

ASPIRIN

Last Update: 3/10/2009 10:57 AM

OBJECTIVE: To synthesize aspirin, isolate the product and perform some tests of its purity.

CONCEPTS:

Synthesis Product Starting Material
Theoretical yield Percent yield
Temperature dependence of solubility
Organic functional groups Condensation Reaction

TECHNIQUES:

vacuum filtration crystallization melting point
quantitative transfer of liquids and solids

EQUIPMENT:

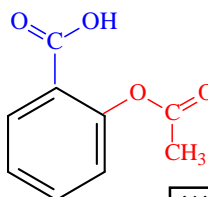
Buchner Funnel Filter Flask
Melting Point Apparatus

QUIZ 2 will be given at the beginning of the **synthesis of aspirin** exercise. It will cover:

SUSB-053, SUSB-017, SUSB-23
& SUSB-028

Possible **STARTING MATERIALS** for the synthesis of **ASPIRIN, C₉H₈O₄** ?

Does the structure of the desired product suggest a starting point for the synthesis?



At this stage, some detailed knowledge of chemistry becomes essential:

What **MATERIALS** are readily available, and

What **REACTIONS** are known that can assemble the required molecular fragments?

READILY ACCESSIBLE MATERIALS:

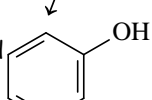
Benzene is produced by **DISTILLATION OF COAL**, or, as a byproduct of **CRACKING OF PETROLEUM (8.4 Mt)**



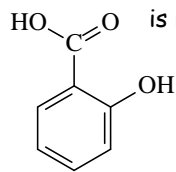
Benzene

Benzene is easily converted to **Phenol**

Phenol is easily converted to **Salicylic Acid** by reaction with **CO₂** and is readily available

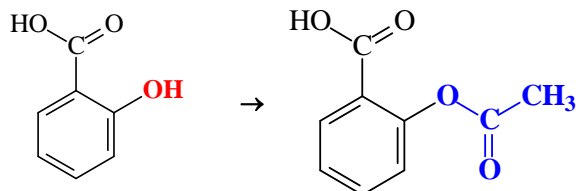


Phenol



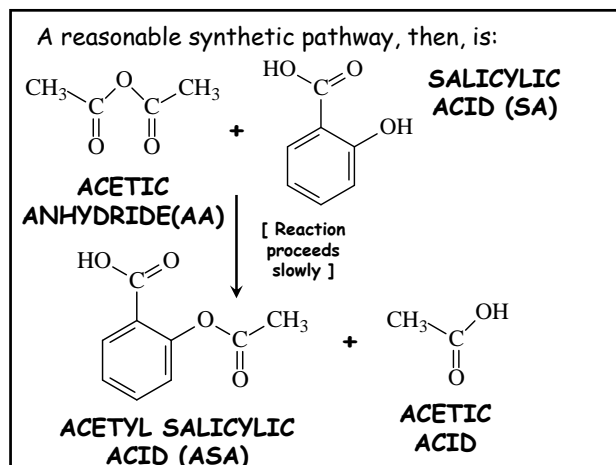
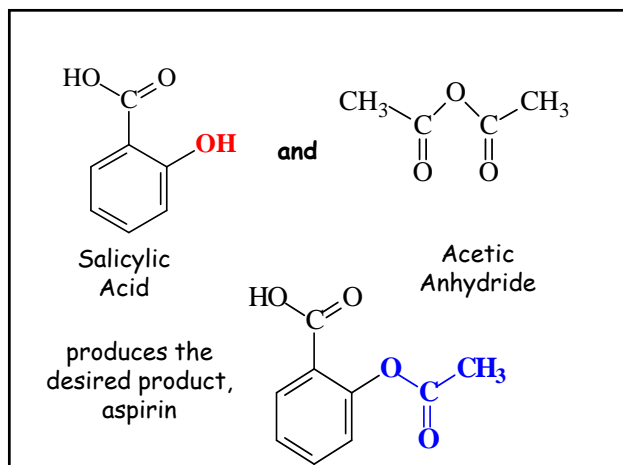
Salicylic Acid

All that would be needed is a **reaction** that converts the **OH** group on the ring to an acetyl group **OOCCH₃**



A reaction which accomplishes such a conversion, is:

The reaction of an **alcohol** or **phenol** with an **acid anhydride**, namely,



IN PRE-LAB, YOU SHOULD HAVE SHOWN THAT:

SALICYLIC ACID is LIMITING REAGENT

IF ALL SA became ASA (YIELD = 100%)

14 mmol SALICYLIC ACID

would react with

14 mmol ACETIC ANHYDRIDE

to produce

14 mmol ASPIRIN

and

14 mmol ACETIC ACID

What is in reaction vessel initially?

| | <u>INIT</u> mmol | <u>FINAL</u> (100%) |
|------------------|---------------------|---------------------|
| ASPIRIN | <u>0.0</u> | |
| ACETIC ANHYDRIDE | 42.0 | |
| SALICYLIC ACID | <u>14.0</u> | |
| ACETIC ACID | 0.0 | |
| SULFURIC ACID | 4.7 | |

ASPIRIN is only *slightly soluble* in water (3.3mg/mL)
We can add water to the reaction vessel to cause ASPIRIN to precipitate which we can then recover by filtration

Some of the water (28 mmol = .5 g) will react with the excess ACETIC ANHYDRIDE to form 28 mmol acetic acid.

Rest of the water will form a solution of all of the final products, insofar as they are soluble.

Since **ASPIRIN** is slightly soluble in water (**3.3 mg/mL**), using a *small* amount of water should cause the loss of very little aspirin.

e.g. If **50 mL** is used,
 $50 \text{ mL} \times 0.0033 \text{ g/mL} = 0.165 \text{ g}$ will be lost
 (out of a maximum of $\sim 2.5 \text{ g}$ produced)

CALCULATION OF YIELD

Suppose you use **2.37 g SALICYLIC ACID**

2.37 g SA

$$\text{-----} = 1.72 \times 10^{-2} \text{ mol SA}$$

138 g / mol SA

If completely converted, you should get

$$1.72 \times 10^{-2} \text{ mol}$$

Suppose the **actual weight** of your (dry) product is **2.49 g**

$$\% \text{ YIELD} = 100 \times \frac{2.49 \text{ g}}{1.72 \times 10^{-2} \text{ mol} \times \text{MM}(\text{ASA})}$$

SUMMARY OF PROCEDURE

Record Data & Observations in Lab Notebook

Use **125 mL ERLLENMEYER FLASK**

- Weigh **SALICYLIC ACID** on top loading balance
- Transfer **ACETIC ANHYDRIDE** using DEWICK PIPET* - IN HOOD
- Add Conc. H_2SO_4 from DROPPER - IN HOOD [Don't handle bottle!]

* *Be sure you know how to use this pipet! If not, ask!*

- Add H_2O to reaction mixture in *Specified amounts*
- Filter using VACUUM FILTRATION apparatus
- Wash with SMALL AMOUNT of water
- Save SOLID!

Need **PRODUCT**
 for one more exercise

Measure total amount of water used for washing

PRECAUTIONS

ACETIC ANHYDRIDE: CORROSIVE,
 FLAMMABLE & LACHRYMATOR

CONC. SULFURIC ACID: VERY CORROSIVE

FERRIC CHLORIDE: CORROSIVE

SALICYLIC ACID & ASPIRIN: IRRITANTS
 DON'T INHALE DUST

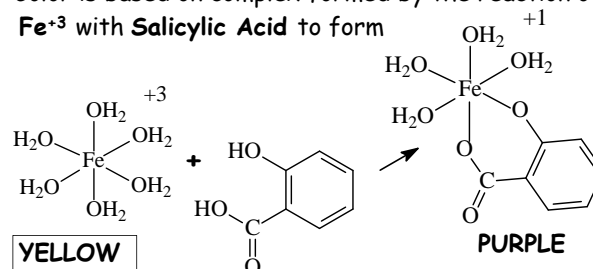
ETHANOL: TOXIC AND FLAMMABLE

DISPOSE OF EXCESS REAGENTS IN
 WASTE CONTAINERS

PERFORM QUALITATIVE TESTS

1. FERRIC CHLORIDE TEST:

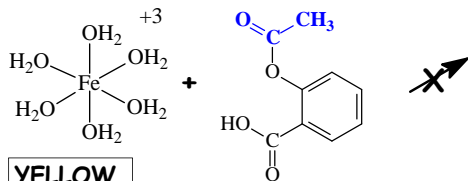
Color is based on complex formed by the reaction of Fe^{+3} with Salicylic Acid to form



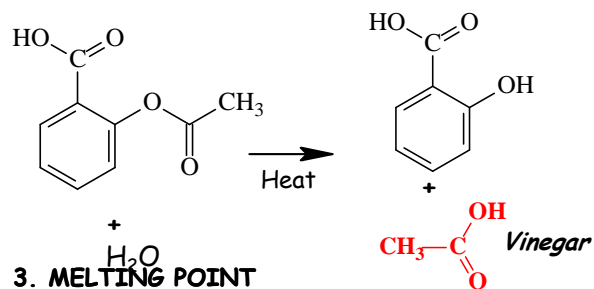
PERFORM QUALITATIVE TESTS

FERRIC CHLORIDE TEST:

The formation of a similar complex of Fe^{+3} with **ASPIRIN** is blocked by the CH_3CO group.



2. HEAT TEST - SMELL WITH CARE:



Dry a ***small amount*** (1/4 spatula-ful) of sample to determine the melting point of the product.

SAVE PRODUCT FOR THE WEEK AFTER NEXT'S EXERCISE !!!!!!!!!

Have data sheet initialed by TA and keep it until next week when you will reweigh the aspirin to get your YIELD

NEXT WEEK

Colorimetric Iron in Multivitamins
SUSB - 015 & SUPL - 005
Do Pre-Labs