47th National Chemistry Olympiad

Utrecht University

PRACTICAL EXAM

Exam booklet

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Instructions/Resources

- This practical exam consists of two parts:
  - The synthesis of the ester 1-pentyl propanoate
  - The determination of the acetic acid content in vinegar using a titration curve
- The practical exam ends after four hours. Within this time:
  - the attached answer sheets must be completed
  - all questions must be answered
- After the practical exam, once you have handed in all materials, the glassware must still be cleaned and placed in the bins for used glassware.
- The maximum score for the practical exam is **80 points**.
- The score is determined by:
  - practical skills, neatness, and safety — maximum 20 points
  - results of the determinations and answers to the questions — maximum 60 points
- Required tools: a (graphical) calculator, ruler/protractor triangle and Binas or ScienceData.
- First, read the introduction and all assignments, and only then begin carrying them out.
- Write your answers to the questions in the boxes on the answer sheets. If you do not have enough space, you may ask for extra paper.

Additional Information:

- This is an examination; consultation with other participants is not permitted.
- If you have a question, you may ask a supervisor.
- If there is a problem with your glassware or equipment, report it to the supervisor as soon as you discover it. Do not borrow equipment from another participant.

Order of the Experiments

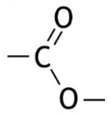
This exam consists of two experiments.

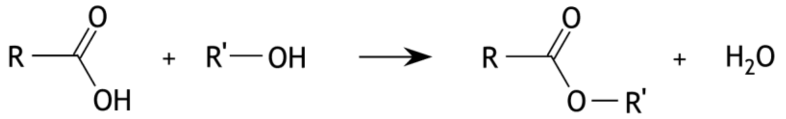
Start with **Experiment 1** (the synthesis of an ester).

During this experiment, the reaction mixture must be heated under reflux for 1.5 hours. Use this time to carry out **Experiment 2** (determining the acetic acid content in vinegar) and answer the associated questions.

Experiment 1 The synthesis of the ester 1-pentyl propanoate 40 points

Introduction

Esters are organic compounds whose molecules contain a  group. Esters can be regarded as the product of a reaction between a carboxylic acid and an alcohol:

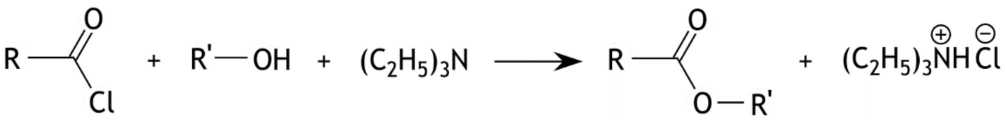


(1)

In everyday life, we come across both natural and synthetic esters everywhere. For example, esters are responsible for the scent of fruits and flowers, fats and oils are esters of glycerol and fatty acids, and ester compounds are commonly found in medicines.

So, esters can be synthesised by a reaction between a carboxylic acid and an alcohol (1). However, these reactions are inefficient and have a low yield. A more efficient way to make esters is by a reaction of an alcohol with an acid chloride, which is much more reactive than a carboxylic acid. When doing this, care must be taken to ensure that the acid chloride does not come into contact with water prior to the reaction, as it will then immediately undergo a violent reaction, forming the corresponding carboxylic acid and hydrogen chloride.

In this type of esterification, an organic base such as triethylamine is often used. This acts as a catalyst, but it also serves to bind the H^+ released during the reaction. Triethylammonium chloride is then produced, which precipitates as a solid from the reaction mixture:



(2)

In this experiment, you will use this reaction to synthesise 1-pentyl propanoate. You'll start with a smelly mixture containing triethylamine and end up with a nicely fruity ester!

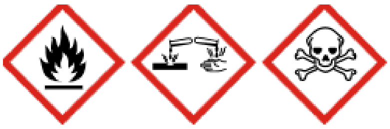
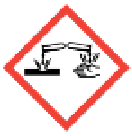
The reactants for the synthesis are pentan-1-ol, propanoyl chloride and triethylamine. A slight excess of each of the latter two substances is used.

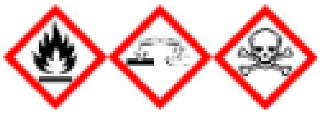
Chemicals

- triethylamine
- diethyl ether (hereafter referred to as ether for convenience)
- pentan-1-ol
- propanoyl chloride
- demineralised water
- 2 M sulphuric acid
- 1 M Na_2CO_3 solution
- $MgSO_4$ powder
- $CaCl_2$ granules

Safety information and data about the chemicals

- wear safety goggles
- if your skin comes into contact with any of the chemicals, rinse it off immediately

<u>Triethylamine</u>  ▪ Formula: $C_6H_{15}N$ ▪ M = 101.191 g mol^{-1} ▪ ρ = 0.726 g mL^{-1} ▪ H301+H311+H331 H314, H335. ▪ P280, P303+P361+P353, P304+P340+P310 P305+P351+P338.	<u>Diethyl ether (Ethoxy ethane)</u>  ▪ Formula: $C_4H_{10}O$ ▪ M = 74.12 g mol^{-1} ▪ ρ = 0.713 g mL^{-1} ▪ H224, H336 ▪ P210, P243, P261 ▪ P303+P361+P353 P304+P340 P312
<u>Sulphuric acid solution 2 M</u>  ▪ Formula: H_2SO_4 solution ▪ H290, H314 ▪ P280, P301+P330+P331+P310 P303+P361+P353, P310, P305+P351+P338.	<u>Calcium chloride granules</u>  ▪ Formula: $CaCl_2$ ▪ M = 111 g mol^{-1} ▪ H319 ▪ P280 P305+P351+P338 P337+P313

Propanoyl chloride	Pentan-1-ol
 ▪ Formula: C_3H_5ClO ▪ M = 92.53 g mol^{-1} ▪ ρ = 1.06 g mL^{-1} ▪ H331, H314, H302, H225 ▪ P305+P351+P338 P303+P361+P353 P280, P210	 ▪ Formula: $C_5H_{12}O$ ▪ M = 88.15 g mol^{-1} ▪ ρ = 0.814 g mL^{-1} ▪ H226, H315, H332, H335 ▪ P210, P260 P302+P352, P312
1-Pentyl propanoate  ▪ Formula: $C_8H_{16}O_2$ ▪ M = 144.2 g mol^{-1} ▪ ρ = 0.873 g mL^{-1} ▪ H226 ▪ P210, P370+P378	

Materials

You'll find the materials you need in the box with clean glassware in your own aisle. Other materials can be found along the edges of the lab room.

Protocol

Read the full protocol before you start!

1. Set up a dry reflux apparatus. This is a reflux apparatus with a calcium chloride tube placed on top of the bulb condenser. We have already filled the calcium chloride tube for you. Use a 250 mL three-necked flask.
2. Place a dropping funnel on one of the "side necks" of the three-neck flask. Make sure the tap is closed.
3. Use a measuring cylinder (and a funnel if you want) to measure out approximately 21.7 mL of pentan-1-ol and approximately 33.7 mL of triethylamine. Write down the measured volumes to the correct number of decimal places.
4. Transfer the alcohol and the triethylamine into the three-neck flask of your reflux set-up. Rinse the measuring cylinder used to measure out the alcohol with two 5 mL portions of diethyl ether (hereafter referred to simply as ether).
5. Add approximately 25 mL of anhydrous ether and a stir bar.
6. Use a pan filled with ice and a little water to cool the reaction mixture.
7. Switch on the stirring motor.
8. Let a supervisor check your set-up.
9. Use a clean, dry measuring cylinder to measure out approximately 19.2 mL of the acid chloride. Write down the measured volume to the correct number of decimal places.
10. Transfer the acid chloride into the dropping funnel. Make sure that the acid chloride cannot accidentally end up in the reaction mixture already.
11. Add the acid chloride slowly, drop by drop, to the reaction mixture. The reaction has to stay under control.
12. Once all acid chloride is added, the ice bath can be removed.
13. Place a heating mantle under the setup, switch on the heater and allow the reaction mixture to reflux for 1.5 hours. (It can sometimes be difficult to tell if it is boiling, but that is no problem.)

Now carry out **Experiment 2**.
After having carried out **Experiment 2**, carry out the rest of **Experiment 1**.

14. After 1.5 hours of refluxing, switch off the heat, lower the lab lift with the heating mantle and allow the flask containing the reaction mixture to cool for about 10 minutes.
15. Add about 40 mL demineralised water to the reaction mixture.
16. Make sure that as much of the material as possible is dissolved. If necessary, swirl gently.
17. Transfer the reaction mixture into a separatory funnel.
18. Remove the aqueous layer.
19. Wash the ether layer by adding 15 mL of 2 M sulphuric acid to the ether layer, shake the mixture (remember to release the gas), and then remove the aqueous layer.
20. Wash the ether layer by adding 10 mL water to the ether layer, shake the mixture (remember to release the gas), and then remove the aqueous layer.
21. Wash the ether layer by adding 15 mL 1 M Na_2CO_3 solution to the ether layer, shake the mixture (remember to release the gas, that is extra important in this step!), and then remove the aqueous layer.
22. Wash the ether layer by adding 10 mL water to the ether layer, shake the mixture (remember to release the gas), and then remove the aqueous layer.

23. Transfer the ether layer into an Erlenmeyer flask
24. Add a few small scoops of dry $MgSO_4$ until you see a fine powder when you swirl the Erlenmeyer flask.
25. Weigh a 100 mL round-bottom flask. Write down the mass.
26. Place some cotton wool at the top of a funnel and filter the mixture through it. Collect the filtrate in the weighed round-bottomed flask.
27. Use a rotavap to evaporate the ether. Keep the water bath at ≤20°C.
28. Weigh the flask with your ester. Write down the mass.
29. Using a Pasteur pipette, transfer 1 drop of your ester into an NMR tube. Seal the NMR tube with a cap and use a “little flag” to mark the tube.
30. Hand in the labelled NMR tube. After the practical test, this will be used to record an NMR spectrum to determine the purity of your product. You do not need to wait for the result.
31. Transfer the rest of your ester into a 20 mL counting vial. Close the vial and write your fume hood number on the vial using a waterproof pen.
32. Hand in the counting vial as well.

Questions – write the answers on the answer sheets

1 Give the name(s) of the substance(s) that are removed from the reaction mixture when water is added (step 15). 3

2 What is the purpose of washing with sulphuric acid (step 19)? 1

3 What is the purpose of adding magnesium sulphate (step 24)? 1

4 Write down:
- the number of mL of alcohol you have used
- the number of mL of triethylamine you have used
- the number of mL of acid chloride you have used 2

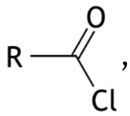
5 Calculate the maximum number of grams of ester that can be produced during the synthesis at 100% conversion. 4

6 Write down:
- the mass of the empty round-bottom flask
- the mass of the round-bottom flask with your ester 3

7 Calculate the yield of the ester synthesis. Express your answer both in grams and as a percentage of the maximum yield at 100% conversion. 2

The triethylamine used in the reaction acts as a catalyst, but also as a base to bind the H^+ that is split off. For this reason, triethylamine is not used in a catalytic amount.

8 Give a possible mechanism for the action of the triethylamine.

Write the acid chloride as 

, the alcohol as R' – OH and the triethylamine as $N(C_2H_5)_3$. Draw the non-bonding electron pairs in this and use curved arrows () to show how electron pairs move. 4

Experiment 2 The determination of the acetic acid content in vinegar using a titration curve. 40 points

Introduction

During an acid-base titration, the pH of the solution being titrated changes. If a (weak) acid is titrated with caustic soda (a solution of sodium hydroxide), the pH rises slowly at first, then strongly and finally slowly again. The pH gradient during a titration can be measured with a pH meter and plotted in a diagram. In this way, a titration curve is obtained.

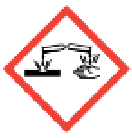
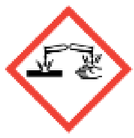
In this experiment, the titration curve for the titration of a solution of acetic acid is determined, and on the basis of this, it is checked whether the vinegar used meets the requirements of the Commodities Act.

Chemicals

- vinegar; density 1,005 g mL^{-1}
- demineralised water
- 0.1 M caustic soda (the exact molarity is given)

Safety

– Wear safety glasses
– If your skin comes into contact with any of the chemicals, rinse it off immediately

Caustic soda 0.1 M	Acetic acid solution <10%
	
▪ Formula: NaOH solution ▪ H290 ▪ P234, P390	▪ Formula: $C_2H_4O_2$ ▪ H226, H314, H318

Materials

You'll find the materials you need in the box with clean glassware in your own aisle.
Other materials can be found along the edges of the lab room.

Protocol

Read the full protocol before you start!

1. Pipette 5.00 mL of vinegar into a beaker.
2. Add 20.00 mL of water with a volumetric pipette.
3. Use a stirring bean to gently stir the mixture during the titration.
4. Remove the cap from the pH electrode. Note: handle it carefully, the electrode is a fragile instrument.
5. Gently rinse the electrode over a large beaker of demineralised water and dab the electrode with a soft tissue to remove the water. Do not touch the electrode with your fingers!
6. Insert the electrode into the solution and press the "read" button to measure the pH. Make sure that the stirring bean does not touch the electrode!
7. When the pH level is stable and no longer blinks, write it down on the answer sheet. Remove the electrode from the solution.
8. Titrate the vinegar solution by adding a little caustic soda at a time, measuring the pH after each addition and writing it on the answer sheet. Also write down how much caustic soda you have added. Rinse and dab the electrode between each measurement. Make sure you have enough measuring points to be able to construct the entire titration curve. Stop when the pH of the solution is about 12.5. Near the equivalence point, the pH changes very quickly!
9. Repeat steps 1 to 8. Use your experience from the first measurements to choose smart step sizes for adding caustic soda, so that you get a good picture of the interesting pH ranges, but don't take unnecessary measurements in the less interesting pH ranges.

Questions: Write down the answers on the answer sheets.

9 Write down the exact molarity of the caustic soda you used.

10 Write down all the pH levels in the tables on your answer sheet and the corresponding amount of caustic soda added. You don't have to use all the rows in the tables. 2

11 Plot the measurement results of both determinations on graph paper to obtain a titration curve of acetic acid for each determination. 10

12 Check whether the acetic acid content in the vinegar used meets the requirement of the Commodities Act (at least 4 per cent by mass). Use the titration curve of your most reliable determination for this. 10

13 In point 2, you added 20.00 mL of water with a volumetric pipette. Could you have used a graduated cylinder for this? Explain your answer. 2

You can also calculate the pH course of an acid-base titration. For example for the titration of 10 mL of 0.10 M acetic acid with 0.10 M caustic soda.

14 For this titration, calculate the pH at the following moments:
- when 5.0 mL of caustic soda is added
- in the equivalence point 6