

A2. Kinetics of Aquation of $\text{Co(en)}_2\text{Cl}_2^+$

The complex that you prepared last week, *trans*-bis(ethylenediamine)cobalt(III) chloride is a complex ion which forms a green solution when dissolved in water. In solution, a water molecule may replace one of the Cl^- ions in this complex, giving $\text{Co(en)}_2\text{Cl}(\text{H}_2\text{O})^{2+}$, which forms a pink solution. The purpose of this experiment is to investigate the kinetics of this aquation reaction. This will include the determination of the order of the reaction with respect to $\text{Co(en)}_2\text{Cl}_2^+$, the rate constant at two different temperatures and the activation energy.

Procedure

You will work in pairs for this experiment.

You will use the $\text{Co(en)}_2\text{Cl}_3$ that you each synthesized last week in lab to prepare 2 aqueous stock solutions at a concentration of 0.045 M, and you will perform a kinetic analysis of these solutions at two different temperatures between 45 and 75 °C (you will be assigned two temperatures to work with in lab). At the end of the lab period the data sets from all of the groups in lab will be pooled to create a complete data set for the kinetic analysis of the aquation reaction.

- Using the volumetric flasks provided, prepare two 25 mL solutions of 0.045 M $\text{Co(en)}_2\text{Cl}_3$. Use *ice-cold distilled water* to make up your solutions and keep the solutions on ice once you have prepared them.
- Briefly familiarize yourself with the operation of an Ocean Optics Spectrophotometer
 - In "scope" mode, insert your reference sample (distilled water) into the cuvette holder
 - click on "reference", then click on "store"
 - Run a dark spectrum, by blocking the light path entirely
 - click on "dark", then click on "store"
 - Switch to "absorbance" mode and insert a sample
 - if continuously scanning, spectrum should appear (if not, click on "scan")
- Remove a 1.000 mL aliquot of one of the solutions you prepared in step one and add it to 2.000 mL distilled water in a test tube. Transfer a small portion (*ca.* 1 mL) of this sample to a cuvette and record a UV/Vis spectrum on the Ocean Optics Spectrophotometers available in the lab. Save the spectrum to the hard drive or your network account. Wrap the test tube with parafilm and place it in a beaker of boiling water (there will be several in the lab). This will be used you to measure A_∞ .
- Prepare two water baths in 1000 mL beakers, one at each of the two temperatures that you have been assigned. Each bath should be filled with enough water to completely cover a 6" test tube when filled with *ca.* 12 mL reaction mixture. It is important that the temperature of these water baths remain fairly constant (within a degree of the target temperature) over the course of the experiment, and the bath should be stirred constantly to insure that there is no temperature gradient present. This will require some attention and creativity on your part (you may want to cool the baths down periodically by addition of cold water or ice). It's also important that the beaker is relatively full so that its temperature is not drastically affected by the sample.
- Label 20 clean dry 3" test tubes, fill with 1.000 mL distilled water, chill in ice.
- Once your water bath has reached the correct temperatures, pour about 12 mL of your stock solutions into a six-inch test tube, and add a small stir bar, and suspend the test tube in the water bath. Start your timer. (Put the rest of your stock solution back on ice in case you need to do any additional runs.)

7. At appropriate intervals (consult your instructor), transfer 0.500 mL of the solution to one of the chilled 3" test tubes.
8. Record spectra of the 20 samples that you collected in step 7. You do not need to save all the spectra, just record the absorbance at 515 nm, noting the time at which the sample was collected.
9. Record a spectrum of the sample prepared in step 3.
10. Repeat steps 5 – 8 for the second water bath.

Calculations

Using your data and Excel or other preferred spreadsheet program (available on the computers in TCL 210) and, *using time in seconds*, determine whether this reaction is zero, first, or second order with respect to $\text{Co(en)}_2\text{Cl}_2^+$. Calculate the rate constant for each temperature you used (Save all data to your network account so that you can access it later). Record your results on a report sheet and hand this in to your lab instructor before you leave the lab. The results from your section will be compiled and e-mailed to the entire class within 24 hours for inclusion in your lab report.