THE PREPARATION OF '1-BROMOBUTANE

Introduction

1-bromobutane can be prepared by reacting butan-1-ol with hydrogen bromide. Unfortunately we cannot get hydrogen bromide 'off the shelf' like we can hydrogen chloride (as hydrochloric acid) and therefore we have to make it in the reaction mixture itself. This is called making it 'in situ'. The hydrogen bromide is made by reacting concentrated sulfuric acid with sodium bromide. The hydrogen bromide then reacts with the butan-1-ol.

You will need

Combostill set up for refluxing at first and then distillation
Collection apparatus
Thermometer
Propettes
Glass micro tubes
Microspatula
Small beaker
Sodium bromide
Concentrated sulfuric acid
Distilled water
Butan-1-ol
Anhydrous calcium chloride
Concentrated hydrochloric acid
Saturated solution of sodium hydrogen carbonate

PROCEDURE

1. Using your Propette, add 14 drops of distilled water to the reaction vessel.

2. Place 0.26 g of sodium bromide into the water and stir using your microspatula.

3. Now add 14 drops of butan-1-ol and mix thoroughly. Place the reaction vessel in a beaker of **cold water**.

4. **Slowly** add 14 drops of concentrated sulfuric acid to the mixture, mixing after each addition by gently shaking the vessel.

5. **Reflux** the mixture for at least 30 minutes. Make observations.

6. After this time, allow the mixture to cool and then rearrange the apparatus for distillation.

7. Set up your fraction collection apparatus so that the micro glass tube is about 1/3 full of water. This will make it easier to see the formation of the halogenoalkane.

8. As the mixture is heated, collect the oily droplets formed in the micro glass test tube. If any droplets settle onto the surface of the water, gently tap the test tube. Any 1-bromobutane will sink to the bottom.

Purification of the 1-bromobutane

1. Using a clean Propette remove the upper layer of water in the test tube.

2. Add 10 drops of concentrated hydrochloric acid to the lower remaining layer and mix thoroughly. This removes any unreacted butan-1-ol.

3. Once again, carefully remove the upper layer.

4. Add 10 drops of the sodium hydrogen carbonate solution and mix carefully.

5. When the effervescence has subsided, remove the upper layer.

6. Using the narrow end of the microspatula, add a few grains of anhydrous calcium chloride and leave overnight.

QUESTIONS ON THE PREPARATION

1. The reactants for the preparation of 1-bromobutane are represented in the equation below. Complete the equation

$HBr(aq) + CH_3CH_2CH_2CH_2OH\ (l) \rightarrow$

Now write the same equation using a) molecular formulae and b) skeletal formulae

a)

b)

2. One of the side reactions taking place is shown in the equation below:

$2H_2SO_4\ (aq) + 2NaBr \rightarrow 2H_2O\ (l) + SO_2\ (g) + Na_2SO_4 + Br_2\ (l)$

or ionically:

$4H^+\ (aq) + SO_4^{2-}\ (aq) + 2Br^-(aq) \rightarrow 2H_2O + SO_2\ (g) + Br_2\ (l)$

Explain why the reaction mixture turned reddy-brown in colour during the preparation

What is meant by the term reflux?

Explain why reflux is required in this preparation

3. The densities of butan-1-ol and 1-bromobutane are 0.81 gcm^{-3} and 1.28 gcm^{-3} respectively. Explain why the upper layer in the test tube is discarded and the lower layer retained.

4. Why is the sodium hydrogen carbonate added to the product. Give an equation to illustrate your answer

EQUATION:

5. Sodium carbonate solution would do the same job as the sodium hydrogen carbonate but its solution is more alkaline.
If it is alkaline, what ion (apart from Na^+ and CO_3^{2-}) must be present in the sodium carbonate solution?

6. Explain (using a diagram/reaction mechanism) why sodium carbonate solution is not used to purify the 1-bromobutane

7. What is the purpose of adding the anhydrous calcium chloride to the product?