INSTRUCTOR'S INFORMATION

Preparing Isopentyl Acetate by the Fischer Esterification

EQUIPMENT

General

balance, 0.001-g
chips, boiling, acid-resistant
foil, aluminum
gas chromatograph, with integrating recorder*
hood, fume
spectrometer, NMR*
spectrophotometer, infrared†

*with instructions for operating and equipment for using
†either dispersive or Fourier-transformed (FT), with instructions for operating and equipment for using

Individual

Semi-microscale

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

*for ice-water bath

Microscale (Using Glassware with Elastomeric Connectors)

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

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

Microscale (Using Glassware with a Hickman Still)

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

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

REAGENTS

(Required for 10 students. Reagent amounts include 30% spillage allowance.)

Semi-Microscale

111 mL glacial acetic acid
71 mL isopentyl alcohol
650 mL 5% sodium hydrogen carbonate solution ($NaHCO_3$)
325 mL saturated sodium chloride solution (NaCl)
19.5 g sodium sulfate, anhydrous
13 mL concentrated sulfuric acid

Microscale *(Using Glassware with Elastomeric Connectors*)

0.65 g Dowex® 50W × 2-100*
26 mL glacial acetic acid
13 mL isopentyl alcohol
*or 2 mL concentrated sulfuric acid
39 mL 5% sodium hydrogen carbonate solution ($NaHCO_3$)
3.9 g sodium sulfate, anhydrous

Microscale *(Using Glassware with a Hickman Still*)

26 mL glacial acetic acid
13 mL isopentyl alcohol
39 mL 5% sodium hydrogen carbonate solution ($NaHCO_3$)
3.9 g sodium sulfate, anhydrous
2 mL concentrated sulfuric acid

PREPARATIONS

Semi-Microscale

1. 5% $NaHCO_3$ solution (~1 L): Dissolve 50 g of $NaHCO_3$ in 950 mL of distilled or deionized water to make ~1 L of solution.

2. Saturated sodium chloride solution (500 mL): Dissolve 180 g of NaCl in 500 mL of distilled or deionized water. The solubility of NaCl in water is 36 g NaCl per 100 g water at 25 °C.

Microscale

1. 5% $NaHCO_3$ solution (50 mL): Dissolve 2.5 g of $NaHCO_3$ in enough distilled or deionized water to make 50 mL of solution.

CHEMICAL HANDLING

Note: The information presented under **CHEMICAL HANDLING** may not conform to the latest Federal and state regulations because the regulations may have changed. Consult the appropriate agency for current information.

Specific information listed here concerning each of the chemicals used in this experiment should be compared to the contents of the corresponding MSDS.

Spillage Cleanup (for ***small*** amounts of liquids and solids in the stockroom)

1. Dowex® 50W × 2-100 ion exchange resin: Sweep up. Package for disposal in landfill.

Or, dispose of by method recommended by local regulations.

2. Glacial acetic acid: Cover with powdered sodium hydrogen carbonate ($NaHCO_3$) until no more reaction occurs. Sweep up. Place in glass container and carefully add water. Dissolve the material completely. Pour into drain with a large amount of running water. Pour a solution of 5% $NaHCO_3$ over the original spill site. Mop up with paper towels. Dispose of in trash.

Or, dispose of by method recommended by local regulations.

3. Isopentyl alcohol: Mop up and prepare for incineration.

Or, use a spill kit to absorb the alcohol, and dispose of by method recommended by local regulations.

4. 5% Sodium hydrogen carbonate solution: Mop up with paper towels. Dispose of in trash.

Or, dispose of by method recommended by local regulations.

5. Sodium sulfate: See **1.** Dowex® 50W × 2-100.

6. Sulfuric acid, concentrated: See **3.** Glacial acetic acid.

Collection Containers

You will need the following appropriately labeled containers:

"Recovered Aqueous Layers"
"Recovered Dowex® 50W × 2-100" (microscale—elastomeric connectors)
"Recovered Isopentyl Acetate"
"Recovered Sodium Sulfate"
"Used Pasteur Pipets"

Disposal

Note: Because of the wide variety of local and state regulations governing the types of reaction mixtures generated in this experiment, consult local authorities for current regulations.

1. Recovered Aqueous Layers: Pour down the drain with a large amount of running water.

Or, dispose of by method recommended by local regulations.

2. Recovered Dowex® 50W × 2-100: Package for disposal in landfill.

Or, dispose of by method recommended by local regulations.

3. Recovered Isopentyl Acetate: Recycle the isopentyl acetate by drying and distilling.

Or, prepare for incineration.

Or, dispose of by method recommended by local regulations.

4. Recovered sodium sulfate: Dry. See **2.** Recovered Dowex® 50W × 2-100.

Hazard Alert

1. Acetic acid [*Registry of Toxic Effects of Chemical Substances* (***RTECS***); 8 vols.; U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, U.S. Government Printing Office: Washington, DC, 1987, #AF1225000]: Corrosive. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting the compound.

2. Isopentyl acetate (***RTECS#*** NS9800000): Flammable and irritant. Fire hazard. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting the compound.

3. Isopentyl alcohol (***RTECS#*** EL5425000): Irritant. Prevent eye, skin, and clothing contact. Avoid inhaling vapors and ingesting the compound.

4. Sodium chloride (***RTECS#*** VZ4725000): Irritant and hygroscopic. Prevent eye, skin, and clothing contact. Avoid inhaling dust and ingesting the compound.

5. Sodium hydrogen carbonate (***RTECS#*** VZ0950000): Moisture-sensitive. Prevent eye, skin, and clothing contact. Avoid inhaling dust and ingesting the compound.

6. Sodium sulfate (***RTECS#*** WE1650000): Irritant and hygroscopic. Prevent eye, skin, and clothing contact. Avoid inhaling dust and ingesting the compound.

7. Sulfuric acid (***RTECS#*** WS5600000): Toxic and oxidizer. Prevent eye, skin, and clothing contact. Prevent contact with combustible materials. Avoid inhaling vapors and ingesting the compound.

DEMONSTRATIONS

1. Set up a reflux apparatus for a demonstration unit, if your students are not acquainted with the reflux procedure.

2. If you use a Hickman still that does not have a side port, demonstrate the preparation and use of a bent-tip Pasteur pipet. Use a microburner or a Bunsen burner to heat a Pasteur pipet 0.5-1 cm from the tip. Use tongs to bend the pipet tip to a 30° angle. Allow the pipet to cool.

3. Weigh 0.5 g of sodium sulfate. Place the sodium sulfate in a vial to serve as a demonstration amount for the microscale drying procedure.

4. Show venting procedure used with a separatory funnel.

5. Demonstrate to students the methods you wish them to use to characterize their product.

COMMENTS ON THE EXPERIMENT

Estimated bench time is 2–3 hr.

1. Bench time will vary depending on the reaction scale.

2. Some types of boiling chips disintegrate in the presence of strong acids. Check to make certain your boiling chips maintain their structure in acid. If they do not, glass beads can be substituted for boiling chips.

3. Removal of isopentyl acetate from the Hickman still is easy if the still is equipped with a side port. If your Hickman still does not have a side port, removal of the product will require removing the heat source from the distillation vial, allowing the apparatus to cool for 1 min, removing of the water-cooled condenser from the top of the Hickman still, and using a bent-tip Pasteur pipet to remove the product. Additional collection of product can be made by reassembling the apparatus and again heating the distillation vial. See the **DEMONSTRATIONS** section for information on preparing the bent-tip Pasteur pipet.

4. You may provide a GC plot showing the retention time for pure isopentyl acetate or you may have students obtain chromatograms of both their product and pure isopentyl acetate. A non-polar (DC-200) column at 130 °C with a flow rate of 60 mL/min works well.

5. If you use IR or NMR to characterize the product, you may wish to provide reference spectra for pure isopentyl acetate.

6. You may substitute glassware where appropriate. Reaction tubes, conical vials, and small round-bottom flasks can be used in microscale reactions. Aqueous extracts can be collected in any container. An air-cooled condenser can replace the water-cooled condenser in Figure 3 for glassware using elastomeric connectors.

7. You may wish to instruct your students to add the drying agent in small portions until the solution is clear or until the drying agent no longer clumps. The amount of sodium sulfate may be more or less than the listed amount, depending on the volume of the ester.

8. Phase separation paper may be used in place of anhydrous sodium sulfate.

9. You may choose to photocopy the attached data sheet for student use.

REPRESENTATIVE STUDENT DATA

The typical student yield for semi-microscale esterification is 60%.
Typical student yields for microscale esterifications are 25–60%.

ANSWERS TO POST-LABORATORY QUESTIONS

1. Answers will vary.

2. isopentyl acetate: C=O, 1740 cm^{-1}; C–O, 1240 cm^{-1}; $(CH_3)_2CH$, 1375 cm^{-1} isopentyl alcohol: O–H, 3200–3500 cm^{-1}; $(CH_3)_2CH$, 1370 and 1380 cm^{-1}, 1450 cm^{-1}

acetic acid: O–H, 2500–3500 cm^{-1}; C=O, 1710–1725 cm^{-1}; C–O, 1200–1300 cm^{-1} and 1400–1450 cm^{-1}

3. Unreacted isopentyl alcohol will elute earlier than isopentyl acetate.

4. Depending on the amount of unreacted alcohol, the spectrum could be very similar to pure isopentyl acetate or it could have a small peak, corresponding to the OH of the alcohol or water.

5. $NaHCO_3(aq) + CH_3COOH(aq) \rightarrow CH_3COONa(aq) + CO_2(g) + H_2O(l)$

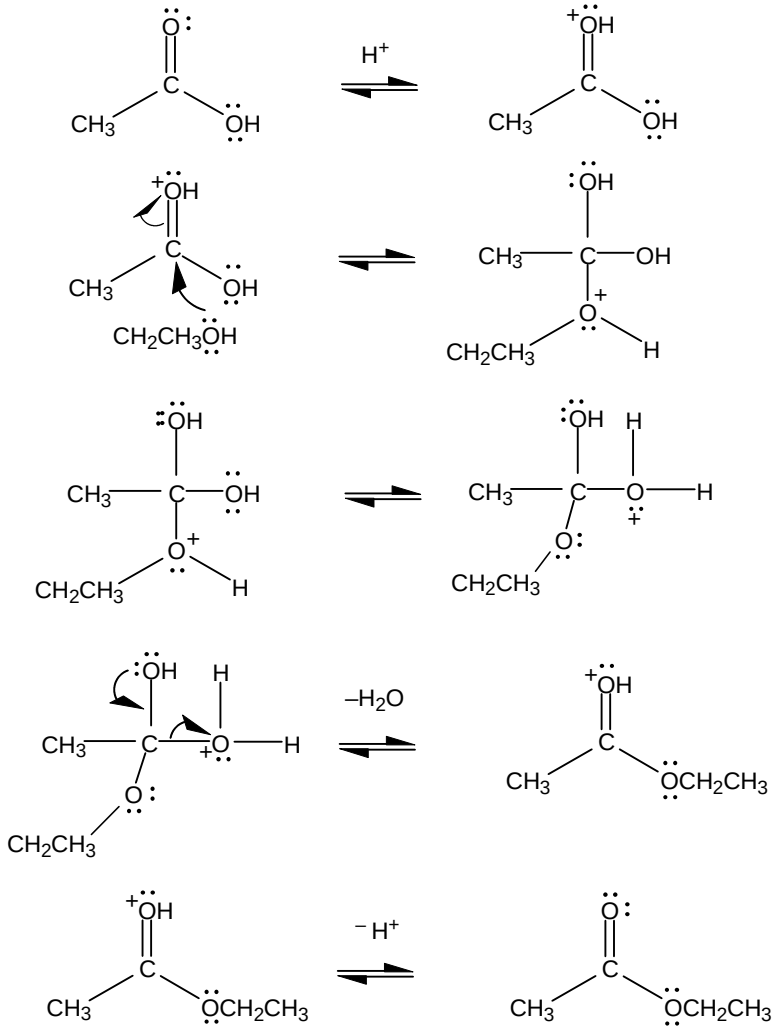
6. 3-methylbutyl ethanoate

ANSWERS TO PRE-LABORATORY ASSIGNMENT

1. (a) Concentrated H_2SO_4 is corrosive and oxidizing. Avoid contact with eyes, skin, clothing, and combustible materials.

(b) Glacial acetic acid is corrosive. Avoid contact with eyes, skin, and clothing.

2. (a)



(b) By protonating the acyl oxygen of the carboxylic acid, the acyl carbon becomes more electrophilic and more susceptible to nucleophilic attack.

3. An excess of either ethanol or acetic acid would shift the equilibrium to the product side. Alternatively, the water produced could be removed by azeotropic distillation or by including a drying agent in the reaction mixture.

4. IAA = isopentyl alcohol; IAAc = isopentyl acetate

Semi-microscale $(4.37\text{ g IAA})\left(\frac{1\text{mol IAA}}{88.15\text{ g IAA}}\right)\left(\frac{1\text{mol IAAc}}{1\text{mol IAA}}\right)\left(\frac{130.19\text{ g IAAc}}{1\text{mol IAAc}}\right) = 6.45\text{ g IAAc}$

Microscale $(0.809\text{ g IAA})\left(\frac{1\text{mol IAA}}{88.15\text{ g IAA}}\right)\left(\frac{1\text{mol IAAc}}{1\text{mol IAA}}\right)\left(\frac{130.19\text{ g IAAc}}{1\text{mol IAAc}}\right) = 1.19\text{ g IAAc}$

name _____ section _____ date _____

DATA SHEET

Fischer Esterification

	mL	*g*	*mol*
amount of isopentyl alcohol used	_____	_____	_____
amount of acetic acid used	_____	_____	_____
product obtained	_____	_____	_____
product theoretical yield		_____	_____

product percent yield, %_____

Write the equation for the reaction.

Product Characterization

	pure isopentyl acetate	*product isopentyl acetate*
GC retention time, min		
major IR bands, cm^{-1}		
NMR bands, ppm		