

Teacher Resource Bank

GCE Chemistry

PSA7A: AS Organic Chemistry

- Distil a Product from a Reaction (Cyclohexene)



AS Organic Chemistry

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Technical Sheet

To prepare cyclohexene by the dehydration of cyclohexanol and to distil the cyclohexene from the reaction mixture.

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

Requirements

- semi-micro distillation apparatus fitted with a thermometer (-10 °C to 110 °C)
- concentrated phosphoric acid
- cyclohexanol
- protective gloves
- stand and clamp
- 10 cm³ measuring cylinder
- 25 cm³ measuring cylinder
- anti-bumping granules
- separating funnel
- 250 cm³ beaker
- 100 cm³ conical flask fitted with a stopper
- saturated sodium chloride solution
- anhydrous calcium chloride (or molecular sieves)
- teat pipette
- acidified potassium manganate(VII) solution (see below)
- sample container

The student sheet requires the use of semi-micro distillation apparatus. Students will need guidance in how to set this up. Students will also need guidance in the correct use of a separating funnel.

The acidified potassium manganate(VII) solution should be made by taking a solution of potassium manganate(VII) of the usual concentration in use in laboratories and then acidifying it with an equal volume of dilute sulfuric acid.

Centres are expected to carry out and be responsible for their own safety risk assessments.

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**Wear Eye
Protection**

Student Sheet

It is the responsibility of the student to carry out and be responsible for their own safety risk assessment before carrying out this experiment. Wear safety glasses at all times. Assume that all of the reagents and liquids are toxic, corrosive and flammable.

Experiment The dehydration of cyclohexanol to form cyclohexene

- Pour approximately 20 cm³ of cyclohexanol into a weighed 50 cm³ pear-shaped flask. Reweigh the flask and record the mass of cyclohexanol.
- Using a test pipette, carefully **and with frequent shaking**, add to the flask approximately 8 cm³ of concentrated phosphoric acid.
- Add a few anti-bumping granules to the flask and assemble the semi-micro distillation apparatus, so that the contents of the flask may be distilled. Heat the flask **GENTLY**, distilling over any liquid which boils below 100 °C.
- Pour the distillate into a separating funnel and add 50 cm³ of saturated sodium chloride solution. Shake the mixture and allow the two layers to separate.
- Carefully run off the lower layer into a beaker (for later disposal) and then transfer the upper layer, which contains the crude cyclohexene, into a small conical flask.
- Add a few lumps of anhydrous calcium chloride (or use molecular sieves, if available) to the crude cyclohexene to remove water. Stopper the flask, shake the contents and allow to stand until the liquid clears.
- Decant the liquid into a clean, dry, weighed sample container.
- Reweigh the container, calculate the mass of dry cyclohexene produced and determine the percentage yield of your product. You should assume that the whole of the dry distillate is cyclohexene.
- Test the distillate as described below, to confirm that it is an alkene.

A test on the product to confirm the formation of an alkene

- To approximately 1 cm³ of the distillate in a test tube, add an equal volume of acidified potassium manganate(VII) solution. Shake the contents of the test tube vigorously from side to side.
- Record your observations.



Teacher Notes and Marking Guidance

The specific marking guidance in the specification is as follows

2 marks: All areas of the task are carried out competently.

The apparatus set-up is safe and appropriate.

Heating is carried out with due care and only as long as necessary.

The yield of product is appropriate.

1 mark: One of the areas of the task is performed poorly.

The apparatus set-up is inappropriate **OR**

Heating is carried out with insufficient care or longer than necessary **OR**

The yield of product is inappropriate.

0 marks: At least two of the areas of the task are performed poorly.

The apparatus set-up is inappropriate.

Heating is carried out with insufficient care or longer than necessary.

The yield of product is inappropriate.

Guidance for Teachers and Students

Teachers are expected to exercise professional judgement in assessing the competence of their candidates in following the instructions.

Candidates should have been given guidance in the correct use of equipment and this guidance **can continue during the practical session** for which this PSA forms a part.

If, however, the guidance required is fundamental or frequent, then the student should **not** be awarded 2 marks.

Judgement of 2 marks, 1 mark or 0 marks will depend on whether the candidate has carried out the activity safely, particularly with regard to

- measuring out and transferring the cyclohexanol to the pear-shaped flask,
- measuring out and adding the concentrated phosphoric acid to the flask,
- heating the flask in a way which is safe and allows the collection of an appropriate volume of distillate.

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It is appropriate to consider whether the candidate has followed the instruction to collect distillate that boils up to 100 °C and whether this distillate actually contains any cyclohexene. The candidate should be judged on ability to carry out the distillation rather than on the quality of product; the purpose of this test is simply to confirm to the candidate and to the supervisor that cyclohexene has actually been made.

It is important to remember when marking these practical exercises that PSA is about student competence and that for a student to score full marks on this exercise **perfection is neither expected nor required.**