

A. Name:

B. Date:

C. Title: Preparation of Isopentyl Acetate (3-Methylbutyl Ethanoate)

D. Introduction and Background

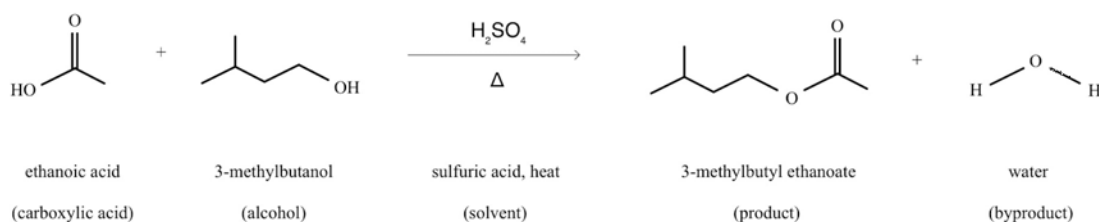
The Fischer esterification reaction is an acid-catalyzed process in which a carboxylic acid and an alcohol react to form an ester and water. This equilibrium-driven reaction proceeds via nucleophilic attack on the protonated carbonyl, forming a tetrahedral intermediate that regenerates the carbonyl upon collapse. To drive ester formation, excess reactants or water removal techniques are employed. Steric hindrance slows the reaction, while electron-withdrawing groups enhance reactivity.

Fischer esterification is essential in organic synthesis, playing a key role in producing pharmaceuticals, fragrances, and biodegradable plastics. In biochemistry, ester bonds are fundamental to lipids, such as triglycerides and phospholipids, which are critical for energy storage and membrane structure. Additionally, ester hydrolysis is central to metabolic processes like lipid digestion, underscoring the biological significance of this reaction.

E. Objective:

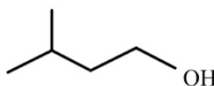
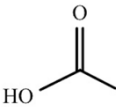
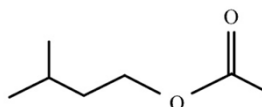
The objective of today's experiment is to synthesize isopentyl acetate (3-methylbutyl ethanoate) through an esterification reaction, by using isopentyl alcohol (3-methylbutanol), acetic acid (ethanoic acid), and sulfuric acid as reagents. This process aims to demonstrate the formation of an ester through the removal of water in the presence of a catalytic amount of sulfuric acid.

F. Graphical Abstract:



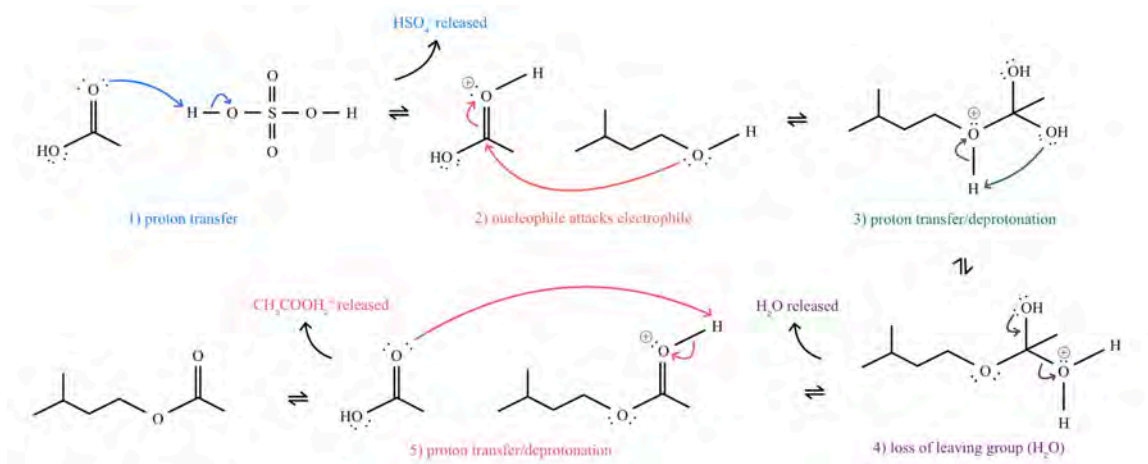
Marmande, M. H., Baxter, B. N., Lawley, H. K., Lippold, C. G., Lopansri, C. N., Mayeaux, K. N., Orr, L. A., Forbes, D. C. (2025). *Preparation of Isopentyl Acetate (3-Methylbutyl Ethanoate)*. Department of Chemistry, University of South Alabama.

G. Table of Physical Properties:

Name	isopentyl alcohol (3-methylbutanol)	acetic acid (ethanoic acid)	sulfuric acid	isopentyl acetate (3-methylbutyl ethanoate)
Structure			H_2SO_4	
ROLE	starting material	solvent	catalyst	desired product (ester)
MF	$C_5H_{12}O$	$C_2H_4O_2$	H_2SO_4	$C_7H_{14}O_2$
MW (g/mol)	88.15	60.1	98.08	130.19
amount	800 μ L (0.8 mL)	1.5 mL	4 drops	
mmol	7.4	26.2		
equiv.	1	3.6	(cat)	
BP ($^{\circ}$ C)	132	116	337	142
Density (g/mL)	0.81	1.05	1.84	0.87
Melting Point ($^{\circ}$ C)	-	16	10	-
Hazard(s)*	Skin, eye, and respiratory irritant. Flammable.	Severe skin burn and eye damage can occur, Respiratory irritant, and corrosive.	Severe skin burns, eye damage, and lung damage if inhaled. Will react violently with water	Flammable, skin and eye irritants, and respiratory irritant.

*Source: ChatGPT using the following prompt: What is listed on the safety data sheets as it relates to the hazards, handling, storage, and emergency response of [insert the name of the chemical HERE].

H. Mechanism:



I. Calculations (Appendix D contains an embedded .xlsx with the key calculations based on the amounts in the procedure, as well as a “what-if” scenario that can be used to revise theoretical yield calculations if needed.)

Limiting Reactant Calculations:

mmol acetic acid (ethanoic acid):

mmol isopentyl alcohol (3-methylbutanol):

mmol sulfuric acid:

Theoretical Yield Calculations:

J. Reaction Procedure

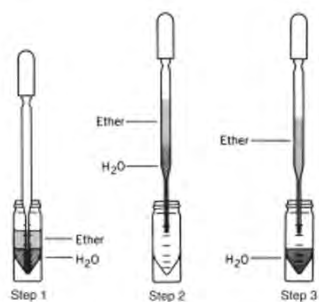
Procedure	Observations/Notes
<p>CAUTION: <u>Cap</u> the vial immediately after addition of each reagent. <u>Dispense</u> the reagents in the hood and <u>note</u>, concentrated acetic and sulfuric acids are <u>corrosive</u>.</p> <ol style="list-style-type: none">1. To a 5.0-mL conical vial placed in a 25-mL beaker (secondary containment), add via syringe 800 μL (7.4 mmol) of isopentyl alcohol (3-methylbutanol).2. Via syringe or a 1-mL plastic transfer pipette, add to the 5.0-mL conical vial 1.5 mL (26.2 mmol) of glacial acetic acid followed by 4 drops using a disposable glass Pasteur pipet of concentrated sulfuric acid.3. Finally, add approximately 100 mg of silica gel beads4. Once all the materials have been introduced to the 5-mL conical vial, equip the vial with the following items as shown in the figure below: magnetic spin vane, condenser, calcium chloride drying tube, and thermometer. With the sand bath, it is important that the height of the sand within the crystallization dish does not exceed 15 mm (between 10 mm and 15 mm should be your target depth). <div data-bbox="272 1234 625 1753"></div> <ol style="list-style-type: none">5. Heat and stir the reaction mixture using a sand bath temperature of 160–180 $^{\circ}\text{C}$ for 1 h.	

6. Cool the resulting mixture to room temperature and remove the spin vane with forceps.

7. Add using a 1-mL plastic transfer pipette around 1.5 mL of *t*-butyl methyl ether to increase the volume of the organic phase.

8. Extract the organic phase with three 2 mL portions of 5% sodium bicarbonate solution, followed by 1 mL of deionized water.

9. During each extraction, cap the vial, shake gently, vent carefully, and then allow the layers to separate.



10. Remove and place the aqueous layer in a small beaker. Be sure, not to discard anything until the final product is confirmed.

11. Dry the organic phase with anhydrous sodium sulfate. (See **Appendix C** for alternative drying technique)

12. Record the weight of a dry/clean 10-mL beaker with boiling stone.

13. Transfer via a 1-mL plastic transfer pipette the ether solution to the 10-mL beaker with boiling stone.

13. Evaporate the ether in the hood using a hot plate. Stop the evaporation process when observing a constant weight of the beaker with boiling stone.

14. Record the final mass of the crude product and obtain the IR of the starting material and the final product (See **Appendix A & B** for representative IR spectra).

Optional:

15. Perform a distillation of the crude product and record boiling point (See **Appendix C** for setup).

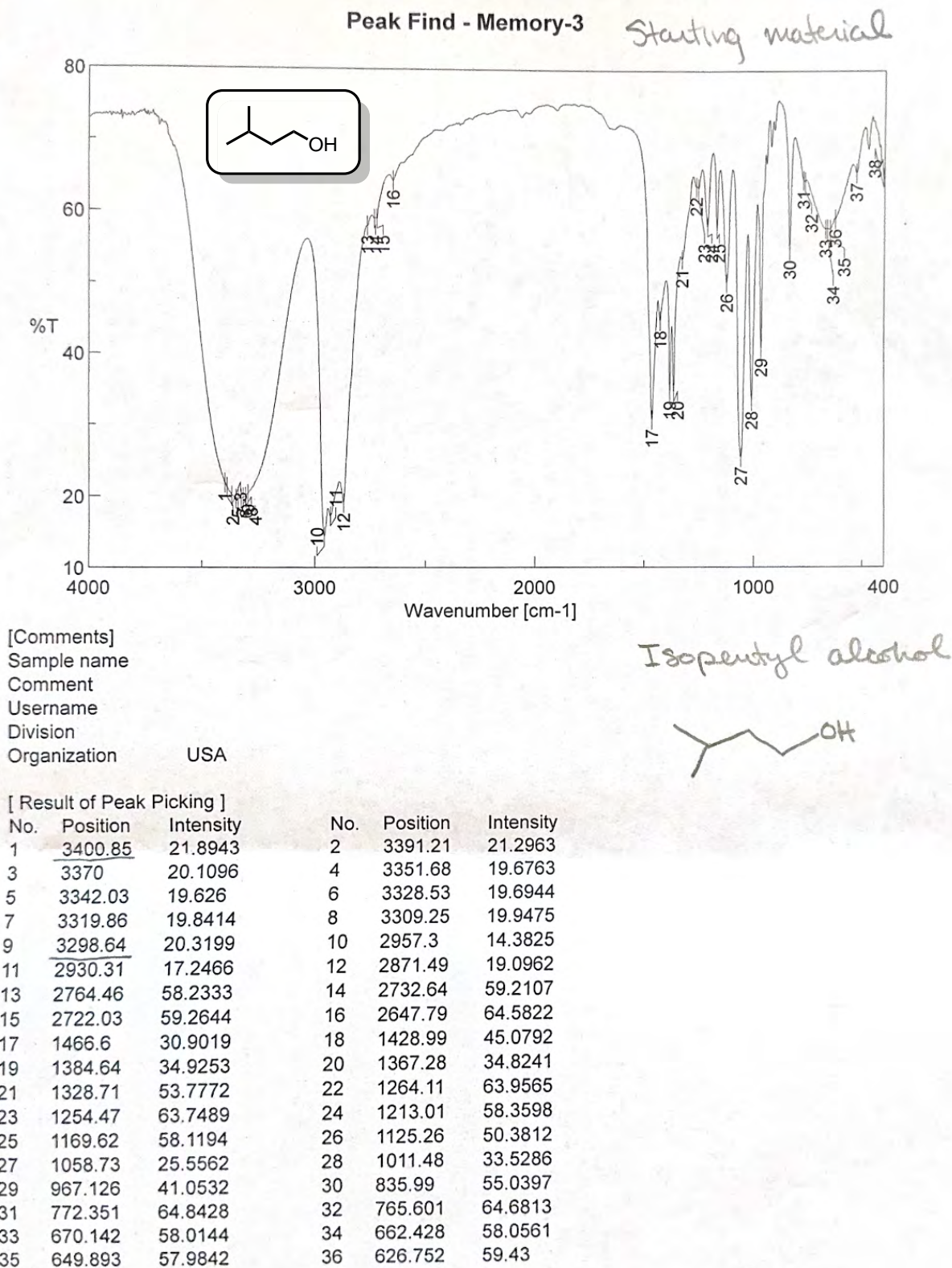
K. Safety

Ensuring safety for all experiments, including the esterification reaction of isopentyl alcohol, is priority #1. Before handling any chemicals, it is essential to review the **Safety Data Sheets (SDS)** for proper handling and disposal of the materials used.

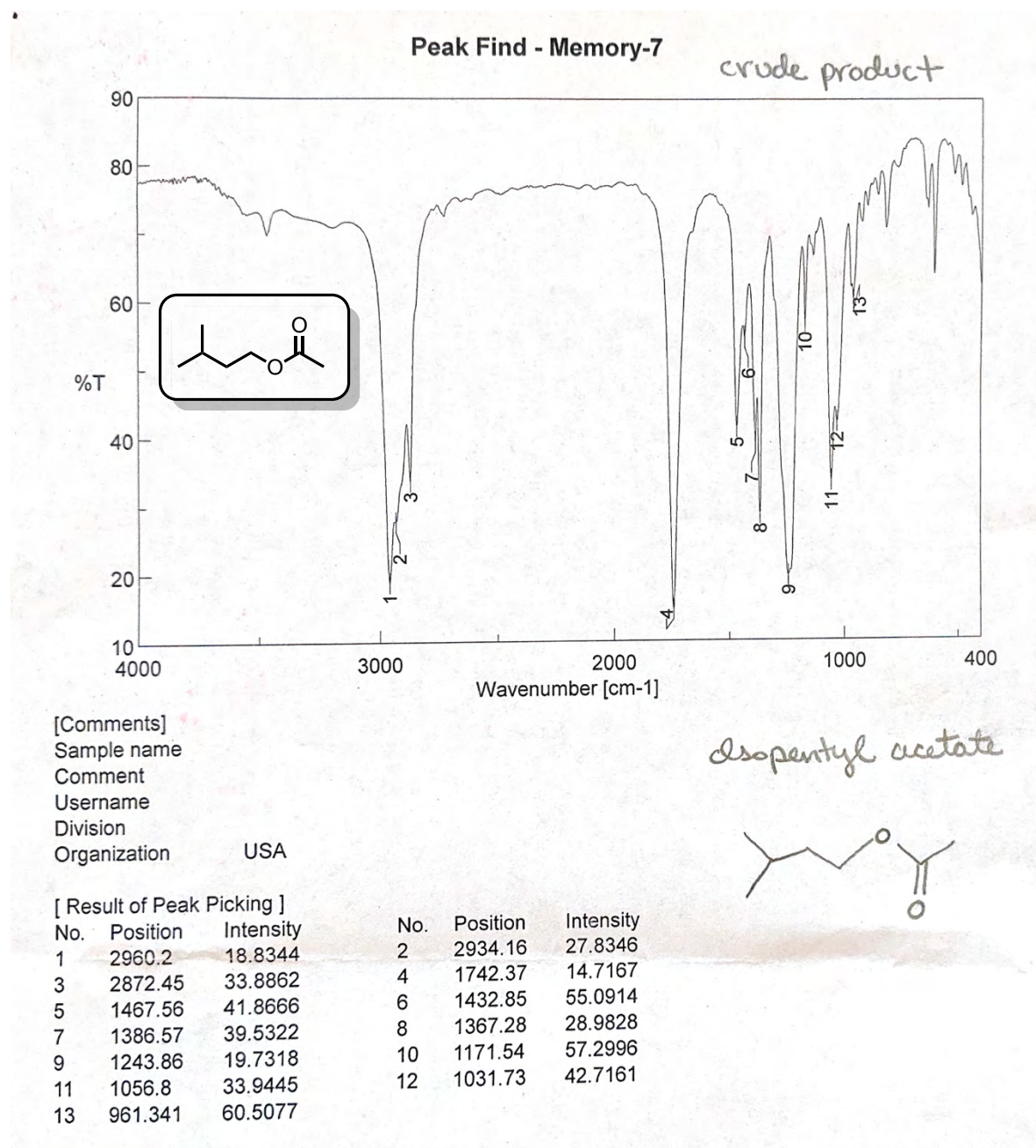
Appropriate **personal protective equipment (PPE)**, including safety goggles and gloves must be worn at all times to minimize risk of exposure to both yourself and your colleagues. **Sulfuric acid**, a highly corrosive substance, should be handled with extreme caution and added carefully. Both **acetic acid (ethanoic acid)** and **isopentyl alcohol (3-methylbutanol)** are irritants and must be dispensed in a properly vented area such as a hood. All **glassware** should be carefully inspected for cracks before use to prevent breakage during the experiment. The **heated stir plate** presents a burn hazard and should not be touched directly when the heating element is engaged. While the **spin vane** will be introduced into the reaction mixture, do proceed with caution to avoid splashing or equipment damage. Emergency measures, including readily available **water and neutralizing agents**, are located in the hoods in the event of a chemical spill. Adhering to these precautions as well as those detailed on the Chemistry's Instructional Laboratories URL ensures a safe and controlled laboratory environment.

L. Appendices

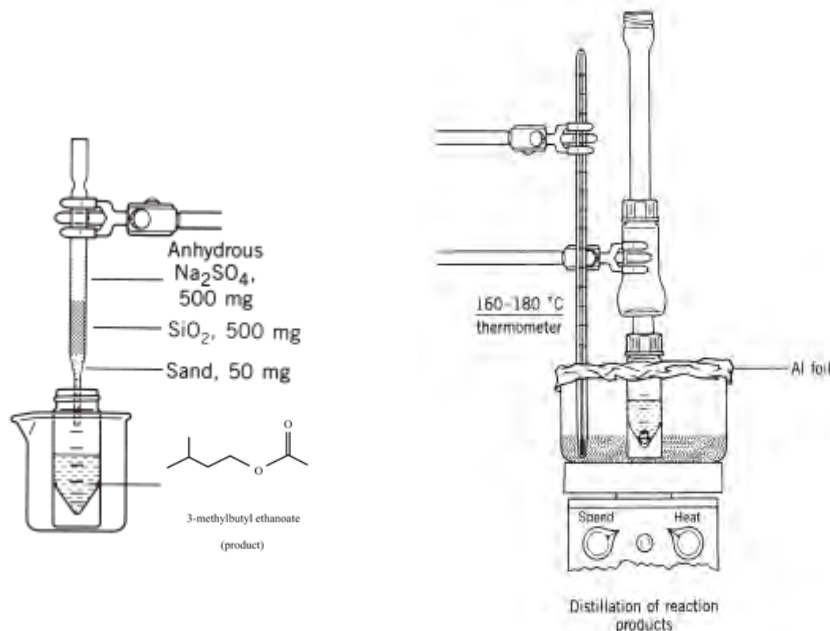
Appendix A: IR spectrum of isopentyl alcohol (3-methylbutanol)



Appendix B: IR spectrum of isopentyl acetate (3-methylbutyl ethanoate)



Appendix C: Alternative drying procedure (left) and distillation setup (right)



Appendix D: Below are two screenshots and a hyperlink. The top screenshot details the calculations based on the procedure whereas the second represents what you will see when acting on the hyperlink: The embedded .xlsx file will recalculate and generate a revised theoretical yield as you may find that the recommended amounts may vary when dispensing the starting material and recording the actual amount of isopentyl alcohol used.

	MW (g/mol)	Units	amount (mL)	density (g/mL)	mol	mmol	equiv.
isopentyl alcohol	88.15		0.80	0.81	0.01	7.35	1.00
acetic acid	60.10		1.50	1.05	0.03	26.21	3.56
isopentyl acetate	130.19						
theoretical yield	957.04	mg				LR	
	0.96	g					

	amount used	Units	mol	mmol
isopentyl alcohol	2.00	mL	0.02	18.38
acetic acid	3.74	mL	0.07	65.42
revised theoretical yield	2392.60	mg		
	2.39	g		Cell unlocked

[Edit excel sheet HERE](#)