A. Name:

B. Date:

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

D. Introduction and Background

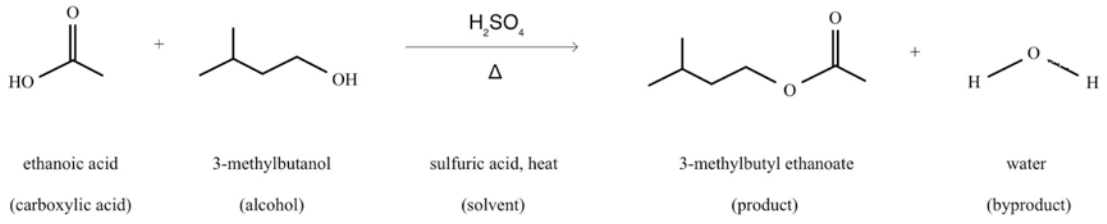
The Fischer esterification reaction is an acid-catalyzed process in which a carboxylic acid and an alcohol react to form an ester and water. This equilibrium-driven reaction proceeds via nucleophilic attack on the protonated carbonyl, forming a tetrahedral intermediate that regenerates the carbonyl upon 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:

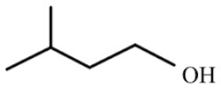
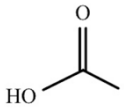
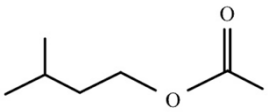
The objective of today's experiment is to synthesize isopentyl acetate (3-methylbutyl ethanoate) through an esterification reaction, by using isopentyl alcohol (3-methylbutanol), acetic acid (ethanoic acid), and sulfuric acid as reagents. This process aims to demonstrate the formation of an ester through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

F. Graphical Abstract:



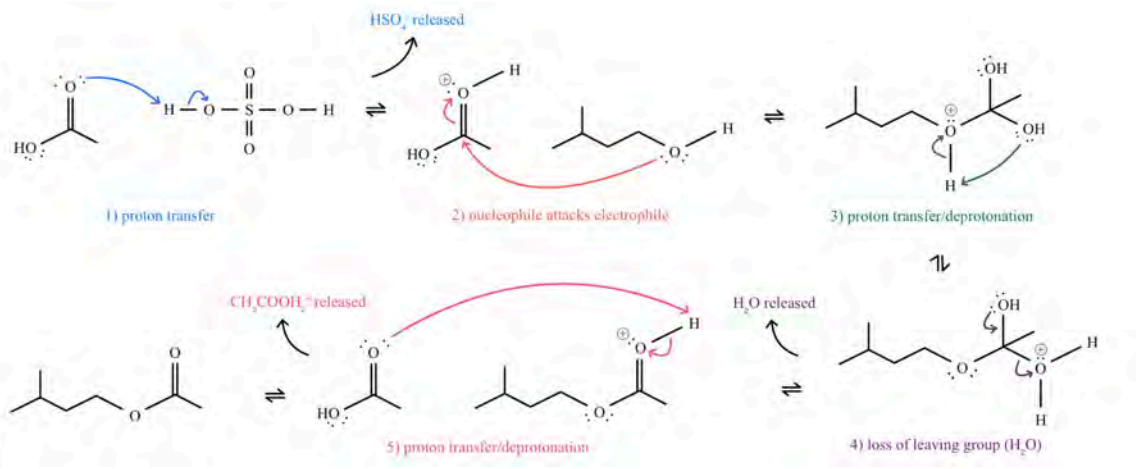
Marmande, M. H., Baxter, B. N., Lawley, H. K., Lippold, C. G., Lopansri, C. N., Mayeaux, K. N., Orr, L. A., Forbes, D. C. (2025). *Preparation of Isopentyl Acetate (3-Methylbutyl Ethanoate)*. Department of Chemistry, University of South Alabama.

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 µL (0.8 mL)	1.5 mL	4 drops	
mmol	7.4	26.2		
equiv.	1	3.6	(cat)	
BP (℃)	132	116	337	142
Density (g/mL)	0.81	1.05	1.84	0.87
Melting Point (℃)	-	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:

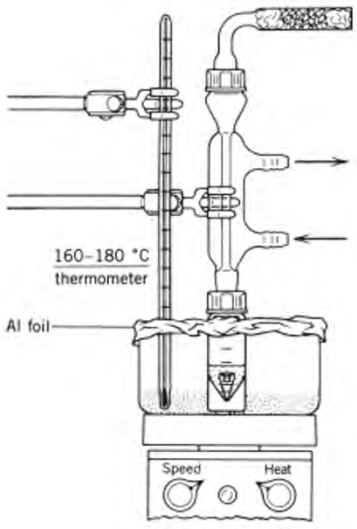
mmol acetic acid (ethanoic acid):

mmol isopentyl alcohol (3-methylbutanol):

mmol sulfuric acid:

Theoretical Yield Calculations:

J. Reaction Procedure

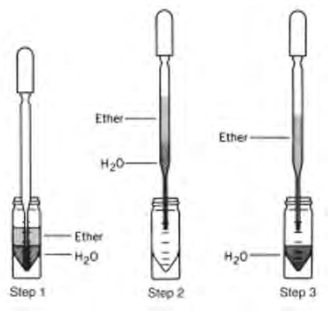
Procedure	Observations/Notes
CAUTION: Cap the vial immediately after addition of each reagent. Dispense the reagents in the **hood** and note, concentrated acetic and sulfuric acids are *corrosive*. 1. To a 5.0-mL conical vial placed in a 25-mL beaker (secondary containment), add via syringe 800 µL (7.4 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 beads 4. 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).  5. Heat and stir the reaction mixture using a sand bath temperature of 160–180 °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).

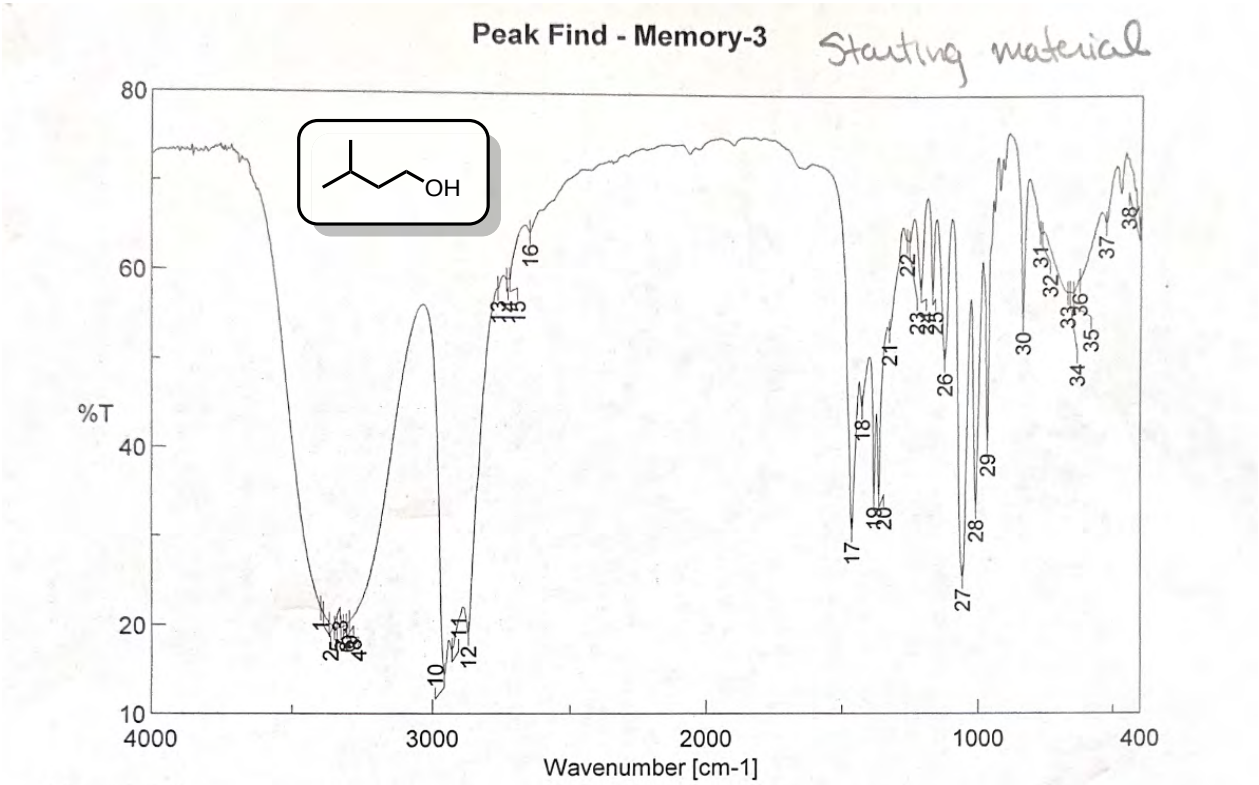
Optional:
15. Perform a distillation of the crude product and record boiling point (See **Appendix C** for setup).

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)



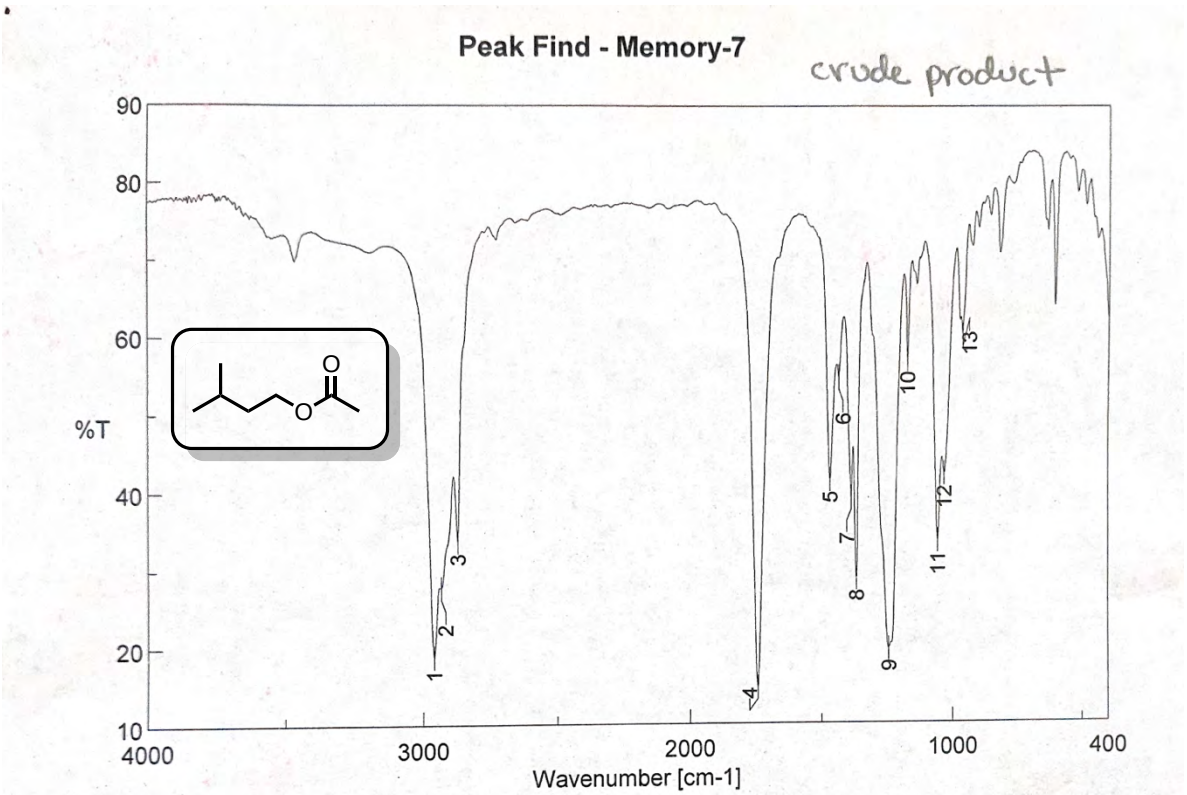
[Comments]
Sample name
Comment
Username
Division
Organization USA

Isopentyl alcohol

[Result of Peak Picking]

No.	Position	Intensity	No.	Position	Intensity
1	3400.85	21.8943	2	3391.21	21.2963
3	3370	20.1096	4	3351.68	19.6763
5	3342.03	19.626	6	3328.53	19.6944
7	3319.86	19.8414	8	3309.25	19.9475
9	3298.64	20.3199	10	2957.3	14.3825
11	2930.31	17.2466	12	2871.49	19.0962
13	2764.46	58.2333	14	2732.64	59.2107
15	2722.03	59.2644	16	2647.79	64.5822
17	1466.6	30.9019	18	1428.99	45.0792
19	1384.64	34.9253	20	1367.28	34.8241
21	1328.71	53.7772	22	1264.11	63.9565
23	1254.47	63.7489	24	1213.01	58.3598
25	1169.62	58.1194	26	1125.26	50.3812
27	1058.73	25.5562	28	1011.48	33.5286
29	967.126	41.0532	30	835.99	55.0397
31	772.351	64.8428	32	765.601	64.6813
33	670.142	58.0144	34	662.428	58.0561
35	649.893	57.9842	36	626.752	59.43

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



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Sample name
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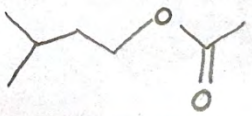
Division
Organization USA

isopentyl acetate

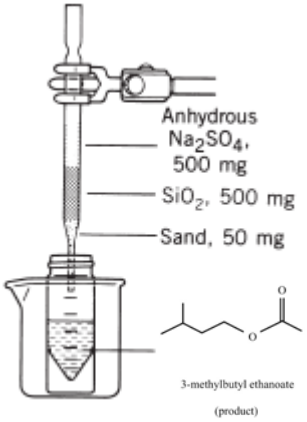


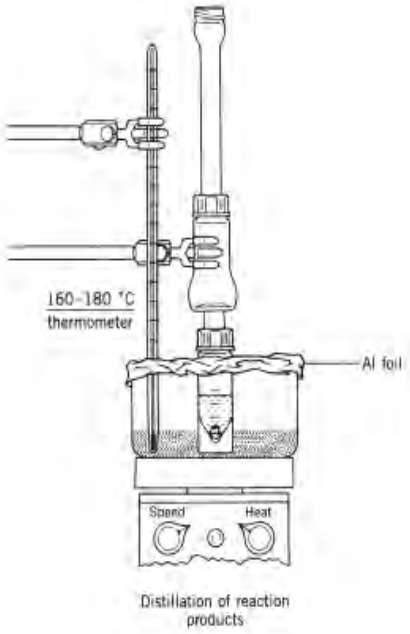
[Result of Peak Picking]

No.	Position	Intensity
1	2960.2	18.8344
3	2872.45	33.8862
5	1467.56	41.8666
7	1386.57	39.5322
9	1243.86	19.7318
11	1056.8	33.9445
13	961.341	60.5077

No.	Position	Intensity
2	2934.16	27.8346
4	1742.37	14.7167
6	1432.85	55.0914
8	1367.28	28.9828
10	1171.54	57.2996
12	1031.73	42.7161

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

