SAMPLE PRELAB AND REPORT FOR AN EXPERIMENTAL PROJECT

INSTRUCTIONS:

- Complete the sections in **blue** for the **prelab**
- Complete the sections in **black** for the **report**. In other words**,** after completing the experimental work

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SYNTHESIS OF ACETYLSALICYLIC ACID (ASPIRIN)

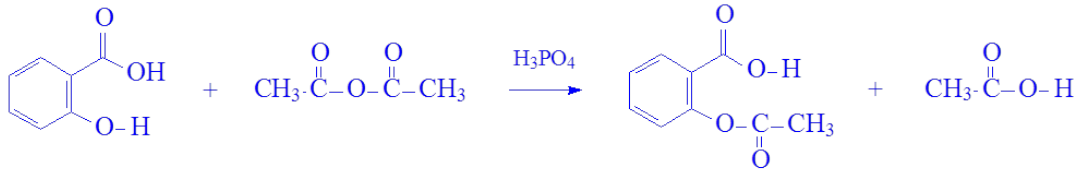
11 Jan 2022
Petra

SPECIFIC EXPERIMENTAL OBJECTIVES [1, 2, 3]

- *To synthesize aspirin (acetylsalicylic acid) by the acid-catalyzed acetylation of salicylic acid with acetic anhydride*
- *To determine the purity and structure of synthesized aspirin using various quantitative analytical techniques (mp, TLC, IR spectroscopy)*
- *To determine the purity of synthesized aspirin using $FeCl_3$ test for the presence of salicylic acid.*

*Depending on the project, you may have to include an **ABSTRACT** and/or an **INTRODUCTION**. Please refer to project document for more detail.*

REACTION: *Products and secondary products formed by the acid-catalyzed (H_3PO_4) esterification (acetylation) of salicylic acid with acetic anhydride.* [1,4]



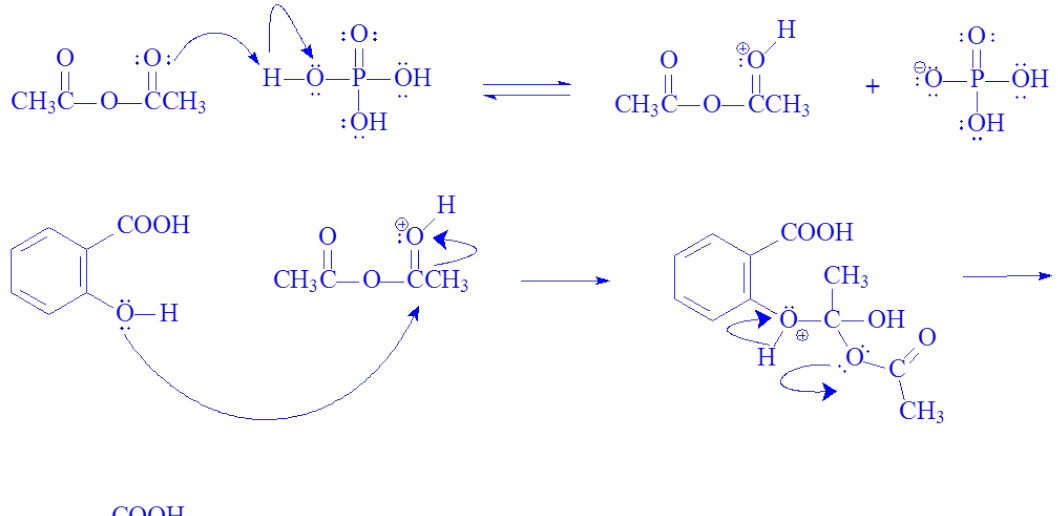
Acide salicylique
Salicylic acid

Anhydride acétique
Acetic anhydride

Aspirine / Aspirin
Acide acétylsalicylique
Acetylsalicylic acid

Acide acétique
Acetic acid

MECHANISM: *Nucleophilic addition-elimination to the carbonyl group of an acyl compound* [5]



Aspirine
Aspirin

Acide acétique
Acetic acid

Acide phosphorique
(catalyseur acide régénéré)
Phosphoric acid
(regenerated acid catalyst)

SAMPLE PRELAB AND REPORT FOR AN EXPERIMENTAL PROJECT

RESULTS

Synthesis and recrystallization of acetylsalicylic acid

Table 1. Quantities (mass, volume, mmol) of reactants used for the synthesis of acetylsalicylic acid. Quantities (masses, theoretical and actual yields, and percentage recovery/yield) of products formed.

	REACTANTS		
Quantities	**Salicylic acid**	**Acetic anhydride**	**85 % Phosphoric acid**
Mass used (g)	1.0545	2.164	0.15 mL (1.685 g/mL) * 0.85 = 0.215
Volume used	---	2.000 mL	3 dps (1 mL/20 dps) = 0.15 mL
Millimoles used (mmol)	7.6346	21.197	0.2148375 g / 97.995 g/mol = 2.19
	limiting reactant	xs reagent	catalyst

	PRODUCT
	Acetylsalicylic acid, ASA, aspirin or 2-(acetyloxy)benzoic acid
Theoretical moles produced	7.6346 mmol
Theoretical mass produced (theoretical yield)	7.6346 mmol * 180.158 g/mol = **1.3754 g**
Mass of Erlenmeyer + label	18.2885 g
Mass of Erlenmeyer + label + crude aspirin	21.3597 g
Actual mass crude aspirin	21.3597 – 18.2885 = **3.0712 g**
Mass of watch glass + filter paper + label	6.6505 g
Mass of watch glass + filter paper + label + recrystallized aspirin	7.2051 g
Actual mass recrystallized aspirin	**0.5546 g**
% Yield crude aspirin	223.30 % = (3.0712 g / 1.3754 g) * 100
% Recovery recrystallization	18.06 % = (0.5546 g / 3.0712 g) * 100
% Yield recrystallized aspirin	40.32 % = (0.5546 g / 1.3754 g) * 100

Calculations

Table 2. Observations for the synthesis and recrystallization of acetylsalicylic acid

Synthesis

- *SA white shiny needles, odour not detected (SA is an ocular and respiratory irritant).*
- *Upon heating all SA dissolved to produce a transparent almost colourless (tinge of yellow) solution.*
- *Upon addition of ice-cold dH_2O, mixture bubbled, and test tube became warmer to the touch – exothermic reaction!!!*
- *Scratching sides of the flask with a glass rod induced crystallization. Crystallization took ~ 30 min.*
- *Crude crystals were brilliant, white, needle-like and wet with dH_2O.*

Recrystallization

- *Hot saturated solution had a light tinge of yellow.*
- *Crystals appeared in the mother liquor, but a 2nd crop of crystals was not collected.*
- *Crystals were dried in air at room temperature.*
- *Recrystallized aspirin crystals were shiny white needle-like crystals. Hint of acetic acid (vinegar) odour. Did not detect an oil of wintergreen scent.*

Setup used for synthesis or extraction

Present a labelled drawing of the setup used to perform the synthesis or extraction.

Analytical methods

Place results for all analysis methods here. Divide into subsections based on the method.

OTHER SECTIONS OF A LAB REPORT

*Depending on the project, the professor may ask you to complete some or all of these sections: **DISCUSSION**, **CONCLUSION** and **EXPERIMENTAL ERRORS**. Please refer to project document for more detail.*

SAMPLE PRELAB AND REPORT FOR AN EXPERIMENTAL PROJECT

REFERENCES *(note that the references are presented in the style given in the Chemistry Guide)*

1. *Williamson KL, Masters KM. Macroscale and Microscale Organic Experiments, 6th edition. Belmont (CA): Brooks/Cole Cengage Learning; 2011. Chapter 41, Acetylsalicylic Acid (Aspirin); p 529.*
2. *Schoffstall AM, Gaddis BA, Druelinger ML. Microscale and Miniscale Organic Chemistry Laboratory Experiments. 2th ed. Montreal: McGraw Hill Higher Education; 2004. Chapter 24, Phenols; p. 480.*
3. *Harwood LM, Moody CJ, Percy JM. Experimental Organic Chemistry. Standard and Microscale. 2nd ed. Malden (MA): Blackwell Science Ltd., a Blackwell Publishing company; 1999. Chapter 4, Qualitative analysis of organic compounds; p. 204*
4. *Lide DR, editor-in-chief, Haynes WM "Mickey", associate editor. CRC Handbook of Chemistry and Physics, 90th ed. Boca Raton: Floride; 2009.*
5. *Graham Solomons TW, Fryhle CB, Snyder SA. Organic Chemistry. 11th ed. Hoboken (NJ): John Wiley & Sons, Inc.; 2014. Chapter 17, Carboxylic Acids and Their Derivatives; p. 771.*

ANSWERS TO CRITICAL THINKING QUESTIONS & ADDITIONAL INFORMATION

Answer any questions included in the protocol or given by the professor. Hand-in one copy per team.
This space is also to be used for additional information, if necessary.

ADDITIONAL DOCUMENTS

Attach supporting documents, such as spectra, chromatograms, etc.