 ml distilled water. Cool the in ice-bath with frequent stirring till it attains a temperature below 5°C. In another beaker, dissolve 3.2 g sodium nitrite in 15 ml water and chill the solution in ice-bath (0– 5°C). Add sodium nitrite solution (2) to the aniline solution (1) in small lots (2 ml) at a time in intervals with vigorous stirring with a glass rod taking care that the temperature of the reaction mixture must not exceed beyond 5°C at any cost. After the complete addition of sodium nitrite solution, it is required to test the reaction mixture for the presence of free nitrite by taking out a drop of it and immediately placing it on KI-starch paper that will distinctly turn blue in the presence of free nitrous acid. Dissolve 6.24 g β -naphthol separately in a 250 ml beaker in 40 ml of sodium hydroxide solution, and cool the naphthol-solution in ice-bath (0-5°C). Slowly add the cold diazonium salt solution to the β -naphthol solution with vigorous stirring. Special care must be taken for not allowing the temperature of the reaction mixture rise beyond 5°C. If need be, crushed ice should be added while the coupling-reaction proceeds. A red colour develops and crystals of crude phenylazo- β -naphthol separate out. Allow the reaction mixture to stand for 30-40 minutes with stirring to complete the reaction. Filter the red product in a Büchner funnel using suction, and wash with ice-cold water. Drain the water by pressing with an inverted glass-stopper.

Calculation of Practical Yield: –

93 g of aniline on reacting with 144 g of β -naphthol yields phenylazo- β -naphthol = 258 g

10 g of aniline shall yield phenyl-azo- β -naphthol = $(258 / 93) \times 10 = 27.74$ g

Therefore, Theoretical yield of phenyl-azo- β -naphthol = 27.74 g

If reported Practical yield = X g

Then, Percentage Practical yield = $(\text{Practical yield} / \text{Theoretical yield}) \times 100$

Expt. No.7**Synthesis of benzoic acid from alkyl benzoate**

Aim: To prepare benzoic acid from alkyl benzoate by Hydrolysis reaction

REQUIREMENTS**Chemicals:**

Ethyl benzoate- 2 ml

Sodium hydroxide solution 10% – 15 ml

Hydrochloric acid sufficient

Apparatus: Round bottomed flask, Reflux condenser, Funnel, Measuring cylinder, Beaker

PROCEDURE

A mixture of ethyl benzoate (2 ml) and sodium hydroxide solution (15 ml, 10%) is refluxed in a round bottomed flask fitted with a water condenser on a water bath (temp. 90-100 C) for about 30 minutes till the ester layer disappears. Then the solution is cooled and acidified with HCl. The resultant acidified solution is cooled in an ice bath. The separated benzoic acid precipitate is filtered and recrystallised from hot water.

Melting point: 122 C.

Weight of ethyl benzoate = 150 g/mole

Weight of benzoic acid = 112 g/mole

Theoretical yield:

150 g of ethyl benzoate gives 112 g of benzoic acid

Therefore, 2 g of ethyl benzoate would give $(112/150) \times 2 = 1.49$ g of benzoic acid.

Practical yield:

% yield of benzoic acid = (Practical yield/ Theoretical yield) X 100.

Conclusion

The % yield of benzoic acid is __%

Expt. No.8**Synthesis of Cinnamic Acid from Benzaldehyde**

Aim: To prepare cinnamic acid from benzaldehyde.

REQUIREMENTS**Chemicals:**

Benzaldehyde – 10.5 g
Potassium acetate – 6 g
Acetic Anhydride – 15 g
Sodium carbonate – 20 g
Conc. Hydrochloric Acid – q.s
Rectified Spirit – 50 ml

Apparatus:

Double naked round-bottomed flask with guard tube system, Reflux condenser set, and Another 500 ml RBF with steam distillation system, Beaker, Measuring cylinder, Buchner funnel etc

PROCEDURE

About 10.5 g (10 ml) of benzaldehyde 15 g (14 ml) of acetic anhydride and 6 g of finely powdered potassium acetate are transferred into a dry 250 ml round bottomed flask, provided with CaCl_2 -guard tube at its top-end. Contents of the RB-flask are mixed thoroughly and the reaction mixture is heated in an oil bath at 160°C for 60 minutes; and further at an elevated temperature of $170\text{--}180^\circ\text{C}$ for about 3 hours. While still hot ($90^\circ\text{--}100^\circ\text{C}$) the contents of the flask is poured into a 500 ml round-bottomed flask containing about 50 ml of water, which is fitted for steam-distillation operation. The contents of the flask-1 is rinsed with a little hot water and poured into the flask-2. The resulting solution in the 500 ml RB-flask is made alkaline by adding gradually a saturated solution of Na_2CO_3 with vigorous shaking. The solution is subjected to steam-distillation until all the 'unreacted benzaldehyde' is removed and the distillate is absolutely clear. The contents of the distillation flask is cooled and filtered by suction to get rid of most resinous unwanted by-products. Carefully, the filtrate is rendered to acidic pH by adding concentrated HCl gradually in small lots at intervals, and with continuous shaking until the evolution of CO_2 ceases completely. The resulting solution is chilled, cinnamic acid gets separated as colourless crystals, filtered in the Buchner funnel, washed with a little cold water, drained well and dried at 100°C .

Calculation of yield:

106.12 g of Benzaldehyde yields Cinnamic Acid = 148.16 g

10.5 g of Benzaldehyde shall yield Cinnamic Acid =

$$(148.16/106.12) \times 10.5 = 14.66 \text{ g}$$

Hence, Theoretical yield of Cinnamic Acid = 14.66 g

If reported Practical yield = X g

Then, Percentage Practical yield = (Practical yield / Theoretical yield) \times 100

CONCLUSION

The percentage yield of the Cinnamic acid is about ___ % of melting point

132 °C

Expt. No.9**Synthesis of Dibenzal Acetone from Benzaldehyde by Claisen Schmidt Reaction**

Aim: To prepare dibenzal acetone from benzaldehyde.

REQUIREMENTS**Chemicals:**

Benzaldehyde – 10 ml

Acetone – 4 ml

Methylated spirit – 10 ml

10% aqueous sodium hydroxide solution- 2 ml

Apparatus:

Conical flask or wide mouthed bottle, Beaker, Measuring cylinder, Buchner funnel etc

PROCEDURE

About 10 ml (10.4 gm) of benzaldehyde and 4 ml of pure acetone is thoroughly mixed with 10 ml of methylated spirit in a conical flask or wide mouthed bottle. 2 ml of 10% aqueous sodium hydroxide solution is diluted with 8 ml of water, and this dilute alkali solution is added to the former solution. The mixture is shaken vigorously in the securely corked flask for about 10 minutes (releasing the pressure from time to time if necessary) and then allowed to stand for 30 minutes, with occasional shaking and finally cooled in ice-water for a few minutes. During the shaking, the dibenzal-acetone separates at first as a fine emulsion which then rapidly forms pale yellow crystals. It is filtered at the pump, washed well with water to eliminate traces of alkali, and then drained thoroughly. The product is recrystallised from hot methylated or rectified spirit.

Calculation of yield:

212 g of benzaldehyde yields dibenzal-acetone = 234 g

10.4 g of benzaldehyde shall yield dibenzal-acetone = $(234 / 212) \times$

10.4 = 11.5 g

Therefore, Theoretical yield of dibenzal-acetone = 11.5 g

If reported Practical yield = X g

Then, Percentage Practical yield = $(\text{Practical yield} / \text{Theoretical yield}) \times 100$

CONCLUSION

The percent yield of dibenzal-acetone is ____ % obtained as pale yellow crystals, m.p. 112°.

Expt. No.10

Synthesis of m-dinitrobenzene from nitrobenzene

Aim: To prepare m-dinitrobenzene from nitrobenzene by Nitration reaction

REQUIREMENTS

Chemicals:

Fuming nitric acid- 15 ml

Conc. sulphuric acid- 20 ml

Nitrobenzene- 12.5 ml

Apparatus: Round bottomed flask, Reflux condenser, Funnel, Measuring cylinder, Beaker.

PROCEDURE

21 ml (37.5 g) of concentrated sulphuric acid and 15 ml (22.5 g) of fuming nitric acid are placed in a 250 ml RBF with a few smaller pieces of porcelain. A reflux condenser is attached and the apparatus is placed in a fuming cupboard. 12.5 ml (15 g) of nitrobenzene in portions of about 3 ml is added slowly and after each addition the flask is shaken thoroughly. The mixture is heated on water bath for 30 min. with shaking. Then it is allowed to cool and poured into about 500 ml of cold water cautiously with vigorous stirring. The dinitrobenzene soon solidifies. The product is filtered, washed with cold water and allowed to drain as much as possible. The product is transferred to a 250 ml flask fitted with a reflux condenser; 80-100 ml of rectified spirit is added and heated on a water bath to dissolve the entire solid. If the solution is not clear, it is filtered using a warm Buchner funnel. About 15 g of m-dinitrobenzene is deposited on cooling as colourless crystal. A second recrystallisation is, usually necessary in order to eliminate traces of o- and p-dinitrobenzene, and thus pure m-dinitrobenzene, m.p. 90° is obtained.

Calculation

Molecular formula of nitrobenzene = $C_6H_5NO_2$

Molecular formula of m-dinitrobenzene = $C_6H_4N_2O_4$

Weight of nitrobenzene = 123 g/mole

Weight of m-dinitrobenzene = 168 g/mole

Theoretical yield

123 g of nitrobenzene gives 168 g of m-dinitrobenzene

Therefore, 15 g of nitrobenzene should give.....? (X) g of m-dinitrobenzene

CONCLUSION

The yield of synthesized m-dinitrobenzene was found to be __ %.

Expt. No.-11**Synthesis of 2, 4, 6-tribromoaniline from aniline**

Aim: To prepare 2, 4, 6-tribromoaniline from aniline.

REQUIREMENTS**Chemicals:**

Aniline – 4 ml

Bromine – 6.4 ml

Dilute hydrochloric acid,

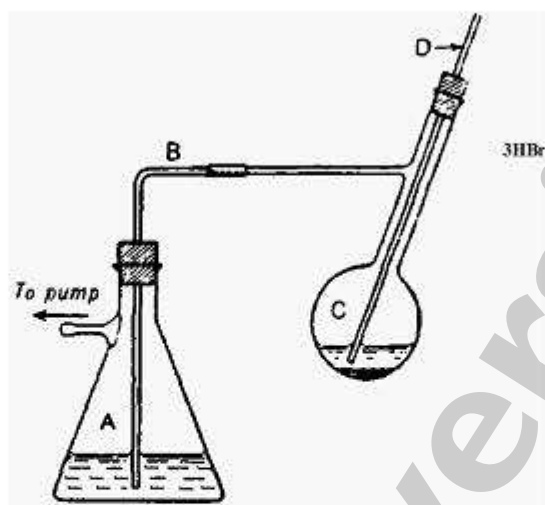
Rectified spirit

Apparatus: Buchner flask set as shown in the figure, Beaker, Filtration set etc.

PROCEDURE**Method: 1**

The apparatus is assembled as shown in fig. A 500 ml. Buchner flask A is fitted with a cork through which passes a glass delivery tube B reaching nearly to the bottom of A. B is then connected by a short piece of rubber tubing to the side-arm of a 150 ml. distilling-flask C, care being taken to ensure that the two glass tubes touch one another inside the rubber connection. A narrow glass tube D is then fitted as shown so that it reaches within 2-3 cm. of the bottom of C.

4 ml aniline is dissolved in 10 ml. of dilute hydrochloric acid in A, and diluted with 200 ml. of water. 6.4 ml. of bromine is placed in C carefully, and it is covered with about 40 ml. of cold water. The position of the tube D is adjusted until it touches the surface of the bromine layer. A is connected to a suction water-pump, so that a steady stream of bromine vapour carries from C into A, where the greyish-white tribromoaniline soon begins to separate. The contents of A are shaken occasionally for even distribution of tribromoaniline. When the water in C becomes almost colourless (about 40 minutes), due to evaporation of bromine, the current of air is stopped, and tribromoaniline is filtered at pump, washed well with water and drained. The product is recrystallised from rectified spirit, using animal charcoal.



Method: 2

A solution is prepared by adding 8.4 ml of bromine in 20 ml glacial acetic acid. Another second solution is prepared adding 5 ml of aniline in 20 ml of glacial acetic acid in a conical flask. The first solution is added drop wise to the second solution from a dropping funnel. During addition, the flask is shaken time to time and cooled in ice. A yellow coloured mass is obtained. It is poured into excess of water. The separated product is filtered, washed with water and recrystallised from rectified spirit.

Calculation of Yield:

93 g of Aniline with 3x159 g of Bromine yields Tribromoaniline = 329.8 g

4.1 g of Aniline shall yield = $(329.8/93) \times 4.1 = 14.54$ g Tribromoaniline
Hence, theoretical yield = 14.54 g.

If reported Practical yield is X g, then, % yield = $(X/14.54) \times 100$.

CONCLUSION

The synthesized 2,4,6-tribromoaniline was obtained as colourless crystals, % yield is and of m.p. 120°.

Expt. No.12**Synthesis of p-bromoacetanilide from acetanilide**

Aim: To prepare p-bromoacetanilide from acetanilide by Bromination reaction

REQUIREMENTS**Chemicals:**

Acetanilide (10 g)
Glacial acetic acid (70 ml)
Bromine (4.2 ml)
Sodium bisulphite (sufficient quantity)
Rectified spirit

Apparatus: Conical flask, Funnel, Beaker, Filter paper, Burette/separating funnel etc.

PROCEDURE

10 g of acetanilide is dissolved in 45 ml glacial acetic acid in a 250 ml conical flask and cooled to below 5 degrees. Then 4.2 ml of bromine is added drop wise in to 25 ml cold acetic acid with constant stirring and the bromine solution is transferred to a burette/separating funnel supported over the flask. The bromine solution is added slowly with constant stirring to acetanilide solution and the flask is placed in cold water as the reaction is exothermic. When addition of all the bromine is complete the solution turns orange due to the presence of slight excess of bromine, then allowed to stay at room temperature for 30 min. Contents of the flask are poured directly into a beaker having 200 ml ice cold water. The conical flask is further rinsed with 50 ml cold water and transferred into the beaker with stirring. Here parabromo acetanilide separates as a white solid. If the colour of the solution is persistently yellow, about 4-5 g of sodium bisulphite is added with constant stirring to bleach coloration. The crude product is filtered with suction, the residue washed with cold water, recrystallized from rectified spirit, dried in an oven at 100 °C and the percentage yield is calculated. The p-bromo acetanilide is obtained as colourless crystals, m.p. 167°C.

Calculation

Molecular formula of acetanilide = C_8H_9NO

Molecular formula of p-bromo acetanilide = C_8H_8NOBr

Weight of acetanilide = 135 g/mole

Weight of p-bromo acetanilide = 214 g/mole

Theoretical yield

135 g of acetanilide gives 214 g of p-bromo acetanilide

Therefore, 10 g of acetanilide would give $(214/135) \times 10 \text{ g} = 15.85 \text{ g}$ of p-bromo acetanilide.

Practical yield

% of p-bromo acetanilide is = $(\text{Practical yield} / \text{Theoretical yield}) \times 100$

CONCLUSION

The yield of synthesized p-bromo acetanilide was found to be __ %

Expt. No. 13.**Determination of Acid value of given oil/fat**

Aim: To determine acid value of given oil/fat

REQUIREMENTS

Ether, 0.1 M potassium hydroxide, Phenolphthalein

Apparatus: Volumetric flask, Pipette, Burette

PROCEDURE

About 10 g (w) of the sample is dissolved, in 50 ml of a mixture of equal volumes of ethanol (95%) and ether, previously neutralized with 0.1 M potassium hydroxide to phenolphthalein solution. If the sample does not dissolve in the cold solvent, the flask is connected with a reflux condenser and slowly warmed, with frequent shaking, until the sample dissolves. 1 ml of phenolphthalein solution is added and titrated with 0.1 M potassium hydroxide until the solution remains faintly pink after shaking for 30 seconds.

Expt. No. 14.**Determination of Iodine value of the given oil or fat**

Aim: To determine Iodine value of the sample (oil or fat)

REQUIREMENTS

Chemical: Carbon tetrachloride, Iodine mono-chloride, 0.1 M sodium thiosulphate, Starch solution.

Apparatus: Volumetric flask, Pipette, Burette

PROCEDURE

(Iodine Mono-chloride (ICl) Method or Wijs Method)

An accurately weighed quantity (w g) of the sample (oil or fat) is taken in a 500 ml iodine flask. 10 ml of carbon tetrachloride (CCl_4) is added to dissolve it. 20 ml of iodine mono-chloride (ICl) solution is added. The mix solution is allowed to stand in the dark at a temperature between $15^\circ - 25^\circ \text{C}$ for 30 minutes, inserting the stopper. Then 15 ml of potassium iodide solution is added. The flask and the stopper are ringed with 100 ml of water, shaken and titrated with 0.1 M sodium thiosulphate solution using freshly prepared starch solution as indicator, which is added towards the end of the titration. The number of ml required is noted as (a). The procedure is repeated without the sample and the number of ml required is noted as (b). Iodine value is calculated from the observed data.

Expt. No. 15.

Determination of Saponification value of the given oil/fat

Aim: To determine saponification value of the given oil/fat

REQUIREMENTS

Sample: Oil or fat

Apparatus: Conical flask

Chemical: 0.5 N HCl, Reflux and condenser, Water bath, Burette and Pipette, Phenolphthalein, 0.5 N alcoholic potassium-hydroxide solutions

CONCLUSION

About 2 g of the given oil or fat is taken in a conical flask but weighed accurately (w g). The oil/fat is dissolved in 25 ml of 0.5 N alcoholic potassium-hydroxide solutions. Then the reaction mixture is refluxed using a water condenser on a water-bath for half an hour. The resulting solution is cooled and titrated against a 0.5 N HCl solution adding 1 ml of phenolphthalein. The number of ml of acid required is noted (a). An exactly identical blank experiment (leaving the fat or oil) is performed. Number of ml of hydrochloric acid required is noted (b).