32

Oct. 1, 2015

Experiment 45 - Synthesis of Aspirin

Pre-Lab Questions

The pre-lab questions should be completed first. It is best to keep them on a page separate from the rest of the experimental writeup.

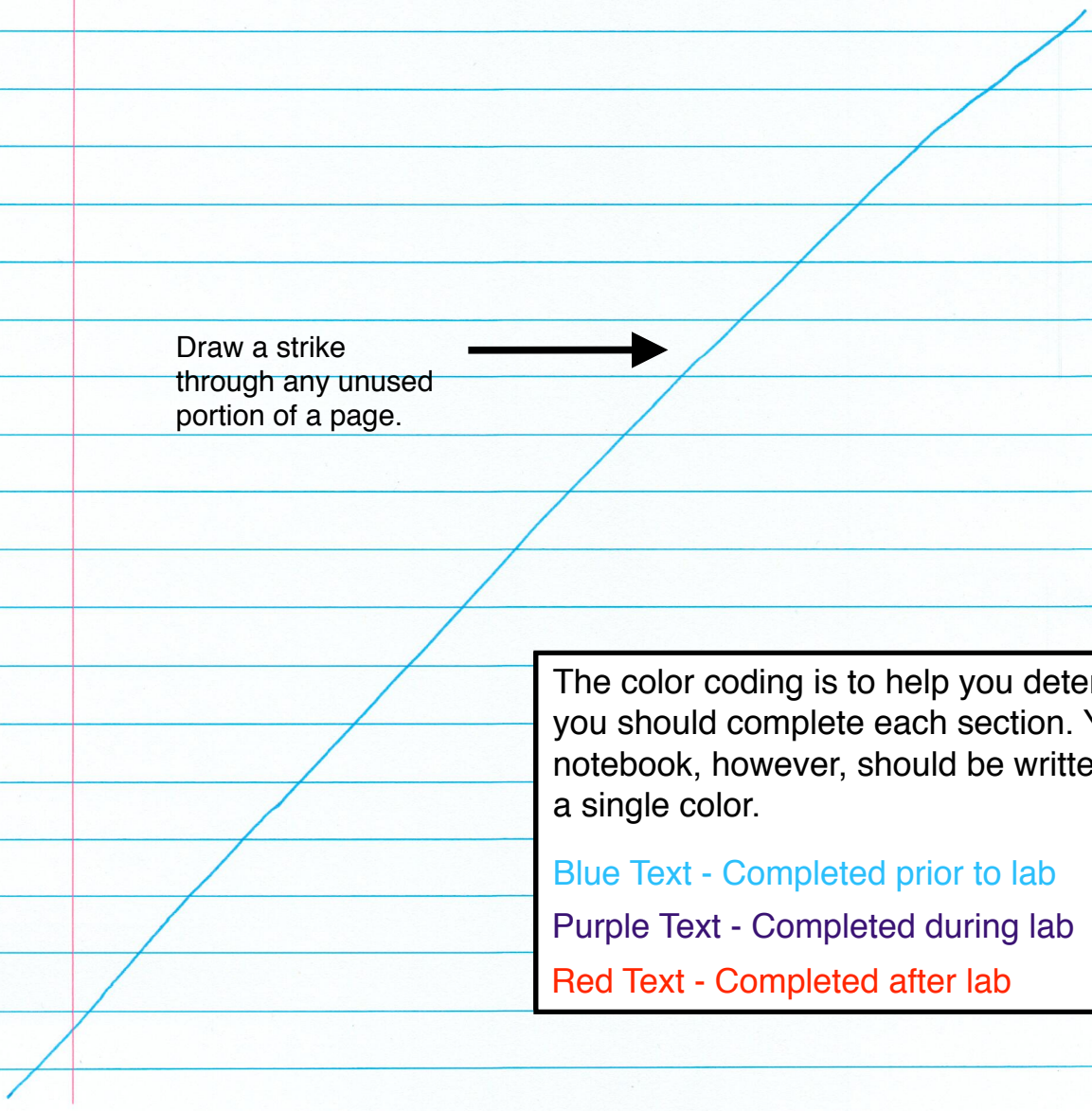
1) . . . . .

2) . . . . .

3) . . . . .

4) . . . . . .

Draw a strike through any unused portion of a page.



The color coding is to help you determine when you should complete each section. You notebook, however, should be written in pen in a single color.

Blue Text - Completed prior to lab

Purple Text - Completed during lab

Red Text - Completed after lab

Date → Oct. 1, 2015

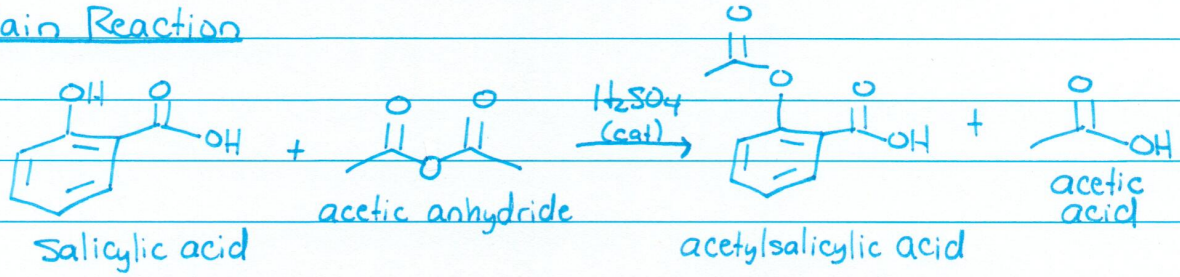
Expt. No. and Title

Experiment 45 - Synthesis of Aspirin

Purpose: Aspirin (acetylsalicylic acid) will be prepared from the reaction of salicylic acid and acetic anhydride in the presence of a catalytic amount of sulfuric acid. It is expected that a solid product will be obtained. This product will be characterized by melting point and IR spectroscopy.

Main Reaction

Write out main reaction. Include names if known.



If desired, you can also include the reaction mechanism here.

Table of Physical Properties

Strike errors with a single line

Physical properties of all reagents and products.

Name	Mol. Wt.	d (g/mL)	BP (°C)	MP (°C)
Salicylic acid	138.1	~~1.08~~	—	157-159
acetic anhydride	102.1	1.08	139	—
Sulfuric acid	98.1	1.84	—	—
acetylsalicylic acid	180.2			

If product is a new compound, the physical properties can not be looked up.

Hazards / Precautions

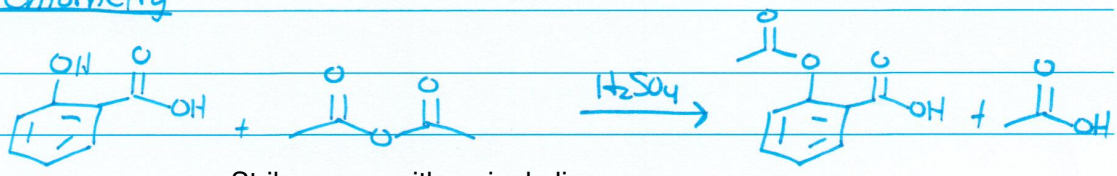
Look up and record any pertinent hazards associated with the reagents.

H_2SO_4: Corrosive; causes skin burns and serious eye damage

acetic anhydride: flammable; causes skin burns and eye damage; toxic if inhaled

Salicylic acid: harmful if swallowed; causes serious eye damage

Rxn Stoichiometry



In microscale, mmol is more convenient than moles. 1 mol = 1000 mmol

Strike errors with a single line

Equiv	1	3	Cat.
mmol	~~202 mg~~ 1.46 mmol	4.38 mmol	–
mass	202 mg	447 mg (0.414 mL)	drop

These are the masses actually used.

Equivalents (equiv.) is the ratio of reactants (i.e. 3 mol of acetic anhydride for every one mole of salicylic acid).

Volume was calculated from the density listed in the table of properties

Experimental Procedure

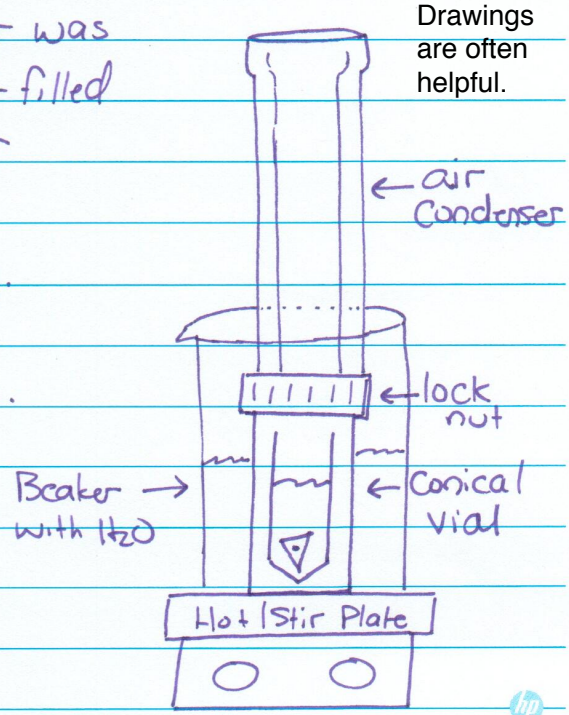
Write out procedure stepwise as it is actually performed.

- Equipped a 5 mL conical vial with a spin vane.
- Added 202 mg of salicylic acid.
- Commenced stirring.
- Added 0.414 mL of acetic anhydride via syringe.
- Added two drops of H_2SO_4 via pipet.
- Equipped vial with an air condenser secured via an O-ring and lock nut (see below).
- The vial with attached condenser was placed in a 100 mL beaker half-filled with water on top of the stir plate.
- The water was heated to boiling.
- Once boiling, the rxn was stirred at that temp for 5 min.
- Removed vial + added 0.5 mL H_2O to quench rxn.

Record any unusual observations → (Some fizzing occurred)

- An additional 2 mL of water was added to the vial.

Drawings are often helpful.



- The soln was then cooled in an ice bath for 5 min.
- Crystallization occurred spontaneously without any initiation.
- The white solid product was collected by filtration using a Hirsch funnel. The vacuum was maintained for 10 min to aid in drying the crystals
- The solid product was further purified by recrystallization from toluene and hexane.
- 2 mL of hot toluene was added to the impure solid in a small erlenmeyer flask. Hexane was then added dropwise until crystallization just began to commence. This took about 0.7 mL of hexane.
- The soln was cooled to room temp and then further cooled in an ice bath for 5 min.
- The crystals were collected on a Hirsch funnel and washed with 1.5 mL of hexane.
- The crystals were then scraped onto a weighed watch glass (15.3751g) and allowed to air dry for 20 min.

Results, Data, and Calculations

Product = white crystalline solid

Mass: 15.5870 g (watch glass + product)
- 15.3751 g (empty watch glass)
0.2119 g => mass of product recovered

Melting point: 132-134 °C

IR Data: 3100 cm^{-1} (broad), 2983 cm^{-1}, 2891 cm^{-1}, 1750 cm^{-1}, 1700 cm^{-1}, 1600 cm^{-1}, 1450 cm^{-1}, 1300 cm^{-1}, 1190 cm^{-1}

Recording IR data will be discussed later in the semester

Theoretical Yield: $1.46\ mmol\ SM \times \frac{1\ mol\ Prod}{1\ mol\ SM} \times \frac{180.2\ mg\ Prod}{1\ mmol\ Prod} = 263\ mg\ Product$

Do calculations such as theoretical yield in your notebook

Percent Yield = $\frac{212\ mg}{263\ mg} \times 100\% = 80.6\%$

Always calculate a percent yield.

Percent yield is different than percent recovery.

Conclusions

Summarize the experiment and the results. Include any potential suggestions for improvement.

Overall the experiment was successful and gave the desired product (acetylsalicylic acid) in 80.6% yield. The structure of the product was confirmed by mp and IR data. The experimental melting point of 132-134 °C is just slightly lower than the literature value (134-136°C), which could be due to the presence of minor impurities. The recorded IR spectrum matches that of the IR reported in the literature. Major absorbances include: 3100 cm^{-1} (OH); 1750 cm^{-1} (C=O); 1700 cm^{-1} (C=O); 1600 (arene C=C). In the future, it might be worth investigating a different solvent pair for recrystallization, which may improve the yield and help to remove ~~the~~ additional impurities.

Post-Lab Questions

1) . . . . . .

Post-Lab Questions can be completed at the very end.

2) . . . . . .

3) . . . . . .

Overworked Student 9/2/15

Sign and date your experiment when finished
You can sign and date before or after the post-lab questions.