# The Wittig reaction of syringaldehyde (3,5-dimethoxy-4-hydroxybenzaldehyde)

A Project Report submitted by

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B24EDC011

Submitted in partial fulfilment of the academic requirements for the degree of

B.Sc.- B.Ed.

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Department Of Chemistry

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# Declaration

I hereby declare that the work presented in this project, titled “The Wittig reaction of Syringaldehyde (98%)”, is a genuine and original record of experiment conducted by me under the supervision of **Dr. Rohan Diliprao Erande**. This Project has been submitted to the Indian Institute of Technology Jodhpur in partial fulfilment of the academic requirements for the degree of Integrated Bachelor of Science and Bachelor of Education (B.Sc.- B.Ed.).

I further declare that this report, in whole or in part, has not been and will not be submitted to any other institution or university, either in India or abroad, for the award of any degree or diploma.

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# Acknowledgements

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My sincere thanks to the Department of Chemistry, IIT Jodhpur for making the advanced instrumental facilities available to me to carry out this study. I would like to thank all my chemistry teachers for their teachings that provided me the knowledge base for carrying out this research work.

I am also thankful to **Ms. Shalu Thakur** for sparing her precious time to guide me through this project.

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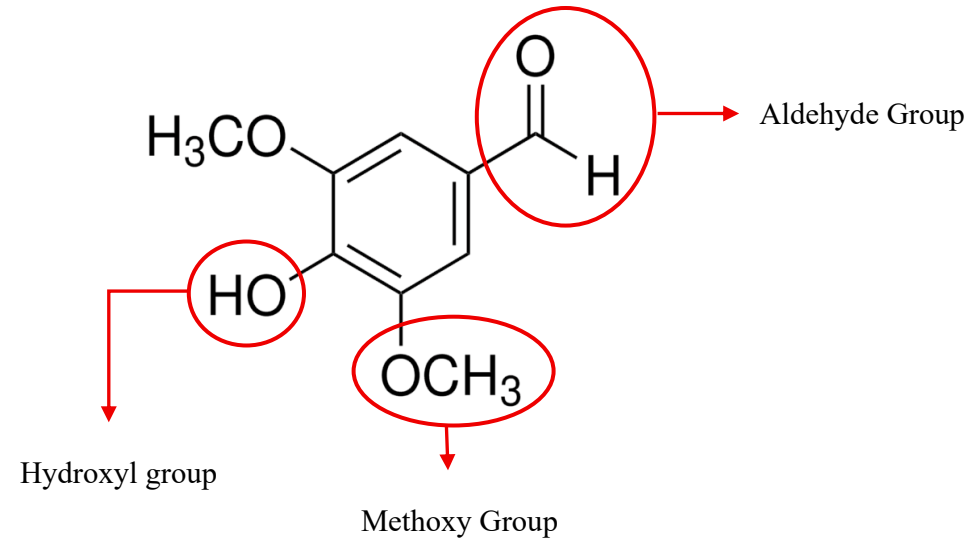
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# Introduction

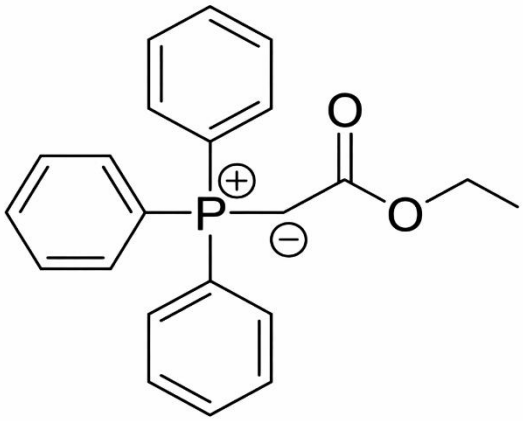
One carbon-carbon bond forming reaction is known as **Wittig reaction.** This reaction is employed for converting aldehydes or ketones into corresponding alkenes by reacting with phosphonium ylide. Wittig reaction was discovered by Georg Wittig. This reaction is well-known for its efficiency and control of producing alkenes. In this reaction, there are two intermediates formed namely betaine and oxaphosphetane which break down to form an alkene along with triphenylphosphine oxide.

**Syringaldehyde** is used as aromatic aldehyde in this experiment. It consists of two electron donating groups namely methoxy and hydroxyl group. These groups influence the reaction activity of carbonyl group by resonance effect. Wittig reaction of syringaldehyde shows how substituted alkenes are prepared and gives knowledge about reaction mechanisms, effect of substituents and various experimental techniques such as refluxing.




- **Syringaldehyde**

**DET Wittig Salt** -



# Motivation

Carbon-carbon double bonds constitute an important target in modern organic synthesis that is widely applied during the synthesis of functional compounds, natural products, and precursors for pharmaceutics and materials science. Wittig reaction represents a very efficient and selective approach to the alkene formation starting from commercially available carbonyls. Still, the ability to precisely tune the structure and properties of alkyl-substituted alkenes, including aromatic ones, containing several different functional groups, is of particular interest in research. Understanding the impact of the substituent on reactivity and selectivity of reaction products can facilitate the design of novel strategies and methods in organic synthesis.

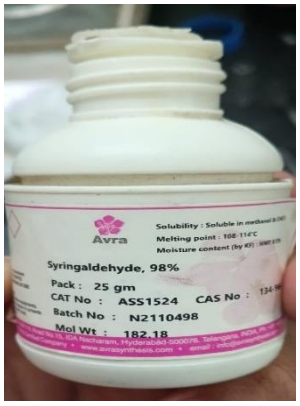
Syringaldehyde is an attractive example of a substituted aromatic aldehyde that contains methoxy and hydroxyl groups that have considerable effects on the reactivity of the aldehyde function. In this regard, the use of syringaldehyde in a Wittig reaction can allow one to explore the possibility to synthesize the desired products via this approach in the presence of such electron-donating substituents. In addition to obtaining valuable compounds, conducting this experiment would provide important data concerning the impact of substituent on the outcome of the reaction.

# Experimental Synthesis

**Preparation Of Phosphonium Ylide:**

First dissolve triphenylphosphine in ethanol in a round bottom flask, then add ethyl bromoacetate slowly (stir continuously). Now heat the mixture under reflux (60°-80°) for 4-12 hours. The crystals of phosphonium salt will form, cool and filter out the crystals and dry the product. Now add the phosphonium salt in DCM and add sodium hydride. Stir the mixture continuously to remove H2 gas. In the last our Phosphonium Ylide (Ph3P=CHCOOEt) is formed





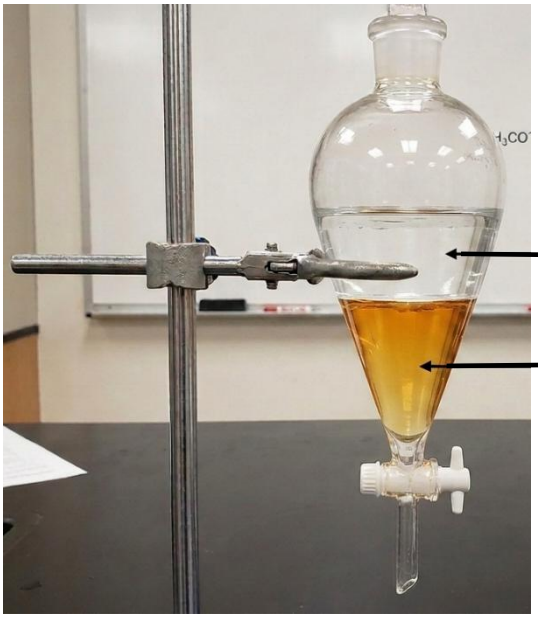

**Reaction Scheme:**

Take 200mg of Syringaldehyde in a cryovial and add DCM (approx. half of the volume of the cryovial). Then add 500mg of DET Wittig salt to the mixture slowly. After adding the complete salt, let the reaction undisturbed for 1-2 days. After 2 days, perform the TLC to check whether the product is formed or not.

**Syringaldehyde (200mg) + DET Wittig salt (500mg) ⟶ Ethyl-4-hydroxy-3,5-dimethoxycinnamte + Ph3P=O**

**Work – Up:**

Take a separatory funnel and add the product formed in it. Take the appropriate solvent pair to make two layers of solvent (aqueous and organic). Take water for the aqueous layer and DCM for the organic layer. Add both the solvents in the separatory funnel. Our product is an organic compound hence it will be soluble in the organic layer. Shake the separatory funnel properly, and hold it stable. After sometime, two separate layers will appear which are easily visible with our naked eyes. As the density of DCM is higher than water then the organic layer will appear at the bottom and the aqueous layer will appear at the top. Now separate out the organic layer in a conical flask by slowly opening the stopcock. Add sodium sulphate to remove water moity from the solution. In the last filter out the mixture by putting a funnel (cotton inside it) on a round bottom flask.

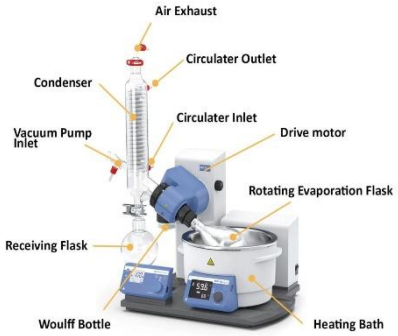


Aqueous Layer (Salts)

Organic Layer (DCM, Wittig product, Ph3P=O)

**Rotary Evaporation:**

It is a scheme to evaporate the solvent. Take the round bottom flask in which we have separated out the organic layer. Start the Rotary evaporator machine and set the pressure at 700mmHg and then start slowly decreasing the pressure till 100mmHg. The solvent will start evaporating at 150mmHg. After complete evaporation of the solvent, take the RB flask and add some silica to further performing Column chromatography.



**Column Chromatography:**

Take the Column tube and prepare the silica gel in a beaker by adding silica and hexane. Now fill the column to 1/3$^{rd}$ of the total volume with the silica gel and add hexane till all the silica settle down in the column. Then add the substance form the RB flask and prepare a 4% solution of ethyl acetate in hexane. Continuously add the 4% solution to the column. After sometime different coloured layer will appear at different levels. Collect all the layers in different test tubes. Take TLC of compound in the test tubes with a gap of 2 test tubes. Identify which one have the product and which one has the biproduct or the reaction mixture. Take the test tubes which contain our product and pour the contents into RB flask. Again, do the Rotor Evaporation of the content that we have collected in the RB flask. Hence when the solvent is completely evaporated, the rest content will be our product. Collect it and send it for Different compound identification techniques like UV, IR, HRMS and NMR, etc.



# Result & Discussion

- **Structure Identification of the Product:**

## 1. Proton NMR

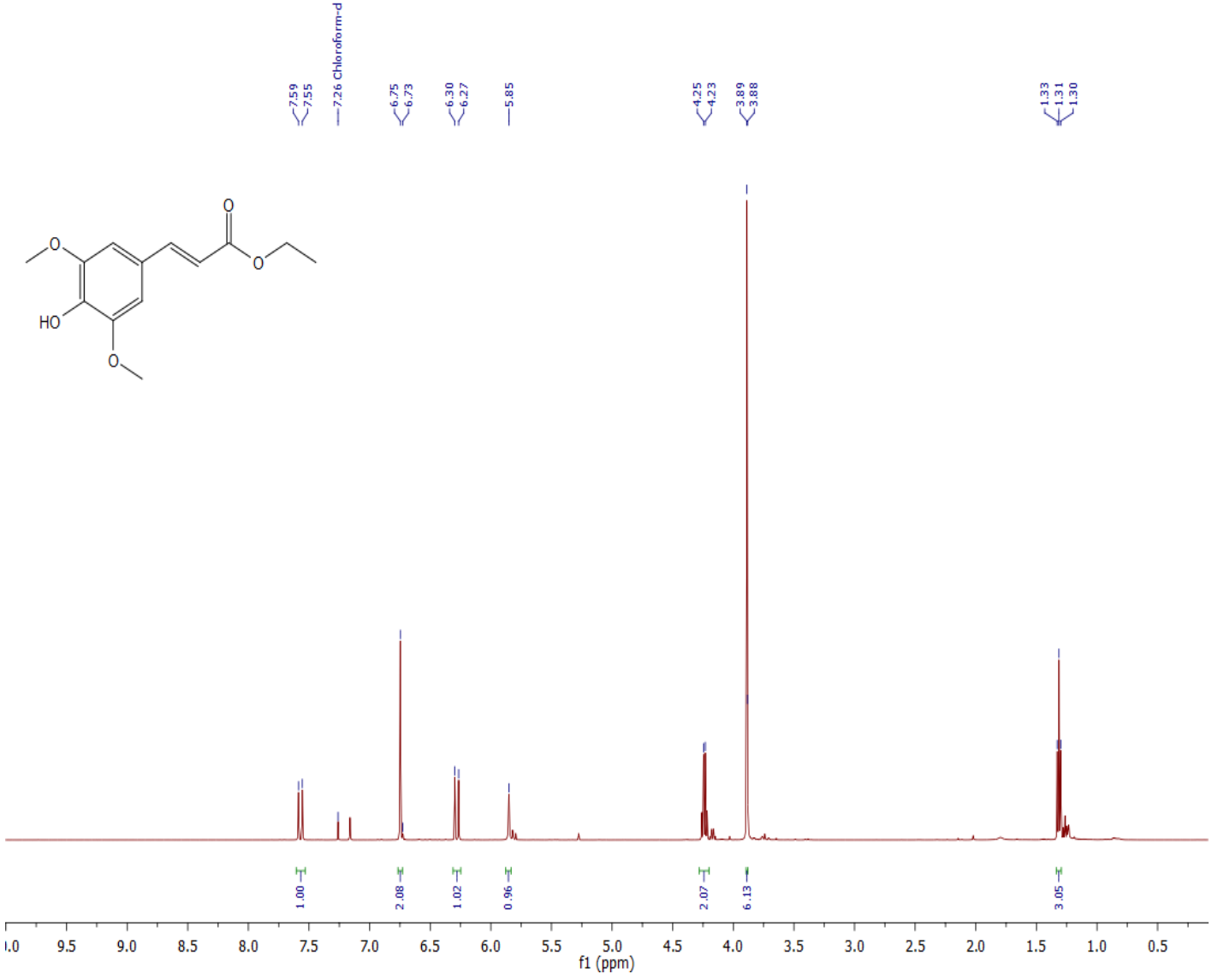



Fig.1: Proton(1H) NMR

**Data Interpretation of 1H NMR:**

| | |
|---|---|
| 7.57 (d, J = 15.9 Hz, 1H) | CH – C |
| 6.75 (s, 2H) | CH2 – C |
| 6.28 (d, J = 15.9 Hz, 1H) | CH – CH2 |
| 5.85 (s, 1H) | - O – H |
| 4.24 (d, J = 7.1 Hz, 2H) | CH2 – CH2 |
| 3.89 (d, J = 2.3 Hz, 6H) | 2* CH3 – O |
| 1.31 (t, J = 7.1 Hz, 3H) | CH3 – CH2 |

## 2. Carbon13 NMR:

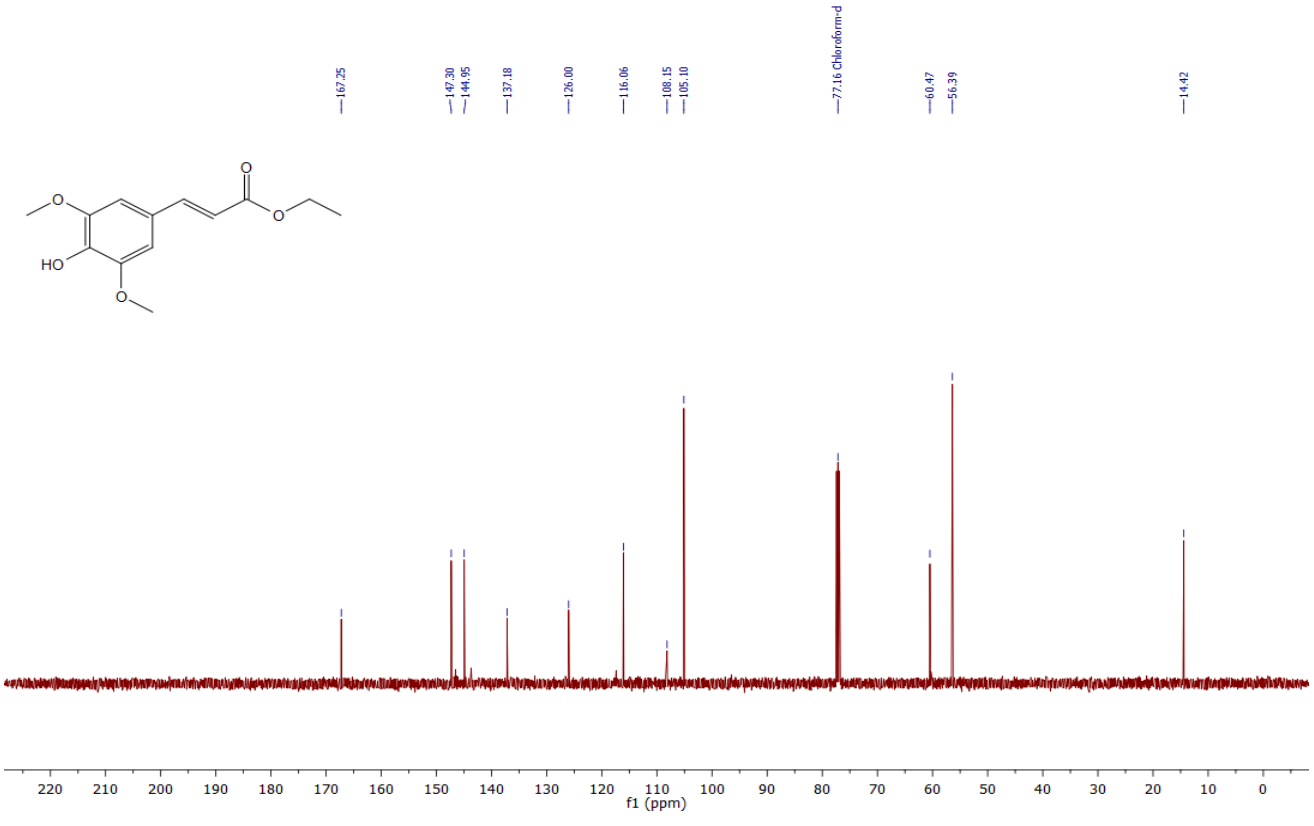



Fig.2: Carbon13 NMR

- **Yield percentage of the Product:**

**Initial compound:** 1. Syringaldehyde (MW = 182.17) = 200 mg, 1.09 mmol

2. DET Wittig salt (MW = 371) = 500 mg, 1.34 mmol

**Limiting Reagent:** Syringaldehyde

**Theoretical Yield:** T.Y. = no. of moles of syringaldehyde * Product Molecular weight (200)

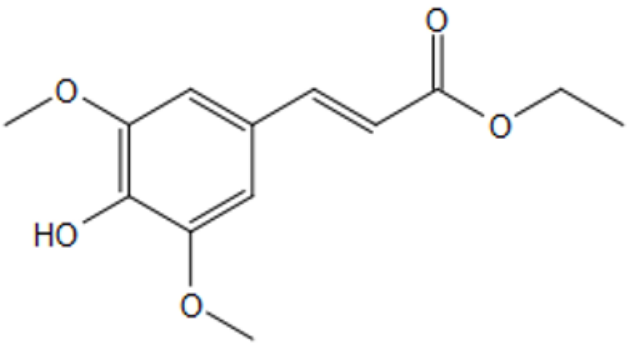
T.Y. = 1.09 * 200 = 218 mg

**Experimental Yield:** E.Y. = 166.4 mg

**Percentage Yield =** (166.4 / 218) * 100

**Yield %** = 76.3 %

- **Final Product:** Ethyl-4-hydroxy-3,5-dimethoxycinnamate




- **By-Product:** Triphenylphosphine Oxide (Ph3P=O)

# Conclusion

The Wittig reaction of syringaldehyde would entail optimizing the reaction conditions through base and solvent screening utilizing Sodium hydride and solvents such as Tetrahydrofuran and Dimethylformamide.

Inclusion of diverse substrates like aldehydes and ylides may be helpful for generalizing the reaction and developing an extensive reaction library.

Green Chemistry techniques such as environmentally friendly solvents and reduction of triphenylphosphine oxide as waste could be explored to make the process sustainable.

Additional modifications of the resultant alkene derivatives can facilitate its use in drugs, material development, and organic natural product synthesis.

# References


1. Wittig Reaction (Wikipedia)
2. ACS Journal of Organic Chemistry
3. PMC Review – Wittig Reaction Conditions
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