46th National Chemistry Olympiad

Symeres, Nijmegen

PRACTICAL TEST

Assignment booklet

Thursday June 5 2025

Symeres
Making Molecules Matter. Together.

57th INTERNATIONAL
CHEMISTRY OLYMPIAD
UNITED ARAB EMIRATES 2025

SCHEIKUNDE
OLYMPIADE

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Instructions/resources

- This practical test consists of two parts:
  - The cracking of PET.
  - The determination of the amount of $NaBO_3.H_2O$ in a sachet of Bikosan.

- The practical test ends after 4 hours. During this time:
  - the attached answer sheets need to be completed;
  - all questions must be answered.

- After the practical test, when you have handed everything in, the glassware still needs to be cleaned and tidied up.

- The maximum score for the practical test is 80 points.

- The score is determined by:
  - practical skills, working cleanly, safety — maximum 20 points
  - results of the determinations and answers to the questions — maximum 60 points

- Required tools: (graphical) calculator, ruler/protractor triangle and Binas or ScienceData.

- First read the introduction and all the assignments before you start working.

- Write your answers to the questions in the boxes on the answer sheets provided. If you don't have enough space, you can request additional paper.

Additional:

- This is a test; it is not permitted to consult with other participants.
- If you have any questions, you can ask the supervisor.
- If there is anything wrong with your glassware or equipment, please report it to the supervisor as soon as you discover it. Don't borrow equipment from others!

Order of the experiments

This test consists of two experiments.

Start with **Experiment 1** (The cracking of PET).
During this experiment, the reaction mixture has to boil for 1.5 hours.
Use this time to perform **Experiment 2** (the titration).

The questions in **Experiment 2** can be answered after both experiments are finished, if necessary.

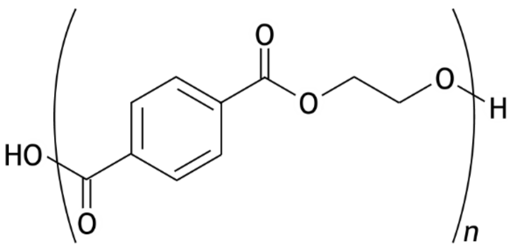
Experiment 1 The cracking of PET **40 points**

Introduction

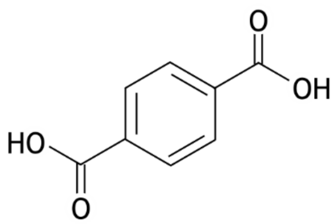
Many bottles for mineral water, soft drinks, liquid detergents and cleaning products are made of polyester PET, **P**oly**E**thylene **T**erephthalate.
It is recognizable by the ‘1’ symbol on the packaging. PET is also used for clothing (‘*fleece*’).
The environmental impact of PET is a major issue.
The advantages of plastic packaging are primarily attributable to its light weight, versatility in applications, and sustainability with regard to food preservation.
The disadvantages primarily relate to production and recycling. PET is easily recycled, better than other plastics, but the disposal of plastic as a separate waste stream could be significantly improved.

PET is primarily recycled by melting it down.
Another, advanced, chemical method for PET recycling could begin with the hydrolysis of PET in a basic environment (saponification). After processing and acidifying the reaction mixture, terephthalic acid can be isolated.

In this experiment you will perform this hydrolysis of PET and determine the yield of terephthalic acid.



PET



terephtalic acid

Chemicals

- piece of PET
- butan-1-ol
- KOH
- demineralized water
- 4 M hydrochloric acid
- ethanol

<u>Butan-1-ol</u> ▪ Formula: $C_4H_{10}O$ ▪ Density: 0.8098 g mL^{-1} ▪ Boiling point: 119 °C ▪ H226, H302, H315, H318, H335, H336 ▪ P261, P280, P302+P352, P304+P340, P305+P351+P338	<u>Potassium hydroxide</u> ▪ Formula: KOH ▪ H290, H302, H314 ▪ P280, P303+P361+P353, P305+P351+P338, P310
<u>Hydrochloric acid 4 M</u> ▪ Formula: HCl solution ▪ H290, H315, H319, H335 ▪ P302+P352, P304+P340, P305+P338+P351, P312	<u>Ethanol (absolute)</u> ▪ Formula: C_2H_6O ▪ Density: 0.789 g mL^{-1} ▪ Boiling point: 78 °C ▪ H225, H319 ▪ P210, P233, P305+P351+P338
<u>Terephthalic acid</u> ▪ Formula: $C_8H_6O_4$ ▪ H315, H319, H335 ▪ P261, P305+P351+P338	

Safety

- wear safety glasses
- if your skin comes into contact with any of the chemicals, rinse immediately with tap water

Materials

- scissors
- a spatula
- two measuring cylinders (25 mL and 50 mL)
- a magnetic stirrer
- a round-bottom flask
- a reflux condenser
- a magnetic bar
- a powder funnel
- a funnel
- a cotton pad
- an oil bath
- an ice bath
- a thermometer
- a lifting platform
- stand with clamp(s) (for separatory funnel)
- a separatory funnel
- two 100 mL Erlenmeyer flasks
- pH paper
- Pasteur pipette
- pipette teat
- glass filter
- a sample jar with a screw lid
- a sample jar with a crimp cap
- a highlighter pen
- a cork ring

Procedure

1. Prepare the setup for a reflux experiment.
2. Cut the PET piece into small pieces and accurately weigh it to approximately 1 g. Record the mass on your answer sheet.
3. Transfer these PET pieces to the round-bottom flask.
4. Add a magnetic bar.
5. Measure out approximately 30 mL of butan-1-ol and add it to the flask.
6. Weigh 0.88 g of potassium hydroxide and add it to the flask.
7. Place the reflux condenser on the flask.
8. Have your setup checked by a supervisor.
9. Place the oil bath on the magnetic stirrer and turn up the lifting platform. Ensure that the oil level remains below the solvent level in the flask.
10. Turn on the magnetic stirrer.
11. Put the thermometer in the oil bath and set the temperature to 120 °C.
12. Turn on the heat and let the reaction mixture reflux for 1.5 hours.
13. **Caution: Be extra careful from now on. The oil is hot!**

<u>Now perform Experiment 2.</u>
<u>After performing Experiment 2, finish the rest of the steps of Experiment 1.</u>

14. After 1.5 hours, turn off the heating, lower the lifting platform, and let the flask cool in the ice bath for about 5 minutes. Do not handle the flask with your hands; use the clamp.
15. Prepare your workspace for an extraction.
16. Add about 20 mL of water to the reaction mixture.
17. Filter the suspension through a funnel with a cotton pad and rinse with approximately 5 mL of water.
18. Transfer the filtrate to a separatory funnel, shake well and collect the aqueous layer in a clean Erlenmeyer flask.
19. Extract the organic layer again with 20 mL of water and collect the aqueous layer in the Erlenmeyer flask containing the aqueous layer from the previous step.
20. Using a Pasteur pipette, acidify the collected aqueous layers with 4 M hydrochloric acid to a pH of approximately 2.
21. Filter the obtained suspension over the glass filter using vacuum suction.
22. Turn off the vacuum suction.
23. Wash the residue as follows: add 5 mL of ethanol to the residue on the glass filter and stir the residue into the ethanol using the spatula. Work carefully.
24. Filter off the ethanol using vacuum suction.
25. Repeat steps 22, 23, and 24 two more times.
26. Turn off the vacuum suction and transfer the filtrate from the vacuum flask into a clean Erlenmeyer flask.
27. Further dry the residue on the glass filter under vacuum suction.
28. When the residue (= terephthalic acid) is dry (it no longer sticks to your spatula when you stir it), you can determine the mass.
29. Take the sample container with the screw cap, mark it with your name + PET, weigh it and record the mass.
30. Transfer the dry residue to this sample container and weigh the contents. Record the mass of the container and its contents.
31. Place approximately 20.0 to 25.0 mg (accurately weighed) of your product in the sample jar with a crimp cap, record the exact mass on the container and on your answer sheet, and sign the container with your name.
32. Close this jar with a crimp cap using the crimping pliers, a supervisor can help you with this.
33. Hand in both sample containers and their contents. The purity of your product will be determined by recording an NMR spectrum. In addition, if necessary, a Karl Fisher determination will be performed on the approximately 20 mg of product to determine the water content. This will be done for you after the practical exam. You do not need to wait for the results.

Tip 1: If the suspension is difficult to filter in step 20, add approximately 5 mL of ethanol first.

Tip 2: If the residue forms a hard lump or multiple lumps during drying in step 25, carefully break it up with the spatula and dry it some more.

Questions – write the answers on the answer sheets

1 Record:
- the mass of the PET
- the mass of the empty sample container
- the mass of the sample container with contents 3

2 Calculate the mass of your product. 1

To ensure complete hydrolysis of PET in alkali conditions, an excess of KOH was used.

3 Write the reaction equation for the complete hydrolysis of PET in alkali conditions. Use structural formulas for the organic compounds and write the structural formula for PET as shown in the introduction. 6

4 Show by calculation that an excess of KOH was used.
- Assume that the piece of PET consists exclusively of polyethylene terephthalate.
- Do not take into account the ends of the PET molecules. 5

Polyethylene terephthalate is the main component of the material used to make PET bottles. Besides polyethylene terephthalate, it also contains a plastisizer, for example.

5 Calculate the mass percentage of polyethylene terephthalate in the PET piece. Assume your product is pure terephthalic acid. Ignore the ends of the PET molecules. 5

Experiment 2 The determination of the amount of sodium perborate monohydrate in one sachet of Bikosan 40 points

Introduction

Bikosan is a solid mixture used as a mouth disinfectant in dissolved form. It consists of two substances. One of these substances is sodium perborate monohydrate ($NaBO_3.H_2O$). When one mole of $NaBO_3.H_2O$ is dissolved in water, one mole of hydrogen peroxide (H_2O_2) is produced. Hydrogen peroxide is the active ingredient in a Bikosan solution.

Bikosan is available in packages containing ten sachets of powder. It is used by dissolving the contents of one sachet in a cup of warm water (approximately 30 mL), and rinsing the mouth with the resulting solution.

In this experiment, the amount of $NaBO_3.H_2O$ in a single sachet of Bikosan is determined. An excess of sulfuric acid and an excess of potassium iodide solution are added to a precisely measured amount of Bikosan solution. The following reaction occurs:

$$H_2O_2 + 2\ H^+ + 2\ I^- \rightarrow I_2 + 2\ H_2O$$

This redox reaction, however, is slow. To speed up the reaction, a catalyst solution is added.

The amount of iodine formed is determined by titration with a sodium thiosulfate solution. The following reaction occurs:

$$I_2 + 2\ S_2O_3^{2-} \rightarrow 2\ I^- + S_4O_6^{2-}$$

The titration is performed in duplicate.

Chemicals

- Bikosan
- demineralized water
- approximately 0.050 M sodium thiosulfate solution (precise molarity is given)
- 0.3 M potassium iodide solution
- ammonium molybdate solution (catalyst)
- 2 M sulfuric acid solution
- starch solution

<u>Sodium thiosulfate solution 0.050 M</u> ▪ Formula: $Na_2S_2O_3$ solution ▪ No H and P phrases	<u>Potassium iodide solution 0.3 M</u> ▪ Formula: KI solution ▪ H372 ▪ P260, P264, P270, P314
<u>Sulphuric acid solution 2 M</u> ▪ Formula: H_2SO_4 solution ▪ H314 ▪ P280, P303+P361+P353, P310, P305+P351+P338.	<u>Starch solution</u> ▪ Formula: $(C_6H_{10}O_5)_n$ solution ▪ No H and P phrases
<u>Ammonium molybdate solution</u> ▪ Formula: $H_8Mo_2N_2O_7$ ▪ No H and P phrases	

Safety

- wear safety glasses
- if your skin comes into contact with any of the chemicals, rinse immediately with tap water

Materials

- a 100 mL volumetric flask
- a funnel
- a 50 mL burette
- a funnel for the burette
- four beakers
- a 10 mL graduated cylinder
- a 25 mL graduated cylinder
- a pipette filler
- a 10 mL pipette
- two 100 mL Erlenmeyer flasks with suitable rubber stoppers
- a magnetic stirrer
- a magnetic bar

Procedure

- ☐1 Weigh the total contents of one sachet of Bikosan. Record the mass on your answer sheet.
- ☐2 Weigh approximately 0.7 g of Bikosan accurately and record the mass on your answer sheet.
- ☐3 Transfer this Bikosan quantitatively into the 100 mL volumetric flask and fill up to the mark with demineralized water.
- ☐4 Transfer 10.00 mL of the Bikosan solution from the volumetric flask to each Erlenmeyer flask.
- ☐5 Add 15 mL of 2 M sulfuric acid solution and 10 mL of 0.3 M potassium iodide solution to both Erlenmeyer flasks.
- ☐6 Add 1 mL of catalyst solution (ammonium molybdate solution) to both Erlenmeyer flasks.
- ☐7 Close both Erlenmeyer flasks with a rubber stopper and wait at least 5 minutes. Swirl the solution occasionally.
- ☐8 While waiting, fill the burette with the sodium thiosulfate solution.
- ☐9 Place the magnetic bar into the solution in one of the Erlenmeyer flasks and begin stirring (do not heat).
- ☐10 Titrate the contents of the Erlenmeyer flask with the sodium thiosulfate solution until the liquid is light yellow.
- ☐11 Then add about 2 mL of starch solution.
- ☐12 Continue titrating until a clear color change occurs.
- ☐13 Remove the magnetic bar from the solution and repeat steps 9 through 12 with the solution in the other Erlenmeyer flask.

Questions – write the answers on the answer sheets

6 Record:

- the mass of the total contents of the Bikosan sachet
- the mass of the Bikosan sample
- all burette readings 8

7 Calculate the amount of $NaBO_3.H_2O$ (in g) in one sachet of Bikosan. 14

8 Closing the Erlenmeyer flask with the rubber stopper is important. Oxygen in the air can convert the iodide into iodine. Give the equation for this reaction and explain whether the calculated amount of $NaBO_3.H_2O$ in a sachet of Bikosan becomes higher or lower. 4

9 Could you also perform the determination by titrating with a solution of the oxidizing agent potassium permanganate?
If you think not, explain why it is not possible.
If you think so, give an outline of how such a determination should be performed. 4