Radical halogenation and gas chromatography essay sample

Science, Chemistry

In radical halogenations lab 1-chlorobutane and 5% sodium hypochlorite solution was mixed in a vial and put through tests to give a product that can then be analyzed using gas chromatography. This experiment was performed to show how a radical hydrogenation reaction works with alkanes. Four isomers were attained and then relative reactivity rate was calculated. 1, 1-dichlorobutane had 2. 5% per Hydrogen; 1, 2-dichlorobutane had 10%; 1, 3-dichlorobutane had 23%; and 1, 4-dichlorobutane had 9. 34% per Hydrogen. Introduction

Alkanes are relatively unreactive. There are only a few types of reactions commonly performed. In this lab, halogenation was performed. In the methane molecule, the carbon-hydrogen bonds are low-polarity covalent bonds. The halogen molecule has a nonpolar covalent bond. UV light contains sufficient energy to break the weaker nonpolar chlorine-chlorine bond, but it has insufficient energy to break the stronger carbon-hydrogen bond. The fracture of the chlorine molecule leads to the formation of two highly reactive chlorine free radicals (chlorine atoms). A free radical is an atom or group that has a single unshared electron.

This initial reaction is called the initiation step of the mechanism. Once the high-energy chlorine free radicals are formed, the energy source (UV light or heat) can be removed. The energy liberated in the reaction of the free radicals with other atoms is sufficient to keep the reaction running. When a chlorine free radical approaches a methane molecule, the chlorine free radical combines with the liberated hydrogen free radical to form hydrogen chloride and a methyl free radical. This is called a propagation step, a step in which both a product and a reactive species, which keeps the reaction going, are formed. When a reaction occurs between free radicals, a product forms, but no new free radicals are formed. This type of reaction is called a termination step because it tends to end the reaction. Experimental Methods

In the hood add 1. 0 mL of 1-chlorobutane, 1. 0 mL of 5% sodium hypochloritesolution, and 0. 5 mL of 3 M hydrochloric acid in 5. 0 mL conical vial. Immediately close the vial with the screw cap. Shake the vial for 30 seconds, or until the yellow chlorine color goes from aqueous to organic layer.

Use unfrosted light bulb to irradiate the vial and its contents for about 30 minutes at about 6 cm distance. Don’t forget to shake the vial often. After irradiation add 100 mg of anhydrous sodium carbonate in several portions. Make sure the foaming stops before continuing addition. After the addition is finished wait until foaming ceases, close the vial and shake intensely. Prepare a Pasteur filter pipette. Remove the lower aqueous layer with the pipette. The top layer should be organic. If in doubt, add water to the layers and whichever layer the water goes to means that layer is aqueous. Then remove the top layer into a 5 ml flask. Add 100 mg of anhydrous calcium chloride to organic layer and allow the solution to dry for 10 minutes. Prepare another filter pipette and use it to transfer the dried solution to sample vial to be tested.

Take the product to the gas chromatograph (GC). Make sure the GC column should be approximately 90-100˚C. Inject 1-µl sample. The first peak will represent the leftover starting material. The last four peaks are the isomers. Upon finishing, dispose of the product mixture in the waste container.

Results

ProductRetentionArea% of ProductsRel. Reactac. RateType of Hydrogen 1, 1-dichlorobutane4. 03. 55%2. 5% per HSecondary (2̊)

1, 2-dichlorobutane5. 813. 420%10% per HSecondary (2̊)

1, 3-dichlorobutane6. 631. 046%23% per HSecondary (2̊)

1, 4-dichlorobutane8. 618. 928%9. 34% per HPrimary (1̊)¬¬¬¬

*Total Product Area= 66. 8 Finding % Product Area

(Area of product/Total Area Product)= % Product Area

Area = 3. 5 (3. 5/66. 8)x100= 5%

Total Product= 66. 8

Relative Reactivity Rate

% Area/ H’s on that carbon = Relative Reactivity

% Area= 5% 5%/2= 2. 5% per H

Hydrogens = 2

Discussion

The results of the GC outcome are not satisfactory. Only three peaks were identified from the data and four were supposed to appear; 1, 4-dichlorobutane was missing on the data. Reading from the handout that was posted online with an example GC run, the fourth peak is not on the graph because the GC was cut off too soon. In the example, the last peak came up around retention time 8. 5. According to this data, GC was cut off around retention time 8. 0. Another issue is that the second peak, 1, 2-dichlorobutane to be precise, is very little. That is it is not even registered in the data. Due to not having the full data, the example GC run is used for this lab report.

1, 1-dichlorobutane has 2 – 2̊ H’s. It is expected from mathematical stand point that 16. 7% of the product will be it. In reality that was not what happened. After the experiment, according to GC data, 1, 1-dichlorobutane had 5% of the product. That is a pretty big difference; it is a lot smaller than expected. It is the first product to show up on the graph, as expected. According to the handout, a major boiling point of 1, 1 is at 114̊. The next three are at higher boiling points, and that is how it is known which peak is which product. The relative reactivity rate is 2. 5% per Hydrogen. Since there is only two Hydrogens and only 5% for this product, each Hydrogen gets 2. 5% chance to be replaced by halogen.

The next product is 1, 2-dichlorobutane which also has 2 – 2̊ H’s. According to statistical factors, 16. 7% of product should appear in the results. According to the GC results, 20% of the product is produced. It is higher than what statistics show. The relative reactivity rate is 10%.

1, 3-dichlorobutane has 2 – 2̊ H’s. Its product production relative to other three is also 16. 7%. According to the GC results 46% of the products are 1, 3. It is almost half of the products. That is a much bigger product than expected. Its relative reactivity rate is 23% for each hydrogen.

The last but not least product is 1, 4-dichlorobutane, 28% of the products. It has 3 – 1̊ H’s. According to mathematical calculations, 25% of the products should be 1, 4. This is pretty close to what it should be. Relative reactivity rate is 9. 34%, because it is divided over three hydrogens.

The results do not co-exist with the mathematical calculations prior to the experiment. This is an interesting situation, is there a problem performing the lab experiment? Everything that was done was done following the handout precisely. One area that requires more precise work is at the end when layers are filtrated. It is easily to contaminate the solution and that could have been the key. Conclusions

In radical halogenations lab 1-chlorobutane and 5% sodium hypochlorite solution was mixed in a vial and put through tests to give a product that can then be analyzed using gas chromatography. This experiment was performed to show how a radical hydrogenation reaction works with alkanes. Four isomers were attained and then relative reactivity rate was calculated. 1, 1-dichlorobutane had 2. 5% per Hydrogen; 1, 2-dichlorobutane had 10%; 1, 3-dichlorobutane had 23%; and 1, 4-dichlorobutane had 9. 34% per Hydrogen.

Question

No they did not give equal amounts of chlorination. The farther 2̊ has better chance of chlorinating the molecule than the closer one. This happens because it is more stable. It does not have to fight the other Cl by repelling each other because they are further away.