Experiment No. 3

**Cyclization of (4-hydroxy)salicylaldehyde with propanoic anhydride**

**to give 7-hydroxy-3-methylcoumarin**

**Aim**: To synthesize 7-hydroxy-3-methylcoumarin by cyclization of (4-hydroxy)   
 salicylaldehyde with propanoic anhydride

**Chemical**: 2,4-dihydroxybenzaldehyde, propanoic anhydride, sodium propanoate,   
 piperidine, hydrochloric acid and sulfuric acid

**Apparatus**: 100 mL round bottom flask, 250 mL conical flask, funnel, filter paper, pH paper,   
 condenser, glass stopper, glass rod, stir bar, hot plate, heating   
 mantle, thermometer, glass column, and test tubes.

**General procedure:**

A mixture of 2,4-dihydroxybenzaldehyde (7.20 mmol), sodium propionate (15.6 mmol), propionic anhydride (19.4 mmol), and piperidine (0.1 mL, 1 mmol) was refluxed for 3 h, and then poured onto ice. The aqueous mixture, made acidic with a 0.1 N solution of HCl, yielded a precipitate that was filtered and treated under stirring with concentrated H2SO4 (2 mL). The resulting mixture was poured onto ice again, the precipitate was filtered, and the residue was purified by column chromatography on silica gel.



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**Result**: The product obtained was:

1.

2.

3.

4.

5.

Yield (%):

Melting point:

IR:

TLC:

Questions:

1. What is the mechanism of this reaction?

2. At the end of the reaction, why we need to use acids? Why not bases?