

Cobalt Coordination Compound Name / date?

Purpose:

- Synthesize trans cobalt coordination compound
 - o Oxidize Co^{2+} to Co^{3+} through the reaction $2\text{Co}^{2+} + 2\text{H}^+ + \text{H}_2\text{O}_2 \rightarrow 2\text{Co}^{3+} + 2\text{H}_2\text{O}$
 - o Bond Co^{3+} to ligand ethylene diamine through reaction $\text{Co}^{3+} + 2\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2 + 3\text{Cl}^- \rightarrow [\text{Co}(\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2)_2\text{Cl}_2]\text{Cl}$
- Isolate trans cobalt coordination compound through vacuum filtration and determine percent yield
- Isomerize solution of trans isomer to cis isomer both partially and fully
- Determine the difference in absorbance of isomers through wavelength of max absorbance

Reference: How did you decide the limiting reagent?

- (1) Kateley, L. J., *Introduction to Chemistry in the Laboratory*, 20th Ed., Lake Forest College, 2021, Experiment Cobalt Coordination Compound, Appendix D_LabEquipment

Synthesis of Trans

- 0.5001g of solid dark pink $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ weighted on analytical balance and added to 25mL Erlenmeyer Flask
- 1mL of deionized water measured in 10mL graduated cylinder and a stir bar added to flask
- Solution is a clear pink red akin to cranberry juice



- 700 μL ethylene diamine solution measured with blue Eppendorf pipet and added to flask while stirring
- Solution turned deep red



- 5mL of 3% H_2O_2 (hydrogen peroxide) added to flask
- Solution turned darker red, akin to soy sauce

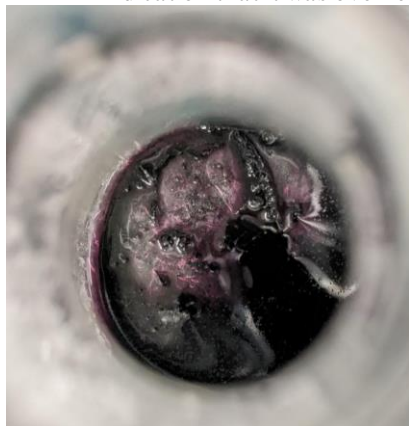


- Mixture stirred for 15 minutes to allow for evaporation

- 3mL of concentrated HCl (hydrochloric acid) measured in graduated cylinder and added to flask
- Solution heated to boiling point for 20 minutes reducing to volume to ~3 mL and turning the solution a dark blue green



- Mixture was cooled on countertop and then in ice bath as it cooled it turned back to a deep red/purple indication that it was overheated into cis isomer, bypassing trans isomer



Isolation of Trans through Vacuum Filtration

- After cooling mixture was filtered through vacuum filtration separating a shiny dark green solid and dark blue filtrate



- Mixture is washed with twice ~2mL of CH_3COCH_3 (acetone), once with ~1 mL of CH_3OCH_3 (diethyl ether), and twice with ~1 mL of acetone again
- Solid is transferred to petri dish in tared analytical balance and weighed in at 0.3082g
- Percent yield = $100 \times \text{experimental/actual} = 100 \times .3082\text{g}/0.482\text{g} = 63.9\%$

CALCULATIONS AND RESULTS						
compound	MM g/mol	conc mass %	density g/mL	volume	mass, g	moles available (mass ÷ MM)
cobalt chloride hexahydrate	237.93	NA	NA	NA	0.50g	.0021mol
ethylenediamine	60.10	30.0	1.00	700 µL	$0.700 \text{ mL} \times 1.00 \text{ g/mL} \times 0.300 = 0.210$.00349 mol
hydrogen peroxide	34.01	3.0	1.00	5.0 mL	$5.0 \text{ mL} \times 1.00 \text{ g/mL} \times .03 = 0.15$.0044mol
trans-dichlorobisethylenediamine-cobalt(III) chloride	275.41	NA	NA	NA	.3082g	.00112 mol

limiting reactant: Ethylenediamine

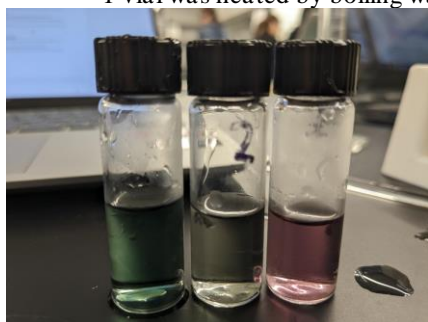
theoretical yield: .00175 mol or 0.482g

actual yield: .3082g or .00112 mol

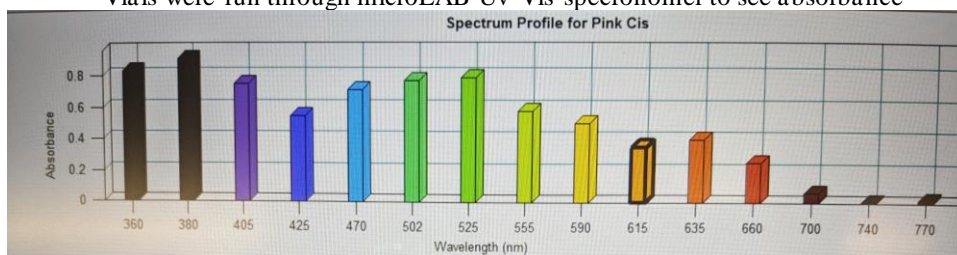
% yield = $100\% \times \frac{\text{experimental yield}}{\text{theoretical yield}} = 63.9\%$

Isomerization and Visible Spectra

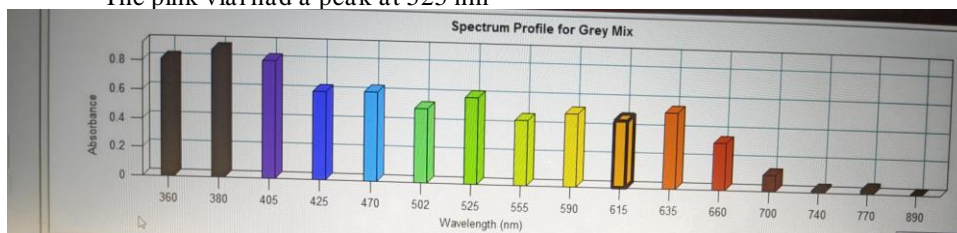
- 50mg (0.0588g) of synthesised trans isomer was dissolved in 10mL of deionized water creating a pale mint green solution
- The solution was equally divided into 3 spectroscopy vials
- 1 vial was packed with ice to make sure it remains as trans isomer, the solution remained green
- 1 vial was heated in a boiling water bath to create cis isomer, the solution turned grey pink and got pinker as it sat
- 1 vial was heated by boiling water for a short time and then put in the ice bath to stop conversion



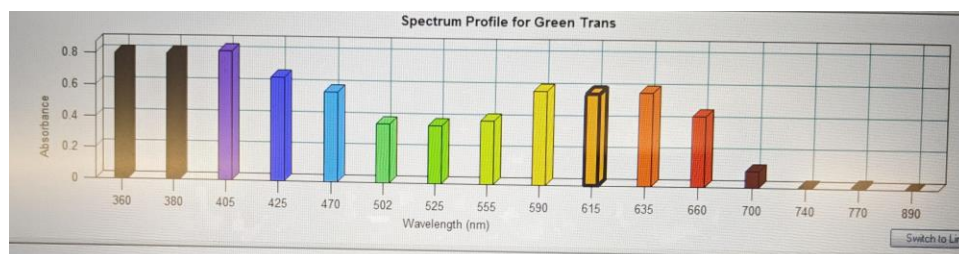
- Vials were run through microLAB Uv-Vis specnomer to see absorbance



- The pink vial had a peak at 525 nm



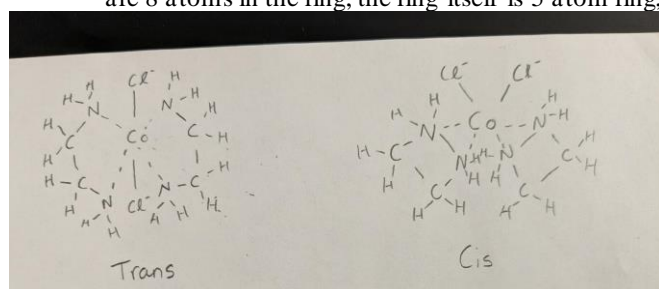
- The grey vial was relatively equal overall



- The green solution vial had a peak around 615 nm, orange red range

Models and Drawing, 2 possible structures for the cis, that are mirror images

- Models B and D are models of the trans isomer. They can be superimposed on each other and have one ring
- Models A and C are cis isomers. They are mirror images of each other and have two rings
- In the models Co is pink, Cl is green, black is C, and blue is N. The rings are made of C and N and there are 8 atoms in the ring, the ring itself is 5 atom ring, Co, N, C, C, N



Conclusion:

- The yield was not 100% because not all of the reactants reacted and some of the product was left behind in various equipment.
- One clear indicator is that the color of the compound is different. We do not have any good indicators of purity.
- Oxidation state change is another possible explanation for color change.
- The pink cis isomer had a larger max absorbance as it has a larger peak at 525 nm [blue green range] then the green trans did at 615 nm, [red orange range]. The grey mix has slightly more cis isomer as it absorbs more green (525 nm) light than orange/red (615 nm) light