

Preparation of 1-bromobutane from 1-butanol by $\text{S}_{\text{N}}2$ reaction assignment



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by SN2 reaction Objective: 1. To study the preparation of 1-bromobutane from 1-butanol by an SN2 reaction 2. To study the method of purification of an organic compound by simple extraction 3. To study the test of

identification of alkyl halide Chemicals and Apparatus: ~18.0 g 1-butanol, ~20. g sodium bromide, 15 ml of concentrated sulfuric acid, anhydrous magnesium sulfate, ~10 ml of 5% aqueous sodium bicarbonate, ~1 ml of sodium iodide ??? acetone reagent, 1 ml of bromine in chloroform, 1 piece of 100 cm³ round-bottomed flask, 1 piece of 50 cm³ of beaker, 1 piece of 50 cm³ of conical flask, 1 piece of 250 cm³ of separating funnel, 1 piece of 10 cm³ of measuring cylinder, 1 piece of electronic hot plate, oil bath, several pieces of anti-bumping granules, ice, filter paper and filter funnel.

Background: Nucleophilic substitution is an important class of organic reaction.

In the experiment, 1-bromobutane was synthesized through the second order mechanism in the presence of concentrated sulphuric acid and sodium bromide. The nucleophile in the experiment is bromide ion (Br⁻) while the leaving group is water. [pic] Purification of the product was then obtained by extraction. The principle of extraction as a purification method was based on the difference in solubility between impurities and product. Identification of the product (1-bromobutane) can be confirmed by carrying out sodium iodide in acetone test. The test involves displacement of bromide by iodide.

Since iodide is a strong nucleophile which can displace the bromide ion from attached carbon. The reaction was carried out in acetone since acetone

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dissolved NaI, but not the products NaBr. So the appearance of precipitate and the time it takes to form is the basis for a qualitative test for alkyl halides. Procedures: The experiment was divided into three parts and worked in pairs. A) Preparation of 1-bromobutane from 1-butanol 1. 18.0 mL of 1-butanol was weighed into a 50 cm³ beaker. The exact amount of 1-butanol used was being marked. 2. The 1-butanol was transferred into a 100 cm³ round-bottomed flask. 3.

The beaker was rinsed with 15 mL deionized water and the rinse was put into the round-bottomed flask. 4. 20 gram of sodium bromide was added into the round-bottomed flask. 5. The mixture was cooled in a ice water bath. 6. To the flask in the ice bath, 15 mL of concentrated sulphuric acid was added gently. The flask was swirled to mix the content and a few anti-bumping granules were added. 7. The flask was fitted with the apparatus for reflux on a hot plat with an oil bath; a condenser was placed with tubing connected on top of the round-bottomed flask as shown in figure 1 of the laboratory menu and the join was wrapped with grease. . Checked that water was flowing through the condenser, and then the mixture was heated to reflux. 9. The mixture was allowed to reflux for 45 minutes, during which time mark the observation of the reaction mixture. 10. After the reflux period, the heat was removed and the mixture was allowed to cool at room temperature. B)

Purification of 1-bromobutane by extraction 1. The cool mixture was decanted into a 250-mL separatory funnel. 2. The round-bottomed flask was rinsed with small amount of water and the rinse was put into the separatory funnel. 3. The two layers were allowed to separate and the aqueous later was drained off. . The organic later was washed in the separatory funnel

successively with 10 mL water, 10 mL of 5 % aqueous sodium bicarbonate and 10 mL of water. 5. The funnel was shaken well in each case. The gas pressure was released regularly during the shaking. 6. The aqueous layer was discarded down the drain. 7. The organic later was collected into a 50 mL conical flask. 8. To the organic later, about 1 gram of anhydrous magnesium sulphate was added. The conical flask was swirled thoroughly. 9. The organic layer was filtered using the filter paper and funnel to remove the used magnesium sulphate. 0. The dry organic extract (1-bromobutane) was collected in a pre-weighed beaker and the exact mass of product was obtained. C) Test for the product i) Sodium iodide in acetone test 1. 5 drops of the product (1-bromobutane) was placed to a test tube 2. A few drops of sodium iodide-acetone reagent were added to the test tube 3. The mixture was shaken and let it stand for 3 minutes. 4. Changes were observed. ii) Bromine test 1. 5 drops of the product (1-bromobutane) was placed to a test tube 2. A few drops of bromine-chloroform reagent were added to the test tube. . The mixture was shaken and let it stand for 3 minutes. 4. Changes were observed. Results: Preparation of 1-bromobutane from 1-butanol Mass of 1-butanol used = 18. 0 g Mass of 1-bromobutane (dried) = 9. 207 g Percentage yield [pic] Observation during the course of reaction: two immiscible liquid formed, the one on the top is yellow colour, and the one on the bottom is colourless. Purification of 1-bromobutane by extraction Observation during the exaction: Two colourless layers formed, the yellow layer was decolourized, and the organic layer has glue-like smell.

Test for the product i) Sodium iodide in acetone test Observation from the test: A small amount of white precipitate formed. ii) Bromine test

Observation from the test: Negative result, no observable change.

Discussion: 1. The purpose of this experiment was to prepare 1-bromobutane by nucleophilic substitution of 1-butanol. The functional group inter-conversion of an alcohol into alkyl halide takes place by a nucleophilic substitution reaction. In the experiment, the alkyl group was substituted on the primary carbon, the reaction proceeds by an SN2 mechanism.

The primary alcohol, 1-butanol, was reacted with a mixture of sodium bromide and the concentrated sulfuric acid and hooked up to a water-cooled reflux condenser. The hydroxyl group of the 1-butanol was protonated by the sulphuric acid. Addition of the strong acid formed an oxonium ion from the 1?? alcohol OH group, changing a poor leaving group (-OH) into a good one (H-O-H). The nucleophile, bromide, attacked the primary carbon and water was released, forming 1-bromobutane. The water that produced was then protonated, forming hydronium ion. [pic] 1-butanol sulfuric acid protonated alcohol pic] 2. When sulphuric acid was added into the reaction mixture, cooling by means of an ice bath was needed. The apparatus must also be swirled at the same time to ensure the acid had reacted. This was to prevent the sulphuric acid from reacting too fast, as it was an exothermic reaction, and might run away splashing the acid everywhere if the reaction went too fast. Also the increased temperature might vaporise more quickly the 1-butanol, which would decrease the yield. Also, hot sulphuric acid would cause significant oxidation of the sodium bromide to bromine, which is useless in this experiment.

The yield of 1-bromobutane could therefore be affected adversely. 3. The oil spreads the heating uniformly over the base of the flask. This reduced the <https://assignbuster.com/preparation-of-1-bromobutane-from-1-butanol-by-sn2-reaction-assignment/>

likelihood of cracking, and of unwanted side reactions occurring (e. g. excessive oxidation either of bromide ions to bromine or of the alcohol to carbon). 4. Apparatus was heated under reflux; it took for approximately 45 minutes to allow the reaction to take place. The anti-bumping granules were added to stop any bubbles from forming and splashing the reactants everywhere.

The need for reflux was because the reaction needed to go to completion, and as with organic liquids it was often necessary to reflux because heat needed to be applied to the reaction without losing reactants through evaporation. The experiment was needed to work in hood because it could absorb any acid fumes (e. g. HBr) or sulphur dioxide gas that might be given off. As the reaction progressed, the liquid became less opaque and a trace of yellow colour was seen. This would take some time because the strong covalent bonds in organic compounds needed to be broken before the reaction could take place.

The reactant also separated into two distinct layers which was not so obvious before. The reason for the multiple layers of products was that there were, several side reactions taking place at the same time in the flask, they are the drawbacks to using sulphuric acid. The acid could cause reactions such as the dehydration of alcohols and/or ether formation to occur which lower the yields of the alkyl bromides. Thus, the probable by-products are 1-butene, dibutyl ether ($C_4H_9OC_4H_9$), and the starting alcohol. (I) Sodium bromide reacted with sulphuric acid to form hydrogen bromide and sodium hydrogen sulphate.

$\text{NaBr} + \text{H}_2\text{SO}_4 \rightarrow \text{HBr} + \text{NaHSO}_4$ (II) Hydrogen bromide was oxidised to bromine molecules as concentrated sulphuric acid was a very good oxidising agent. The sulphuric acid reacted to form sulphur dioxide gas. $\text{HBr} + \text{H}_2\text{SO}_4 \rightarrow \text{SO}_4\text{Br}_2 + 2\text{SO}_2$ (g) (III) Hydrogen bromide dissociated and the bromide ion from it attacked the carbon atom with the -OH function group in 1-butanol and displaced the -OH function group forming a bromo function group and a hydroxide ion, which then associated itself with another H_3O^+ ion to form water. $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{Br}^- \rightarrow \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{OH}^-$ (IV) A molecule of sulphuric acid attacked the lone pair on an -OH function group, releasing a molecule of water, and a mixture of butoxybutane and but-1-ene was formed, along with the regenerated sulphuric acid. $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{H}_2\text{SO}_4 \rightarrow \text{CH}_3\text{CH}_2\text{CH}=\text{CH}_2 + \text{H}_2\text{O} + \text{H}_2\text{SO}_4$ 2 $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{H}_2\text{SO}_4 \rightarrow \text{CH}_3(\text{CH}_2)_3\text{O}(\text{CH}_2)_3\text{CH}_3 + \text{H}_2\text{O} + \text{H}_2\text{SO}_4$ [pic] 6. To identify the organic layer in the separatory funnel, carefully draw off about 0.25 mL of the lower layer into a test tube containing about 1 mL of water and mix vigorously. If the liquid from the funnel dissolves in the water as you mix it, the lower layer is aqueous.

If two immiscible layers form in the test tube, the lower layer in the funnel is organic (in this case return the contents of the test tube to the funnel). 7. In the experiment, especially the extraction process, it was a good practice to save all layers until the product was surely in hand. 8. In the part C of the experiment, sodium iodide in acetone provides reaction conditions favourable to $\text{S}_{\text{N}}2$ reactions. The test tubes used must be totally dry because the anhydrous acetone reaction made the reaction forward, from alkyl

bromide to alkyl iodide. Sodium iodide was soluble in this solvent, whereas sodium chloride and sodium bromide were not.

When the reaction occurred, a precipitate of sodium bromide would form.

[pic] Conclusion: 1-butanol went through an SN2 reaction by using sodium bromide in water to create hydrobromic acid in the presence of excess sulfuric acid. Thus, water was the leaving group and bromide ion was the nucleophile in this in situ reaction. This displaced the alcohol's -OH group with a bromine creating 1-bromobutane. The excess sulfuric acid pushed the reaction far to the right corresponding with LeChatelier's principle which states that more reactants yield more products.

The extractions further isolated the product, the substance which has higher density was always the bottom layer and the lighter was on top. It turned out that the organic layer was always on the bottom of each extraction.

Anhydrous magnesium sulphate was a very good drying solution. This allowed it to soak up the remaining water to a great extent. The amount collected was 9.207 gram of 1-bromobutane. The percent yield of 1-bromobutane obtained was 31%. The sodium iodide/acetone classification tests proved positive.

This reaction must be a SN2 reaction as the product was almost entirely 1-bromobutane, if by an SN1 mechanism, the product should consist of mostly 2-bromobutane derived from the sec-butyl cation formed via a 1,2-hydride shift. [pic] Such an interpretation of the product distribution must be made with caution. The presence of small amounts of 2-bromobutane in the reaction product did not necessarily mean that it was produced from 1-

butanol via an SN1 mechanism because an alternative sequence of an E2 elimination of the protonated 1-butanol to give 1-butene followed by HBr addition to the 1-butene would also produce 2-bromobutane. pic] [pic]

Hazard and precaution: Precautions were taken to prevent the highly inflammable and volatile alcohol from catching fire or lost through evaporation. The pressure of the separatory funnel must be periodically released to avoid the stopper being pushed out and product being lost and sprayed. The pressure is due to liberated carbon dioxide. All substances must be handled in the hood, wearing gloves and eye protection. Avoid contact with eyes and inhalation.

Skin contact procedures for these substances: Wash affected area with plenty of soap and water. Bromobutane is a lachrymator. Avoid breathing vapors and skin contact. Wear gloves when handling sulfuric acid or pour very carefully! Be wary of drips on the outside of containers. It is too viscous for a Pasteur pipet, so pour a small amount into a small beaker. A graduated cylinder wasn't necessary since it's used in excess Eye contact with sulphuric acid required immediate attention. Wash with plenty of waster and see a physician as soon as possible.