

# [The synthesis and optical resolution of co(en)33+ isomers essay sample](https://assignbuster.com/the-synthesis-and-optical-resolution-of-coen33-isomers-essay-sample/)

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The tris(ethylenediamine)cobalt ion is an optically active compound. The enantiomers can be separated and disguised from each. This is done first through the synthesis of Cl and then the debased from this complex. The separation results with I3 and (-)-Co(en)3]I3. The two are disguised from each other by using a polarimeter and obtaining the optical angle of rotation. From this further calculations are done to further identify and determine if the enantiomers were separated and isolated synthetically. The results found that the isomers were separated and a great deal was recovered according to the theoretical yield that was calculated. It was also proven that the complex can be racemized by boiling and the presence of an activated carbon.

Introduction:

The purpose of this experiment is to synthesis and learn about Co(en)33+ optical isomers. The tris(ethylenediamine)cobalt ion is an optically active compound. Werner in 1912 was the first to do the resolution of tris(ethylenediamine)cobalt ion. 1 He did this through Second-order Asymmetric Induction. Second-order Asymmetric Induction is when one of the enantiomers can react with a chiral ion. This changes the solubility properties of the one enantiomer making it more soluble then the other.

The other enantiomer then crystallizes out first, making it easy to filter one from the other.(2, 3) The Cl is first synthesized and then is converted to I3 H2O by reaction with NaI. The other optical isomer, I3, is obtained by adding NaI to the filtrate from which CI 5H2O was previously precipitated. The solid that precipitates with NaI is a mixture of crystals of the racemate, (+) and (-)-I3 H2O, and crystals of pure I3 H2O.

The I3 H2O is much more soluble in warm water than the racemate and may be extracted into solution, which on cooling precipitates the desired enantiomer, I3 H2O. The optical purities of the isolated (+) and (-) enantiomers are to be evaluated by measuring their specific rotations from a polarimeter. Finally, it will be shown that the resolved compound may be racemized by boiling an aqueous solution of one of the enantiomers in the presence of activated charcoal.

Experimental:

Tris(ethylenediamine)cobalt(III) Chloride, Cl3\*1/2 NaCl\*3H2O

In a small beaker, dissolve 6. 00g (6. 02g) (25. 0mmol) of CoCl2. 6H2O and 13. 3g (13. 3g) (100. mmol) of ethylenediamine dihydrochloride in 25 mL water. Stir for one minute or so to dissolve the cobalt salt. Note that the color should be cloudy pink. Add 8. 00g (8. 02g) (200. mmol) of NaOH pellets and stir. The solution should be cloudy orange/brownish red. Stir until the NaOH is dissolved. Carefully, a little at a time, add 20mL (20mmol) of 3% H2O2 with swirling. The solution should thicken and darken upon the addition of the peroxide. Dilute the mixture to 50. 0 mL if it is not already around that volume.

Heat to a boil for a few minutes to remove the cloudiness from the solution. Place beaker in an ice bath for 30 minutes to cool. Using a suction filter, collect the orange to yellow/orange needles that have formed. Wash the crystals with 50. 0 mL of 95. 0% ethanol and 20. 0 mL of diethyl ether. Pull air through the crystals until they have dried and warmed to room temperature. The yield recorded was 5. 52g of Cl3\*1/2 NaCl\*3H2O. The filtrate is discarded.

Resolution of Tris(ethylenediamine)cobalt(III) Ion

In a small beaker, put 6. 00g(14mmol) of Cl3\*1/2 NaCl\*3H2O, 2. 60g (17. 4 mmol) of (+)tartaric acid, and a stir bar. Add 20. 0mL of water. Then add 1. 40g(35mmol) of NaOH and cover the beaker with a watch glass. Stir and heat until the solids completely dissolve. Remove the stirring bar and cool the solution to room temperature overnight.

Collect the dark orange crystals through filtration using a 60. 0 mL glass frit. Transfer the filtrate into a medium sized beaker and save the solution, which contains the isomer. After the filtrate is transferred, wash the crystals first with 20. 0mL of a 1: 1 ratio of acetone and water and then with 20. 0mL of acetone. Pull air through the crystals. Cl 5H2O is formed. The yield measured was 2. 87g. The theoretical yield was 4. 23. The percent yield was 69. 7%.

To determine the specific rotation of the compound, dilute approximately 0. 50g of the sample to a solution with a volume of 10. 0mL. Pipette the solution into a 1-dm polarimeter tube. Tilt the tube so that the air bubble is not in the path of the light. Blank the polarimeter with air so that the angle of rotation is zero. Place the polarimeter tube into the polarimeter. The sign and magnitude of the rotation was measured. The rotation was +2. 73 at a fixed wavelength of 589. 3nm.

To convert the tart salt to (+)-Co(en)3I3 H20, put 2. 00g (3. 90mmol) of Cl 5H2O in a 50. 0mL beaker. The crystals were broken up because they were too large. One pellet of NaOH, a stir bar, and 15. 0mL of distilled water was added. The solution was heated on a hot plate while stirred until all the solids dissolved. Heating the solution longer than 5 minutes would cause the cobalt complex to racemize. Added to the solution was 3. 60g(24mmol) of NaI.

Heating was continued for 1 minute longer and then the solution was cooled in an ice bath to room temperature. The solution was then filtered and washed with 3. 00g of NaI in a 10. 0mL water solution to remove the tartrate. The crystals were washed with 10. 0mL of ethanol and then 10. 0mL of distilled water. The (+)-Co(en)3I3 H20 complex was dried through the filter frit. The mass collected was 2. 49g. The theoretical yield was 2. 48g. The percent yield was 100%. Polarimetry was done on the collected compound.

To isolate the (-)-Co(en)3I3 H20 isomer, the filtrate that was collected and saved from earlier was diluted to 30. 0mL. One pellet of NaOH was added and the solution was heated. With stirring, 8. 50g(57mmol) of NaI was added. The solution was cooled in an ice bath and then filtered through to collect the impure (-)-Co(en)3I3 H20. The precipitate was washed with a solution of 3. 00g of NaI dissolved in 10. 0mL of distilled water. To purify the (-)-Co(en)3I3 H20, the compound was dissolved in 35. 0mL of distilled water at 50 degrees Celsius. The undissolved racemate was filtered off and 5. 00g of NaI was added to the filtrate. The crystallization of the (-)-Co(en)3I3 H20 occurred with cooling in an ice bath. The precipitate was collected through filtration and then washed with ethanol and acetone. Lastly the complex was air-dried. The mass collected was 2. 88g. The theoretical yield was 3. 16g. The percent yield was 91. 1%. Polarimetry was done on the collected compound.

Racemization of (+)-Co(en)33+ or (-)-Co(en)33+

The last thing was to racemize one of the enantiomers. A small amount of I3ï¿½ H2O (1. 00g) was dissolved in about 10mL of water. Activated carbon, charcoal, was added to the beaker and it was heated for 30 minutes. The hot solution was filtered to get rid of all the activated carbon. About 2 grams of NaI was added to the filtrate and the I3ï¿½ H2O precipitated out and was collected by filtration. It was then dried and weighted. The mass collected was 0. 33g. Polarimetry was done on the collected compound.