

Honors Cup Synthetic Proposal

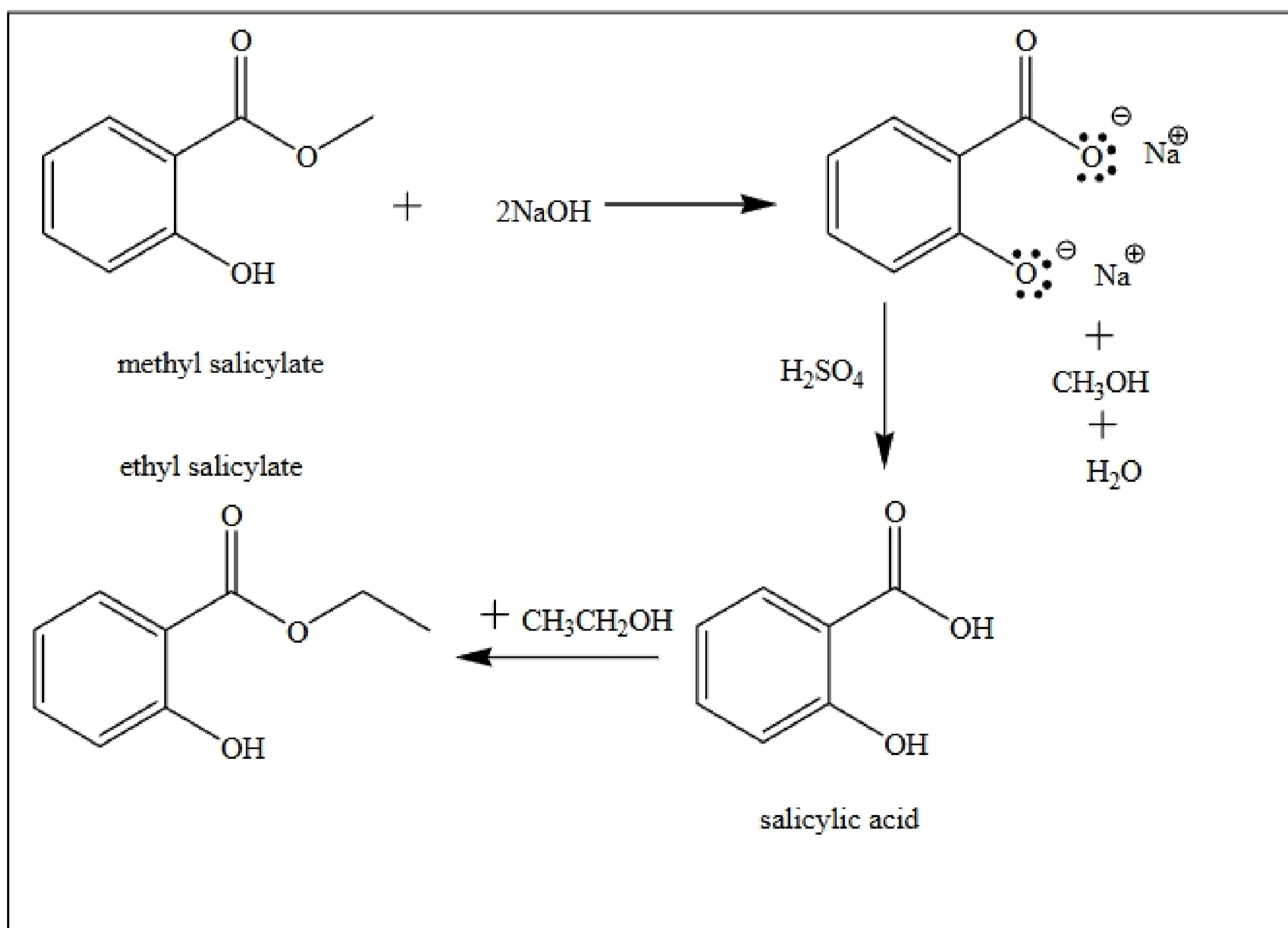
Section: 210 .

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Title: Synthesis of Ethyl Salicylate

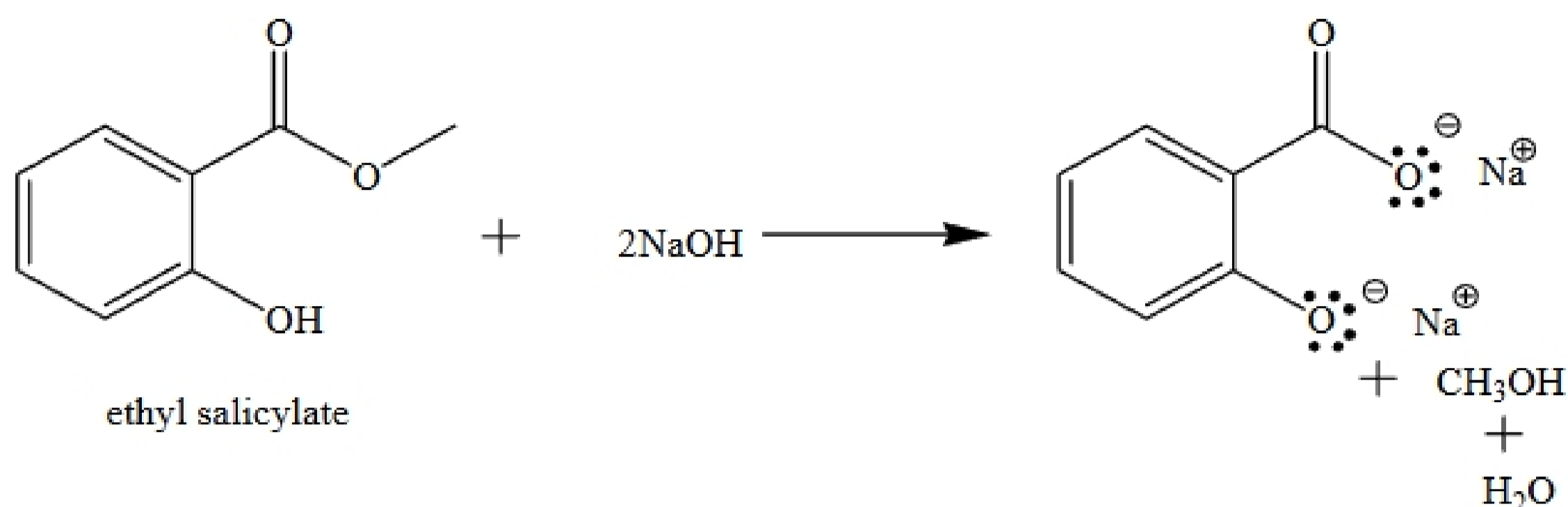
Introduction: Ethyl salicylate is a molecule that is naturally occurring in plants, fruits & vegetables. It is a natural preservative that is used in perfume and cosmetics and also has uses as an analgesic, antiseptic, and antipyretic. It has the odor of wintergreen/mint and is used in sweet floral, tutti frutti and spicy balsam essences. Ethyl salicylate is an ideal synthetic product because of the ease with which it can be identified due to its distinctive smell. Methyl salicylate also has a distinctive smell and the olfactory characteristics of these salicylate esters will help to reassure researchers that they are following the procedure correctly.

Overall synthetic reaction scheme:



Step 1

Synthetic transformation 1:



Experimental 1

Dissolve 16 g of NaOH in 80 mL of water. Allow the solution to cool to room temperature and add it to 8 g of methyl salicylate in a 200 mL round-bottom flask. Add two boiling chips and attach a reflux condenser with greased joints. Using a heating mantle, heat the solution slowly at first, then at vigorous boil for 10 to 20 minutes until the oily ester appears to be gone. Set aside one quarter of the solution for characterization.

To characterize the resulting ionic intermediate of this step, distill the solution to remove excess ethanol. Prepare the sample for IR, and for H-NMR use D₂O as the solvent. (Lambert & Mazzola 2004)

The scale has been doubled so that there will be enough product for characterization and further steps.

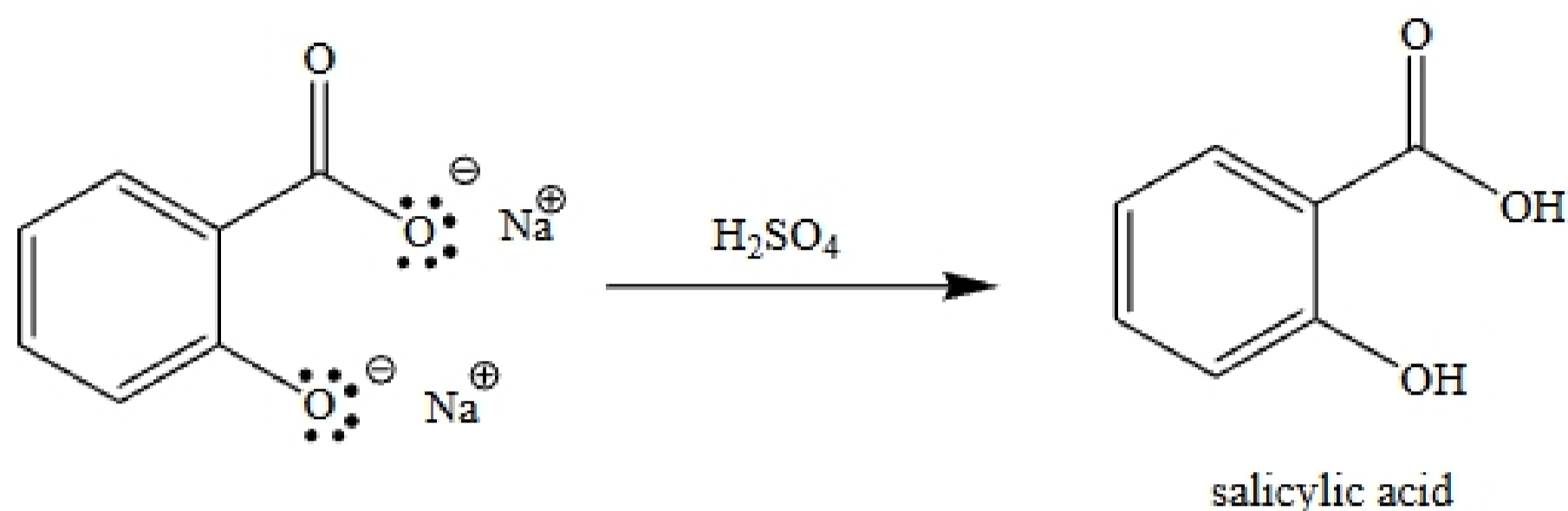
Expected yield: Ideal: 100% yield, 7.16 g. Due to error 60% yield expected, 4.30 g.

Safety, disposal and green issues 1:

Distillation will result in methanol as a byproduct. This can be disposed in the waste hood.

Step 2

Synthetic transformation 2:



Experimental 2

Cool the reaction solution from experiment 1 in an ice water bath. Pour it into a 100 mL beaker and cautiously add with stirring enough 3 M sulfuric acid to make the pH of the solution about 1. This will be approximately about 5 to 6 mL. With occasional stirring, cool the mixture of liquid and precipitate in an ice water bath to about 5°C. Collect the precipitate by vacuum filtration, rinse with ice cold water. Recrystallize the impure product from water in a 100 mL Erlenmeyer flask. Characterize the product using IR, NMR and TLC.

Expected yield: Ideal 100% 5.45 g. Due to error, 60% yield expected, 3.27 g.

Note: These molar amounts will not correspond to those of step 1 due to the fact that one quarter of the solution from step 1 was removed prior to beginning step 2.

Safety, disposal and green issues 2:

Excess methanol will be filtered out as a byproduct. This can be disposed in the waste hood.