

Pure enantiomers of phenylethylamine mixture



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Introduction

The purpose of this laboratory was to resolve the pure enantiomers of (\pm)- α -phenylethylamine (racemic) mixture, by separating their diastereomeric derivatives using (+)-tartaric acid. The differing enantiomers form different salts with acids. Two molecules that are enantiomers have nearly identical physical and chemical properties although this may be true, the salts that are formed after the reaction with acid have distinct properties. Some salts are less soluble [(+)(-)] than others, and therefore crystallize from the mixture in a nearly pure stereoisomeric form. When using NaOH as a strong base to treat the salt, it allows for the isolation of the enantiomer (Lab Manual, 2007). Polarimetry is a common method used to distinguish between enantiomers, based on their ability to rotate the plane of polarized light in opposite directions (+ and -). This allows the observer to determine the enantiomeric purity, and therefore the composition of the mixture (Wade, 2007)

Chemical Reaction:

(-)-amine (+)-amine less soluble salt [(+)(-)]:
crystallizes more soluble salt [(+)(+)] remains in solution

2NaOH

+ 2H₂O

(-)- α -phenylethylamine (Lab Manual, 2007)

Procedure:

Instead of using a 50 mL beaker to boil the amine solution in, we used a 50 mL Erlenmyer flask

For the rest of the procedure refer to pg. 18, 22-24 (Lab Manual, 2007)

Observations:

The crystals were given a 4 week crystallization period and afterward, the (-)- α -phenylethylamine- (+)-hydrogen tartrate salt was observed to be a white crystalline solid, and the methanol was a transparent liquid. Two very distinct layers were visible following the reaction with the NaOH (strong base) and addition of the methylene chloride (CH_2Cl_2). The top layer was translucent in some places and opaque in others, very cloudy, white liquid, while the bottom layer was transparent and also liquid. The resultant mixture following the three separate extractions was close to transparent

Discussion:

When the (+)-tartaric acid was added to the racemic mixture, (\pm)- α -phenylethylamine, (-)-amine-(+)-hydrogen tartrate, and (+)-amine-(+)-hydrogen tartrate salts were formed. The (-)-amine-(+)-hydrogen tartrate was much less soluble in methanol, and therefore crystallized out of the solution (Lab Manual, 2007). This method of separation was proven to be quite successful, as the percent yield of this crystallization was 73.1%, which is relatively high. The presence of impurities, as well as the inability to completely crystallize the salt from methanol most likely attributed to any discrepancies. It is also possible that although the (-)(+) salt is less soluble than the other salts, it still has some sort of solubility, and therefore crystallizes rather slowly (hence the mandatory 2 week waiting period, in our <https://assignbuster.com/pure-enantiomers-of-phenylethylamine-mixture/>

case it was 4 weeks). As well, the other salts, despite their high solubility in methanol, may have still crystallized very slightly over the long waiting period, adding to impurities

Addition of NaOH resulted in the formation of two distinct layers: a white, cloudy aqueous layer (top), and a clear amine layer (bottom), and allowed for the isolation of (-)- α -phenylethylamine (Lab Manual, 2007). The addition of 5 mL of water to the flask confirm that the top layer was the aqueous layer, since it increased relative to the bottom layer and the water was absorbed here (Lab Manual, 2007). The aqueous layer consisted of the (-)-amine, along with sodium tartrate, and water, while the amine layer included any impurities. The sodium tartrate readily dissolved in water, while methylene chloride (CH_2Cl_2) was added to dissolve (-)- α -phenylethylamine (boiling point $\sim 186^\circ\text{C}$), since it had a lower boiling point (40°C), and could easily be removed through heating (Synthesis and resolution of α -phenylethylamine).

After a filtration process, including a series of extractions, there was percentage yield of 61.3% for the (-)- α -phenylethylamine, which is a lower yield than the original 73.1%, indicating that there was a loss of amine during the second part of the experimental procedure. The main cause of this error was the accidental disposal of much of the amine layer, in which a small amount of (-)- α -phenylethylamine was still present. The presence of some impurities may have also affected results, however, they would have instead increased the yield and lead to misleading results. Another possible cause of error is the slight leakage out of the glass stopper on the separatory funnel when the solution was shaken. There was a bit of solution that leaked

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out the bottom or squirted out the top when releasing the pressure in the funnel. Subsequently, the error that substantially lowered the yield of the product greatly increases the optical purity of the mixture. The observed rotation of the final sample was -31.8° (levorotary, left hand rotation) and the specific rotation was -33.8° compared with the empirical specific rotation of $-40.4^\circ \pm 0.2^\circ$ (Lab Manual, 2007). The resultant optical purity was 83.7%, which is considerably high. Aside from the previously mentioned disposal of the organic layer, numerous other errors, such as the presence of impurities may have contributed to deviations in the optical purity. The negative (counter clockwise) rotation essentially confirmed that the enantiomer being isolated was the (-)- α -phenylethylamine, and the high optical purity demonstrated that the extraction was accomplished with much success and considerable accuracy, since the final product was mainly (-)-amine, despite the relatively low yield.