TECHNOCRATS

Lab Work Book of

Medicinal Chemistry – I

(BP - 406 P)

Department of Pharmacy

Lab Manual of

Medicinal Chemistry – I

(BP - 406P)

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TECHNOCRATS
PUBLICATIONS

Lab Work Book
of

Medicinal Chemistry – I

(BP-406 P)

(Strictly According to RGPV Syllabus)

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 synthesis of medicinal compounds.
- CO2: Analyze and predict the principles of chemical reactions.
- CO3: Apply 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.

Index

Experiment No:- 1

AIM

To perform the synthesis and characterization of Phenothiazine

REFERENCES

Fitton AO and Smallery A. Benzo derivatives of six membered systems- above one heteroatom. In: Practical Heterocyclic Chemistry, Academic Press: London, 1968, pp 127

Vogel AI. Qualitative Organic Analysis, In: Vogel's Textbook of Practical organic chemistry, Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR (Ed), 5th ed, Addison Wesley Publication: England, 1998, pp75, 1055-1056

REQUIREMENTS

Chemicals: Sulfur, diphenyl amine, iodine, ethanol, silica gel, chloroform, acetone, paraffin wax

Glassware and Equipments: Round bottom flask, sand bath, mortar and pestle, beaker, test tubes, test tube holder, petridish, microscopic slides, hot air oven, thermometer, burner etc.

REACTION

THEORY

Phenothiazine, abbreviated PTZ, is an organic compound that has the formula $S(C_6H_4)_2NH$ and is related to the thiazine-class of heterocyclic compounds. Although the parent compound has no uses, derivatives of phenothiazine are highly bioactive and have widespread use and rich history. The derivative chlorpromazine revolutionized the field of psychiatry and allergy treatment. An earlier derivative, methylene blue, was one of the first antimalarial drugs, and derivatives are under investigation as possible anti-infective drugs

PROCEDURE

In a 100 mL round bottom flask, heat an intimate mixture of 3.2g sulfur, 8.4g diphenyl amine and 0.15g iodine in a Wood's metal bath (use sand bath instead), maintaining the temperature between 190-200°C for 30 min. Cool the molten mixture and grind the residual mass to fine powder with a mortar and pestle. Crystallize the product obtained from ethanol (6 mL/g).

Weigh the dried product and determine the percentage yield obtained. Determine the solubility characteristic of the phenothiazine synthesized in solvents of varying polarity. Determine the melting point using melting

point apparatus or thiele's tube. Determine the retention factor of the product and compare it with the reactants using appropriate solvent system by TLC.

RESULTS

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
1		
2		
3		
4		
5		

VIVA QUESTIONS

Q.-1. What is the use of Phenothiazine ?

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Q.-2. How is melting point determined ?

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Q.-3. What solvents are used to determine solubility ?

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Q.-4. What is Rf value ?

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Q.-5. How is recrystallization performed?

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Experiment No:- 2

AIM

To perform the synthesis and characterization of 5,5-diphenyl hydantoin (Phenytoin)

REFERENCES

Fitton AO and Smallery A. Benzo derivatives of six membered systems- above one heteroatom. In: Practical Heterocyclic Chemistry, Academic Press: London, 1968, pp 19

Vogel AI. Qualitative Organic Analysis, In: Vogel's Textbook of Practical organic chemistry, Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR (Ed), 5th ed, Addison Wesley Publication: England, 1998, pp 75-80, 1053-1055

REQUIREMENTS

Chemicals: Benzil, urea, sodium hydroxide, ethanol, waer, conc. HCl, industrial spirit, silica gel, chloroform, acetone, paraffin wax

Glassware and Equipments: Round bottom flask, reflux condenser, electric heating mantle, test tubes, test tube holder, petridish, microscopic slides, hot air oven, thermometer, burner etc.

REACTION

THEORY

PROCEDURE

5.3g benzyl, 3.0g urea, 15 mL 30% aqueous NaOH solution and 7.5 mL of ethanol are placed in a 100 mL round bottom flask. A reflux condenser is attached and the mixture is boiled under reflux on a heating mantle for at least 2 h. The reaction mixture is cooled to room temperature and the product is poured into 125 mL water and mixed thoroughly. The mixture is allowed to stand for 15 min and then filtered under suction to remove the insoluble product. The filtrate is then rendered strongly acidic with conc. HCl, ooled in ice water and immediately the precipitated product is filtered under suction. It is recrystallized from industrial spirit to obtain the pure phenytoin.

Weigh the dried product and determine the percentage yield obtained. Determine the solubility characteristic of the phenothiazine synthesized in solvents of varying polarity. Determine the melting point using melting point apparatus or thiele's tube. Determine the retention factor of the product and compare it with the reactants using appropriate solvent system by TLC.

RESULTS

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
1		
2		
3		
4		
5		

VIVA QUESTIONS

Q.-1. What is the use of Phenytoin ?

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Q.-2. Why conc HCl is added in this reaction ?

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Q.-3. Write the numbering of Phenytoin structure ?

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Q.-4. What is Rf value ?

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Q.-5. How is recrystallization performed?

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Experiment No:- 3

AIM

To perform the synthesis and characterization of Benzimidazole

REFERENCE

Vogel AI. Qualitative Organic Analysis, In: Vogel's Textbook of Practical organic chemistry, Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR (Ed), 5th ed, Addison Wesley Publication: England, 1998, pp 75-80, 1053-1055

REQUIREMENTS

Chemicals: o-phenylenediamine, formic acid, sodium hydroxide, ethanol, waer, conc. HCl, industrial spirit, silica gel, chloroform, acetone, paraffin wax, charcoal.

Glassware and Equipments: Round bottom flask, reflux condenser, electric heating mantle, test tubes, test tube holder, petridish, microscopic slides, hot air oven, thermometer, burner etc.

REACTION

THEORY

Benzimidazole are formed by the action of an acylating agent on o-phenylenediamine under reflux conditions. The reaction proceeds via an acyl derivative which cyclizes under the influence of excess acid.

Disconnection of the two carbon- nitrogen bonds in benzimidazole reveals the presence of o-phenylenediamine and formic acid in the structure. The synthesis is affected by simply heating the two reagents together.

PROCEDURE

In a 50 mL round bottom flask, a solution of 2.7 g o-phenylenediamine and 10 mL of 98% formic acid is heated under reflux for 2 hours. The reaction mixture is cooled, and basified using a solution of 10% NaOH. The crude product obtained in filtered off under suction and washed repeatedly with cold water to remove impurities.

The product obtained in recrystallized from hot water using activated charcoal.

RESULTS

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
1		
2		
3		
4		
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VIVA QUESTIONS

Q.-1. What is the use of Benzimidazole ?

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Q.-2. Why charcoal is used in recrystallization ?

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Q.-3. How is percentage yield calculated ?

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Q.-4. What is Rf value ?

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Q.-5. How is solubility performed ?

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Experiment No:- 4

AIM

To perform the synthesis and characterization of Benzotriazole

REFERENCE

Vogel AI. Qualitative Organic Analysis, In: Vogel's Textbook of Practical organic chemistry, Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR (Ed), 5th ed, Addison Wesley Publication: England, 1998, pp 75-80, 1053-1055

REQUIREMENTS

Chemicals: o-phenylene diamine, glacial acetic acid, sodium nitrite, benzene, sodium hydroxide, ethanol, waer, conc. HCl, industrial spirit, silica gel, chloroform, acetone, paraffin wax

Glassware and Equipments: Round bottom flask, reflux condenser, electric heating mantle, test tubes, test tube holder, petridish, microscopic slides, hot air oven, thermometer, burner etc.

REACTION

THEORY

Diazotization of o-phenylenediamine in dilute acid solution yields benzotriazole. The reaction proceeds via the mono diazonium salt which then couples with the adjacent amino group to give the internal diazo amino compound, benzotriazole.

PROCEDURE

To a solution of o-phenylene diamine (5.4g) in a mixture of glacial acetic acid (12 mL) and water (25 mL) contained in a 250 mL round bottom flask, add a solution of sodium nitrite (3.5 g in 10 mL water). Cool the resulting solution which will have attained a temperature of around 70°C in an ice bath and neutralize to pH 7 by addition of 2N NaOH solution.

Stir the mixture until a solid product is obtained and then filter the brown residue under suction and wash with cold water.

Recrystallize the product using benzene and activated charcoal to obtain colorless needles of benzotriazole

RESULTS

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
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2		
3		
4		
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VIVA QUESTIONS

Q.-1. What is the use of Benzotriazole ?

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Q.-2. What is diazotization ?

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Q.-3. What is the role of sodium nitrite in this reaction ?

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Q.-4. Why is refluxing done ?

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Q.-5. How is recrystallization performed?

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Experiment No:- 5

AIM:

To perform the synthesis and characterization of Benzocaine

REFERENCE

Vogel AI. Qualitative Organic Analysis, In: Vogel's Textbook of Practical organic chemistry, Furniss BS, Hannaford AJ, Smith PWG, Tatchell AR (Ed), 5th ed, Addison Wesley Publication: England, 1998, pp 75-80, 1053-1055

REQUIREMENTS

Chemicals: p-amino benzoic acid, conc. Sulphuric acid, methylene chloride, ethanol, sodium carbonate, ethanol, waer, conc. HCl, industrial spirit, silica gel, chloroform, acetone, paraffin wax

Glassware and Equipments: Round bottom flask, reflux condenser, electric heating mantle, test tubes, test tube holder, petridish, microscopic slides, hot air oven, thermometer, burner etc.

REACTION

THEORY

PROCEDURE

To a 100-mL round bottom flask fitted with a stir bar, add 2.50 g of p-aminobenzoic acid and 20 mL of ethyl alcohol. While stirring, add 2.0 mL of concentrated H_2SO_4 dropwise. The precipitate that forms upon the addition of sulfuric acid should dissolve when the solution is heated. Reflux, with stirring, for 1 hour. If you notice that the white suspension persist, add 1 additional mL of concentrated H2SO4 dropwise. Cool to room temperature. Neutralize cautiously with dropwise addition of 10% Na2CO3 until the pH is approximately 8. (Gas evolution will be vigorous.) Extract with two 10-mL portions of methylene chloride. Wash the combined methylene chloride layers with two 8-mL portions of water. Dry the methylene chloride solution over anhydrous sodium sulfate. Gravity filter into a clean Erlenmeyer flask containing a boiling stone. Evaporate the methylene chloride using the rotary evaporator. Recrystallize the whitish residue using as a solvent pair, ethyl alcohol and water. Suction filter the product, and let air dry. Weigh the white crystals and take the melting point

Weigh the dried product and determine the percentage yield obtained. Determine the solubility characteristic of the benzocaine synthesized in solvents of varying polarity. Determine the melting point using melting point apparatus or thiele's tube. Determine the retention factor of the product and compare it with the reactants using appropriate solvent system by TLC.

RESULTS

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
1		
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3		
4		
5		

VIVA QUESTIONS

Q.-1. What is the use of Benzocaine ?

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Q.-2. What is the chemical class to which benzocaine belongs ?

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Q.-3. What is the role of H_2SO_4 in this reaction ?

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Q.-4. What is Rf value ?

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Q.-5. How is recrystallization performed?

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Experiment No:- 6

AIM

To synthesize & Characterized Aspirin from salicylic acid.

REFERENCE

Kar Ashutosh, "Advanced Practical Medicinal Chemistry" Published by New Age international Publishers, New Delhi no. 79.

REQUIREMENTS

Salicylic acid, acetic acid, sulphuric acid, Round bottom flask, Beaker, Funnel, Condenser, Water bath, Litmus paper, Glass rod, Hot air oven.

THEORY

Aspirin, also known as acetylsalicylic acid (**ASA**), is a medication, often used to treat pain, fever, and inflammation. Aspirin is also used long-term, at low doses, to help prevent heart attacks, strokes, and blood clot formation in people at high risk of developing blood clots. Low doses of aspirin may be given immediately after a heart attack to reduce the risk of another heart attack or the death of heart tissue. Aspirin may be effective at preventing certain types of cancer, particularly colorectal cancer.

The main side effects of aspirin are gastric ulcers, stomach bleeding, and ringing in the ears, especially with higher doses. While daily aspirin can help prevent a clot-related stroke, it may increase risk of a bleeding stroke (hemorrhagic stroke). In children and adolescents, aspirin is not recommended for flu-like symptoms or viral illnesses, because of the risk of Reye's syndrome

Aspirin is part of a group of medications called non steroidal anti-inflammatory drugs (NSAIDs), but differs from most other NSAIDs in the mechanism of action. The salicylates have similar effects (antipyretic, anti-inflammatory, analgesic) to the other NSAIDs and inhibit the same enzyme cyclooxygenase (COX), but aspirin does so in an irreversible manner and, unlike others, affects the COX-1 variant more than the COX-2 variant of the enzyme. Aspirin also has an antiplatelet effect by stopping the binding together of platelets.

The synthesis of aspirin is classified as an esterification reaction. Salicylic acid is treated with acetic anhydride, an acid derivative, causing a chemical reaction that turns salicylic acid's hydroxyl group into an ester group (R-OH → R-OCOCH$_3$). This process yields aspirin and acetic acid, which is considered a by product of this reaction. Small amounts of sulfuric acid(and occasionally phosphoric acid) are almost always used as a catalyst

PROCEDURE

1. Take salicylic acid (10 g), acetic anhydride (14 ml) and concentrated suphuric (2 ml) and warm the reaction mixture for half an hour at 50-60^0 with frequent stirring.
2. Pour the contents into cold water (150 ml) with occasional stirring and filter off the crude aspirin.
3. Recrystallization , dissolve the product in alcohol (30 ml) and pour the solution into warm water of a

solid separates, warm the mixture of dissolve the solid.

4. The clear solution is allowed slowly to get needle shaped crystals of aspirin, m.p. 136-137^0, yield 13 g.

OBSERVATION:

Theoretical yield: 13 gm

Practical yield:.....

% yield:.....

Melting Point: .....

RESULTS

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
1		
2		
3		
4		
5		

VIVA QUESTIONS

Q.-1. What is the category of aspirin

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Q.-2. What is the MAO of aspirin

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Q.-3. How to recrystalize aspirin

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Q.-4. What is Chemical name of aspirin

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Q.-5. What is COX-1 & COX-2

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Experiment No:- 7

AIM

To synthesize Butamben from *p*-amino benzoic acid.

REFERENCES

Delgado N Jaime and Remers A William. Wilson and Gisvold's Textbook of Organic Medicinal and Pharmaceutical Chemistry, Tenth edition-1998. Lippincott Raven Publishesr, USA, 2008, Page no- 646-649.

Lemke L Thomas and Williams A David, Foye's principles of medicinal chemistry, Sixth edition-2008, Wolters Kluwer Publications Pvt. Ltd. New Delhi, Page no-462-471.

REQUIREMENTS

p-amino benzoic acid, n-butanol, H_2SO_4, Sodium carbonate, Ether**,** Round bottom flask, Beaker, Funnel, Heating mantel, Condenser, Litmus paper, Conical flask, Separating funnel, Glass rod, Hot air oven

THEORY

Butamben is obtained by esterification of *p*-amino benzoic acid by using n-butyl alcohol in presence of conc. H_2SO_4. Butamben is a local anesthetic. It is the ester of 4-aminobenzoic acid and butanol. A white, odourless, crystalline powder that is soluble in water and soluble in alcohol, ether, chloroform, fixed oils, and dilute acids. It slowly hydrolyses when boiled with water. Synonyms include Butamben, Butil aminobenzoato, and Butoforme. Proprietary names includes Alvogil in Spain and Alvogyl in Switzerland. It is one of three components in the topical anesthetic cetacaine. *Butambin* is an analgesic agent that was developed as a single epidural injection for the treatment of patients in surgical management of pain. Treating pain in certain areas eg, mouth, throat, ears, vagina, rectum. It may also be used to numb these areas before medical procedures. Butamben, tetracaine, benzocaine liquid in a local anesthetic. It works by numbing sensitive and painful areas.

PROCEDURE

1. 2 gm of *p*-amino benzoic acid was placed in round bottom flask.
2. 26 ml of n-butanol was added and stirred gently to help the dissociation of the solid.
3. The mixture was cooled in ice both.
4. 2 ml of conc. H_2SO_4 was added slowly condenser was attached and heat for 2 hr.
5. The content of flask was stirred in 15 min interval, then transfer it into 500 ml beaker and neutralize it with 10% sodium carbonate solution and maintain Ph 9.
6. Filter if necessary to remove any solid material, extract the aqu. layer with 25 ml ether.
7. Collect the ether layer, dry it, evaporate until oil is visible

OBSERVATION:

Theoretical yield: 1.7 gm

Practical yield: .....

% yield: .....

Melting Point: .....

RESULT:

Product Name:

IUPAC Name:

Yield:

Melting Point:

Rf value:

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SOLUBILITY

S.No.	Solvent	Observed Solubility
1		
2		
3		
4		
5		

VIVA QUESTIONS

Q.-1. What is the use of butamben ?

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Q.-2. Which principle involve in the synthesis of butamben ?

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Q.-3. What are local anesthetics ?

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Q.-4. What is the use of H_2SO_4 in the synthesis of butamben ?

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Q.-5. What is the use of sodium carbonate solution in the synthesis procedure?

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Experiment No:- 8

AIM

To perform the Assay of Aspirin.

REFERENCE

Rao G. Devala., Practical Medicinal Chemistry, First edition-2008, Birla Publications Pvt. Ltd. Delhi, Page no-

REQUIREMENTS

Aspirin (Bulk or Formulation)

Sodium hydroxide Solution ------ 0.5 N

Hydrochloric acid ------ 0.5N

Phenol red indicator

PRINCIPLE

Aspirin or Acetyl salicylic acid is an example of analgesic and antipyretic, which is widely used in the management of pain. It is estimated by acidimetry and alkalimetry. Its determination depends upon the alkaline hydrolysis of aspirin to acetic acid and salicylic acid (sodium salts are formed immediately), followed by back titration of the excess alkali using phenol red as indicator, A blank determination is needed in this assay. The reactions involved are as follows :

REACTION

PROCEDURE

Procedure for standardization: Standardize the sodium hydroxide solution using standard solution of oxalic acid (0.5N) and phenolphthalein indicator. Latter, Standardize the hydrochloric acid, using the standardized sodium hydroxide solution (indicator is phenolphthalein).

Procedure for assay : Weigh accurately about 1.5 gm of Acetyl salicylic acid or aspirin and add 50 ml of 0.5N sodium hydroxide solution and boil gently for ten minutes. Titrate the excess alkali with 0.5N hydrochloric acid using solution of phenol red as indicator. Repeat the experiment with the same quantities of the same reagents in the same manner omitting the acetyl salicylic acid.

EQUIVALENT FACTOR

Each ml of 0.5 N NaOH = 0.04504 gm of Aspirin.

From the volume of 0.5 N sodium hydroxide consumed, calculate the quantity of Aspirin in the given sample.

RESULTS

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VIVA QUESTIONS.

Q.-1. What is Normality

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Q.-2. What is equivalent factor

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Q.-3. What is Assay

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Q.-4. Write the category of aspirin

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Q.-5. Explain the MOA of Aspirin

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Experiment No:-9

AIM

To perform the Assay of Paracetamol

REFERENCE

Rao G. Devala., Practical Medicinal Chemistry, First edition-2008, Birla Publications Pvt. Ltd. Delhi, Page no-

REQUIREMENTS

Paracetamol (Bulk or Formulation)

Sulphuric acid - 2N

Hydrochloric acid - 2N

Ceric ammonium Sulphate Solution - 0.1 N

Ferroin indicator

PRINCIPLE

Paracetamol is an example of analgesic and antipyretic drug widely used in the management of pain and fever. It is official in Indian Pharmacopoeia (1985). The official method is cerimetry (Redox titration). When paracetamol is refluxed with dilute sulphuric acid, p-aminophenol is obtained. This is titrated with a standard solution of ceric ammonium sulphate (0.1 N) using ferroin indicator.

PROCEDURE

1. Procedure for Standardization of ceric ammonium sulphate :
2. Standardize the ceric ammonium sulphate using standard solution of ferrous ammonium sulphate (0.1 N) and ferroin indicator.

Procedure for assay : Dissolve 0.3 gm of paracetamol in a mixture of 10 ml of water and 30 ml of 2N sulphuric acid. Boil under reflux for one hour, cool and dilute to 100 ml with water. To 20ml of the solution add 40ml water, 40 gm of ice,15 ml of 2 N hydrochloric acid, 0.1 ml of ferroin sulphate solution and titrate with 0.1 N ceric ammonium sulphate . Repeat the procedure without the substance being examined.

EQUIVALENT FACTOR

Each ml of 0.1N ceric ammonium sulphate = 0.00756 gm of paracetamol.

From the volume of 0.1 N ceric ammonium sulphate consumed, calculate the amount of ascorbic acid present in the given sample solution.

RESULTS

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VIVA QUESTIONS.

Q.-1. How to prepare 0.1 N Solution.

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Q.-2. What is primary & Secondary standard.

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Q.-3. Category of paracetamol.

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Q.-4. Define redox tration.

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Experiment No:-10

AIM

To perform the Assay of Ibuprofen.

REFERENCE

Rao G. Devala., Practical Medicinal Chemistry, First edition-2008, Birla Publications Pvt. Ltd. Delhi, Page no-

REQUIREMENTS

Ibuprofen (Bulk or formulation)

Sodium hydroxide Solution - 0.1 N

Phenolphthalein indicator

PRINCIPLE

Ibuprofen is an example of non- steroidal anti- inflammatory drug (NSAID). It is also having analgesic and anti- pyretic activity. It is widely used in the management of pain and fever. Ibuprofen is aryl acetic acid derivative and is weakly acidic in nature. It is estimated by alkalimetry. In this method, the alcoholic solution of Ibuprofen is titrated against a standard solution of sodium hydroxide (0.1 N) using solution of phenolphthalein as indicator. The reactions involved are as followss:

REACTION

PROCEDURE

1. **Procedure for Standardization of Sodium Hydroxide solution :**Standardize the sodium hydroxide solution using a standard solution of oxalic acid (0.1 N) and Phenolpthalein indicator.
2. **Procedure for Assay :** Weight accurately, about 0.5 gm of Ibuprofen and dissolve in 100 ml of alcohol and titrate with 0.1 N sodium hydroxide solution using phenolphthalein solution as indicator.

EQUIVALENT FACTOR

Each ml of 0.1 N Sodium hydroxide solution = 0.02063 gm of Ibuprofen. From the volume of the standard sodium hydroxide consumed, calculate the amount of Ibuprofen present in the given sample.

RESULT

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VIVA QUESTIONS.

Q.-1. What is NSAID.

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Q.-2. Define indicator

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Q.-3. How to prepare 0.1 N Solution of NaOH

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Experiment No:-11

AIM

To perform the Assay of Indomethacin.

REFERENCE

Rao G. Devala., Practical Medicinal Chemistry, First edition-2008, Birla Publications Pvt. Ltd. Delhi, Page no-

REQUIREMENTS

Indomethacin (Bulk or formulation)

Sodium hydroxide Solution - 0.1 N

Phenolphthalein indicator

PRINCIPLE

Indomethacin is an example of Non- steroidal anti- inflammatory drug (NSAID). It is also having analgesic and anti- pyretic activity. It is an example of aryl acetic acid derivative. Therefore, it is acidic in nature. It can be estimated by alkalimetry. Alcoholic solution of indomethacin is titrated against standard solution of sodium hydroxide using phenolphthalein indicator. The reactions involved are as follow:

REACTION

PROCEDURE

1. Procedure for Standardization of Sodium hydroxide solution :
2. Standardize the sodium hydroxide solution using a standard solution of oxalic acid (0.1 N) and Phenolphthalein indicator.

PROCEDURE FOR ASSAY

Weight accurately, about 0.45 gm of indomethacin and dissolve in 75 ml of methanol. Add 75 ml of carbon-dioxide free water and titrate with 0.1N sodium hydroxide solution using solution of phenolphthalein as indicator. Repeat the experiment without indomethacin. The difference between the titrations represents the amount of alkali required by the indomethacin.

EQUIVALENT FACTOR

Each ml of 0.1 N Sodium hydroxide =0.03578 gm of indomethacin.

From the volume of the standard solution of sodium hydroxide (0.1 N) consumed, calculate the amount of indomethacin present in the given sample.

RESULT

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VIVA QUESTIONS

Q.-1. What is Assay

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Q.-2. Define standard & test solutions

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Q.-3. What is molarity.

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Q.-4. What is Equivalent point.

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Experiment No:-12

AIM

To perform the Assay of Furosemide (Frusemide)

REFERENCE

Rao G. Devala., Practical Medicinal Chemistry, First edition-2008, Birla Publications Pvt. Ltd. Delhi, Page no-

REQUIREMENTS

- Furosemide (Bulk or formulation)
- Dimethyl formamide (DMF)
- Sodium hydroxide solution - 0.1N
- Bromothymol blue solution (indicator)

PRINCIPLE

1. Frusemide is an example of diuretic. It is also known as furosemide.
2. It is widely used in the management of oedema. It is official in I.P. 1985 and is estimated by alkalimetry. In this method, the solution of fursemide in DMF is titrated against 0.1 N sodium hydroxide using bromothymol blue as indicator. The reactions involved are as follows.

REACTION

PROCEDURE

1. Procedure for the standardization of sodium hydroxide solution :
2. Standardize the sodium hydroxide solution (0.1 N) and using a standard solution of oxalic acid (0.1N) and phenolphthalein indicator.

PROCEDURE FOR ASSAY

Weight accurately about 0.5 gm of frusemide and dissolve in 40 ml of dimethyl formamide and titrate with 0.1 N sodium hydroxide solution using bromothymol blue solution as indicator. Repeat the operation

without the substance being examined. The difference in titration represents the amount of sodium hydroxide required by frusemide.

EQUIVALENT FACTOR

Each ml of 0.1 N sodium hydroxide solution =0.03308gm of frusemide, from the volume of the standard sodium hydroxide solution consumed, calculate the amount of the frusemide present in the given sample solution.

RESULT

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VIVA QUESTIONS

Q.-1. Write the ues of Frusemide.

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Q.-2. What is Titration.

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Q.-3. How to prepare 0.1M HCl Solution

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