The resolution of phenylethylamine biology essay

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Enantiomers are isomers that are non-superimposable mirror images of each other and contain an asymmetric carbon atom and hence are chiral compounds. They have identical physical and chemical properties such as boiling and melting point, solubilities, and reactivity with symmetric reagents. They only differ in the direction of rotation of plane of polarized light, which is called optical activity. 1 The direction and degree of rotation of the plane depends on the nature of the substance. Though, the magnitude of direction of rotation by the enantiomers is the same, but the direction is opposite of each other. Clockwise rotation is referred to as dextrorotatory (+) and counter clockwise rotation is referred to as levorotatory (-). 2 A polarimeter is a commonly used instrument that is used to measure the optical activity of a sample. A sample's purity is determined by the comparison of observed rotation to accepted rotation of the sample. 3

The purpose of this experiment was to resolve (±)-α-phenylethylamine, which is dissymmetric, by reacting it with (+)-tartaric acid to separate the compound's diasteriomeric derivatives. (±)-α-phenylethylamine is a racemic mixture and contains both enantiomers in equal quantities. The diastereomers that are produced due to the resolution are salts that are either (+)(+) or (+)(-) as the acid is (+) and the enantiomers are either (+) or (-). These vary in their physical traits and hence can easily be separated via fractional crystallization. The hydrogen tartrate in (+)(-) form is less soluble in methanol than (+)(+) salt and hence, it crystallizes in pure stereoisomeric form. In the end, the (-)-α-phenylethylamine enantiomer was separated in the solution after treatment with strong base. 1

2NaOH

+ 2H2O

(+)-tartaric acid

(-)-amine

(+)-amine

Figure 11: The reaction of the racemic mixture of (±)-α-phenylethylamine with (+)-tartaric acid produced two salts. After the solution was treated with excess NaOH, only the (-) enantiomer remained.

Experimental:

The experimental procedure carried out for this lab followed the steps listed in the lab manual. Refer to Organic Chemistry Lab Manual Fall 2010 – Winter 2011 pages 11-17.

The only alteration to the procedure was that, instead of extracting the aqueous phase twice more with 5mL of CH2Cl2 after the initial extraction, the aqueous phase was extracted once more with 10mL of CH2Cl2.

Results:

Table1: Shows the mass of each compound in reaction and the corresponding number of moles. Since (±)-α-Phenylethylamine has lower amount of moles than tartaric acid, it was the limiting reactant of this reaction. Refer to the appendix for calculation of the number of moles.

Product	Amount (g)	Molar mass (g/mol)	Number of Moles
Tartaric Acid	12. 0	150. 09	0. 0799
(±)-α-Phenylethylamine	9. 4	121. 18	0. 0776
(-)-α-phenylethylamine-(+)-hydrogen tartrate	10. 5	271. 27	0. 0388

Table2: Shows the obtained products from the experiment and their observed masses against the theoretical masses. Percent yield is calculated

to show how well the experimental process was carried out. Refer to the appendix for calculations.

Product

Observed Mass (g)

Theoretical Mass (g)

% Yield

(-)-α-phenylethylamine-(+)-hydrogen tartrate

6. 13

10. 50

58. 4%

(-)-α-phenylethylamine

Beaker: 29. 68

Product:

2. 82

4. 70

60. 0%

Beaker + product: 32. 50

Table3: Shows the observed rotation of (-)-α-phenylethylamine measured via PerkinElmer instrument at optical rotation at 589nm. Refer to the appendix for calculations.

Observed rotation

Calculated specific rotation of (-)-α-phenylethylamine

Reported specific rotation of (-)-α-phenylethylamine

Optical purity

-31. 90

-33. 94

-40. 40

84%

Qualitative observations:

(-)-amine-(+)-hydrogen tartrate looked white in color and was hard in nature (crystals)

The solution in the seperatory funnel had two distinct layers. The top layer was white and opaque while the bottom layer was relatively clear.

Discussion:

In the experiment, the addition of (+)-tartaric acid to (±)-α-phenylethylamine formed (+)-amine-(+)-hydrogen tartrate, and (-)-amine-(+)-hydrogen tartrate salts. These salts were separated as they had varying solubilities in methanol. The (-)-amine-(+)-hydrogen tartrate was able to be crystallized as it was less soluble. 1 The separation of salt was fairly successful as the

percentage yield of (-)-amine-(+)-hydrogen tartrate, a (-)(+) salt, was 65. 2%. There could have been many experimental errors that led to only a satisfactory percentage yield of the salt. A major factor of error could have been the fact that not all of the (-)(+) salt was crystallized. Also, even though seed crystals were added to the mixture so that only clear prisms are formed, some fine white needles might have also formed, disrupting the pure nature of amine. Additionally, despite all possible attempts to scrape off every bit of the crystallized salt, there was a minute amount that remained stuck to the Buchner funnel and Erlenmeyer flask. All of which directly correlated to a lower percentage yield of the (-)(+) salt.

In the second part of the experiment, Addition of 2M NaOH strong base to the (-)(+) salt lead to abstraction of a proton from (-)-amine to produce water, sodium tartrate, and (-)-α-phenylethylamine. The separatory funnel showed formation of two distinct layers. The top layer was white and opaque, while the bottom layer was relatively very clear. In order to determine which layer was organic and which was inorganic, a bit of water was run through the funnel. The water did not travel further than the top layer and as a result, it was determined that the top layer was inorganic whereas the bottom layer was organic. That was a result of varying densities of the layers, the top being less dense. 4 The sodium tartrate dissolved in water but (-)-α-phenylethylamine is insoluble in water and required addition of methylene chloride, CH2Cl2, to dissolve it. Methylene chloride was the choice of organic solvent as it has a low boiling point of 40°C and (-)-α-phenylethylamine has a much higher boiling point of about 188°C. 5, 6 Thus, methylene chloride could be easily removed from the solution through

boiling without having any of the (-)-α-phenylethylamine evaporate. Before boiling, one gram of anhydrous K2CO3 was added to remove the water and the sodium tartrate dissolved in the water.

After the extracting couple times, and removing the K2CO3 through filtration, the percentage yield of the (-)-α-phenylethylamine was determined again. Though, this time the yield was of 60. 0%. The decrease in yield indicated that there was a loss of (-)-α-phenylethylamine in the part B of the experiment as initially there was a 65. 2% yield. One of the sources of errors for this could have been excess disposal of the bottom amine layer from the funnel as it could have contained some of the (-)-α-phenylethylamine. That could not have been controlled since it was hard to distinguish a clear boundary between the two layers as there was a slight gradient instead. Additionally, when the funnel was shaken, a small amount of solution poured out of the glass stopper. This could have critically lowered the percentage yield. Furthermore, some of the (-)-α-phenylethylamine might not have made it through the filtration caused by the cotton.

The observed rotation was -31. 90° (the negative sign indicating a counterclockwise rotation) which calculated to a specific rotation of -33. 94°. Since the theoretical specific rotation is -40. 4±0. 2o, the optical purity of the sample was determined to be 84. 0%. 2 This is a fairly high yield. This means that the experiment was carried out fairly properly. Also, the negative (levorotory rotation) showed that it was the (-)-α-phenylethylamine enantiomer that was isolated. Some errors could have still been associated with the experiment. The slight deviation in the optical purity might have been a result of presence of impurities, such as mixed compounds from the

two layers in the funnel. Also, when the solution was boiled in order to remove methylene chloride, some of it might not have evaporated even if the observer did not see any major visible boiling. Hence, some of the methylene chloride might have been left in the solution, and so the optical purity could have been a result of amine and methylene chloride instead of just amine. Furthermore, there could have been fingerprints or air bubbles in the sample cell while obtaining the optical rotation reading.

Questions:

Only dissymmetrical acids, which are chiral structures and have enantiomers, can be used for the resolution of an amine as the resolution is expected to produce enantiomers of that specific amine. 7 Since glutaric, glyoxylic, and gluataconic acids are not chiral structures, they are not useful in the resolution of amine. The dissymmetrical acids include glutamic, glucaric, and gluconic acids and these can be used to resolute an amine. The meso-tartaric acid is optically inactive as it is not chiral even though it contains chiral centers. Hence, meso-tartaric acid would not contain enantiomers and as a result, it would not be able to resolute an amine.

Glyoxylic Acid8 Glutamic Acid9

(achiral structure) (Dissymmetrical)

Glucaric Acid10 Glutaric Acid11

(Dissymmetrical) (Symmetrical; achiral structure)

Gluconic Acid12 Glutaconic Acid13

(Dissymmetrical) (achiral structure)

The value presented by the student is incorrect. The values the student must present should be exactly ones that he obtained and not changed around. In this case, since the student changed the value to being positive, it falsely gives an indication that the rotation of light was drextrorotatory instead of the actual levorotatory rotation. 2 The student should have reported the value as -239. 7°±1. 3°. The true uncertainty is obtained through the standard deviation of a data set.

Mean = [(-238. 5°) + (-241. 0°) + (-239. 7°)] / 3 = -239. 7°

Standard Deviation: = (((-238. 5°-239. 7°)2)+((-241. 0°-239. 7°)2)+((-239. 7°-239. 7°)2)) / 2 = 1. 3

a) If the amine had been already partly resolved to 75% optical purity then:

d + l = 100%

d – l = 75%

2d = 175% à d = 87. 5% ; l = 12. 5%

87. 5% – 12. 5% = 75%

It can be inferred that the maximum yield of optically pure amine in this experiment would then be 75%.

b) If the tartaric acid used with 75% optically pure amine was itself only 95% pure than the maximum optical purity of the amine sample would be 86. 6%.

d + l = 100%

d – l = 95%

2d = 195% à d = 97. 5%; l = 2. 5%

Since the 97. 5% refers only to when 95% pure tartaric acid is concerned, it has to be multiplied by 75% in order to also take into account the optically pure amine.

d + l = 100%

d – l = 73. 1%

2d = 173. 1% à d = 86. 6%; l = 13. 45%

It can be inferred that the maximum yield of optically pure amine in this experiment would then be 86. 6%.