

Experiment No. 02

AIM: To prepare and submit 7-hydroxy-4-methyl coumarin from Resorcinol.

REFERENCES:

1. Vogel's Textbook of Practical Organic Chemistry by Brian S. Furniss, Antony J. Hannaford, Peter W. G. Smith & Austin R. Tatchell; Fifth Edition; Page No. 1193.

REQUIREMENTS

Chemicals: Conc. sulphuric acid, Resorcinol, Ethyl acetoacetate, Ice, Sodium hydroxide solution (5%), Sulphuric acid (2 M), Ethanol (95%).

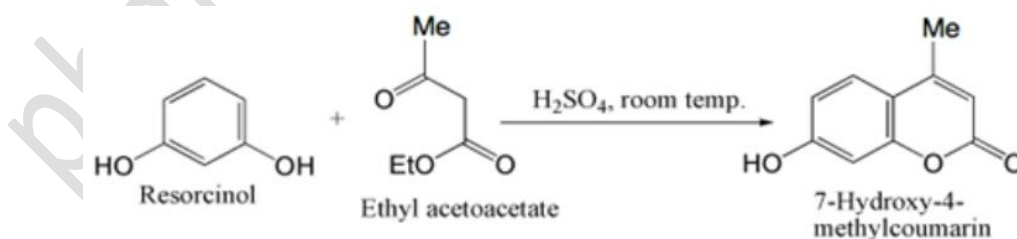
Apparatus: Three necked flask – 3 liters, Thermometer, Mechanical stirrer, A dropping funnel, Beaker, Buchner funnel, Measuring cylinder, Filter paper.

PRINCIPLE:

The synthesis of coumarins generally involves the reaction of a phenol with a β -ketoester in the presence of an acid-condensing agent, known as the Pechmann reaction. Concentrated sulfuric acid is typically used as the condensing agent for simple monohydric phenols and β -ketoesters, though phenol reacts more effectively with aluminum chloride.

The reaction mechanism is believed to start with the formation of a β -hydroxy ester, which then cyclizes and dehydrates to produce the coumarin. Polyhydric phenols, especially those with meta-oriented hydroxyl groups, react very readily. For these phenols, sulfuric acid is used as the condensing agent, with careful temperature control to ensure a good yield.

REACTION:



Use:

It is used commercially as a laser dye and as the starting material for producing the insecticide hymecromone.

PROCEDURE:

1. Take 1 liter of concentrated sulfuric acid in a 3-liter, three-necked flask fitted with a thermometer, mechanical stirrer, and a dropping funnel. Immerse the flask in an ice bath.
2. When the temperature falls below 10°C, add a solution of 100 g (0.91 mol) of resorcinol in 134 g (130.5 ml, 1.03 mol) of redistilled ethyl acetoacetate dropwise with stirring. Maintain the temperature below 10°C using an ice-salt bath during the addition (2 hours).
3. Keep the mixture at room temperature for 18 hours and pour the mixture into 2 kg of crushed ice combined with 3 liters of water, stirring vigorously.
4. Collect the precipitate by suction filtration and wash it with three 25 ml portions of cold water.
5. Dissolve the solid in 1500 ml of 5% sodium hydroxide solution, then filter. Add dilute 2 M sulfuric acid (about 550 ml) with vigorous stirring until the solution is acidic to litmus.
6. Filter the crude 4-methyl-7-hydroxycoumarin using suction filtration and Wash the product with four 25 ml portions of cold water.
7. Dry the product at 100°C and Recrystallize from 95% ethanol.
8. The pure compound will separate as colorless needles with a melting point of 185°C.

CALCULATION:

Here, the limiting reagent is resorcinol; hence, the yield should be calculated from the amount taken.

Molecular formula of resorcinol = $C_6H_6O_2$

And molecular formula of 7-hydroxy-4-methyl coumarin = $C_{10}H_8O_3$

Molecular weight of resorcinol = 110 g/mole

And molecular weight of 7-hydroxy-4-methyl coumarin = 176 g/mole

Theoretical yield:

110 g resorcinol forms 176 g 7-hydroxy-4-methyl coumarin

Therefore, 100 g resorcinol will form? (X) g 7-hydroxy-4-methyl coumarin

= 160 g

Theoretical yield = 160 g

Practical yield = _____ g

% Yield = (Practical Yield)/(Theoretical Yield) × 100

RESULT:

7-hydroxy-4-methyl coumarin was synthesized from acetanilide and submitted.

Name of Compound	7-hydroxy-4-methyl coumarin
Theoretical yieldgm
Practical yieldgm
% Practical yield%
Melting point°C

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