A-level Chemistry exemplar for required practical 5 – alternative a

Distillation of a product from a reaction:

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

Student sheet

Requirements

You are provided with the following:

- semi-micro distillation apparatus OR Quickfit apparatus fitted with a thermometer and collection vessel
- concentrated phosphoric acid
- cyclohexanol
- protective gloves
- stand and clamp
- micro-burner
- 10 cm^3 measuring cylinder
- 25 cm^3 measuring cylinder
- anti-bumping granules
- separating funnel
- 250 cm^3 beaker
- 100 cm^3 conical flask fitted with a stopper
- saturated sodium chloride solution
- anhydrous calcium chloride (or molecular sieves)
- plastic graduated dropping pipette
- acidified potassium manganate(VII) solution
- sample container
- access to a digital mass balance (reading to ±0.1 g).

Suggested method

The dehydration of cyclohexanol to form cyclohexene

This experiment must be carried out in a fume cupboard.

a) Pour about 20 cm^3 of cyclohexanol into a weighed 50 cm^3 pear-shaped flask. Reweigh the flask and record the mass of cyclohexanol.

b) Using a plastic graduated dropping pipette, carefully **and with frequent shaking**, add to the flask approximately 8 cm^3 of concentrated phosphoric acid.

c) 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.

d) Pour the distillate into a separating funnel and add 50 cm^3 of saturated sodium chloride solution. Shake the mixture and allow the two layers to separate.

e) 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.
f) Add a few lumps of anhydrous calcium chloride or anhydrous sodium sulfate(VI) or anhydrous magnesium sulfate (or use molecular sieves (4A), if available) to the crude cyclohexene to remove water. Stopper the flask, shake the contents and allow this to stand until the liquid becomes clear.
g) Decant the liquid into a clean, dry, weighed sample container.
h) 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.
i) Test the distillate as described below, to confirm that it contains an alkene.

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

a) To approximately 1 cm^3 of the distillate in a test tube, add an equal volume of acidified potassium manganate(VII) solution. Shake the contents of the test tube well.
b) Record your observations.

A-level Chemistry exemplar for required practical 5 – alternative b

Distillation of a product from a reaction:

To prepare ethanal by the oxidation of ethanol and to distil the ethanal from the reaction mixture.

Student sheet

Requirements

You are provided with the following:

- simple distillation apparatus OR Quickfit apparatus
- acidified sodium dichromate(VI)
- protective gloves
- stand and clamp
- 10 cm^3 measuring cylinder
- 25 cm^3 measuring cylinder
- anti-bumping granules
- test tube
- thermometer
- two 250 cm^3 beakers
- ethanol
- teat pipette
- 0.05 mol dm^{-3} silver nitrate solution
- 2 mol dm^{-3} dilute ammonia solution
- 2 mol dm^{-3} sodium hydroxide solution
- 1 mol dm^{-3} dilute sulfuric acid.

Suggested method

The oxidation of ethanol to ethanal

a) Using a 25 cm^3 measuring cylinder, carefully measure out 12 cm^3 of the solution of acidified sodium dichromate(VI). Pour this oxidising agent into a boiling tube. You should wear protective gloves when handling the corrosive oxidising agent.
b) Cool the boiling tube in cold water in a beaker.
c) Using a 10 cm^3 measuring cylinder, carefully measure out 2 cm^3 of ethanol.
d) Using a teat pipette, **slowly** add the 2 cm^3 of ethanol **dropwise**, to the oxidising agent in the **cooled boiling tube** (immersed in cold water in a beaker), shaking the tube gently to mix the contents.
e) After the addition of ethanol, add a few anti-bumping granules to the boiling tube and attach to it a bung fitted with a right-angled glass delivery tube.
f) Clamp the boiling tube at about 45° in a beaker of water. Heat this beaker of water gently and **slowly** distil off approximately 5 cm^3 of liquid distillate into a test tube **which is immersed in cold water in a beaker.** Keep the test tube cool to avoid loss of the volatile ethanal.
g) Carry out the test described below on the distillate to confirm that ethanal has been formed in this reaction.

Test on the distillate to confirm the formation of ethanal

Tollens' silver mirror test:

a) Prepare a sample of Tollens' reagent by adding 5 drops of sodium hydroxide solution to 2 cm^3 of silver nitrate solution in a test tube.
b) To this test tube add **just enough** dilute ammonia solution to dissolve the brown precipitate completely.
c) Using a beaker of hot water (50–60 °C), **gently warm** approximately 5 cm^3 of this test reagent in a test tube.
d) Add 10 drops of the distillate containing ethanal to the warmed Tollens' reagent in the test tube. Wait a few minutes and note what happens. You should have produced a silver mirror on the walls of the tube.

Make sure that you dispose of the Tollens' reagent thoroughly by rinsing it away with plenty of water and then rinsing any glassware that has contained the reagent with a little dilute sulfuric acid when you are finished.