

Werner complexes: synthesis and resolution of Δ/Λ -[Co(en)₃]³⁺

Week 1: synthesis of [Co(en)₃]Cl₃·H₂O, [Co(en)₃][tart]Cl, and Δ/Λ -[Co(en)₃]I₃

1) brief prelab lecture/intro to the experiment over zoom.

2) dividing into teams. Group 1 will “carry out the synthesis” using (+)-tartrate, while Group 2 will carry out the synthesis using (-)-tartrate. Note, the coprecipitating salts and waters of hydration are not listed above; make sure to include them in your molecular weights! The procedure for this experiment is based on that published in Girolami, Rauchfuss and Angilici (Synthesis and Technique in Inorganic Chemistry, 3rd edition) with the modifications suggested by McClellan and Cass (*J. Chem. Educ.*, **2015**, *92*, 1766-1770).

group 1a: use (+)-tartrate

group 1b: use (+)-tartrate

group 2a: use (-)-tartrate

group 2b: use (-)-tartrate

3) watch the videos for the synthesis using your enantiomer of tartaric acid. From the videos, determine the balanced chemical reaction for each step, the purpose of each reagent, and the yield. Finally, as a team, determine the characterization data you *most* want to have in order to characterize your complexes. Remember, the point of characterization is to determine the identity (in this case, to *verify* the identity) and purity of the complex. Submit your proposed characterization methods, including a rationale. We will discuss this as a class at the beginning of week 2.

Video playlist on YouTube:

https://www.youtube.com/playlist?list=PLJb-VFt_wYH69DG9zHdPRSi48F99g9rN-

Each group has a total of 4 videos to watch. Everyone watches “Synthesis of [Co(en)₃]Cl₃” and then there are 3 videos for each of the (+)- and (-)-tartaric acid.

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Week 2: Characterization of Co(en)₃ complexes

1) brief prelab lecture/intro to the experiment.

2) collecting back into teams. Each group will characterize their complexes with the data provided. There will be 4 groups, same as last week.

group 1a: use (+)-tartrate

group 1b: use (+)-tartrate

group 2a: use (-)-tartrate

group 2b: use (-)-tartrate

3) The videos last week included characterization by polarimetry. Other data is available depending on the data you chose in your group. Interpret the data you are given. Remember, the point of characterization is to determine the identity (in this case, to *verify* the identity) and purity of the complex. The instructor will help, and will provide additional data as necessary (in case your data is insufficient to confirm the identity and purity).

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Week 3: Characterization of Co(en)_3 complexes by single crystal X-ray diffraction

- 1) The instructor will describe the principles of X-ray diffraction, including what data is available, and what can be learned. Then the class will be divided into 3 teams.
- 2) Each team will spend about one hour interpreting the data for one of the Co(en)_3 complexes. They will then prepare a short (5-10 minute, 3-5 slide) presentation to the class.
- 3) Each team will then present their data to the class. The presentations will be recorded or made available so that the data can be included in your final report for this experiment.