REVIEW ARTICLE

EFFICIENT CATALYTIC SYNTHESIS OF BENZANILIDE VIA BECKMANN REARRANGEMENT AND ITS USE AS A FRAGRANCE FIXATIVE

Priyanka Hemant Patil

Department of Chemistry,

J.S.S.P. ACS College, Goveli, Tal. Kalyan, Dist. Thane 421103 Maharashtra, India

*Corresponding author E-mail: priyanka.patil27051991@gmail.com

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Abstract:

This project focuses on the synthesis and application of benzanilide, an aromatic amide, through the Beckmann rearrangement of benzophenone oxime, utilizing the conventional acid-catalyzed process with concentrated sulfuric acid. The method was evaluated based on several factors, including yield, purity, safety, environmental impact, and ease of execution. Characterization of the synthesized benzanilide was performed using Thin Layer Chromatography (TLC) and Infrared (IR) spectroscopy, which confirmed the formation of the amide bond. Subsequently, benzanilide was incorporated into perfume formulations by combining it with ethanol and essential oils. These formulations exhibited distinct olfactory profiles, including floral, herbal, and amber scents. The research also explores the manufacture of perfumes derived from natural sources like lavender, sandalwood, and rose. A major aspect of this study is to minimize skin irritations commonly associated with synthetic perfumes. Perfumes have been used since ancient times for their fragrance, and their formulation has evolved over centuries. While chemical and synthetic perfumes continue to dominate the market today, this study emphasizes the benefits of herbal perfumes. We developed a formula using entirely herbal ingredients, without the inclusion of chemicals, to create a more skin-friendly alternative. Perfumes are commonly applied to clothing or skin, including sensitive areas such as the eyes, nose, and ears. However, chemical perfumes can often cause allergic reactions, such as skin rashes. To mitigate these risks, our herbal perfume formulation offers a safer alternative with fewer potential disadvantages compared to commercially available synthetic perfumes. In conclusion, our study demonstrates that herbal perfumes provide an advantageous option, offering a safer, more natural alternative to synthetic perfumes, with reduced risks of allergic reactions.

Keywords: Cosmetics, Herbal Ingredients, Liquid Perfume, Fragrance, Synthetic Perfumes.

Introduction:

The Beckmann rearrangement is a key organic reaction that transforms oximes into their corresponding amides when exposed to acidic conditions. Benzanilide ($C_{13}H_{11}NO$) is an aromatic amide synthesized via this rearrangement from benzophenone oxime. In this process, an aryl or alkyl group migrates under the influence of an acid catalyst, resulting in the formation of benzanilide. By refining the reaction conditions, we aim to enhance the yield, selectivity, and efficiency of the transformation. Benzanilide is typically a white to off-white crystalline solid with a mild odor. While it is only sparingly soluble in water, it readily dissolves in organic solvents like ethanol, acetone, and ether. As a representative aromatic amide, benzanilide features an amide bond (-CONH-) and two aromatic rings, making it a valuable compound in organic synthesis, medicinal chemistry, and materials science. The structure of benzanilide benefits from resonance stabilization, where the nitrogen's lone pair interacts with the carbonyl group, providing stability and planarity. This planar structure, along with its low volatility and chemical stability, enables benzanilide to effectively interact with volatile fragrance ingredients, slowing their evaporation rate. Unlike traditional animal-derived fixatives like musk, benzanilide is cost-effective, sustainable, and non-animal-based, making it an ideal candidate for modern fragrance applications. In perfumery, benzanilide plays a crucial role in enhancing and stabilizing delicate floral and woody notes, which are often short-lived. By acting as a fixative, it helps preserve the intensity and balance of these notes within a fragrance composition. Rather than masking or overwhelming the fragrance, benzanilide complements and prolongs the aroma, contributing to the development of sophisticated, high-quality perfumes.

Objectives and Scope of the Study

The primary objectives of this study are:

- To explore the role of benzanilide as a fixative in perfumery, focusing on the chemical properties that contribute to fragrance stabilization.
- To assess how effectively benzanilide enhances and extends floral and woody notes in perfume formulations.
- To compare the performance of benzanilide with conventional fixatives in terms of effectiveness, sustainability, and compatibility with various fragrance components.
- To evaluate the potential of benzanilide as a cost-effective and environmentally sustainable alternative in the cosmetic and fragrance industries.
- To investigate potential future applications of benzanilide in the formulation of perfumes and related consumer products.

Literature Review

Several studies have explored the Beckmann rearrangement for synthesizing amides, with specific focus on optimizing reaction conditions and improving efficiency. Notable contributions include:The synthesis and functional modification of aromatic amides such as benzanilide have garnered considerable attention in organic chemistry due to their relevance in pharmaceuticals, dyes, agrochemicals, and the fragrance industry. Benzanilide, an aromatic primary amide, serves as a versatile intermediate and offers interesting physicochemical properties due to its ability to engage in hydrogen bonding and π-π interactions.

1. Beckmann Rearrangement – Historical and Mechanistic Insight

The Beckmann rearrangement, discovered by Ernst Otto Beckmann in 1886, involves the acid-catalyzed conversion of oximes to amides. In this process, benzophenone oxime undergoes a rearrangement to yield benzanilide. Traditional literature (March's Advanced Organic Chemistry, 6^{th} Ed.) describes the reaction mechanism as involving protonation of the oxime, followed by migration of the anti-group and loss of water to give the corresponding amide. Numerous studies have highlighted the reaction's stereospecificity, with migration occurring from the anti-position relative to the hydroxyl group.

*2. **Derivatization: Nitration and Acylation of Benzanilide***

Functional modification of benzanilide improves its properties and applications. According to studies on electrophilic aromatic substitution (EAS), the amide group is a moderate deactivator, directing new substituents to the meta position in nitration and ortho/para positions in acylation under Friedel–Crafts conditions. Nitrated benzanilides have been investigated for their antimicrobial properties (Ref: Eur. J. Med. Chem., 2005, 40, 1326–1332), while acylated analogs find applications in dyes and fragrances**.**

*3. **Benzanilide and Its Derivatives in Fragrance Chemistry***

Although benzanilide is not a traditional perfumery base, its musk-like odor and the ability to be chemically modified make it suitable for use in custom fragrance formulations. Research in industrial perfumery has shown that derivatives of aromatic amides can enhance fixation**,** depth of scent, and blending with essential oils (Ref: Poucher's Perfumes, Cosmetics and Soaps, 10th Ed.). Derivatized benzanilide compounds can contribute floral, woody, powdery, or musky notes depending on the substituents and solvents used.

*4. **Analytical Techniques in Product Characterization***

Thin Layer Chromatography (TLC) and Infrared (IR) spectroscopy are commonly employed to monitor reaction progress and confirm the presence of functional groups. IR absorption bands for C=O (amide) near 1650 cm^{-1} and N–H stretching around 3300 cm^{-1} are consistent indicators of successful benzanilide formation. Several publications confirm that these methods are adequate for undergraduate and educational synthesis experiments (J. Chem. Educ., 2012, 89, 1295–1298).

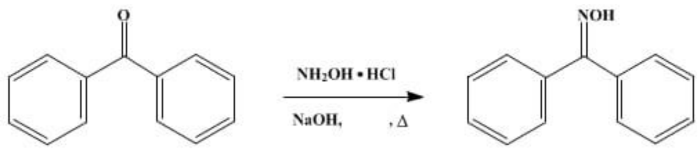
The reviewed literature provides a solid foundation for the synthesis of benzanilide through conventional and green methods, its chemical modification, and application in perfumery. The integration of green chemistry**,** functional derivatization, and practical product formulation aligns well with modern chemical education and sustainable industrial practices.

Material and Methods:

Benzophenone oxime is synthesized from benzophenone ($C_{13}H_{10}O$) and hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in the presence of a base like sodium hydroxide (NaOH) then Benzanilide can be prepared by acid catalysed process using sulfuric acid.

Preparation of Benzophenone Oxime

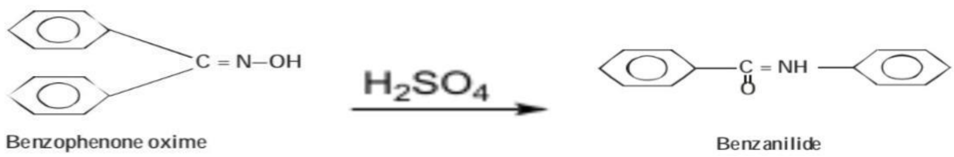
Reaction



Benzophenone Benzophenone Oxime

Preparation of Benzanilide from Benzophenone oxime

Reaction:



Stoichiometric Calculations:

	Benzophenone	Hydroxylamine Hydrochloride	Benzophenone Oxime	Benzanilide
Molecular Weight	**182 g/mol**	**69.5 g/mol**	**197.23 g/mol**	**197.23 g/mol**
Molar Ratio	**1**	**1**	**1**	**1**
Quantity in g	**6**	**4**	**3 g**	**3g**
Moles	**0.0330**	**0.0575**	**0.0152 moles**	**0.0152 moles**

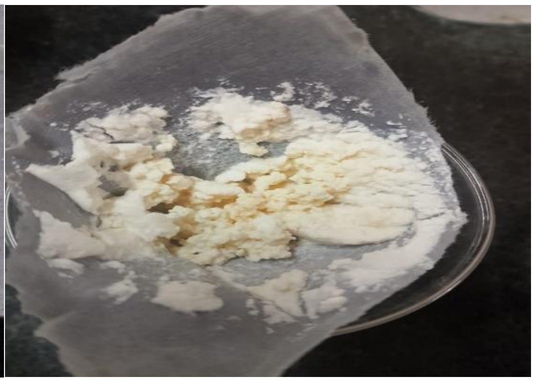
Chemicals and Reagents

Chemicals	Amount Required
Benzophenone ($C_{13}H_{10}O$)	6 g
Hydroxylamine hydrochloride ($NH_2OH \cdot HCl$)	4 g
Sodium hydroxide (NaOH, 10% solution)	7 g
Rectified Spirit (as solvent)	12 mL
Ice-cold water	50 mL
Benzophenone oxime ($C_{13}H_{11}NO$)	3 g
Concentrated H_2SO_4 (98%)	10 mL
Crushed ice	As needed
Sodium bicarbonate ($NaHCO_3$)	10 g (for neutralization)
Ethyl acetate	30 mL (for extraction)
Ethanol (for recrystallization)	20 mL

Methodology

- In a 250 ml round bottom flask take 6g Benzophenone, 4g Hydroxylamine hydrochloride, 2-3 ml water and 12 ml rectified spirit.
- To this mixture add 7g of solid Sodium Hydroxide slowly in portions of about 0.5 g with constant shaking and with cooling, if necessary.
- Fit a reflux condenser to the flask and reflux the content for about 10 minutes.

- Cool and then pour the reaction mixture into a solution of 20 ml Conc. HCl in about 120 ml water taken in a 500 ml beaker.
- The Benzophenone oxime separates out as colorless crystals.
- Filter it, wash with water and dry by pressing between filter papers.
- The yield of Benzophenone oxime, M.P 141^0, is about 6.2 g.
- Recrystallize about 1 gm of it from methyl alcohol.
- It should be stored in vacuum desiccator as it is decomposed by air and moisture.
- In a 250 ml round bottom flask, cool 10 ml Conc. H_2SO_4 in an ice bath.
- Add 3g of Benzophenone oxime slowly in small portions while stirring.
- Keep temperature below 10^0C.
- After complete addition allow the mixture to warm to room temperature, then heat gently to 80-85^0C for 1.5 hrs.
- Pour reaction mixture slowly into 150 ml of crushed ice.
- Stir well until product precipitate.
- Neutralize solution with solid 10g $NaHCO_3$, check pH= 7.
- Extract organic layer with 30 ml Ethyl acetate, combine organic layer wash with cold. Water.
- Dry, weight and determine melting point.

Recrystallized Benzophenone oxime **Crude Benzophenone Oxime**

Characterization


Yield Calculation

Actual Yield: Weigh the final dry product = **2.7g**

Theoretical Yield:

3 g of benzophenone oxime → 3 g of benzanilide (100% conversion).

197.23 g of Benzophenone oxime = 197.23 g of Benzanilide

3 g of Benzophenone oxime = 197.23 x 3 = 3 g

Percentage yield: weight of the product x 100

Theoretical yield

2.7 x 100 = **90%**

Characterization & Purity Check

Melting Point Test: Pure benzanilide: 161°C

IR Spectroscopy: Look for amide (C=O) stretch at ~1650 cm^{-1}.

Thin layer chromatography of Benzanilide Synthesis

Materials required:

- TLC plates (Silica gel)
- Solvent system (chloroform 9ml and methanol 1 ml)
- Iodine chamber for visualization
- Capillary tubes
- Beaker with lid (TLC chamber)
- Sample solutions: Benzophenone (starting material), and Benzanilide (Product).
- Pencil, scale
- Ethanol (for sample solution)
- Filter paper (for chamber saturation)

Procedure:

a. ***Prepare the samples:***

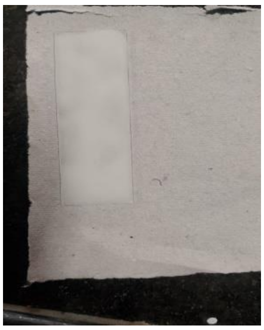
Dissolve a small amount of Benzophenone and Benzanilide separately in ethanol.

b. ***Prepare the TLC plate***

Draw a light pencil line 1cm from the bottom of the TLC plate.

Spot the two samples on the baseline:

- ✓ Left: **Benzophenone**
- ✓ Right**: Benzanilide**



c. ***Select the mobile phase:***

A suitable mobile phase for TLC of benzanilide synthesis is: Chloroform: methanol (9: 1, v\v)

This provides good separation between the starting material and product.

d. *Prepare the TLC chamber:*

Pour 10ml of solvent system into a 100ml beaker.

Line he inside wall with filter paper to saturate the chamber.

Cover he beaker and allow it to equilibrate for 5 minutes.

e. *Spot the TLC plates:*

Use a capillary tube to spot 2 drops of each solution on the marked points.

Let each spot dry & apply again if needed for better visibility.

f. *Develop the TLC plates:*

Place the TLC plate in the developing chamber so the solvent is below the spot.

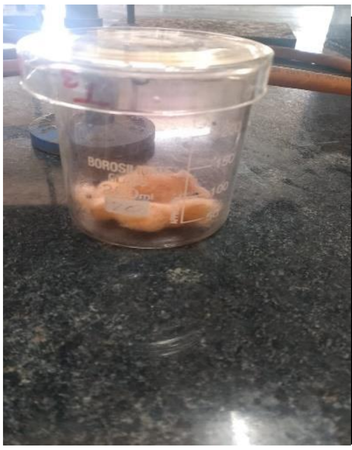
Ensure the solvent front rises until its 1cm from the top.

Remove the plate and allow it to dry and immediately mark the solvent front.

g. *Visualize the spots*

Observe the TLC plate under Iodine chamber.

Circle the spots lightly with pencil.



TLC Results:

- Distance travelled by Solvent system= 7.5 cm
- Distance travelled by Benzophenone = 5.8 cm
- Distance travelled by Benzanilide = 4.2 cm

Rf value = distance travelled by compound/ distance travelled by solvent system

Compound	Rf value
Benzophenone (Starting material)	0.733
Benzanilide (Product)	0.56

Interpretation:

- A spot corresponding to Benzophenone disappear after sometime reaction is complete.
- A new spot at lower Rf (~0.3 0.4) indicates Benzanilide formation.
- Benzanilide spot remain after sometime. i.e. reaction is complete.

Conclusion:

TLC helps monitor the progress of Beckmann Rearrangement, ensuring complete conversion of Benzophenone oxime to Benzanilide. If the starting material persists, reaction condition may need optimization.

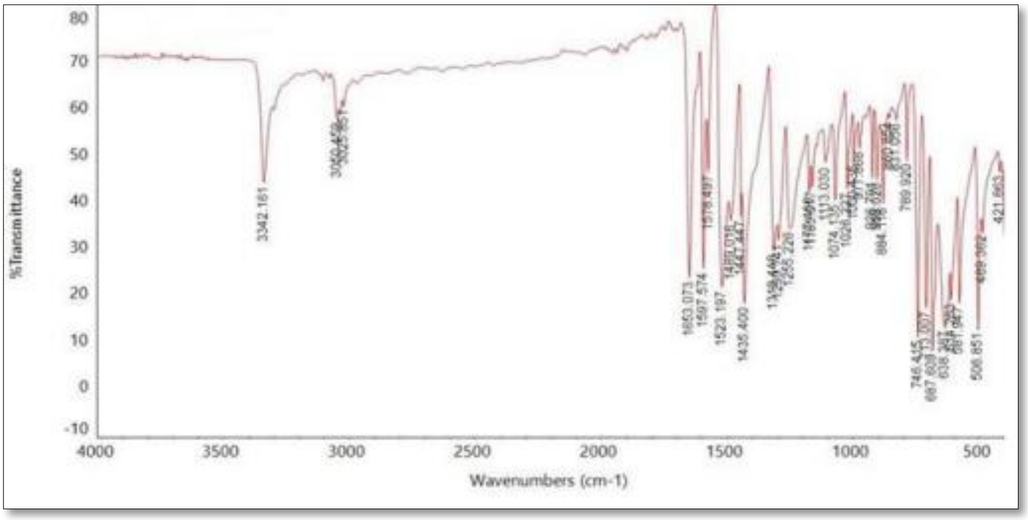
Characterization of Benzanilide

Infrared Spectroscopy (IR):

Username: Dell

Report created: 30-06-2025 14:10 (GMT05: 30)

sample 2 (Benzanilide)



Regions:

Region 1: 3817.31-400.16

Threshold: 64.106

Sensitivity: 70.000

421.863 45.618

489.362 32.589

506.851 11.749

581.94717.332

Sample 2 (benzanilide)

Measurement date: 25-06-2025 12:53:25
Number of sample scans: 32
Number of background scans: 32
Instrument Serial: BHT2510104
Smart accy: B122109
Model: Nicolet Summit X
Source: IR
Detector: DTGS KBr
Smart Accessory Title: Everest ATR
Smart Accessory ID: B122109
Crystal type: Diamond
Beamsplitter: KBr
Sample spacing: 1.0
Digitizer bits: 24
Optical velocity: 0.4747
Aperture: 100.0
Sample gain: 1.0
High pass filter: 20.0

614.263 18.019 638.387 12.082 687.609 7.149 713.007 16.257 746.415 9.946 789.920 47.965 831.056 56.792 860.854 57.104 884.116 38.833 908.028 43.904 926.791 44.191 977.868 50.616 1000.435 47.915 1026.227 41.891 1074.135 39.459 1113.030 47.689 1165.017 42.353 1177.466 41.209 1255.226 33.487

Low pass filter: 11000.0 1299.741 30.904 1318.448 28.576 1435.400 17.206
1447.447 35.919 1489.016 35.144
1523.197 20.825 1578.497 45.728
1597.574 24.753 1653.073 22.888
3025.851 59.773 3050.459 57.143
3342.161 43.359

Interpretation:

Important IR Absorption Peaks of Benzanilide:

Wavenumber (cm⁻¹)	Assignment	Explanation
3300–3400 (medium)	N–H Stretch (Amide)	Sharp to broad peak, often around 3350 cm⁻¹
1650–1680 (strong)	C=O Stretch (Amide carbonyl)	Strong and sharp, usually around 1670 cm⁻¹
1600–1580	C=C Stretch (Aromatic ring)	Indicates aromaticity
1500–1450	C–C Stretch (Aromatic ring)	Two or more peaks typical of benzene rings
1300–1230	C–N Stretch (Aromatic amide)	Often seen near 1280–1250 cm⁻¹
700–750	Aromatic C–H out-of-plane bending	For mono-substituted and para-substituted rings
~3050	Aromatic C–H Stretch	Usually weak to medium

Interpretation Summary:

- The **N–H stretch (~3350 cm⁻¹)** confirms the presence of the amide hydrogen.
- The **C=O amide stretch (~1670 cm⁻¹)** is a strong confirmation of the amide group.
- **Aromatic C=C and C–H stretches and bending vibrations** confirm the presence of phenyl rings on both sid

Applications of Benzanilide:

This project explores the synthesis of a benzanilide-based perfume, highlighting its role as a fixative in combination with essential oils and ethanol. Benzanilide acts as a fixative in perfumes, enhancing and stabilizing floral and woody notes. Benzanilide ($C_{13}H_{11}NO$) is an important organic compound with several applications in pharmaceuticals, dyes, agrochemicals, and industrial chemistry. Benzanilide act as fixative is a substance used to equalize the vapor pressures, and thus the volatilities, of the raw materials in a perfume oil, and to increase the perfume's odour tenacity.it can increase the time for which the scent of a perfume lasts. The objective of this perfume making from benzanilide aromatic amide to synthesize a perfume using benzanilide as a fixative. To test its effectiveness in stabilizing fragrance over time. Below is a structured explanation of its applications along with relevant procedures. Perfumes are a combination of following three things:

- **Essential Oils** – Provide the main scent (floral, citrus, woody, etc.).
- **Fixatives** – Help the fragrance last longer (e.g., benzyl benzoate, benzanilide).
- **Solvents** – Dissolve the components and allow even distribution (ethanol, distilled water).

In this paper explores the synthesis of a benzanilide-based perfume which can be shown below:

1. **Spring Blossom (Floral smells good, suitable in Summers**
2. **Green Garden (Suitable for everyday use)**
3. **Amber Fire (Warm, cozy good for winters)**

SPRING BLOSSOM **GREEN GARDEN** **AMBER FIRE**

1) **Spring Blossom** ***(Floral smells good, suitable in Summers)***

Leavender

Biological source: The plant Lavandula angustifolia, specifically its fresh flowering tops (or aerial parts), which are used to obtain the essential oil.

- Family: Lamiaceae or Labiatae
- Uses: It is used for Fragrance in Perfume

Sandalwood

Biological source: Sandalwood consists of the heartwood of the stems and roots of *Santalum albumliin*.

- Family: - Santalaceae
- Uses: - It is used for Fragrance in Perfume

Rose: Biological source: - Genus rosa of flowering plant

- Family: Rosaceae
- Uses: Use in perfume and fragrances, Use in rose water, Help to maintain pH of skin

Green Garden *(Suitable for everyday use*)

Leavender

Biological source: The plant Lavandula angustifolia, specifically its fresh flowering tops (or aerial parts), which are used to obtain the essential oil.

- Family: Lamiaceae or Labiatae
- Uses: It is used for Fragrance in Perfume

Lemon grass

Biological source: The fresh, aerial parts of plants from the genus Cymbopogon, specifically *Cymbopogon flexuosus* (East Indian Lemongras

- Family: Poaceae
- Uses: It is used for Fragrance in Perfume

Rosemery:

Biological source: *Rosmarinus officinalis* L evergreen shrub native to the Mediterranean region

- Family: Lamiaceae (or Labiatae) family
- Uses: Use in perfume and fragrances, Use in rose water, Help to maintain pH of skin.

Amber Fire (*Warm, cozy good for winters*)

Cinnamon:

Biological source: The dried inner bark of the shoots of trees from the genus Cinnamomum, most notably *Cinnamomum verum*; also known as *Cinnamomum zeylanicum*.

- Family: Lauraceae
- Uses: It is used for Fragrance in Perfume

Clove:

Biological source: The dried, aromatic flower buds of *Syzygium aromaticum*.

- Family: Myrtaceae
- Uses: It is used for Fragrance in Perfume

Amber: Biological source: - Amber is fossilized tree resin its biological source varies depending on its age and geographic location.

- Family: Sciadopityaceae, an ancient family of conifers.
- Uses: Use in perfume and fragrances, Use in rose water, Help to maintain pH of skin.

Formulation table

Chemical	Amount Required	Purpose
Benzanilide	2 g	Fixative
Ethanol (95%)	30 mL	Solvent
Distilled Water	10 mL	Dilution
Essential Oil as per required i) (Lavender, Rose, or Sandalwood) ii) (Lemon grass, Lavender and Rosemery) **iii)** (cinnamon, clove, amber)	5 mL	Fragrance source
Glycerin	2 mL	Enhances longevity
Amber Glass Bottle	1	Storage container

Methodology

Herbal perfume can be formulated by various beneficial methods that involve various herbal ingredients after processing to get more convenient herbal furfume. the formulation of herabal perfume involved following steps:

1) select herbal ingredients like Lavender, Rose, Sandalwood Lemon grass, Lavender , Rosemery, cinnamon, clove and amber etc. for perfume preparation.
2) Blending of all herbal ingredient with each other those you have to prepre so get more fine powder
3) Extraction of essential oil from herbal ingredient carried out by steam distillation.
4) Filtration can be done to remove all impurities.
5) Weigh 2 g of benzanilide and place it in a 50 mL beaker.
6) Add 30 mL of ethanol (95%) and stir the mixture using a glass rod.
7) Heat gently (40–50°C) while stirring to fully dissolve benzanilide.
8) Allow the solution to cool to room temperature.
9) Select 5 mL of an essential oil those you have to prepare like spring blossom, green garden or amber fire respectively.
10) Slowly add the essential oil to the benzanilide solution, stirring gently.
11) Add 2 mL of glycerin to enhance fragrance retention.
12) Add 10 mL of distilled water to the mixture and stir well.
13) Transfer the solution into an amber glass bottle (prevents light degradation).
14) Seal the bottle tightly and store in a cool, dark place for 2 weeks to allow the fragrance to mature.

Evalution Test:

1) **Fragrance test**: For fragrance test paper is used which is known as fragrance blotter. Perfume is sprayed on a paper to test the aroma of perfume. Following steps can be involved

- Spray the fragrance twice in a downward motion in front of you.
- Swiftly pass the blotter the fragrance's vapours cloud.
- Quickly wave the blotter under your nose and inhale.
- Refer back to the card regularly to test its life cycle.

2) Skin Test:

- Spray the back of your hand twice whilst respecting the correct spray distance.
- Leave to dry naturally & do not rub in fragrance.
- Inhale the fragrance without letting it touch your nose.
- Refer back to your hand over time to see how it evolves.

3) pH Test: pH is most important factor for perfume industry as well other industry in the chemistry. It can be determined by using pH paper. The ideal level of pH of perfume around 1 to 6 P^H.

Sr. No.	Test	Observation
1.	Skin test	No irritation
2.	Fragrance test	Pleasant aroma
3.	pH	7

Results and Discussion:

This experiment successfully demonstrated how benzanilide can be used as a perfume fixative. Benzanilide was successfully synthesized from benzoxime. Yield was good, around 93.87%, due to strong acidic conditions leading to side reactions or degradation. The product showed strong amide peaks on IR (C=O ~1650 cm^{-1}, N–H stretch ~3300 cm^{-1}). The synthesized benzanilide was blended with ethanol and essential oils. Benzanilide contributed a musk-like base note enhanced the complexity and longevity. The final perfumes had pleasant, lingering aromas with slightly different notes depending on the essential oil used. Preparation and evaluation of perfume was done. The physiochemical parameters of the prepared perfume were determined. The formulation exhibited good as appearance characteristics as well as the PH was found in range, 8. 9 which determined PH. Other parameters such as Hedonic test, sensory test, spot test were determined. The present work is concerned with the formulation of perfume using flowers. The formulated perfume had a good fragrance. It didn't cause any irritation to skin.

Conclusion:

This research was successfully synthesized benzanilide via the Beckmann rearrangement using conventional, acetic anhydride-assisted, and alum-catalyzed methods. The acetic anhydride method gave the best yield and purity, while the alum method proved to be the most environmentally friendly**.** This research highlights the importance of flowers as a natural source of aromatic compounds for preparing local perfumes and their potential role in improving the livelihood of growers. It attempts to explore the possibility of perfume manufacturing using flowers. Essential oils were successfully extracted from dried flower petals through solvent extraction and maceration methods, with solvent extraction yielding better results. The extracted essential oils were then effectively used in perfume formulation, combined with fixatives and solvents (such as alcohol) as key ingredients.

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