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Preparing Isopentyl Acetate by the Fischer Esterification

prepared by **Ronald J. Wikholm**, University of Connecticut

PURPOSE OF THE EXPERIMENT

Prepare isopentyl acetate from isopentyl alcohol and acetic acid by the Fischer esterification.

EXPERIMENTAL OPTIONS

BACKGROUND REQUIRED

You should be familiar with the techniques for extraction and distillation. You should be familiar with gas chromatography, infrared spectroscopy, and/or nuclear magnetic resonance spectroscopy for characterizing the product and assessing its purity.

BACKGROUND INFORMATION

Esters are derivatives of carboxylic acids in which the acyl carbon bears an alkoxy substituent (–OR) rather than the hydroxyl substituent (–OH) of the acid. Simple esters tend to have pleasant, fruity odors and are widely used as flavors and fragrances. Table 1 on page 2 shows the flavors or fragrances associated with some esters.

The volatile compounds in natural fruits and flowers are usually complex mixtures of compounds, where esters frequently predominate. Many artificial flavorings contain esters or mixtures of esters. For example, the volatile oil of ripe pineapple contains several esters, as shown in Table 2 on page 3.

Isopentyl acetate is known as banana oil because of its characteristic odor. This ester has also been shown to be one of the active substances in the alarm pheromone of the honeybee. When a honeybee worker stings an intruder, an alarm pheromone is secreted along with the venom. The pheromone causes other bees to become aggressive and attack the intruder.

Esterification generally refers to the formation of esters from alcohols and carboxylic acids, as shown in Equation 1.

$$\underset{\substack{\text{carboxylic}\\ \text{acid}}}{\mathrm{RCOOH}} + \underset{\text{alcohol}}{\mathrm{R'OH}} \rightleftharpoons \underset{\text{ester}}{\mathrm{RCOOR'}} + \mathrm{H_2O} \qquad \text{(Eq. 1)}$$

02 01 00 99 98 — 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1

Table 1 Fragrances of esters

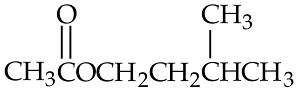
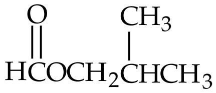
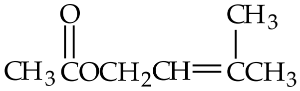
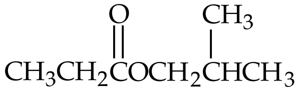
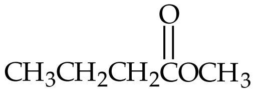
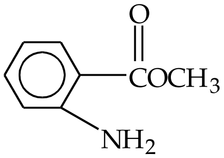
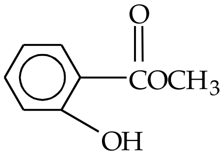
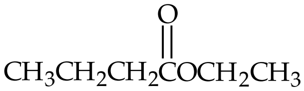
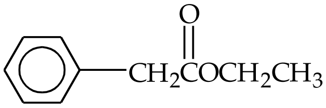
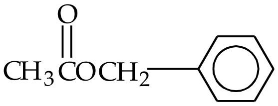
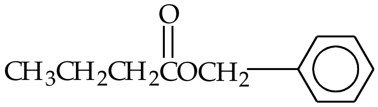
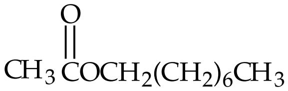
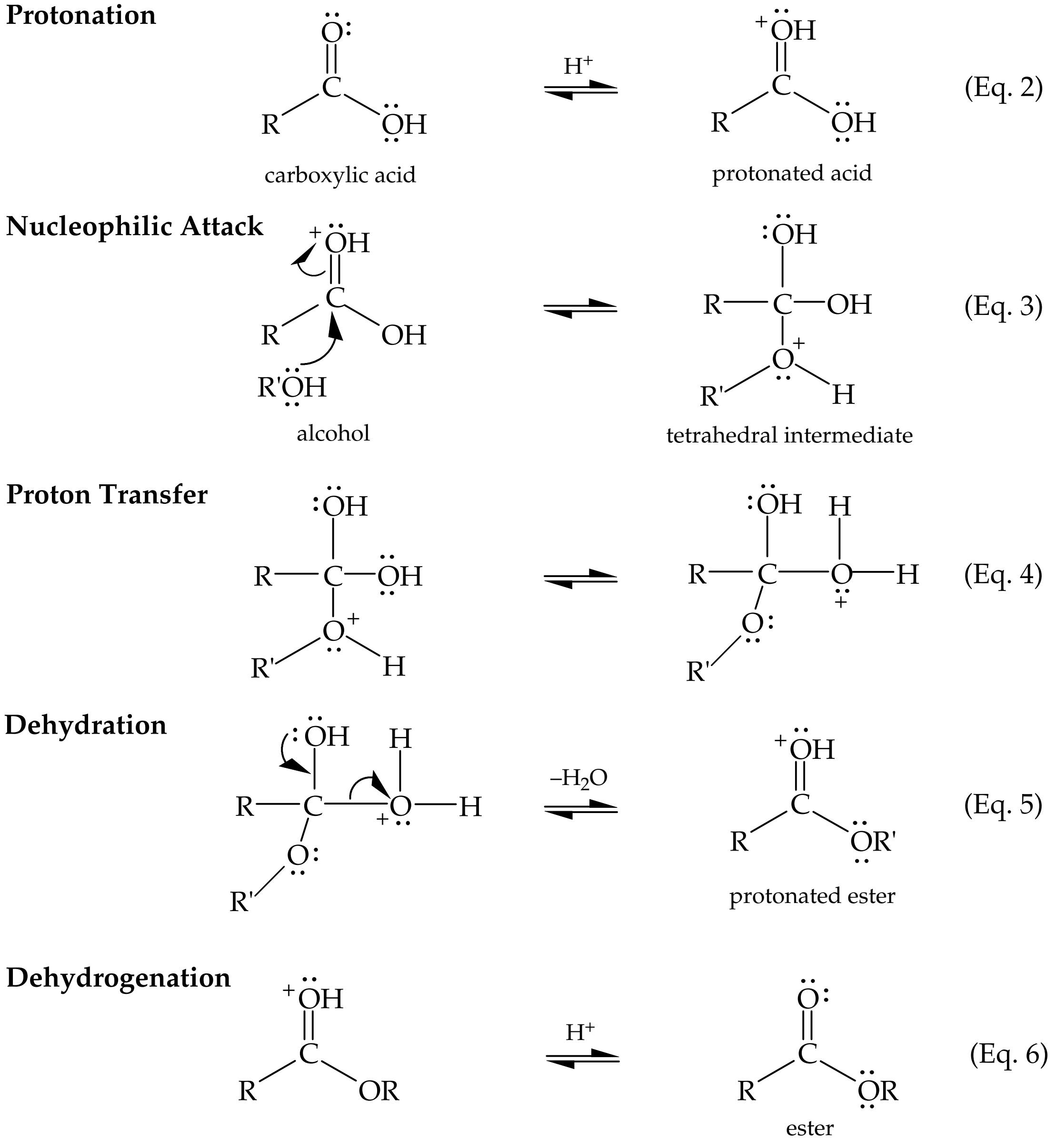
ester	*formula*	*fragrance*
isopentyl acetate	 $CH_3\overset{O}{\overset{\Vert}{C}}OCH_2CH_2\overset{CH_3}{\overset{\vert}{C}}HCH_3$	banana
isobutyl formate	 $H\overset{O}{\overset{\Vert}{C}}OCH_2\overset{CH_3}{\overset{\vert}{C}}HCH_3$	raspberry
isopentenyl acetate	 $CH_3\overset{O}{\overset{\Vert}{C}}OCH_2CH{=}\overset{CH_3}{\overset{\vert}{C}}CH_3$	juicy fruit
isobutyl propionate	 $CH_3CH_2\overset{O}{\overset{\Vert}{C}}OCH_2\overset{CH_3}{\overset{\vert}{C}}HCH_3$	rum
n -propyl acetate	 $CH_3\overset{O}{\overset{\Vert}{C}}OCH_2CH_2CH_3$	pear
methyl butyrate	 $CH_3CH_2CH_2\overset{O}{\overset{\Vert}{C}}OCH_3$	apple
methyl anthranilate		grape
methyl salicylate		wintergreen
ethyl butyrate	 $CH_3CH_2CH_2\overset{O}{\overset{\Vert}{C}}OCH_2CH_3$	pineapple
ethyl phenyl acetate		honey
benzyl acetate		peach
benzyl butyrate		cherry
octyl acetate	 $CH_3\overset{O}{\overset{\Vert}{C}}OCH_2(CH_2)_6CH_3$	orange

Table 2 Composition of the volatile oil of ripe pineapple

constituent	*percent, %*
total volatile oil	0.1900
ethyl acetate	0.1196
ethyl alcohol	0.0605
acetaldehyde	0.0014
ethyl acrylate	0.0008
ethyl *i*-valerate	0.0004
ethyl *n*-caproate	0.0008

The reaction proceeds by way of a nucleophilic substitution at the acyl carbon of the carboxylic acid. When catalyzed by a strong acid, usually sulfuric acid or *p*-toluenesulfonic acid, the reaction is called the **Fischer esterification**.

The reaction mechanism is shown in Equations 2–6. Equation 2 shows the protonation of the acyl oxygen of the carboxylic acid. The protonation activates the acyl carbon toward nucleophilic attack. Equation 3 shows the nucleophilic attack at the acyl carbon by the alcohol oxygen atom to form a tetrahedral intermediate. Equation 4 shows a proton transfer to the hydroxyl oxygen of the carboxyl group. This protonation converts the hydroxyl group into the good leaving group, water. Equation 5 shows the loss of water forming the protonated ester. Finally, Equation 6 shows the proton transfer to a base, resulting in the formation of the ester.



Each step of the reaction mechanism is reversible and, therefore, the reaction reaches an equilibrium. **Le Chatelier's principle** suggests that the amount of ester produced in an equilibrium reaction might be increased either by using an excess of one of the reactants or by removing one of the products. In practice, an excess of carboxylic acid or alcohol, whichever is more readily available, is added, and/or water is removed as the reaction proceeds. A water-absorbing substance such as molecular sieves might be included in the reaction mixture, or the water might be removed as part of an azeotrope with benzene or toluene.

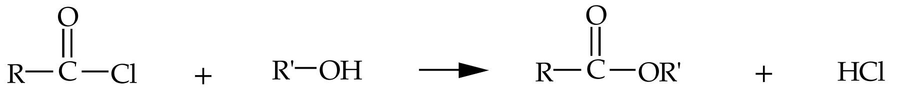
The mechanism suggests that steric effects might be important in Equation 3, the step involving the attack by the alcohol at the acyl carbon of the carboxylic acid. Indeed, alkyl-group branching at the α or β carbon of the acid slows the rate of esterification. For example, the relative rates of esterification with methanol, CH_3OH, follow the order

$$CH_3CO_2H > CH_3CH_2CO_2H > (CH_3)_2CHCO_2H > (CH_3)_3CCO_2H$$

Sterically hindered alcohols also react more slowly in the esterification reaction. The relative rates for esterification of alcohols with acetic acid, CH_3CO_2H, follow the order

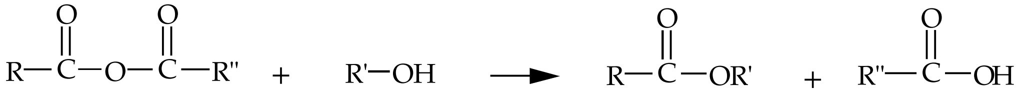
$$CH_3OH > CH_3CH_2OH > (CH_3)_3CCH_2OH$$

Fischer esterification is an example of an acyl transfer reaction. The acyl group from the acid is transferred to the alcohol. Acid chlorides and anhydrides also serve as acylating agents. Because acid chlorides and anhydrides contain good leaving groups, these compounds are very reactive toward nucleophilic substitution by an alcohol, as shown in Equations 7 and 8.



$$\mathrm{R{-}\overset{O}{\overset{\|}{C}}{-}Cl \;+\; R'{-}OH \longrightarrow R{-}\overset{O}{\overset{\|}{C}}{-}OR' \;+\; HCl}$$

(Eq. 7)



$$\mathrm{R{-}\overset{O}{\overset{\|}{C}}{-}O{-}\overset{O}{\overset{\|}{C}}{-}R'' \;+\; R'{-}OH \longrightarrow R{-}\overset{O}{\overset{\|}{C}}{-}OR' \;+\; R''{-}\overset{O}{\overset{\|}{C}}{-}OH}$$

(Eq. 8)

The Fischer esterification reaction is conducted at reflux. The purpose of **reflux** is to heat a reaction mixture at its boiling temperature to form products, without losing any of the compounds in the reaction flask.

In practice, a condenser is set vertically into the top of the reaction flask. Any compound that vaporizes will condense when it enters the cool environment of the reflux condenser and will then drain back into the reaction flask. Reflux apparatus using glassware for semi-microscale and microscale techniques are shown in their appropriate sections of the Procedure.

In this experiment, you will prepare isopentyl acetate by reacting an excess of acetic acid with isopentyl alcohol. After the reaction is complete, you will remove the excess acetic acid from the reaction mixture by extraction with sodium hydrogen carbonate ($NaHCO_3$).

You will use either sulfuric acid or Dowex® 50 ion exchange resin to catalyze the reaction. **Dowex® 50** is a polystyrene resin containing sulfonic acid groups on some of the styrene units that compose the polymer. A representative section of Dowex® 50 is shown in Figure 1.

SEMI-MICROSCALE FISCHER ESTERIFICATION

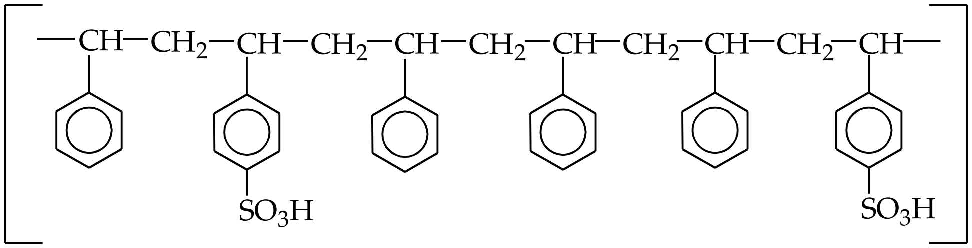


Figure 1 A representative section of Dowex® 50

Equipment

aluminum foil
150-mL beaker
400-mL beaker*
boiling chips, acid resistant
electric flask heater,
  with heat controller
50-mL Erlenmeyer flask
50-mL graduated cylinder
microspatula
standard taper glassware:
  condenser, with adapter and tubing
  distilling head
  2 round-bottom flasks, 25-mL
  thermometer, –10 to 260 °C, with adapter
125-mL separatory funnel
Pasteur pipet, with latex bulb
10-mL graduated pipet, with bulb
10-mL product vial
support ring
2 support stands
25 × 150-mm test tube
2 utility clamps

*for ice-water bath

Reagents and Properties

substance	*quantity*	*molar mass (g/mol)*	*bp (°C)*	*d (g/mL)*
glacial acetic acid	8.5 mL	60.05		1.049
isopentyl acetate*		130.19	142	0.876
isopentyl alcohol	4.37 g	88.15	130	0.809
sodium chloride, saturated solution	25 mL			
sodium hydrogen carbonate, 5%	50 mL			
sodium sulfate, anhydrous	1.5 g	142.04		
sulfuric acid, concentrated	1.0 mL	98.08		

*product

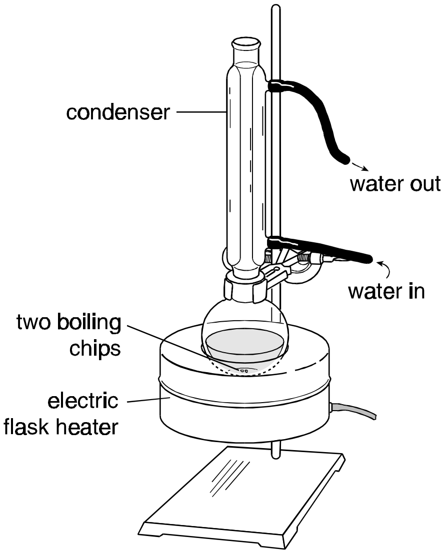


Figure 2 Reflux apparatus for semi-microscale glassware

Preview

- Assemble the reflux apparatus for semi-microscale glassware
- Add the isopentyl alcohol, acetic acid, and H_2SO_4 to the flask and reflux the mixture for 1–1.5 hr
- Cool the reaction mixture
- Place cold water into the separatory funnel and add the reaction mixture
- Separate the aqueous layer
- Wash the organic layer with two 25-mL portions of 5% $NaHCO_3$
- Wash the organic layer with 25 mL saturated NaCl solution
- Dry the organic layer over anhydrous Na_2SO_4
- Assemble a simple distillation apparatus
- Distill the product, collecting the 134–143 °C fraction
- Weigh the product
- Characterize the product for purity using GC, IR, and/or NMR

PROCEDURE

Chemical Alert

glacial acetic acid—*corrosive*
isopentyl acetate—*flammable and irritant*
isopentyl alcohol—*irritant*
sodium sulfate—*irritant and hygroscopic*
sulfuric acid—*corrosive and oxidant*

Caution: Wear departmentally approved safety goggles at all times while in the chemistry laboratory.

1. Refluxing the Reaction Mixture

Caution: Isopentyl alcohol (3-methyl-1-butanol) is irritating. Glacial acetic acid is corrosive. Concentrated sulfuric acid (H_2SO_4) is corrosive and oxidizing. Prevent eye, skin, clothing, and combustible material contact. Avoid inhaling vapors and ingesting these compounds. Use a ***fume hood.***

Assemble the reflux apparatus shown in Figure 2.

Place 4.37 g (5.4 mL) of isopentyl alcohol into the 25-mL round-bottom flask. Add 8.5 mL of glacial acetic acid and 1 mL of concentrated H_2SO_4. Add two *acid-resistant* boiling chips. Attach the round-bottom flask to the reflux apparatus.

Start the flow of water through the reflux condenser. Heat the reaction mixture to boiling and reflux the mixture gently for 1–1.5 hr. [NOTE 1]

NOTE 1: A slightly higher yield is obtained with the longer reaction time.

Turn off the flask heater and lower the heater away from the flask. Allow the flask to cool to room temperature. Turn off the water to the condenser.

Prepare an ice-water bath in a 400-mL beaker. Remove the flask from the reflux apparatus.

Cool the flask in the ice-water bath. Place 30 mL of distilled or deionized water in a 25 × 150-mm test tube. Cool the test tube in the ice-water bath.

2. Separating and Washing the Product Layer

Pour 20 mL of the cold water into a 125-mL separatory funnel. Carefully pour the reaction mixture from the round-bottom flask into the funnel containing the water. Rinse the reaction flask with the remaining 10 mL of cold water and pour the rinse water into the separatory funnel. Swirl the mixture gently, stopper the separatory funnel, and shake once. [NOTE 2] Vent the funnel carefully.

NOTE 2: Vigorous shaking may result in an emulsion.

Label a 150-mL beaker "Aqueous Layers". Drain the lower aqueous layer from the funnel into the beaker. [NOTE 3]

NOTE 3: It is a good practice to keep all extraction layers in a labeled container until the conclusion of the experiment.

Caution: Neutralizing acids with sodium hydrogen carbonate ($NaHCO_3$) generates CO_2 gas. Vent the funnel frequently so that pressure does not build up in the stoppered separatory funnel.

Carefully add 25 mL of 5% $NaHCO_3$ to the separatory funnel. Swirl the funnel gently until bubbles no longer appear. Stopper the funnel and shake it cautiously. Vent the funnel. Shake the funnel vigorously and vent it immediately.

Allow the layers to separate. Remove the stopper and drain the lower aqueous layer into the same labeled beaker.

Wash the organic layer in the funnel with a second 25-mL portion of 5% $NaHCO_3$. Again, drain the lower aqueous layer into the labeled beaker.

Finally, wash the organic layer with 25 mL of saturated NaCl solution. Drain the lower aqueous layer into the labeled beaker.

Weigh 1.5 g of anhydrous sodium sulfate (Na_2SO_4) and place it into a 50-mL Erlenmeyer flask [NOTE 4]. Pour the organic layer into the Erlenmeyer flask. Dry the organic layer over anhydrous Na_2SO_4 for 15 min.

NOTE 4: Your laboratory instructor may instruct you to add the anhydrous Na_2SO_4 gradually until the solution is no longer cloudy or until the Na_2SO_4 no longer clumps.

3. Distilling the Product

Caution: Isopentyl acetate is flammable and irritating. Keep away from flames or other heat sources. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting this compound.

Assemble a simple distillation apparatus. Tare a clean 10-mL product vial for a receiver.

Decant the crude ester from the flask containing the Na_2SO_4 into a clean 25-mL round-bottom flask. Add a boiling chip to the flask. Attach the round-bottom flask to the distillation apparatus. Wrap the distilling head with aluminum foil to minimize temperature fluctuations during distillation. Start the flow of water through the condenser. Heat the flask.

Collect the compound that distills from 134–143 °C in the tared receiver. Measure and record the mass of the product.

4. Characterizing the Product [NOTE 5]

NOTE 5: Use the product characterization techniques designated by your laboratory instructor.

Analyze the purity of your product by gas chromatography. Compare the retention time of your product with that of pure isopentyl acetate under identical conditions.

Obtain an infrared spectrum of your product. Compare your spectrum to that of pure isopentyl acetate, provided by your laboratory instructor. Analyze your spectrum for absorption bands consistent with the structure of isopentyl acetate.

Obtain a nuclear magnetic resonance spectrum of your product. Compare your spectrum to that of pure isopentyl acetate, provided by your laboratory instructor.

5. **Cleaning Up** Place your recovered materials in the appropriate labeled collection containers as directed by your laboratory instructor. Clean your glassware with soap or detergent.

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.

MICROSCALE FISCHER ESTERIFICATION

Using Glassware with Elastomeric Connectors

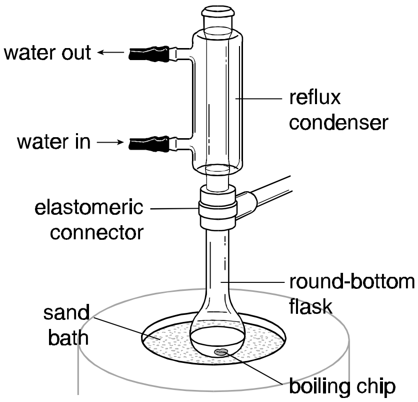


Figure 3 Reflux apparatus for glassware using elastomeric connectors

Equipment

- 100-mL beaker
- boiling chip, acid-resistant
- condenser, with tubing
- distilling head–air condenser
- elastomeric connectors
- microspatula
- 3 Pasteur pipets, with latex bulb
- 2-mL pipet
- 5-mL product vial
- 2 reaction tubes, with stoppers
- 5-mL round-bottom flask
- sand bath*
- stirring rod
- support ring
- support stand
- 13 × 100-mm test tube
- thermometer, –10 to 260 °C, or equivalent, with adapter
- utility clamp

*sand in crystallizing dish on electric hot plate or sand in electric heating well with heat controller

Reagents and Properties

substance	*quantity*	*molar mass (g/mol)*	*bp (°C)*	*d (g/mL)*
Dowex® 50W 2–100*	50 mg			
glacial acetic acid	2 mL	60.05		1.049
isopentyl acetate†		130.19	142	0.876
isopentyl alcohol	0.809 g	88.15	130	0.809
sodium hydrogen carbonate, 5%	3 mL			
sodium sulfate, anhydrous	0.3 g	142.04		

*or 3 drops (0.15 mL) concentrated sulfuric acid

†product

Preview

- Assemble the reflux apparatus using glassware with elastomeric connectors
- Add the reagents to the flask and reflux the mixture for 1 hr
- Cool the reaction mixture
- Separate the mixture from the ion-exchange resin
- Add 5% $NaHCO_3$
- Separate the aqueous layer

- Wash the organic layer with two additional portions of 5% $NaHCO_3$
- Wash the organic layer with water
- Dry the organic layer over anhydrous Na_2SO_4
- Transfer the product to a 5-mL round-bottom flask
- Assemble a simple distillation apparatus
- Distill the product, collecting the 134-143 °C fraction
- Weigh the product
- Characterize the product for purity using GC, IR, and/or NMR

PROCEDURE

Chemical Alert

glacial acetic acid—*corrosive*
isopentyl acetate—*flammable and irritant*
isopentyl alcohol—*irritant*
sodium sulfate—*irritant and hygroscopic*

Caution: Wear departmentally approved safety goggles at all times while in the chemistry laboratory.

1. Refluxing the Reaction Mixture

Caution: Isopentyl alcohol (3-methyl-1-butanol) is irritating. Glacial acetic acid is corrosive. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting these compounds. Use a ***fume hood.***

Weigh a 5-mL round-bottom flask supported in a small beaker and record the tare mass. Place 0.809 g (1.0 mL) of isopentyl alcohol in the flask. Reweigh the container to obtain the precise mass of the alcohol.

Add 2 mL of glacial acetic acid. Add 50 mg of Dowex® 50W 2–100 ion-exchange resin. Finally, add an *acid-resistant* boiling chip.

Use the round-bottom flask containing the alcohol to assemble the reflux apparatus shown in Figure 3.

Use a sand bath to heat the mixture to boiling. Reflux the mixture for 1 hr. Turn off the heat and lower the sand bath away from the flask. Allow the flask to cool to room temperature. Turn off the water to the condenser.

Use a Pasteur pipet to separate the product mixture from the ion-exchange resin. Place the mixture into a clean reaction tube.

2. Separating and Washing the Product Layer

Slowly add 1 mL of 5% sodium hydrogen carbonate ($NaHCO_3$) to the reaction tube. Carefully stir the mixture until bubble evolution is no longer vigorous. Stopper the reaction tube and shake it gently. Vent the reaction tube repeatedly until no more bubbles appear.

Label a test tube "Aqueous Layers". Use a Pasteur pipet to remove the lower aqueous layer. Place the layer into the labeled test tube. [NOTE 1]

Repeat the extraction of the upper organic layer two more times with new 1-mL portions of 5% $NaHCO_3$. Each time place the lower aqueous layer into the labeled test tube.

Finally, wash the organic layer with 1 mL of distilled or deionized water. Again, remove the lower aqueous layer.

NOTE 1: It is a good practice to keep all extraction layers in labeled containers until the conclusion of the experiment.

Add 0.3 g of anhydrous sodium sulfate (Na_2SO_4) to the organic layer to dry the product [NOTE 2]. Stopper the reaction tube and allow it to stand for 15 min.

3. Distilling the Product

Caution: Isopentyl acetate is flammable and irritating. Keep away from flames or other heat sources. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting this compound.

NOTE 2: Your laboratory instructor may direct you to add the anhydrous Na_2SO_4 gradually until the solution is no longer cloudy or until the Na_2SO_4 no longer clumps.

Use a Pasteur pipet to transfer the dried ester into a 5-mL round-bottom flask. Add a boiling chip. Assemble a simple distillation apparatus. Tare a product vial for a receiver.

Heat the flask. Collect the compound that distills from 134–143 °C in the tared receiver. Measure and record the mass of the product.

4. Characterizing the Product [NOTE 3]

Analyze the purity of your product by gas chromatography. Compare the retention time of your product with that of pure isopentyl acetate under identical conditions.

Obtain an infrared spectrum of your product. Compare your spectrum to that of pure isopentyl acetate, provided by your laboratory instructor. Analyze your spectrum for absorption bands consistent with the structure of isopentyl acetate.

NOTE 3: Use the product characterization techniques designated by your laboratory instructor.

Obtain a nuclear magnetic resonance spectrum of your product. Compare your spectrum to that of pure isopentyl acetate, provided by your laboratory instructor.

5. Cleaning Up

Place your recovered materials in the appropriate labeled collection containers as directed by your laboratory instructor. Clean your glassware with soap or detergent.

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.

MICROSCALE FISCHER ESTERIFICATION

Using Glassware with a Hickman Still

Equipment

aluminum foil
3-mL conical vial
5-mL conical vial, with cap
forceps
Hickman still
magnetic spin vane
microspatula
4 Pasteur pipets, with latex bulb
2-mL pipet
5-mL product vial
sand bath*
13 × 100-mm test tube
thermometer, –10 to 150 °C,
  small size to fit Hickman still
thermometer, –10 to 260 °C,
  or equivalent
3 utility clamps
water-cooled condenser,
  with tubing

*stirring hot plate with crystallizing dish filled with sand or magnetic stirrer and electric flask heater filled with sand

Reagents and Properties

substance	*quantity*	*molar mass (g/mol)*	*bp (°C)*	*d (g/mL)*
glacial acetic acid	2 mL	60.05		1.049
isopentyl acetate*		130.19	142	0.876
isopentyl alcohol	0.809 g	88.15	130	0.809
sodium hydrogen carbonate, 5%	3 mL			
sodium sulfate, anhydrous	0.3 g	142.04		
sulfuric acid, concentrated	0.15 mL	98.08		

*product

Preview

- Weigh a 5-mL conical vial
- Add isopentyl alcohol and reweigh the vial
- Add glacial acetic acid and sulfuric acid to the vial and reflux the mixture for 1 hr
- Cool the reaction mixture
- Add 5% $NaHCO_3$
- Separate the aqueous layer
- Wash the organic layer with two additional portions of 5% $NaHCO_3$
- Wash the organic layer with water
- Dry the organic layer over anhydrous Na_2SO_4
- Transfer the product to a clean conical vial
- Assemble a distillation apparatus
- Distill the product
- Weigh the product
- Characterize the product for purity using GC, IR, and/or NMR

PROCEDURE

Chemical Alert

glacial acetic acid—*corrosive*
isopentyl acetate—*flammable and irritant*
isopentyl alcohol—*irritant*
sodium sulfate—*irritant and hygroscopic*
sulfuric acid—*corrosive and oxidant*

Caution: Wear departmentally approved safety goggles at all times while in the chemistry laboratory.

1. Refluxing the Reaction Mixture

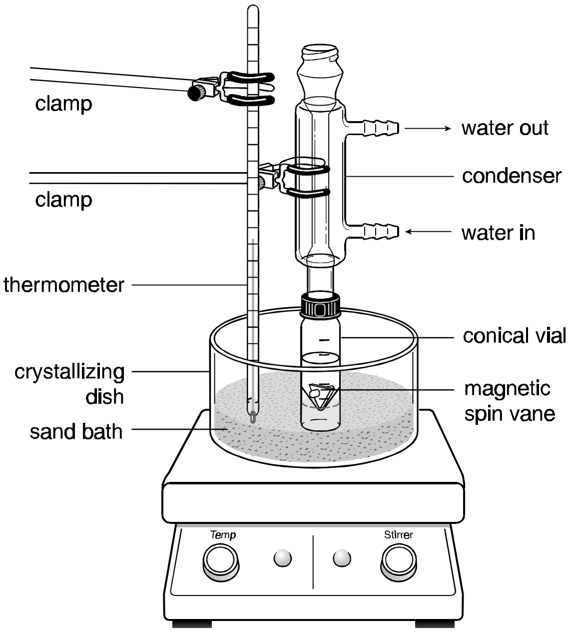
> **Caution:** Isopentyl alcohol (3-methyl-1-butanol) is irritating. Glacial acetic acid is corrosive. Concentrated sulfuric acid (H_2SO_4) is corrosive and oxidizing. Prevent eye, skin, clothing, and combustible material contact. Avoid inhaling vapors and ingesting these compounds. Use a ***fume hood***.

Weigh a 5-mL conical vial and record the tare mass. Place 0.809 g (1.0 mL) of isopentyl alcohol in the vial. Reweigh the vial to obtain the precise mass of the alcohol.

Add 2 mL of glacial acetic acid to the vial. Finally, use a Pasteur pipet to add 3 drops of concentrated H_2SO_4 to the vial.

Add a magnetic spin vane. Attach a water-cooled condenser, as shown in Figure 4.

Figure 4 Reflux apparatus for glassware using a conical vial



Turn on the magnetic stirrer. Start the flow of water through the condenser. Heat the sand bath to 150–160 °C. Reflux the mixture for 1 hr.

Turn off the heat and the magnetic stirrer. Raise the reflux apparatus out of the sand bath. Allow the reaction mixture to cool to room temperature. Turn off the water to the condenser.

2. Separating and Washing the Product Layer

Use forceps to remove the spin vane from the vial. *Slowly* add 1 mL of 5% sodium hydrogen carbonate ($NaHCO_3$) to the reaction mixture in the conical vial. Swirl the vial gently, then cap the vial. Shake the vial gently, with venting, until bubbles are no longer produced.

Label a test tube “Aqueous Layers”. Use a Pasteur pipet to remove the lower aqueous layer from the conical vial. Place the aqueous layer into the labeled test tube. [NOTE 1]

NOTE 1: It is a good practice to keep all extraction layers in labeled containers until the conclusion of the experiment.

Repeat the extraction of the upper organic layer two more times with new 1-mL portions of 5% $NaHCO_3$. Each time, place the lower aqueous layer into the labeled test tube.

Finally, wash the organic layer with 1 mL of distilled or deionized water. Again, remove the lower aqueous layer.

Add 0.3 g of anhydrous sodium sulfate (Na_2SO_4) to the vial to dry the product [NOTE 2]. Allow the mixture to stand for 15 min.

NOTE 2: Your laboratory instructor may instruct you to add the anhydrous Na_2SO_4 gradually until the solution is no longer cloudy or until the Na_2SO_4 no longer clumps.

3. Distilling the Product

Caution: Isopentyl acetate is flammable and irritating. Keep away from flames or other heat sources. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting this compound.

Use a Pasteur pipet to transfer the dried product to a 3-mL conical vial. Add a magnetic spin vane to the vial. Attach a Hickman still and a water-cooled condenser, as shown in Figure 5.

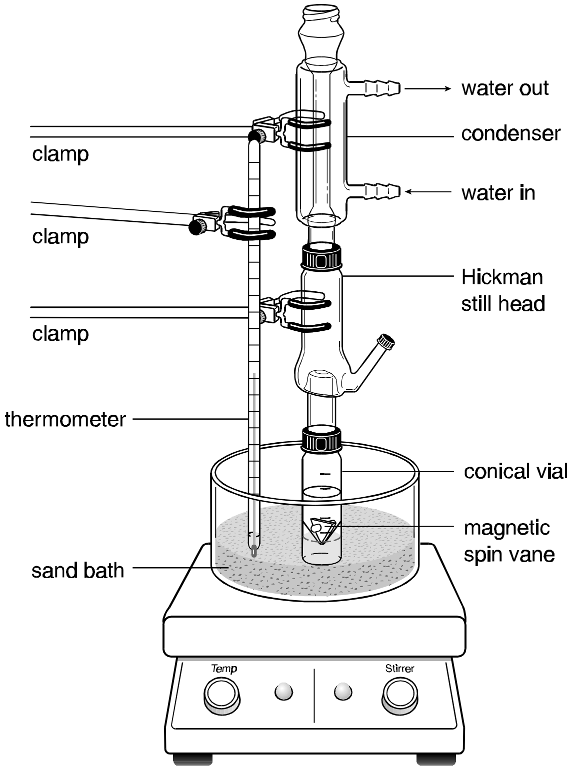


Figure 5 Distillation apparatus using Hickman still

Tare a product vial. Turn on the magnetic stirrer. Start the flow of water through the condenser. Distill the product by heating the sand bath to 150–160 °C. Cover the top of the sand bath with aluminum foil.

As distillate collects in the Hickman still, use a Pasteur pipet to transfer the distillate to the tared product vial. Weigh the product.

4. Characterizing the Product

[NOTE 3]

Analyze the purity of your product by gas chromatography. Compare the retention time of your product with that of pure isopentyl acetate under identical conditions.

Obtain an infrared spectrum of your product. Compare your spectrum to that of pure isopentyl acetate, provided by your laboratory instructor. Analyze your spectrum for absorption bands consistent with the structure of isopentyl acetate.

Obtain a nuclear magnetic resonance spectrum of your product. Compare your spectrum to that of pure isopentyl acetate, provided by your laboratory instructor.

NOTE 3: Use the product characterization techniques designated by your laboratory instructor.

5. Cleaning Up

Place your recovered materials in the appropriate labeled collection containers as directed by your laboratory instructor. Clean your glassware with soap or detergent.

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.

POST-LABORATORY QUESTIONS

1. Calculate your percent yield of isopentyl acetate.
2. List the distinctive features of the IR spectra of isopentyl acetate, isopentyl alcohol, and acetic acid and assign the absorption bands to the appropriate functional groups.
3. Check your gas chromatogram for the presence of compounds other than isopentyl acetate. Explain the presence of any additional compounds.
4. Compare the NMR spectrum for your product with pure isopentyl acetate. Explain any differences.
5. Write the equation for the reaction of 5% $NaHCO_3$ with acetic acid.
6. What is the IUPAC name for isopentyl acetate?

NAME SECTION DATE

713/Preparing Isopentyl Acetate by the Fischer Esterification

Pre-Laboratory Assignment

1. What are the hazards you should be aware of when you work with the following reagents?
   (a) concentrated H_2SO_4

   (b) glacial acetic acid

2. (a) Write a detailed mechanism for the formation of ethyl acetate from ethanol and acetic acid with H_2SO_4 as catalyst.

   (b) How does concentrated H_2SO_4 catalyze the esterification reaction? Briefly explain.

3. Give two methods by which the Fischer esterification equilibrium of the reaction described in Assignment 2 can be shifted to produce more of the ester.

4. Calculate the theoretical yield of isopentyl acetate for the esterification reaction described in the Procedure.

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