



S.A.RAJA PHARMACY COLLEGE

VADAKKANGULAM 627 116

SUBJECT:

**PHARMACEUTICAL ORGANIC
CHEMSITRY**

III SEMESTER B.PHARM

PRACTICAL LAB MANUAL

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RECRYSTALLISATION

Recrystallisation is a very important technique often employed for the purification of solids. It is based on the fact that all organic solids are more soluble in hot than in cold solvent, so that the solid dissolved on heating can be obtained back on cooling.

The following steps should be performed for purification by crystallization.

1. Selection of a solvent or a mixture of solvents.
2. Dissolution of the solute in required amount of the solvent
3. Decolorisation for removal of colored impurities
4. Crystallization, filtration and drying of the solid.

Selection of a solvent

The solvent chosen for recrystallisation should be such that the solid to be purified must be insoluble or nearly insoluble in the cold and fairly soluble in the hot solvent. The boiling point of the solvent should be lower than the melting point of the solid so that it can be removed during the drying of the solid.

The rule of like dissolves like should be kept in mind while selecting a solvent. Water, 95% ethanol, methanol, petroleum ether, THF, CCl₄, ethyl acetate, acetone and acetic acid are some of the common solvents employed for recrystallisation.

Procedure

for the selection of the solvent, take about 20- 30 mg of the solid in a clean dry tube and add about 0.5- 1 ml of the solvent, stir well, if the solid is soluble at room temperature, the solvent can't be used for crystallization. If the solid is insoluble, heat the test tube in a hot waterbath or directly on a flame. If the solid dissolves in the boiling solvent, filter and then cool the solution. If crystals appear on cooling, the solvent is suitable for recrystallisation.

If a pure (single) solvent is found unsuitable, a mixture of solvents may be used, the two solvents should be miscible with each other. Eg: water- ethanol, ethyl acetate- benzene.

Dissolve the solute in the hot solvent (A) in which it is freely soluble and add slowly the solvent (B) in which the compound is insoluble till a turbid solution is obtained. Add dropwise the solvent (A) to get back a clear solution and allow the solution to cool undisturbed for the separation of crystals.

Dissolution of the solute in required amount of the solvent

After selecting a suitable solvent for crystallization, take the impure solid in a clean test tube or a conical flask. Add few milliliters of the solvent and heat the solution on a water bath

with continuous stirring or swirling (do not heat inflammable liquids directly on a flame). If the solid does not dissolve, add a few more milliliters of the solvent and heat again (do not add excess of solvent). If near the end of dissolution process, it appears that an additional amount of the solvent is not dissolving any more of the solid. Do not add any more solvent but filter the hot solution through a fluted filter paper and allow the filtrate to stand undisturbed for crystallization.

Decolorisation

If the resultant solution is highly colored, add a small amount of charcoal, boil the solution for some more time (the colored impurities are adsorbed on the surface of charcoal), filter the hot solution through a fluted filter paper and allow the filtrate to cool for the separation of crystals.

Crystallisation

Do not cool the solution obtained above rapidly, allow it to slowly cool to room temperature. If no crystals form, cool the solution in an ice bath. Scratch the sides of the tube with glass rod, just above the surface of the solution to initiate crystallization or add a very small amount of solid compound to induce crystallization or add a very small amount of solid compound to induce crystal formation.

If an oil separates on cooling (1) either scratch it against the sides of the tube to solidify, (2) add a few drop of the solvent, heat, allow to solidify, or (3) add a few crystals of the solute to initiate the crystal formation.

Filtration, drying etc.

Filter the crystals by vacuum filtration and collect the mother liquor in a clean dry flask or a filter tube. Cool the mother liquor in an ice bath or concentrate and cool to obtain the second crop of the crystals. Do not mix with the first crop until their purity has been confirmed by melting point determination. Transfer the crystals from the filter funnel on to a filter paper and air dry or in an air oven maintained at a much lower temperature than the melting point of the solid or by keeping in a vacuum desiccator.

REFERENCE

1. Comprehensive Practical Organic Chemistry – Preparation and Qualitative Analysis; V K Ahluvalia and Renu Aggarwal; page no: 47-48

EXTRACTION OF EUGENOL FROM CLOVE BUDS BY STEAM DISTILLATION

Aim: To carry out the extraction of eugenol from clove buds by steam distillation.

Reference:

1. Natural products chemistry practical manual, Anees A Siddiqui and Seemi Siddiqui
pg no: 193-195

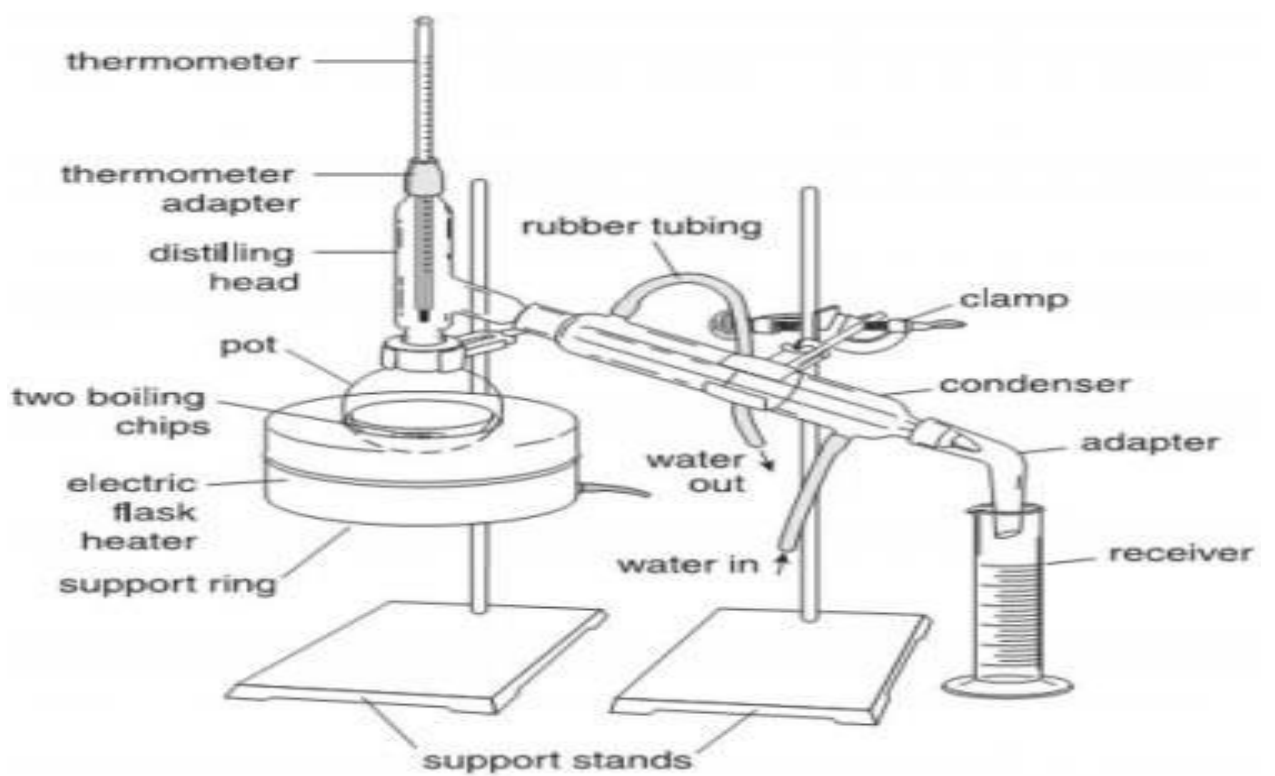
Requirements: clove buds, water

Apparatus: 100 ml round bottom flask, Distillation head, Heating mantle, Thermometer, Condenser, Adapter, Rubber tubings

Principle:

Steam distillation is used in the extraction of Essential Oil from the plant material. It is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The fundamental nature of steam distillation is that it enables a compound or mixture of compounds to be distilled at a temperature substantially below that of the boiling point(s) of the individual constituent(s).

Clove oil belongs to the essential oils. Many of these compounds are used as flavorings and perfumes and, in the past, were considered to be the “essence” of the plant from which they were derived. Steam distillation of freshly ground cloves results in clove oil, which consists of several compounds. Eugenol is the major compound, comprising 85-90 %. Eugenol acetate comprises 9-10 %.



Distillation set up for clove oil extraction

PROCEDURE:

Weigh 5.00 g of freshly ground cloves to the 100 ml round bottom flask (distillation flask). Add 50 ml of distilled water and couple of boiling chips to the above distillation flask. Set up the distillation apparatus according to Figure 3 using 50 ml measuring cylinder as the receiving flask. Start the flow of water through the condenser according to the indicated direction in Figure. Turn on the heating and continue the distillation until little liquid (less than 5 ml) remains in the distillation flask. If foaming occurs reduce the heat. Continue heating until 35 ml distillate has been collected. After the distillation, cool the distillate up to room temperature. Transfer the distillate quantitatively to the separatory funnel. Shake the contents and allow the organic and aqueous layers to separate. Carefully transfer the organic layer in to a pre weighed container. Measure the weight of the container with clove oil.

REPORT

Clove oil is isolated from clove buds by steam distillation.

ANALYSIS OF OIL

Experiment No:

Date:

DETERMINATION OF ACID VALUE

AIM

To determine the acid value of the given sample of oil and report

REQUIREMENTS

APPARATUS: Weight box, weighing bottle, measuring cylinder, burette, conical flask, pipette.

CHEMICALS: Oil sample, alcohol, ether, 0.1N KOH, phenolphthalein

REFERENCE

2. Indian pharmacopoeia 1996; vol. II; page no. A-212
3. Practical pharmaceutical chemistry IVth edition; Part I by A. H Beckett, J. B Stenlake; Page no 146-147.
4. Practical pharmaceutical chemistry by Harkishan Singh and V. K. Kapoor Page no. 182-183.

PRINCIPLE

Acid value is the number of mg of KOH required to neutralize the free acid present in 1g of fat /oil.

Acid value is determined by dissolving the sample in ether-alcohol mixture and titrating it with 0.1N KOH using phenolphthalein indicator. High acid value indicates that the given sample is rancified.

Rancidity: On long exposure to air and moisture, fats and oils develops a disagreeable smell and taste. This is termed as rancidity.

Acid value is useful for determining the freshness, adultration, rancidity, purity of oil/fat

PROCEDURE

Standardization of 0.1N KOH

Dissolve about 6g of potassium hydroxide in sufficient carbon dioxide free water to produce 1000 ml. standardize the solution in the following manner.

Titrate 20 ml of the solution with 0.1 N hydrochloric acid using 0.5 ml phenolphthalein as indicator.

Determination of acid value

Weigh accurately about 5g of sample into a clean 250ml conical flask and add 25ml alcohol ether mixture (1:1) which has been previously neutralized with 0.1N KOH to phenolphthalein indicator. If the sample doesn't dissolve in the solvent reflux for 10minutes with frequent shaking, in a water bath. Add 1ml phenolphthalein indicator and titrate with 0.1N KOH until the solution becomes pink. Calculate acid value using the formula

$$\text{Acid value} = N/W \times 5.61$$

Where, N = No. of ml of 0.1N KOH required

W = Weight of sample in gm.

REPORT

The acid value of the given sample was found to be

Experiment No:

Date:

DETERMINATION OF SAPONIFICATION VALUE

AIM

To determine the saponification value of the given sample of oil and report.

REQUIREMENTS

APPARATUS: Weight box, weighing bottle, measuring cylinder, burette, conical flask, pipette, round bottom flask, condenser.

CHEMICALS: Oil sample, alcoholic potassium hydroxide, 0.5N HCl, phenolphthalein.

REFERENCE

1. Indian pharmacopoeia 1996; vol. II; page no. A-50
2. Practical pharmaceutical chemistry IVth edition; Part I by A. H Beckett, J. B Stenlake; Page no 158.
3. Natural products chemistry practical manual, Anees A Siddiqui and Seemi Siddiqui pg no:39-40

PRINCIPLE

Saponification value is the number of milligram of potassium hydroxide required to neutralize the fatty acid resulting from the complete hydrolysis of 1g of oil or fat. It is determined by boiling a weighed amount of substance with a measured volume of standard alcoholic KOH and back titrating with 0.5M HCl.

Saponification value is a measure of the size of the fat molecule or the size or molecular weight of fatty acids in the fat. It also indicates the quantity of alkali which must be used to convert a blend of fats to soap. Saponification value is also useful for detecting adulteration of a given fat by one of the higher or lower saponification value.

Some average accepted saponification values for common oils:

Olive oil =185-196

Linseed oil =192-195

Coconut oil= 246-260

Palm oil= 242-250

PROCEDURE

Standardization of 0.5M HCl

Weigh accurately about 0.75g anhydrous sodium carbonate, previously heated at about 270°C for 1 hour. Dissolve it in 100ml of water and add 0.1ml of methyl red solution. Add the acid slowly from the burette with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool and continue the titration. Heat again to boiling and titrate further as necessary until the faint pink colour is no longer affected by continued boiling.

Determination of saponification value

Weigh accurately about 2g of sample into a clean 250ml round bottom flask. To this add 25ml alcoholic potassium hydroxide and reflux for 30 minutes. Cool and add 1ml phenolphthalein indicator. Immediately titrate with 0.5M HCl. End point is the appearance of white colour. This titre value can be considered as 'a'. Repeat the operation omitting the substance being examined. The titre value obtained can be considered as 'b'.

Each ml of 0.5M HCl \equiv 28.05g of KOH

Calculate saponification value using the formula

Saponification value = $[(b - a) \times \text{equivalent weight factor}] / W$

Where, W = Weight of oil.

REPORT

The saponification value of the given sample was found to be

Experiment No:

Date:

DETERMINATION OF IODINE VALUE

AIM

To determine the Iodine value of the given sample of oil and report.

REQUIREMENTS

APPARATUS: Weight box, weighing bottle, measuring cylinder, burette, conical flask, pipette, round bottom flask, condenser.

CHEMICALS: Oil sample, carbon tetra chloride, iodine monochloride solution, potassium iodide solution, 0.1 M sodium thiosulphate, starch solution

REFERENCE

1. Indian pharmacopoeia 2010, volume 1 page no: 86
2. Practical pharmaceutical chemistry IVth edition; Part I by A. H Beckett, J. B Stenlake; Page no 158.
3. Natural products chemistry practical manual, Anees A Siddiqui and Seemi Siddiqui pg no:41-43

PRINCIPLE

The iodine value is the number which express in grams the quantity of halogen, calculated as iodine, which is absorbed by 100g of substance under the described conditions. It may be determined by three methods.

1. Iodine monochloride method or Wijs method.
2. Iodine monobromide method or Hanus method
3. Pyridine monobromide method

Iodine value give indication of proportion of unsaturated acid present in fat or oils. If the fatty acid is more unsaturated it will give a high iodine value. Solid fat have low iodine value. Non drying oils have iodine value of 80-100.; whereas drying oil have high iodine value.

Iodine value of some oils;

Castor oil -82-90

Olive oil- 79-88

Cod liver oil- 145-180

Linseed oil 175-200

PROCEDURE:

Standardisation of 0.1 M sodium thiosulphate

Dissolve accurately weighed potassium dichromate in 25 ml of water in a 25l of erlenmayer flask. Add 10 ml of hydrochloric acid and 2 g of potassium iodide, stopper, shake and keep dark for 15 minutes. Add 100ml of water to the above mixture and titrate with sodium thiosulphate using starch as indicator. Near end point the color will be changed from dark blue to bottle green. Each ml of 0.1 M sodium thiosulphate is equivalent to 0.04904g of potassium dichromate.

Iodine monochloride method or Wijs method.

Place an accurately weighed quantity of the substance in a dry 500ml iodine flask, add 10 ml carbon tetrachloride and dissolve. Add 20 ml iodine monochloride solution, insert the stopper and allow to stand in the dark at a temperature between 15- 25 for 30 minutes. Place 15 ml potassium iodide solution in the cup top, carefully remove the stopper, rinse the stopper and sides of the flask with 100 ml of water, shake and titrate with 0.1 M sodium thiosulphate using starch solution, added towards the end of the titration, as indicator. Note the no. of ml required (a). Repeat the operation without the substance under examination and note the no of ml required (b).

Calculate the iodine value from the expression

$$\text{Iodine value} = 1.269(b-a)/w$$

Where w = weight in gm of the substance.

REPORT

The iodine value of the given sample was found to be

PREPARATIONS

Expt No:

Date:

PREPARATION OF PHENYL BENZOATE

AIM:

To prepare and submit phenyl benzoate and to find out its percentage yield.

REFERENCES:

1. Practical organic chemistry; F.G.Mann and B.C.Saunders;4th edition; Page no:243-244.
2. Elementary practical organic chemistry. Part-I: Small scale preparations; Arthur.I. Vogel; 2nd edition; Page no:347.

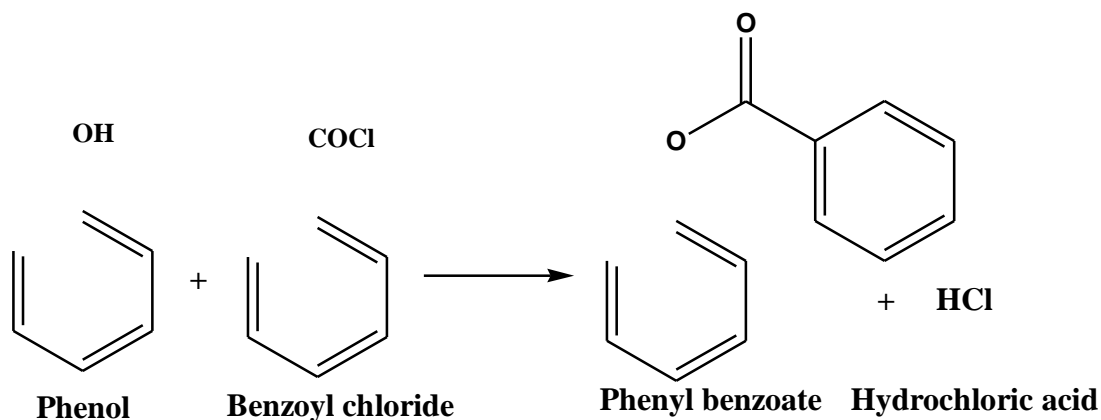
REQUIREMENTS:

Reagents: Phenol-1g, Benzoyl chloride-2ml, Sodium hydroxide (10%)-15ml.

Apparatus: Iodine flask /Erlen Meyer flask with a cork, measuring cylinder, funnel, filter paper, glass rod etc.

PRINCIPLE:

Phenyl benzoate is prepared by Schotten- Baumann method of benzoylation. In this method of benzoylation phenols are reacted with benzoyl chloride in presence of aqueous sodium hydroxide and the reaction mixture is shaken vigorously. Benzoylation proceeds smoothly under these conditions and the solid benzoyl compound being insoluble in water separates out. Phenols when subjected to the Schotten- Baumann benzoylation, first dissolves in sodium hydroxide to give sodium phenoxide, which then undergoes benzoylation to give phenyl benzoate.



PROCEDURE:

Dissolve phenol in 10% sodium hydroxide solution contained in an Iodine flask or Erlen Meyer flask with a cork of about 250ml capacity. Then add benzoyl chloride, cork the flask securely and shake the mixture vigorously for 15 minutes. The solid product obtained is filtered off, washed with water, dried and submitted.

USES:

1. Phenyl benzoate can be used in a variety of polyesters.
2. As a starting material for the production of optical components, particularly high quality lenses for still and motion picture cameras.

REPORT:

Phenyl benzoate is prepared from phenol. The product is dried and submitted.

Practical yield of phenyl benzoate is found to be = g

Theoretical yield of phenyl benzoate is found to be = g

Percentage yield of phenyl benzoate is found to be= % w/w.

Expt No:

Date:

PREPARATION OF BENZANILIDE

AIM:

To prepare and submit benzanilide and to find out its percentage yield.

REFERENCES:

1. Practical organic chemistry; F.G.Mann and B.C.Saunders;4th edition; Page no:243-246.
2. Elementary Practical Organic chemistry. Part-I: Small Scale Preparations; Arthur .I. Vogel; 2nd edition; Page no: 269-270.

REQUIREMENTS:

Reagents: Aniline-1ml, Benzoyl chloride-1.5ml, Sodium hydroxide(10%)-15ml.

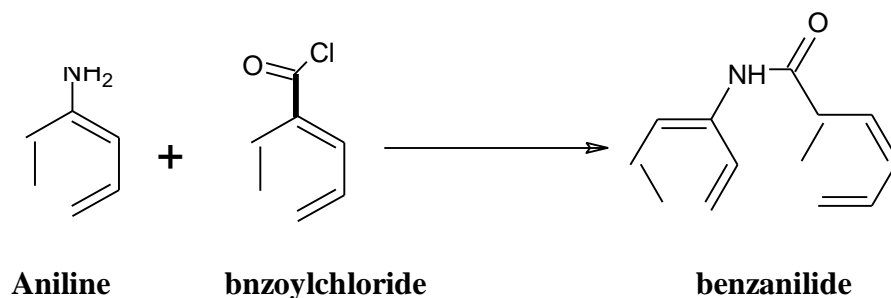
Apparatus: Erlen Meyer flask with a cork/Iodine flask, beaker, measuring cylinder, funnel, filter paper, glass rod etc.

PRINCIPLE:

The substitution of hydrogen atom of –OH, -NH₂ or >NH group by benzoyl group is known as Schotten Baumann reaction of Benzoylation. Benzoylation is usually preferred to acetylation because of following two facts.

1. Since acetic anhydride and acetyl chloride are very readily hydrolysed by water, acetylation is usually carried out in the absence of water, while benzoylation may be achieved even in aqueous solution.
2. Benzoyl derivatives are insoluble in water and very much less soluble in other solvents than the corresponding acetyl derivatives.

In this method benzoylation of amine is carried out with benzoyl chloride in presence of aqueous sodium hydroxide. Benzoylation proceeds smoothly with formation of sparingly soluble derivative which is usually insoluble in water gets separated out. Sodium hydroxide hydrolyses the excess of benzoyl chloride yielding sodium benzoate and sodium chloride which remains in the solution. Aniline when subjected to the Schotten-Baumann reaction undergoes benzoylation to give benzoyl aniline or phenyl benzamide.



PROCEDURE:

Dissolve aniline (1ml) in 10% sodium solution hydroxide (1.5ml) contained in an Erlen Meyer flask/Iodine flask with a cork of about 250ml capacity. Then add benzoyl chloride cork the flask securely and shake the mixture vigorously for 10-15 minutes. The solid product obtained was filtered off, washed with water, dried and submitted.

USES:

Benzanilide is used in the manufacture of dyes and perfumes.

REPORT:

Benzanilide is prepared from aniline. The product is dried and submitted.

Practical yield of Benzanilide is found to be = g

Theoretical yield of Benzanilide is found to be = g

Percentage yield of Benzanilide is found to be = % w/w

Expt No:

Date:

PREPARATION OF p-BROMO ACETANILIDE

AIM:

To prepare and submit p-bromo acetanilide, and to find out its percentage yield.

REFERENCES:

1. Practical Organic Chemistry; F G Mann and B C Saunders; 4th edition; page no. 166-167.
2. Elementary Practical Organic Chemistry. Part 1: Small scale preparation; Arthur I. Vogel; 2nd edition; page no: 267.

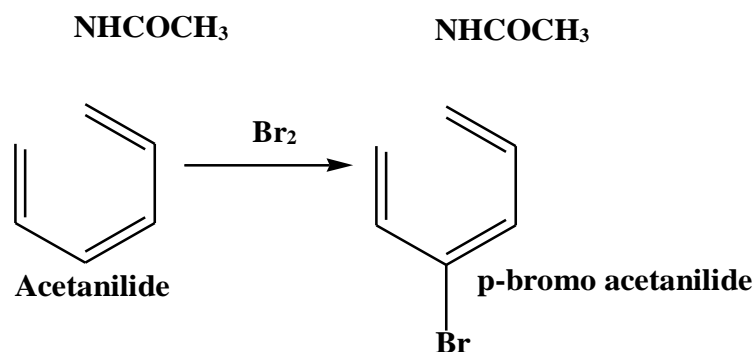
REQUIREMENTS:

Reagents: Acetanilide-1g, glacial acetic acid-1ml, bromine-0.42ml.

Apparatus: Conical flask, beaker, measuring cylinder, funnel, filter paper, glass rod etc.

PRINCIPLE:

Mono-substitution products of primary amines cannot be prepared by direct action of appropriate reagent; it is necessary to protect the amino group. Acetanilide undergoes ready bromination with bromine, giving chiefly p-bromo acetanilide.



PROCEDURE:

Dissolve finely powdered acetanilide in 5ml of cold glacial acetic acid contained in a 250ml conical flask. Prepare a solution of 0.42ml of bromine in 6ml of glacial acetic acid and add slowly to the acetanilide solution with shaking. Mix well and allow to stand for 15 minutes at room temperature. Pour the contents into a beaker containing 60 ml of cold water. Stir the mixture well to eliminate acetic acid, unchanged bromine etc. Filter off the precipitated p-bromo acetanilide washed with cold water to remove acid dried and submitted.

USES:

p-bromo acetanilide is used as an analgesic and antipyretic.

REPORT:

p-Bromo acetanilide is prepared from acetanilide. The product is dried and submitted.

Practical yield of p-bromo acetanilide is found to be = g

Theoretical yield of p-bromo acetanilide is found to be = g

Percentage yield of p-bromo acetanilide is found to be = % w/w.

Expt No:

Date:

PREPARATION OF NITRO BENZENE

AIM:

To prepare and submit Nitro benzene from benzene .

REFERENCE:

1. Advanced practical organic chemistry by O P Agarwal pg no: 313-314.
2. Practical Organic Chemistry; F G Mann and B C Saunders; 4th edition; page no. 157-158

REQUIREMENTS:

Reagents: Nitrobenzene, Conc. Nitric acid, Conc. Sulphuric acid.

Apparatus: RB flask, reflux condenser, waterbath.

PRINCIPLE:

The replacement of active hydrogen atom by nitro group is known as nitration. It is a characteristic reactions of aromatic compounds. Nitration is usually carried out by conc. Nitric acid and conc. Sulphuric acid; although it may also be affected by nitric acid alone. The function of sulphuric acid is to produce the more nitrating species. The number of nitro group introduced in the nucleus depends upon the temperature of the experiment and nature of the compound to be nitrated.

PROCEDURE:

Place 3.5 ml conc. Nitric acid in a 50 ml RB flask and add slowly with cooling 4ml of conc. Sulphuric acid. Add slowly from a burette 2.9 ml of benzene , shaking the mixture well during the addition; the temperature must be kept at 45-50⁰C. fit the water condenser to the flask and heat in a waterbath maintained at 60oc. For 10 minutes. During this period, shake the flask from time to time and pour the mixture in to 30 ml of cold water. Use only 2-3 pieces of granular calcium chloride, and dry for 10 minutes. If a clear liquid is obtained, omit the filtration, and decant the solution.

USES: Synthetic intermediate

REPORT:

Nitro benzene is prepared, dried and submitted.

Practical yield of Nitro benzene is found to be = g

Theoretical yield of Nitro benzene is found to be = g

Percentage yield of Nitro benzene is found to be = %w/w

Expt No:

Date:

PREPARATION OF BENZOIC ACID

AIM:

To prepare and submit Benzoic acid and to find out its percentage yield.

REFERENCE:

1. Practical Organic Chemistry; F.G.Mann and B.C.Saunders; 4th edition; Page no: .
2. Comprehensive Practical Organic Chemistry-Preparation and Qualitative analysis; V.K.Ahulwalia and Renu Agarwal; Page no: 3-4.
3. Advanced practical organic chemistry; O.P Agarwal, page no: 321

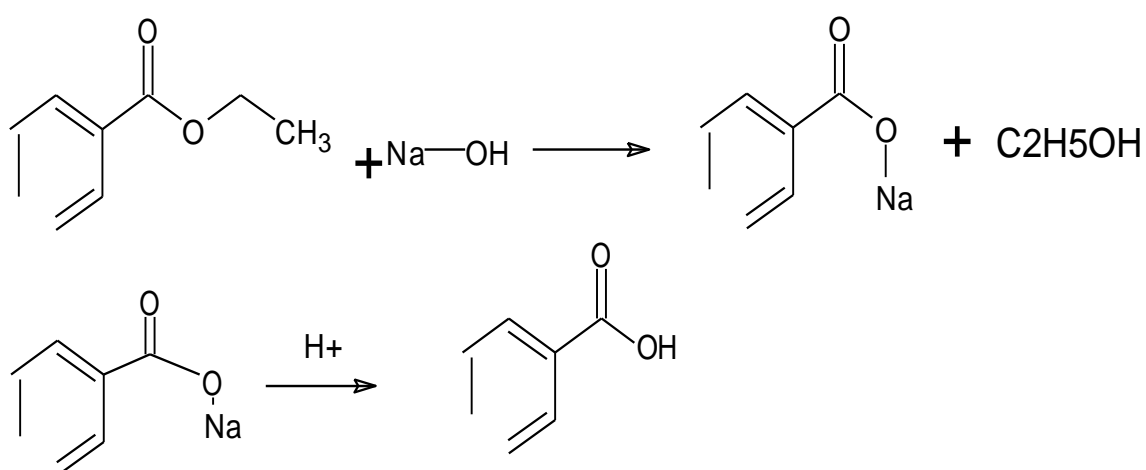
REQUIREMENTS:

Reagents: Ethyl benzoate, Hydrochloric acid, sodium hydroxide(10%)

Apparatus: R.B flask, measuring cylinder, beaker, funnel, filter paper, glass rod etc.

PRINCIPLE:

Esters are hydrolysed either by acid or base. Alkaline hydrolysis of ester is irreversible, which is also called saponification. Acid hydrolysis of ester is a reversible reaction. The alkaline hydrolysis, which occurs through nucleophilic acyl substitution. Here ethyl benzoate on hydrolysis with sodium hydroxide gives benzoic acid and ethyl alcohol where OH⁻ ion of sodium hydroxide acts as a nucleophile.



PROCEDURE:

A mixture of ethyl benzoate (2ml) and sodium hydroxide solution (15 ml, 10%) is refluxed in a round bottom flask fitted with a water condenser on a water bath (90-100⁰ C) for about 30 minutes till ester layer disappears. Then the solution is cooled and acidified with hydrochloric acid. The resultant acidified solution is cooled in an ice bath. The separated benzoic acid is filtered and recrystallized in hot water.

USES:

Benzoic acid is used as food preservatives

REPORT:

Benzoic acid is prepared, dried and submitted.

Practical yield of Benzoic acid is found to be = g

Theoretical yield of Benzoic acid is found to be = g

Percentage yield of Benzoic acid is found to be = % w/w

Expt No:

Date:

PREPARATION OF PHENYL AZO-β-NAPHTHOL

AIM:

To prepare and submit phenyl azo-β-naphthol and to find out its percentage yield.

REFERENCES:

1. Practical Organic Chemistry: F.G Mann and B C Saunders; 4th edition; page no: 210.
2. Comprehensive Practical Organic Chemistry – Preparation and Qualitative Analysis; V K Ahluvalia and Renu Aggarwal; page no: 65.

REQUIREMENTS:

Reagents: Aniline-4.5ml, concentrated hydrochloric acid-10ml, sodium nitrite-4g, β-naphthol-7g, 10% sodium hydroxide solution-60ml, water-20ml.

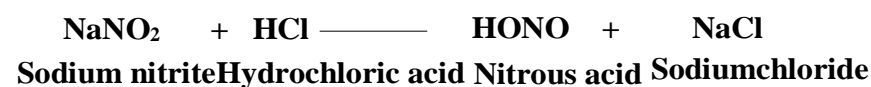
Apparatus: Conical flask, measuring cylinder, beaker, funnel, filter paper, glass rod etc.

PRINCIPLE:

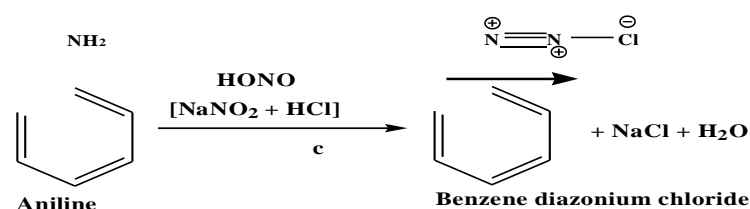
The principle involved in the preparation of phenyl azo-β-naphthol is coupling reaction. Aromatic primary amines react with nitrous acid in the presence of hydrochloric acid at 0-5⁰C to give diazonium salt and the reaction is called diazotization reaction. Diazonium salt is highly reactive compound and couple with alkaline solution of certain aromatic compounds (β-naphthol / phenol) to give highly colored compounds with general formula Ar-N=N-Ar called azo-compounds. This process is called coupling reaction.

REACTIONS INVOLVED:

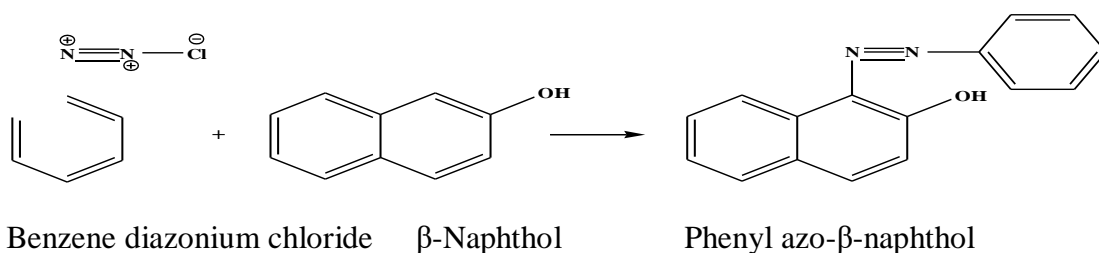
Preparation of Nitrous acid



Diazotization reaction



Coupling Reaction



PROCEDURE:

Dissolve aniline (4.5ml) in a mixture of concentrated hydrochloric acid (10ml) and water (20ml). Cool the solution to 5^oC and diazotize by the addition of sodium nitrite (4g) dissolved in water (20ml) drop wise with continuous shaking and maintaining a temperature below 5^oC. Dissolve β-naphthol (7g) in 10% sodium hydroxide solution (60ml) in a beaker and cool this solution to below 5^oc by the external cooling, aided by the direct addition of about 20-30g of crushed ice. Now add the diazotized solution very slowly to the Naphthol solution with constant stirring. The mix solution immediately and it develops a deep red colour and the formed phenyl azo- β-naphthol (Benzene azo-2-naphthol) is allowed to stand in an ice-salt mixture for 30 minutes with occasional stirring when the addition of diazo solution is complete. Then filter the solution, wash with water, dried and submitted.

USE:

Benzene azo-2-Naphthol is used as dye.

REPORT:

Benzene azo- 2-naphthol was prepared by the coupling reaction of aniline. The product was dried and submitted.

Practical yield of Benzene azo- 2-naphthol is found to be =

Theoretical yield of Benzene azo- 2-naphthol is found to be =

Percentage yield of Benzene azo- 2-naphthol is found to be = % w/w.

Expt No:

Date:

PREPARATION OF BENZIL

AIM:

To prepare and submit benzil from benzoin and calculate the percentage yield.

REFERENCE:

1. Advanced practical organic chemistry by O.P Agarwal pg no 327-328.
2. Practical organic chemistry, 4th edition by F. G Mann and B C Saunders, pg no: 234.

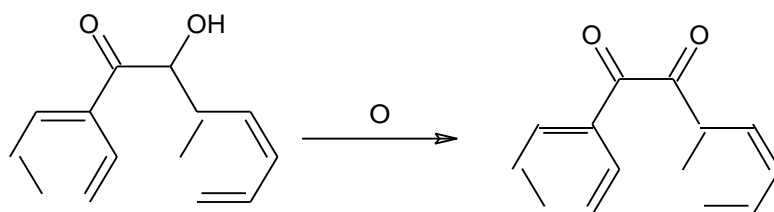
REQUIREMENTS:

Reagents: benzoin, conc. Nitric acid

Apparatus: R.B flask, ice bath

PRINCIPLE:

The secondary alcoholic group $-\text{CH}(\text{OH})-$ in benzoin is readily oxidized to a ketone group, and thus benzil, a diketone is obtained. For this purpose, the usual oxidizing agent is nitric acid, which in this circumstances does not nitrate the benzene ring.



Benzoin

Benzil

PROCEDURE:

Take Benzoin(10gm), conc. Nitric acid(25ml) in a R B flask and shake well and reflux for 2 hrs on a boiling waterbath. Cool the flask in icebath, and pour the mixture in to a beaker of cold water. Mix thoroughly with water and allow the precipitate to settle. Recrystallize the product in methanol.

USES:

Most benzil is used in the free radical curing of polymer networks. Benzil is a relatively poor photoinitiator, and is seldom used.

REPORT:

Benzil is prepared from benzoin by oxidation, dried and submitted.

Practical yield of Benzil is found to be = g

Theoretical yield of Benzil is found to be = g

Percentage yield of Benzil is found to be = % w/w

Expt No:

Date:

PREPARATION OF DIBENZAL ACETONE

AIM:

To prepare and submit Dibenzalacetone and to find out its percentage yield.

REFERENCE:

1. Practical Organic Chemistry; F.G. Mann and B.C. Saunders; 4th edition; Page no: 231
2. Comprehensive Practical Organic Chemistry-Preparation and Qualitative analysis; V.K. Ahulwalia and Renu Agarwal; Page no: 3-4.
3. Advanced practical organic chemistry; O.P Agarwal, page no: 365-366.

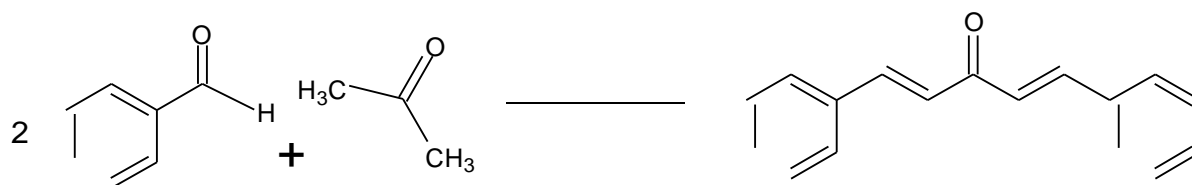
REQUIREMENTS:

Reagents: Benzaldehyde, alcohol, acetone, sodium hydroxide.

Apparatus: R.B flask, measuring cylinder, beaker, funnel, filter paper, glass rod etc.

PRINCIPLE:

Reaction involved in the preparation of Dibenzalacetone is Claisen- Schmidt reaction. It involves the condensation of aromatic aldehydes, having no α -hydrogen, with aliphatic aldehydes, ketones or esters, having active hydrogen, in the presence of 10% alkali solution to give α , β - unsaturated aldehydes or ketones. Here in the presence of dil. NaOH two molecules of benzaldehyde along with one molecule of acetone undergo condensation to give dibenzal acetone.



Benzaldehyde

Acetone

Dibenzal acetone

PROCEDURE:

Dissolve 10gm of sodium hydroxide in 80 ml of water and 100ml of alcohol in a flask. Cool the flask in an icebath to about 20-25^o, stir vigorously and add a mixture of 10 ml of benzaldehyde and 4 ml of acetone slowly in two installments with an interval of 15 minutes but with constant stirring. Cork the flask and shake it vigorously for 30 minutes. Filter the product, wash with water and recrystallize from ethyl alcohol.

USES:

Dibenzalacetone is used as a component in sunscreens and as a ligand in organometallic chemistry.

REPORT:

Dibenzalacetone is prepared from benzaldehyde. The product is dried and submitted.

Practical yield of Dibenzalacetone is found to be = g

Theoretical yield of Dibenzalacetone is found to be = g

Percentage yield of Dibenzalacetone is found to be = %w/w