**STAGE 2 CHEMISTRY**

**ASSESSMENT TYPE 1: Investigation Folio**

**Practical 2: Ester Preparation**

## Purpose

This assessment task provides you with the opportunity to prepare, purify and collect an ester, and in particular, to demonstrate your ability to:

* perform a reflux using the appropriate apparatus,
* perform a distillation using the appropriate apparatus,
* use a separating funnel to purify the ester
* evaluate results, form a conclusion, and communicate your understanding of concepts relating to an organic preparation.

## Description of the Assessment

*Esters are organic compounds found in many fruits. They often have distinctive odours that are responsible for the characteristic smell and flavour of fruit such as bananas and apples.*

*The ester being prepared in this experiment is a common solvent, is found in perfumes and is used as a food flavouring. This clear, colourless liquid is known by its characteristic odour of pears. Due to this fact, it is commonly used as a flavouring additive. Further information about the health risks and safety data associated with this ester can be found by reading the* ***Materials Safety Data Sheet*** *for this ester. This can be obtained from the teacher on request.*

**SAFETY NOTE**

**Concentrated sulfuric acid is highly corrosive. Handle this substance with extreme care and carry out the mixing of sulfuric acid in the fume cupboard.**

**Ethanol is a volatile liquid and is flammable.**

**Propanoic acid is corrosive and causes burns. It is harmful by ingestion, inhalation and through skin contact and is a respiratory irritant.**

You are to prepare and purify an ester by carefully following the given procedure.

There are two STAGES to this investigation:

See page 4-5 for specific details of the steps required for Stages 1, 2 and 3.

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| **STAGE 1** **Preparation and purification of the ester.** This stage will involve the use of reflux,  distillation and the use of the separating funnel.  **STAGE 2** Individually, write a **practical report** using the suggested format provided. Assessment Conditions For this practical investigation, students plan and carry out the investigation in pairs, under teacher supervision. The practical, and the practical report are completed in a two-hour lesson in the laboratory.  **STAGE 1**  Perform the practical preparation in the laboratory.  **STAGE 2**  Use the results from the practical investigation to write the report.  In the report the following are assessed:   * the use of appropriate chemical terms, conventions, formulae and equations * communication of knowledge and understanding of chemistry in different forms. |  |

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| ***Learning Requirements*** | ***Assessment Design Criteria*** | ***Capabilities*** |
| 1. demonstrate and apply knowledge and understanding of chemical concepts and interrelationships  2. formulate questions, manipulate apparatus, record observations in practical chemical activities, and design and undertake chemistry investigations  3. demonstrate an understanding of how knowledge of chemistry can be used to draw informed conclusions or make informed decisions, taking into account social and environmental contexts  4. develop possible solutions to a variety of problems in chemistry, in new or familiar contexts  5. critically analyse and evaluate chemical information and procedures from different sources  6. communicate in a variety of forms using appropriate chemical terms and conventions. | Investigation  The specific features are as follows:  I1 Design of chemistry investigations.  I2 Selection and acknowledgment of information about chemistry and issues in chemistry from different sources.  I3 Manipulation of apparatus and technological tools using safe and ethical investigation procedures.  I4 The obtaining, recording, and display of findings of investigations using appropriate conventions and formats.  Analysis and Evaluation  The specific features are as follows:  AE1 Analysis and evaluation of data and other evidence to formulate conclusions and make relevant predictions.  AE2 Evaluation of procedures, with suggestions for improvements.  Application  The specific features are as follows:  A1 Application of chemistry concepts and evidence from investigations to solve problems in new and familiar contexts.  A2 Use of appropriate chemistry terms, conventions, formulae, and equations.  A3 Demonstration of skills in individual and collaborative work.  Knowledge and Understanding  The specific features are as follows:  KU1 Demonstration of knowledge and understanding of chemistry concepts.  KU2 Use of knowledge of chemistry to understand and explain social or environmental issues.  KU3 Communication of knowledge and understanding of chemistry in different forms. | Communication  Citizenship  Personal Development  Work  Learning  (delete those which do not apply to this assessment) |

**Investigation Folio: Practical – Ester Preparation**

## STAGE 1

**Materials**: • Quickfit apparatus • pumice

• propanoic acid (pure) • measuring cylinders (10 mL, 25 mL)

• ethanol • concentrated sulfuric acid

• 20 mL sodium carbonate solution (0.1 M) • fused calcium chloride

• small conical flask and stopper • 2000C thermometer

### Procedure 1 Measure 15 mL of the alcohol provided and 10 mL propanoic acid (pure) into the

### 50 mL pear-shaped Quickfit flask.

### 

2 Slowly add about 1 mL concentrated sulfuric acid and a few rice grains of pumice.

3 Assemble the glassware for reflux.

4 Reflux the mixture for 30 minutes, adjusting the flame for smooth boiling.

5 Allow the flask and contents to cool.

6 Pour the **cooled** mixture into a separating funnel, which contains about 20 mL of

distilled water. Shake and allow the layers to separate.

7 Discard the lower aqueous layer. Retain the upper organic layer in the separating funnel.

***Take care with this step.***

***Take extra care with the following steps.***

8 Add about 20 mL of sodium carbonate solution. Shake gently, releasing any gas that

builds up.

Repeat this step until no more effervescence occurs. Take care with this step as

pressure is built up due to carbon dioxide release.

9 Discard the aqueous layer. Retain the organic layer and wash it with about

20 mL of distilled water.

10 Collect the organic layer in a small conical flask containing fused calcium chloride (a

dehydrating agent). Stopper, swirl and allow to stand for 15 minutes.

11 Decant the organic liquid into a clean, dry 50 mL pear-shaped Quickfit flask and add a

few grains of pumice.

12 Assemble the glassware for distillation.

13 Distil the ester, recording the boiling range of the fraction collected.

*Do not distil all of the liquid*.

The boiling points of the possible components of the mixture to be distilled are:

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|  | Boiling Point (0C) |
| propanoic acid | 140.8 |
| ethanol | 78.3 |
| pure ester | 99.1 |
| sulfuric acid | 142 |

14 Record the appearance and odour of the ester.

# STAGE 2: Report Writing

When you have completed your practical you are to write a report, using the two headings:

“***Results***” and “***Discussion***”.

A guide to what is to be included under each of these heading is indicated below.

When you submit your report, attach to it any laboratory notes you may have written.

**Results:**

1 (a) Describe your observations for the following two stages of the experiment:

(i) Reflux: before, during and after reflux.

(ii) Distillation: include the physical characteristics of the product.

(b) Record the boiling *range* of your distillate. (ie the boiling temperature when the ester was collected?)

**Discussion:**

2 (a) Write an equation for the esterification reaction.

1. State the systematic name of the ester formed.

3 For this experiment, explain the *purpose* of the reflux and distillation procedures.

4 (a) (i) Identify the possible species present in the pear-shaped flask

at the *end of the reflux*.

1. Hence explain the purpose of adding 20 mL of water in Step 6.

(b) With the aid of an equation, explain the reason for adding the sodium carbonate

solution in Step 8.

5 This method of preparation often results in a poor yield of ester.

Identify possible reasons for a poor yield of ester.

6 Identify different types of hazards associated with this preparation and

*describe* the precautions that could be taken to ensure the safety of the experimenter.

**Material Safety Data Sheet**

**Ethyl Propanoate**

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| Section 1 - Chemical Product and Company Identification |

**MSDS Name:** Ethyl Propanoate, 99+% (gc)   
**Catalog Numbers:** AC150500000, AC150500010, AC150500025, AC150500250, AC150505000  **Synonyms:** Propanoic acid ethyl ester; Ethyl propanoate

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| Section 2 - Hazards Identification |

**EMERGENCY OVERVIEW**

Appearance: clear, colorless liquid. Flash Point: 12 deg C.  
**Warning! Flammable liquid and vapor.** Causes eye, skin, and respiratory tract irritation.   
**Target Organs:** Respiratory system, eyes, skin.   
  
**Potential Health Effects**   
**Eye:** Causes eye irritation.   
**Skin:** Causes skin irritation.   
**Ingestion:** May cause irritation of the digestive tract.   
**Inhalation:** Causes respiratory tract irritation. Vapors may cause dizziness or suffocation.   
**Chronic:** No information found.

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| Section 4 - First Aid Measures |

**Eyes:** Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid imme diately.   
**Skin:** Get medical aid. Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes.   
**Ingestion:** Do not induce vomiting. If victim is conscious and alert, give 2-4 cupfuls of milk or water. Never give anything by mouth to an unconscious person. Get medical aid immediately.   
**Inhalation:** Get medical aid immediately. Remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.   
**Notes to Physician:** Treat symptomatically and supportively.

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| Section 6 - Accidental Release Measures |

**General Information:** Use proper personal protective equipment as indicated in Section 8.   
**Spills/Leaks:** Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container. Wash area with soap and water. Clean up spills immediately, observing precautions in the Protective Equipment section. Remove all sources of ignition. Use a spark-proof tool. Isolate area and deny entry. Provide ventilation.

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| Section 7 - Handling and Storage |

**Handling:** Wash thoroughly after handling. Avoid contact with eyes, skin, and clothing. Empty containers retain product residue, (liquid and/or vapor), and can be dangerous. Keep away from heat, sparks and flame. Avoid ingestion and inhalation. Use only in a chemical fume hood. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose empty containers to heat, sparks or open flames.   
**Storage:** Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Flammables-area.

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| Section 8 - Exposure Controls, Personal Protection |

**Engineering Controls:** Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use only under a chemical fume hood.

**Exposure Limits**

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| **Chemical Name** | **ACGIH** | **NIOSH** | **OSHA - Final PELs** |
| Ethyl propionate | none listed | none listed | none listed |

**Personal Protective Equipment**   
**Eyes:** Wear appropriate protective eyeglasses or chemical safety goggles as described by OSHA's eye and face protection regulations in 29 CFR 1910.133 or European Standard EN166.   
**Skin:** Wear appropriate protective gloves to prevent skin exposure.   
**Clothing:** Wear appropriate protective clothing to prevent skin exposure.   
**Respirators:** Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

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| Section 9 - Physical and Chemical Properties |

**Physical State:** Liquid   
**Appearance:** clear, colourless   
**Odour:** fruity odour

**Vapour Pressure:** 44 mm Hg @ 27.2C   
**Vapour Density:** 3.5 (Air = 1)   
**Viscosity:** Not available.   
**Boiling Point:** 99 deg C @ 760.00mm Hg   
**Freezing/Melting Point:** -73 deg C   
**Solubility:** Soluble.   
**Specific Gravity/Density:** 0.8910g/cm3   
**Molecular Formula:** C5H10O2   
**Molecular Weight:** 102.13

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| Section 10 - Stability and Reactivity |

**Chemical Stability:** Stable under normal temperatures and pressures.   
**Conditions to Avoid:** Incompatible materials, ignition sources, excess heat.   
**Incompatibilities with Other Materials:** Strong oxidizing agents, acids, bases.   
**Hazardous Decomposition Products:** Carbon monoxide, carbon monoxide, carbon dioxide.   
**Hazardous Polymerization:** Has not been reported.

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| Section 15 - Regulatory Information |

**Risk Phrases:**  
     R 11 Highly flammable. R 2 Risk of explosion by shock, friction, fire or other sources of ignition.   
     R 36/37/38 Irritating to eyes, respiratory system and skin.  **Safety Phrases:**  
     S 16 Keep away from sources of ignition - No smoking.   
     S 23 Do not inhale gas/fumes/vapour/spray.   
     S 24/25 Avoid contact with skin and eyes.   
     S 26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.   
     S 36/37/39 Wear suitable protective clothing, gloves and eye/face protection.

**Skills checklist – manipulating apparatus, quality of product, safety, collaboration (Teacher use only)**

**GROUP: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

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| **Stage 1:** | **Teacher observations:** | **Highly effectively constructive/focused** | **Mostly effectively mostly constructive/focused** | **Generally effective**  **Generally constructive/focused** | **Inconsistent** | **Limited** |
| Performing reflux |  |  |  |  |  |  |
| Use of separating funnel |  |  |  |  |  |  |
| Performing distillation |  |  |  |  |  |  |
| Clarity of ester product |  |  |  |  |  |  |
| Area cleanliness |  |  |  |  |  |  |
| Safe conduct |  |  |  |  |  |  |
| Cooperation |  |  |  |  |  |  |
| Other |  |  |  |  |  |  |