TECHNOCRATS

Lab Work Book of

Pharmaceutical Organic Chemistry-II

(BP- 305 P)

Department of Pharmacy

Lab Manual of

Pharmaceutical Organic Chemistry-II

(BP- 305P)

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TECHNOCRATS
PUBLICATIONS

Lab Work Book
of

PHARMACEUTICAL ORGANIC CHEMISTRY-II

(BP-305P)

Name : .....

Enrollment No. : .....

Institute : .....

Academic Session : .....

Department of Pharmacy

TECHNOCRATS
PUBLICATIONS

Vision of the Institute

To grow as an institute of Excellence for Pharmacy Education and Research and to serve the humanity by sowing the seeds of intellectual, cultural, ethical, and humane sensitivities in the students to develop a scientific temper, and to promote professional and technological expertise.

Mission of the Institute

M 1: To inculcate ethical, moral, cultural and professional values in students

M 2: To provide state of art infrastructure facilities to the staff and students so as to enable them to learn latest technological advancements

M 3: State of art learning of professionalism by the faculty and students

M 4: To produce well learned, devoted and proficient pharmacists

M 5: To make the students competent to meet the professional challenges of future

M 6: To develop entrepreneurship qualities and abilities in the students

PROGRAM OUTCOMES (POs)

1. **Pharmacy Knowledge:** Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.
2. **Planning Abilities:** Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.
3. **Problem analysis:** Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions.
4. **Modern tool usage:** Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of thelimitations.
5. **Leadership skills:** Understand and consider the human reaction to change, motivationissues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and well- being.
6. **Professional Identity:** Understand, analyze and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).
7. **Pharmaceutical Ethics:** Honour personal values and apply ethical principles in professional and social contexts. Demonstrate behavior that recognizes cultural and personal variability in values, communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions.
8. **Communication:** Communicate effectively with the pharmacy community and with society at large, such as, being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions.
9. **The Pharmacist and society:** Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.
10. **Environment and sustainability:** Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
11. **Life-long learning:** Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-assess and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

PEOs

PEO 1: To inculcate quality pharmacy education and training through innovative Teaching Learning Process.

PEO 2: To promote professionalism, team spirit, social and ethical commitment with effective interpersonal communication skills to boost leadership role assisting improvement in healthcare sector.

PEO 3: To enhance Industry-Institute-Interaction for industry oriented education and research, which will overcome healthcare problems of the society.

PEO 4: To adapt and implement best practices in the profession by enrichment of knowledge and skills in research and critical thinking

PEO 5: To generate potential knowledge pools with interpersonal and collaborative skills to identify, assess and formulate problems and execute the solution in closely related pharmaceutical industries and to nurture striving desire in students for higher education and career growth.

Course Outcomes (COs):

Student will be able to:

- CO1: Apply the basic knowledge of organic chemistry in identification of functional groups and synthesis of organic compounds.
- CO2: Analyse and predict the principles of chemical reactions.
- CO3: Analyse and interpret the mechanism of chemical reactions.
- CO4: Apply the concept of moles in calculating theoretical yield.
- CO5: Calculate and estimate the percentage purity of the compounds synthesized.

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Experiment No.- 1

OBJECT:

To perform the simple distillation of single solution

REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

REQUIREMENTS:

Water (H20)

20% Sodium Chloride (NaCl)

THEORY:

Distillation is a process where a liquid is vaporized, recondensed, and collected. Distillation is used to purify liquids and to separate one liquid from another. The liquids have different volatilies which is the relative ease with which the molecules of a liquid escape from the surface. Volatility is normally the opposite of the boiling point of a liquid. The higher the volatility of a liquid, the lower the boiling point. The lower the volatility of a liquid, the higher the boiling point. Vapour pressure is a measure of the force a liquid exerts on the surface for its molecule to escape.

The liquids will give off molecules, until the atmosphere above the liquid has a vapour pressure equal to the respective temperature. For example, if the vapour pressure of a liquid was 760 mm Hg at 60° C, molecules would escape from the surface until there was a pressure of 760 mm Hg in the atmosphere vapour exerting pressure back at the liquid. When a solvent is enclosed, the liquid will evaporate until the partial pressure of the gas above the liquid equals the vapour pressure of the liquid. If some of the gaseous vapour is removed, more liquid will evaporate in order to equalize the vapour pressure and partial pressure. This is the principle behind distillation.

A liquid is heated in a distilling flask. The temperature of the liquid will increase (specific heat) until the vapour pressure/temperature of the first liquid is reached. At this point, all the heat energy (heat of vaporization) is used to evaporate the liquid. The hot vapour travels upward and reaches a condensing column which removes heat from the vapour. The gas recondenses back to a liquid and is collected in a receiving flask. Since this reduces the vapour pressure over the liquid in the distilling flask, more liquid is converted to vapour to equalize, which in turn recondenses. This is the general idea behind distillation. If there is only one liquid in the receiving flask, it will be separated from any nonvolatile solid. These solids would remain in the distillation flask. Thus, the liquid is purified.

PROCEDURE:

1. Add 100 ml of aqueous 20% sodium chloride (NaCl) to a 200 ml distilling flask. In picking a certain size of distilling flask, the volume of the flask should be about twice the size of the volume of liquid.

2. Add 3 or 4 boiling chips to the flask to avoid bumping.
3. Assemble the distillation apparatus. Remember to put a film of silicon lubricant between the ground glass joints to prevent freezing of the joints. The thermometer bulb should be slightly below the sidearm opening.
4. The upper outlet on the condenser should be for exiting cooling water. This will prevent the accumulation of air in the condenser.
5. Turn on the cooling water.
6. Increase the heat to the distilling flask until the rate of distillate (liquid) into the receiving flask is about 2-3 drops per second.
7. Record the temperature of the vapour every 5 ml of distillate collected.
8. Continue to distill until only a small amount of residue remains in the distilled flask. Do not distill to dryness.

VIVA QUESTIONS

Q.1. Define distillation

Ans- .....

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Q.2. Define vapor pressure

Ans- .....

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Q.3. What is volatility of liquid?

Ans- .....

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Q.4. Why are boiling chips added to the solution while carrying out distillation?

Ans- .....

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Q.5. Why is silicon film added on the ground joints of boiling or freezing apparatus?

Ans- .....

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Experiment No. -2

OBJECT:

To perform the recrystallization of given sample

REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

REQUIREMENTS:

acetanilide decolorizing carbon, 2 - erlenmeyer flask (250 ml), hot plate, filter paper, watch glass, Hirsch funnel, vacuum flask

THEORY:

Most organic substances are initially produced in an impure form. The substance is mixed with unreacted reagents, side products, and impurities. If the substance is a solid, a process called recrystallization can purify it.

Recrystallization is a process in which the solid of interest is dissolved in a hot solvent that is then slowly cooled. The crystals of the purified product are slowly and selectively precipitated. The impurities remain dissolved in the solution or are removed from the hot solution (before recrystallization occurs) by decolorizing carbon. The crystals are then separated from the solution by filtration.

The solvent selected is based on the solubility of the product to be recrystallized. The product should be highly soluble at high temperatures but only slightly soluble at room temperatures. If the solubility of the product at room temperature is high, the yield of product will be greatly reduced. The selection of solvent is usually on a trial and error basis unless a chemical reference can be found which recommends a solvent.

Many times a mixture of solvents is used. The product is dissolved in a small amount of solvent which it is very soluble in. While still hot, a second solvent that the product is not very soluble in is slowly added until cloudiness appears (products begin to precipitate out). The first solvent is then slowly added until the cloudiness just disappears. The mixture is then slowly cooled and the product recrystallizes.

The boiling point of the solvent should be lower than the melting point of the product. Otherwise, the product may melt in the solvent rather than dissolve. This is called "oiling out". The melted product often contains a great deal of impurities and if allowed to cool, will recrystallize in an impure state.

Decolorizing carbon is used to remove colored impurities from the solution. The carbon has a large active surface area which attracts and absorbs impurities. The carbon is added to the hot solution which prevents recrystallization of the product while the carbon isabsorbing impurities.. A process called hot filtration removes the carbon. The solution is kept hot during the filtration of the carbon to prevent recrystallization and loss of product.

Repeated recrystallization may be necessary to obtain the desired purity.

PROCEDURE:

1. Boil approximately 200 ml of DI water.
2. Slowly add the hot water to 5 grams of acetanilide in a 250 ml Erlenmeyer flask with constant stirring until the acetanilide is dissolved in the minimum amount of boiling solvent. If needed, place the flask in a hot water bath to keep the solution hot and prevent recrystallization. Do not place directly onto the hot plate because this might cause *oiling out*.
3. Using a minimum amount of solvent will return a greater yield of acetanilide upon cooling. Also remember that the solid may dissolve slowly. Do not add the hot solvent too rapidly.
4. Allow the solution to slowly cool to room temperature. Crystals will begin to form as the solution cools. It is recommended to leave the solution standing overnight. If the solvent is volatile, cover it with paraffin or a watch glass. In this experiment, we will just wait until room temperature.
5. Place the flask into an ice bath and allow cooling.
6. Filter the cold solution by vacuum filtration using a Hirsch funnel.
7. Rinse the collected crystals with COLD solvent.
8. Continue to pull air over the crystals using the filtration device.
9. Remove the crystals and filter and allow drying in a covered watch glass.
10. Weigh and calculate % yield.

VIVA QUESTIONS

Q.1. Define distillation

Ans- .....

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Q.2. Define vapor pressure

Ans- .....

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Q.3. What is volatility of liquid?

Ans- .....

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Q.4. Why are boiling chips added to the solution while carrying out distillation?

Ans- .....

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Q.5. Why is silicon film added on the ground joints of boiling or freezing apparatus?

Ans- .....

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Experiment No.-3

OBJECT:

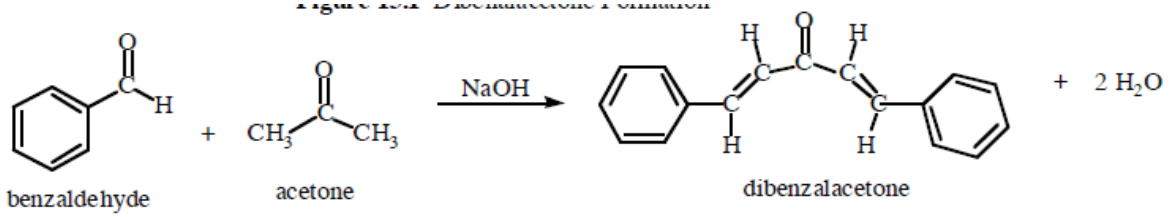
To perform the synthesis and characterization of dibenzalacetone

REFERENCE:

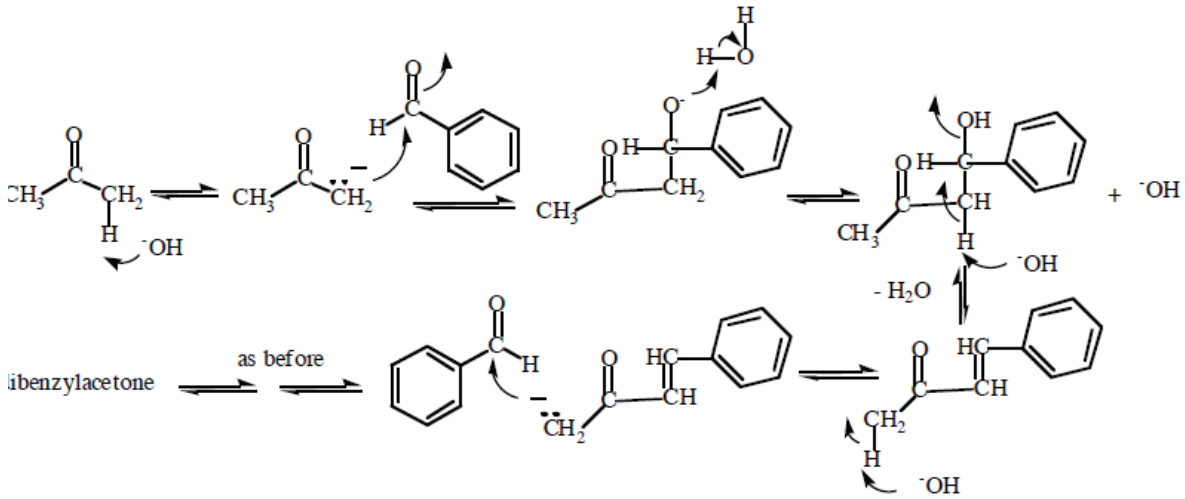
Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

THEORY:

In this experiment we will prepare dibenzalacetone from acetone and two equivalents of benzaldehyde under basic conditions. This is an example of a crossed-aldol (or mixed-aldol) reaction.



Acetone enolizes in the strongly basic conditions. Note that benzaldehye cannot enolize and so it must act as the electrophile. The nucleophilic alpha carbon then attacks the carbonyl of benzaldehyde. After proton transfer there is loss of water to give the α,β-unsaturated carbonyl that is stabilized by conjugation with the phenyl substituent. Notice how the π-electrons of the phenyl ring are delocalized all the way onto the carbonyl and onto the other carbonyl in the final dibenzylacetone product.



PROCEDURE:

1. In a 125 mL Erlenmeyer flask, dissolve 0.020 moles sodium hydroxide (pellets) in 4.0 mL of water. Solid sodium hydroxide is hygroscopic (absorbs water from the air) and you must close the bottle containing it immediately after using it. The dissolution is exothermic and the contents of the Erlenmeyer will get warm. Allow the solution to cool before using it.
2. In a 50 ml Erlenmeyer flask weigh out accurately 0.0160 moles benzaldehye and weigh into the same flask 0.0080 moles acetone.
3. Add 10 ml of 95% ethanol and pour this mixture into the prepared solution of sodium hydroxide.
4. Mix and swirl occasionally for fifteen minutes. A yellow, flocculent precipitate should form.
5. Filter the solid product by vacuum using your spatula to transfer as much of the solid as possible to the Buchner funnel.
6. After no more liquid is coming through the filter paper, disconnect the filter flask from the vacuum line, wash the solid with 10 mL water and, after about one minute, reconnect to the vacuum.
7. Repeat the wash in the same way using 5 mL chilled 95% ethanol. Allow air to be sucked around the crystals for about 2 minutes.
8. Recrystallize your product from ethyl acetate using a water bath and hot plate to heat the solvent.
9. Allow the solution to cool slowly to room temperature and then cool in an ice bath.
10. Collect the final product on the Buchner funnel by suction filtration.

VIVA QUESTIONS

Q.1. What is aldol condensation?

Ans- .....

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Q.2. Why basic conditions are needed for the crossed aldol condensation reaction?

Ans- .....

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Q.3. Why one mole of acetone is taken in comparison to two moles of benzaldehyde?

Ans- .....

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Q.4. Why suction filtration is used to filter the final product?

Ans- .....

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Q.5. How many grams of benzaldehyde should be taken for 0.0160 moles?

Ans- .....

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Experiment No. - 4

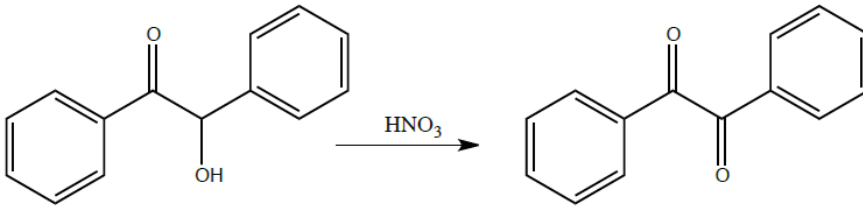
OBJECT:

To perform the synthesis and characterization of Benzil from Benzoin

REFERENCE:

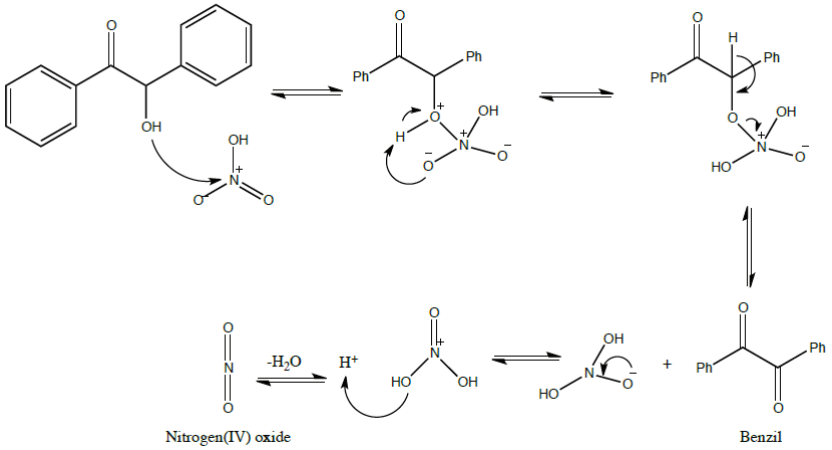
Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

REQUIREMENTS: Benzoin, concentrated nitric acid, three neck flask, acetic acid, magnetic stirrer



Benzoin Benzil

THEORY: Mechanism involved in the reaction is nitric acid mediated oxidation of benzoin leading to the formation of benzyl.



PROCEDURE:

1. In a 250mL triple-neck round bottom flask equipped with a refrigerator, 45mL of nitric acid, 30mL of acetic acid and 6g of benzoin were added.
2. The solution was heated at 100°C and agitated until the red fumes were gone.
3. Then, the solution was transferred in a flask containing 150mL of cold water and agitated until the precipitate was formed.
4. The solid product (yellow crystals) were filtered and washed with cold water and then recrystallized in ethanol.

VIVA QUESTIONS

Q.1. How many moles of nitric acid per mole of benzoin are needed for oxidation to benzyl.

Ans- .....

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Q.2. How many steps are involved in the mechanism

Ans- .....

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Q.3. What is an extended conjugated system.

Ans- .....

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Q.4. Which oxidizing agent is used for conversion of benzoin to benzyl.

Ans- .....

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Q.5. The extended conjugated system in benzyl involves how many carbons.

Ans- .....

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Experiment No. 5

OBJECT:

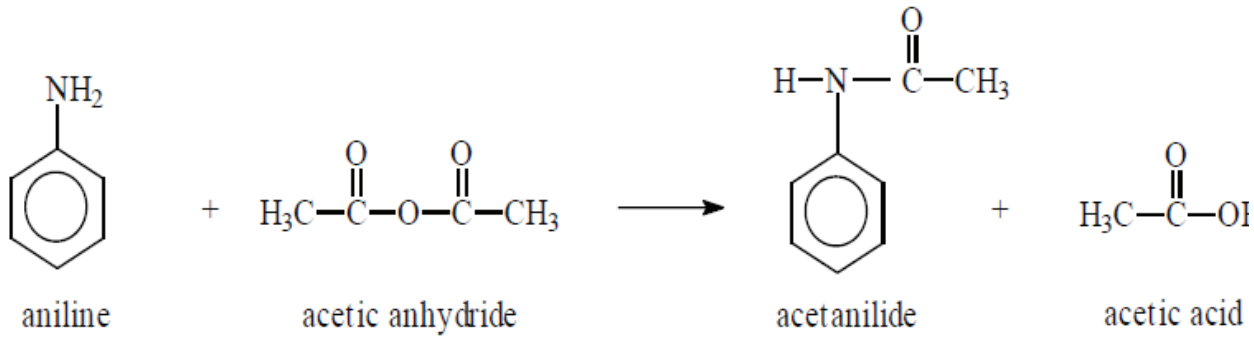
To perform the synthesis and characterization of acetanilide

REFERENCE:

Requirements: aniline (10 grams) graduated cylinder (10 ml), acetic anhydride (10 ml) hot plate, zinc dust (pinch) vacuum distillation apparatus, erlenmeyer flask (500 ml)

THEORY:

Acetanilide can be prepared from the acetylation of aniline by the following reaction:



PROCEDURE:

1. Using a 10 ml graduated cylinder, add 4.0 ml of aniline (0.04 moles) to a 500 ml Erlenmeyer flask.
2. Add 30 ml of DI water and a pinch of zinc dust to the flask.
3. With constant stirring, slowly add 6 ml of acetic anhydride (0.06 moles). This should be done in several small portions.
4. Crude acetanilide should slowly begin to precipitate. Continue to stir the solution for at least 20 minutes.
5. Dissolve the precipitant with a minimal amount of hot water. Remember that the zinc dust will not dissolve.
6. Remove the zinc by performing a hot filtration.
7. Recrystallize the acetanilide.
8. Calculate the % yield using aniline as the limiting reagent.

VIVA QUESTIONS

Q.1. Explain the mechanism of the reaction

Ans- .....

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Q.2. What is the rate limiting reagent in the reaction?

Ans- .....

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Q.3. How many moles of acetanilide per mole of aniline are used in the reaction?

Ans- .....

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Q.4. What is the recrystallization solvent used?

Ans- .....

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Q.5. What catalyst is used for carrying out the reaction?

Ans- .....

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Experiment No. 6

OBJECT:

To perform the synthesis of Phenyl benzoate from Phenol.

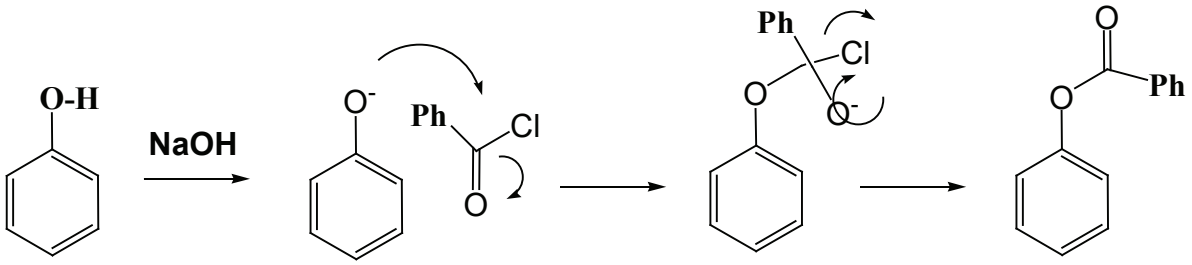
REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

Requirements:

THEORY:

Many phenols yield crystalline benzoyl derivatives with benzoyl chloride in the presence of sodium hydroxide (Schötten-Baumann method).



Benzoyl chloride has the formula C6H5COCl. The -COCl group is attached directly to a benzene ring. It is much less reactive than simple acyl chlorides likeethanoyl chloride.The phenol is first converted into the ionic compound sodium phenoxide (sodium phenate) by dissolving it in sodium hydroxide solution. The phenoxide ion reacts more rapidly with benzoyl chloride than the original phenol does, but even so you have to shake it with benzoyl chloride for about 15 minutes. Solid phenyl benzoate is formed.

PROCEDURE:

1. To the phenol (0.5 g) is added 5% sodium hydroxide (10 mL) in a well-corked boiling tube or a small conical flask.
2. Benzoyl chloride (2 mL, density 1.21 g cm^{-3}) is added in small quantities at a time, and the mixture shaken vigorously with occasional cooling under the tap or in ice water.
3. After 15 minutes the solid benzoate separates out: the solution should be alkaline at the end of the reaction; if not alkaline, or if oily, add a solid pellet of sodium hydroxide and shake again.
4. Collect the benzoate, wash thoroughly with cold water, and recrystallise from ethanol

VIVA QUESTIONS

Q.1. Explain Schotten-Baumann reaction

Ans- .....

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Q.2. Why a corked flask is used for this reaction?

Ans- .....

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Q.3. Why benzoyl chloride is added slowly in small volumes?

Ans- .....

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Q.4. Why is phenol acidic?

Ans- .....

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Q.5. Why NaOH is used for carrying out the reaction?

Ans- .....

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Experiment No. 7

OBJECT:

To perform the synthesis of para bromo acetanilide from acetanilide

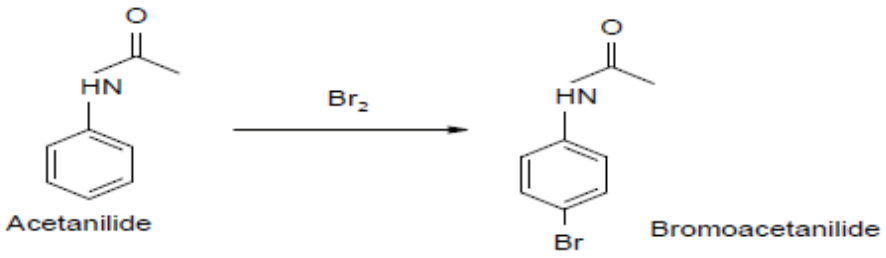
REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

Requirements

THEORY

This mechanism is a classic example of electrophilic aromatic substitution. An amine may lead to di- and tri- substituted products. If an amide is used in place of the amine, monosubstitution usually predominates (the electron-withdrawing carbonyl group makes the benzene ring less nucleophilic). This ortho-, para-directing group will tend to only add groups para- to itself because of the steric bulk of the amide group



PROCEDURE

1. Place 3 g (0.022 mol) of acetanilide into a 100 mL conical vial. Add 10 mL of glacial acetic acid. Stirring with a glass rod may be necessary to help dissolve the acetanilide (r.t). - Now, in the hood,prepare the bromine solution by adding 1.5 mL of bromine into 10 mL of acetic acid (first but 10 mL of the acid then 1.5 mL of bromine using dropper "Fast") - in the hood,add bromine-acetic acid solution to acetanilide solution with stirring then leave the mixture 15 min.

2. Transfer the mixture into beaker contain 100 mL of water with stirring. - Collect the product by vacuum filtration using Büchner funnel. - Purify the product by crystallization method using ethanol. - Collect the white crystals by vacuum filtration, dried and weigh and calculate the percent yield.

VIVA QUESTIONS

Q.1. What is a fume hood?

Ans- .....

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Q.2. Why reactions involving bromine should be carried out in fume hood?

Ans- .....

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Q.3. What are the uses of bromo acetanilide?

Ans- .....

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Q.4. Explain the mechanism of reaction involved in synthesis of bromoacetanilide

Ans- .....

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Q.5. Why bromination occurs on para position in acetanilide?

Ans- .....

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Experiment No. - 8

OBJECT:

To perform the synthesis of nitro salicylic acid from salicylic acid.

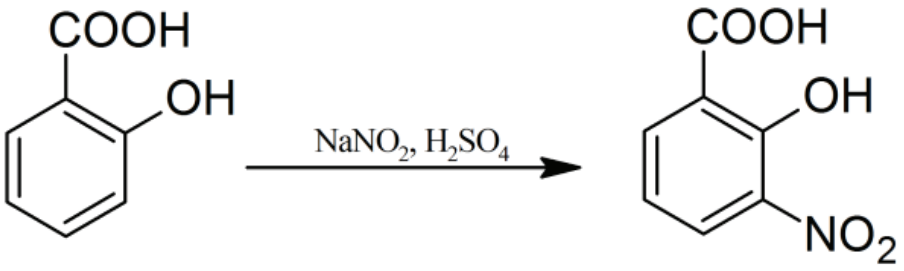
REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

REQUIREMENTS:

Calcium nitrate tetrahydrate - 1.5 g Acetic acid - 5 ml Salicylic acid - 1 g

THEORY:



PROCEDURE:

Calcium nitrate (1.5 g) was dissolved in warm acetic acid (5 ml) and salicylic acid (1 g) was added to it. Then the mixture was heated in a boiling water bath (maintained at > 80 o C) for 1 min. Salicylic acid was dissolved completely and the solution became dark red. It was immediately poured into a 10 ml of ice cold water. The resultant turbid dark red solution was placed in a refrigerator. After four hours, the yellow crystals that separated were washed free of acid with minimum amount of ice cold water and then dried.

VIVA QUESTIONS

Q.1. Explain the mechanism of reaction in preparation of nitro salicylic acid

Ans- .....

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Q.2. What are the uses of salicylic acid?

Ans- .....

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Q.3. Why nitration occurs at meta position of salicylic acid?

Ans- .....

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Q.4. Why acetic acid is used in the reaction?

Ans- .....

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Q.5. Explain the balanced reaction involved in this synthesis

Ans- .....

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Experiment No. 9

OBJECT:

To perform the synthesis of Sudan –I

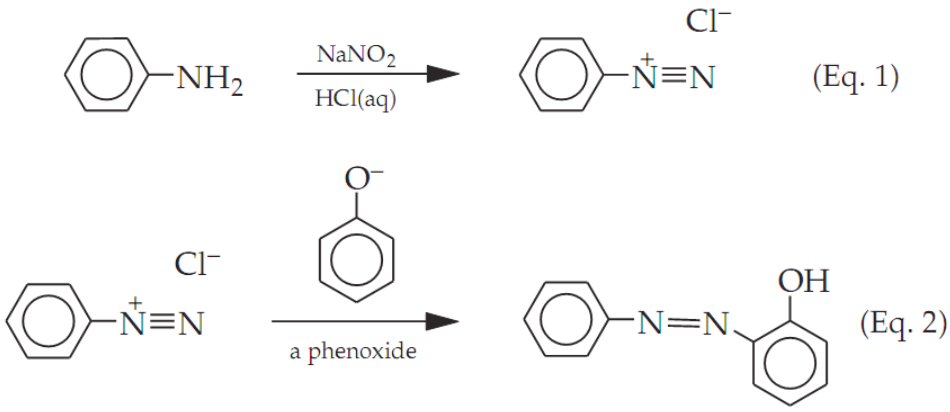
REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

Requirements:

THEORY:

An azo dye is defined by having an azo linkage (-N=N-) as part of its chromophore. Azo dyes are made in two steps. First, a primary aromatic amine is reacted to give a diazonium salt, as shown in Equation 1. Second, the diazonium salt is reacted or coupled with a strongly activated aromatic system, such as phenoxide, as shown in Equation 2.



An **azo coupling** is a reaction between a diazonium compound and an aniline, phenol or other aromatic compound which produces an azo compound. In this reaction the diazonium salt is an electrophile and the activated arene is a nucleophile in an **electrophilic aromatic substitution**. In most cases, including the example in Equation 2, the diazonium compound is also aromatic. The product absorbs longer wavelengths of light than the reactants because of increased conjugation.

PROCEDURE:

1. Place about 0.2 g of aniline in a pre-weighed 10 mL round-bottom flask. Record the actual weight of aniline.
2. Add 1 mL of distilled water and 10 drops of concentrated HCl. Swirl the flask in an icewater
3. bath.
4. To a clean and dry test tube, place 1 mL of 10% NaNO2 (from burette). Chill this solution in the icewater bath.
5. Weigh 0.15 g of β-naphthol in a 50 mL beaker. Add 1 mL of 10% NaOH and 2 mL of distilled water. Stir the mixture with a glass rod until a homogeneous solution occurs. Chill this solution in the ice-water bath.
6. When all three mixtures are cooled to about 0 °C, use a dropper to transfer the NaNO2 solution into the round-bottom flask containing the aniline solution. Stir the mixture thoroughly. **Do not add the NaNO2 solution too fast because the internal temperature should be below 10 °C.**
7. Transfer the reaction mixture from the round-bottom flask into the beaker containing β-naphthol. Stir the mixture to avoid aggregation of red precipitates.
8. Stir the mixture in the ice-water bath for 3-5 minutes.
9. Vacuum filter the precipitates and wash the filtrate with cold water.
10. Allow the precipitates to dry on the vacuum filtration set for a few minutes. Transfer this product into a 50 mL Erlenmeyer flask.
11. Recrystallize the crude product in ethanol.
12. Vacuum filter the crystals and wash them with cold ethanol.
13. Dry the crystals on a pre-weighed watch glass.
14. Weigh and record the yield of the recrystallized product.
15. Determine the melting point of the product.

VIVA QUESTIONS

Q.1. What is an azo dye?

Ans- .....

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Q.2. Explain the mechanism of azo coupling

Ans- .....

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Q.3. What is electrophilic aromatic substitution?

Ans- .....

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Q.4. Why azo coupling is done in ice-water bathy?

Ans- .....

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Q.5. What is the use of Sudan-I?

Ans- .....

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Experiment No. -10

OBJECT:

To determine the saponification value of the given oil.

REFERENCE:

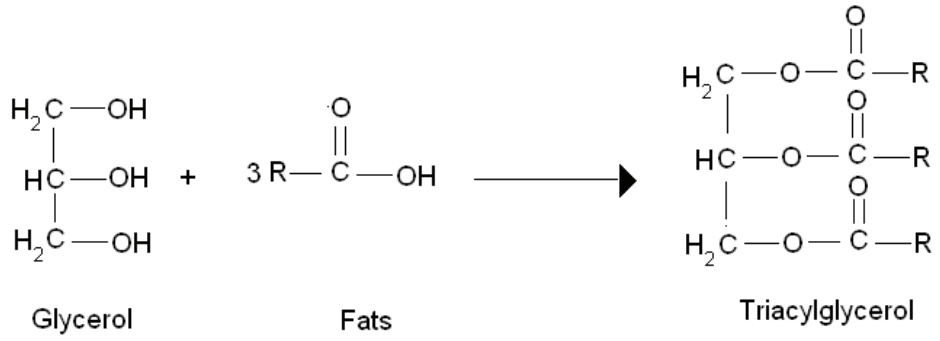
Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

REQUIREMENTS:

Oil, Ethanolic KOH, Potassium hydroxide [0.5N], Fat solvent, Hydrochloric acid[0.5N], Phenolphthalein indicator

THEORY:

Saponification is the hydrolysis of fats or oils under basic conditions to afford glycerol and the salt of the corresponding fatty acid. Saponification literally means "soap making".



The amount of free fatty acid is estimated by determining the quantity of alkali that must be added to the fat to render it neutral. This is done by warming a known amount of the fat with strong aqueous caustic soda solution, which converts the free fatty acid into soap. This soap is then removed and the amount of fat remaining is then determined. The loss is estimated by subtracting this amount from the amount of fat originally taken for the test.

The saponification number is the number of milligrams of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1g of fat . It gives information concerning the character of the fatty acids of the fat- the longer the carbon chain, the less acid is liberated per gram of fat hydrolysed. It is also considered as a measure of the average molecular weight (or chain length) of all the fatty acids present. The long chain fatty acids found in fats have low saponification value because they have a relatively fewer number of carboxylic functional groups per unit mass of the fat and therefore high molecular weight.

PROCEDURE:

1) Weigh 1g of fat in a tared beaker and dissolve in about 3ml of the fat solvent [ethanol /ether mixture].

2) Quantitatively transfer the contents of the beaker three times with a further 7ml of the solvent.

3) Add 25ml of 0.5N alcoholic KOH and mix well, attach this to a reflux condenser.

4) Set up another reflux condenser as the blank with all other reagents present except the fat.

5) Place both the flasks in a boiling water bath for 30 minutes .

6) Cool the flasks to room temperature .

7) Now add phenolphthalein indicator to both the flasks and titrate with 0.5N HCl .

8) Note down the endpoint of blank and test .

9) The difference between the blank and test reading gives the number of millilitres of 0.5N KOH required to saponify 1g of fat.

10) Calculate the saponification value using the formula:

Saponification value or number of fat = mg of KOH consumed by 1g of fat.

Weight of KOH = Normality of KOH * Equivalent weight* volume of KOH in litres

Volume of KOH consumed by 1g fat = [Blank – test]ml

VIVA QUESTIONS

Q.1. Define saponification value

Ans- .....

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Q.2. What is the product of hydrolysis of fats and oils?

Ans- .....

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Q.3. What do you mean by tared beaker?

Ans- .....

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Q.4. How will you prepare 0.5 N NaOH solution?

Ans- .....

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Q.5. How will you prepare 0.5 N HCl solution?

Ans- .....

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Experiment No. -11

OBJECT:

To determine the acid value of the given oil sample.

REFERENCE:

Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR. (Eds). In Vogel's Textbook of Practical Organic Chemistry, 5th Edition, Longman Scientific and Technical, UK

REQUIREMENTS:

Theory: Acid value indicates the proportion of free fatty acid present in an oil or fat and may be defined as the number of milligrams of caustic potash required to neutralize the acid in 1 gm of the sample. The normal acid value for most samples lies within 0.5. If any titrable acid other than a fatty acid is present in the sample, it will be an error. A high acid value indicates a stale oil or fat stored under improper conditions.

PROCEDURE:

Standardization of KOH

1. Take 20 ml of 0.1 N oxalic acid solution in a 250 ml conical flask.
2. Add 1 or 2 drops of phenolphthalein indicator to this solution.
3. Titrate this solution against KOH taken in a burette.
4. The appearance of pink color indicates the end point.
5. From the volume of the KOH solution in burette, find the normality of KOH.

Determination of Acid Value

1. Weigh 5 gm of oil and transfer it into 250 ml conical flask.
2. Add 50 ml of neutralized alcohol solution to the oil solution.
3. Heat this mixture for 10 minutes by using the heater.
4. Take the solution after 10 minutes and add 1 or 2 drops of phenolphthalein indicator.
5. Titrate this against the KOH solution from the burette.
6. The appearance of pink color indicates the end point

Calculation
Titration I: Standardisation of Potassium hydroxide

- Burette solution : KOH
- Pipette solution: Oxalic acid

- Indicator: Phenolphthalein
- End point: Appearance of pink colour
- Volume of oxalic acid (V1)=
- Normality of oxalic acid (N1)=
- Volume of KOH consumed (V2)=
- Normality of KOH consumed (N2) = V1 N1 / V2
- Normality of KOH (N2) = _____.

Titration II: Estimation of acid value

- Burette solution: KOH
- Pipette solution: Oil + 50 ml of neutralized alcohol
- Indicator: Phenolphthalein
- End point: Appearance of pink colour

Acid value = (Volume of KOH x Normality of KOH x Eq. wt x 1000) / Weight of Oil sample
Acid Value = _____.

VIVA QUESTIONS

Q.1. What is acid value?

Ans- .....

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Q.2. How will you prepare 0.5 N oxalic acid?

Ans- .....

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Q.3. What is the role of Phenolphthalein indicator in the titration?

Ans- .....

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Q.4. Define titration

Ans- .....

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Q.5. How will you prepare 0.5 N KOH solution?

Ans- .....

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