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Synthesis And spectral Analysis of 7-hydroxy-4-methyl coumarin7-Hydroxy-4Styryl-2H-chromen-2-one

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Abstract: *Based on the observed biological activities of Coumarin and chalcones, we have synthesized Coumarin -chalcone hybrids with the aim of evaluating their anti-oxidant properties. All derivatives have been proved to be good anti-oxidants. These preliminary findings encourage us to future structural optimization of these kind of compounds. Various Coumarins were synthesized and converted to Coumarin - Chalcone hybrids. Coumarins were synthesized by treating Salicylaldehyde and Ethyl acetoacetate in presence of piperidine as base and ethanol as solvent. Then Coumarins were converted to Chalcones by reacting with 3-acetyl Coumarin with aldehydes to give Coumarin Chalcone hydrides. A solution of 1.1 gram of resorcinol and 1.2 gram of EAA was added drop wise with stirring to 10ml of conc.H_2SO_4. So that the temperature of reaction mixture did not raise above the 10°C the reaction on complete addition mixture was kept at ambient temperature for 18 hours and then poured with vigorous stirring to reaction mixture of ice and water. A mixture of 7-Hydroxy-4-methyl-coumarin (1eq.) and corresponding benzaldehyde (1.2eq.) in EtOH was stirred with a few drops of piperidine under reflux during 2-12 hours. Mixture was cooled and the resulting solid was obtained.Analysized by FTIR and H^1NMR.*

Keywords: *Synthesis of 7-hydroxy-4-methyl coumarin, 7-Hydroxy-4Styryl-2H-chromen-2-one, FTIR, H^1NMR.*

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I. Introduction

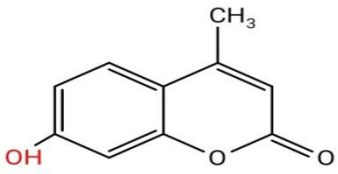
Coumarins are one of the important structural units and widely found in nature they show diverse biological and pharmacological activities ranging from antimicrobial, anti-arrhythmic, antitumor, antifungal, anti-HIV, anti-osteoporosis to anti-inflammatory. Apart from their pharma Chemotherapy, is the treatment of diseases by chemicals especially by killing micro-organisms or cancerous cells. Nowadays are known a wide range of different chemotherapeutic agents. Drug Discovery plays an important role in day today life. Lot of drugs have been synthesized for various biological activities such as anti-cancer, anti-diabetic, anti-microbial, anti-oxidant, anti-malarial, anti-fungal. Chalcones or benzylidene acetophenone are the important constituents of natural sources. It was first isolated from Chinese liquorice. It has 1, 3-diaryl-1-ones skeletal system. Which was recognized as the main pharmacophore for chalcones. From plants, stable chalcone synthetase (CSH) which immediately converts chalcone into flavanone. Various Coumarin were synthesized and converted to Coumarin Chalcone hybrids. Coumarins were synthesized by treating salicylaldehyde and ethyl-acetoacetate. In presence of piperidine as base and ethanol as solvent. Then coumarin chalcone hybrids. Pharmaceutical application, coumarins have been used as additives in foods, perfumes and cosmetics as well as in the preparation of optical brighteners, laser dyes, fluorescent labels and nonlinear optical chromophores.

Synthesis:
All reactions were carried out under dry and deoxygenated argon Atmosphere. Solvents were used as anhydrous by reflux of each solvent over an appropriate dryer agent and further distillate under argon atmosphere.

2.1) Synthesis of 7-hydroxy-4-methyl coumarin by conventional method with conc. Sulphuric acid as catalyst:

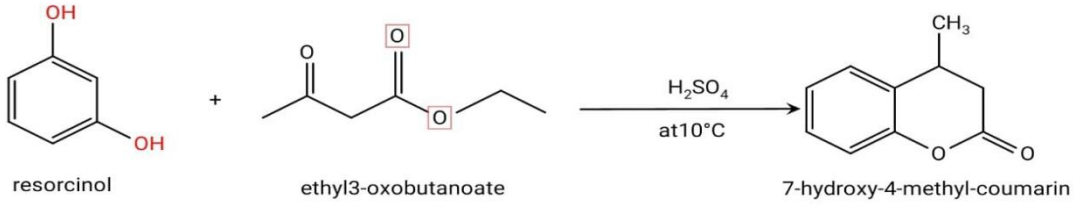
A solution of 1.1 gram of resorcinol and 1.2 gram of EAA was added drop wise with stirring to 10ml of conc.H_2SO_4. So that the temperature of reaction mixture did not raise above the 10°C the reaction on complete addition mixture was kept at ambient temperature for 18 hours and then poured with vigorous stirring to reaction mixture of ice and water. The precipitate was filter off and wash with cold water then dried under reduced pressure offered the crude solid mass. On recrystallized from aq. Alcohol gives final compound.

Chemicals: Resorcinol (1eq.), Ethyl-acetoacetate (1.2eq.), conc.H_2SO_4, Ethanol.
Structure:



7-hydroxy-4-methyl-2H-chromen-2-one

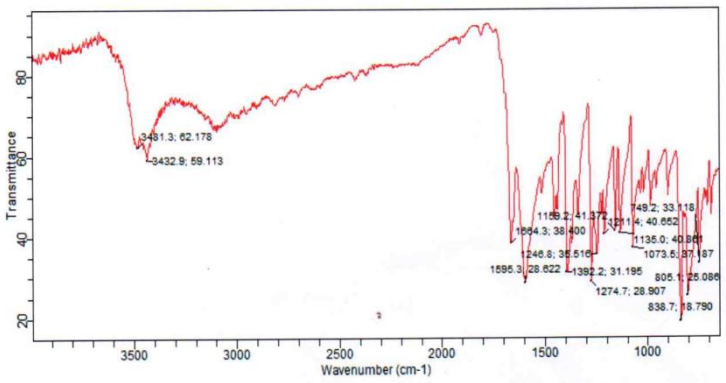
Reaction:



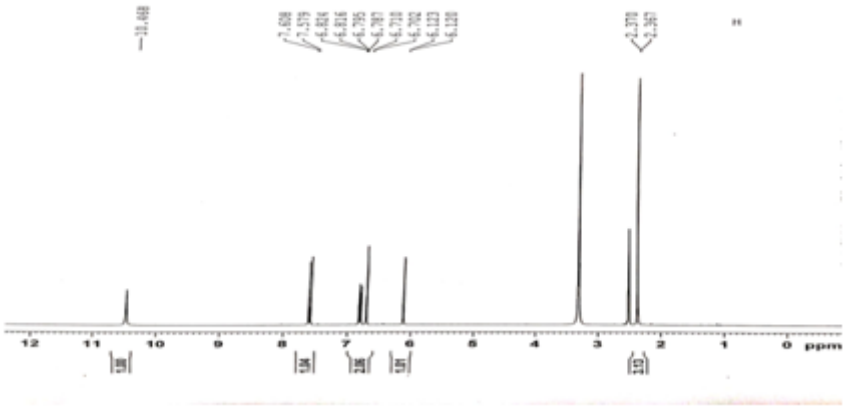
resorcinol ethyl3-oxobutanoate 7-hydroxy-4-methyl-coumarin

- **Tabular Column:**

Compound	Molecular weight	Quantity	Moles	Equivalent weight
Resorcinol	110.1 gram/mole	1.1gm	0.001198	1eq
EAA	130.1 gram/mole	1.55gm	0.01198	1.2eq
H_2SO_4	10ml	-	-	-



- C=C stretching at 1595cm^{-1}
- C-O-C stretching at 1159cm^{-1}
- C=O stretching at 1884cm^{-1}
- C-H stretching at 3481cm^{-1}
- O-H stretching at 3432cm^{-1}



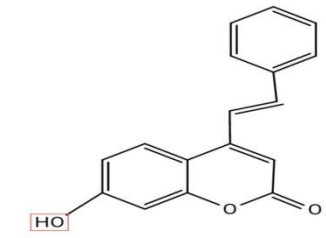
- δ2.45 (s, 3H) of ($–CH_3$)
- δ6.8 (d, 1H) at (6 position)
- δ6.1 (s, 1H) at (8 position)
- δ7.8 (d, 1H) at (4 position)
- δ10.6 (s, 1H) at (-OH)

Characterization:
- **Yield**: 36.08%
- **Melting Point**: 183°C

Synthesis of 7-Hydroxy-4Styryl-2H-chromen-2-one:

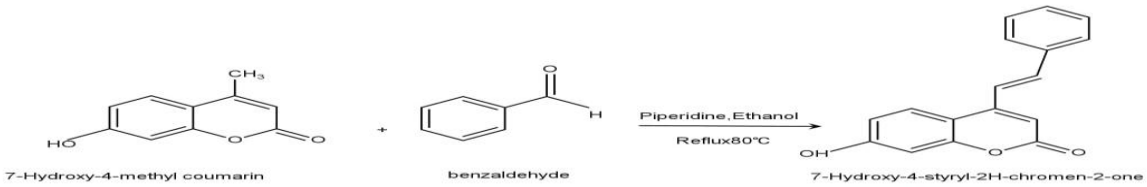
A mixture of 7-Hydroxy-4-methyl-coumarin (1eq.) and corresponding benzaldehyde (1.2eq.) in EtOH was stirred with a few drops of piperidine under reflux during 2-12 hours. Mixture was cooled and the resulting solid was filtered and purified by recrystallization. Purification of compounds was made by recrystallization in MeOH.

Structure:



7-hydroxy-4-styryl-2H-chromen-2-one

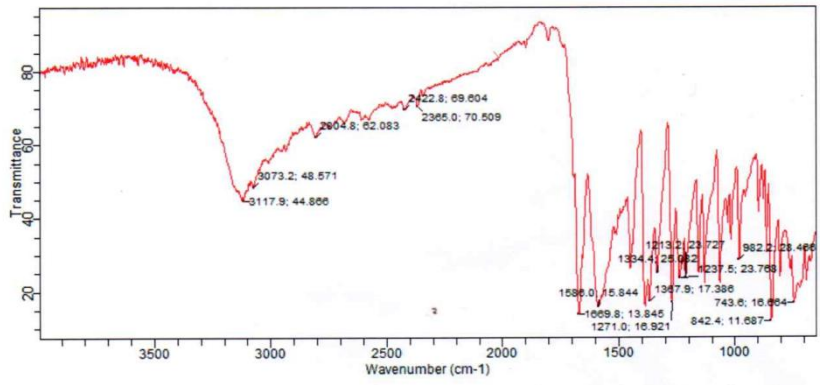
Reaction:



7-Hydroxy-4-methyl coumarin benzaldehyde 7-Hydroxy-4-styryl-2H-chromen-2-one

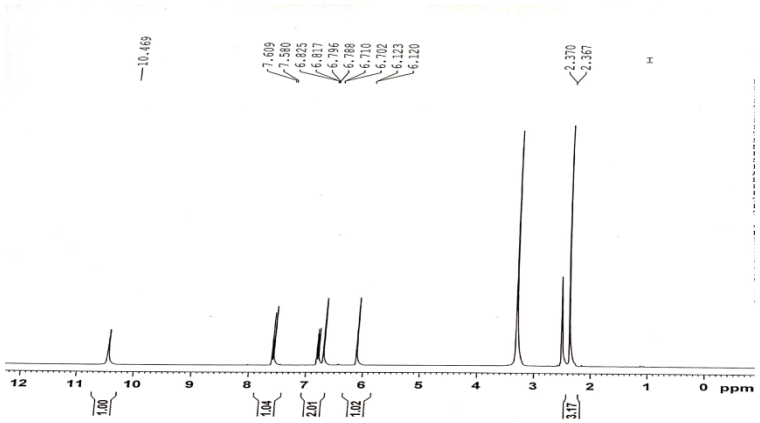
Tabular Column:

Compound	Molecular weight	Quantity	Moles	Equivalent weight
7-Hydroxy-4-methyl coumarin	194.18 gram/mole	2.21gm	0.0108147	1eq
Benzaldehyde	106.13 gram/mole	1.377gm	0.0129776	1.2eq
Piperidine	2-3 drops	-	-	-
Ethanol	10ml	-	-	-



- C=C stretching at 1588cm^{-1}
- C-O-C stretching at 1237cm^{-1}
- C=O stretching at 1889cm^{-1}

- C-O stretching at 1213cm^{-1}
- C-H stretching at 3073cm^{-1}



- δ2.2 (d, 2H) of (-2H)
- δ6.1 (s, 1H) at (8 position)
- δ6.8 (d, 1H)
- δ7.8 (d, 1H)
- δ10.6(s,1H) of (-OH)

Characterization:

- **Yield**: 40.48%
- **Melting Point: 224°C**

II. Result & Discussion

Sl. No.	Name of the Product	Practical yield	M.P
1.	7-hydroxy-4-methyl coumarin	36.08%	183°C
2.	7-hydroxy-4-styryl-2H-chromen-2-one	40.48%	224°C

7-Hydroxy 4-methyl Coumarin is Synthesized from Resorcinol and Ethyl aceto acetate was added drop wise. With stirring to 10ml concentrated sulphuric acid. Didn't 10°c further 7-hydroxy 4-methyl Coumarin is converted to Coumarin Chalcone Hybride. (7-hydroxy-4-styryl-2-one). It was prepared by refluxing 7-hydroxy 4-methyl Coumarin and Benzaldehyde in presence of piperidine using Ethanol as a solvent.

III. Conclusion

In this study, we synthesized various substituted Coumarins and they were converted to Coumarin-Chalcone hybrids. All the compounds that we synthesized were characterized by H1NMR, IR and their melting points.

Reference

[1]. Basangouda, M.; Kulkarni, M. V.; Sharma, D.; Gupta, V. K.; Sandhyarani, P. and Sasal, V. P. J. Chem. Sci. 2009, 121, 485-495.
[2]. Liu, X.; Dong, M.; Chen, X.; Jiang, M.; Lv, X. and Zhou, J.Appl Microbiol Biotechnol. 2008, 78, 241-247.

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