# Prelab: Lab 3. Cr(III) Kinetics

## CHEM 310 Week 2, Day 1-2

*Microscale Inorganic Chemistry* pages 263-271
Groups of 2 students, 4 hours × 2 days

### Learning Objectives:

- Successfully synthesize and analyze $Cr(EDTA)^-$, following the reaction with a pH probe and buffer solutions
- Demonstrate effective plotting and analysis of the UV-Vis data to determine the kinetics of the reaction
- Write a clear and concise abstract and introduction that effectively communicates an overview of experimentation, modeled after the ACS Journal *Inorganic Chemistry*

### Essential Lab Techniques:

- Measurement of pH with a pH probe
- UV-Vis data collection and analysis
- Graphical determination of reaction order

### Modifications from *MIC*:

- The first day of lab will be used to make all the solutions and adjust the pH. On the second day of lab we will immediately mix the EDTA and chromium solutions and start obtaining the UV-Vis spectra.
- You will be paired up with your fume hood partner for this experiment.
- Set water to warm on a hot plate as soon as you begin on Day 1.

There will be slight modifications to the amounts we are working with.

- Prepare a 50 mL volumetric flask for the EDTA solution, rather than a 250 mL flask.
- When adjusting the pH, use 10 mL for each aliquot instead of 25.
- Use the complete 10 mL of the pH adjusted EDTA solution added to 10 mL of the chromium nitrate solution. This means that some values must be recalculated. First, recalculate the concentration of each solution given in the lab manual. Using the new volumes, adjust the mass to match the concentration at this new volume.
- Please save the lowest pH complex for a future lab. Store it in a labeled scintillation vial.

For this lab we will be modifying the time points for each UV-Vis spectrum obtained. Instead of every 10 minutes for 3 hours as instructed in the text, we will do the following:

- Every 10 minutes for the first hour (6 measurements x 4 samples)
- Every 15 minutes for the second hour (4 measurements x 4 samples)
- Every 20 minutes for the third hour. (3 measurements x 4 samples)
- The last time point ($t_\infty$) will be provided via canvas. (Total of 14 measurements per pH unit)

## I. Pertinent Information:

This lab is focused on synthesizing and analyzing $Cr(EDTA)^-$, and applying analytical techniques to determine the reaction kinetics. Students will also complete a writing assignment to draft and edit a clear abstract and introduction following ACS standards. This lab reinforces and applies the information regarding kinetics and polydentate ligands learned in CHEM 112 and 310 to CHEM 411W, using absorption spectroscopy as a method to determine the kinetics of a reaction. To prepare for this lab, please read pages 263-271 in *Microscale Inorganic Chemistry (MIC).* During your reading, take notes on the chemistry and how this reaction works.

This lab will be an introduction on how to visually represent data to make it easy to read. We will be using a data plotting program called Veusz, which is free to download at the following link: https://veusz.github.io/download/. During the lab you will be given a brief introduction on how to use this program. There will be resources on Canvas to help with your data analysis journey. To aid in data analysis, the use of coding is highly encouraged. On Canvas there will be several files that will process your raw data and make it easier to analyze and plot into Veusz. If you need help setting up an integrated development environment such as Spyder, Visual Studio Code, or Jupyter please follow the instructions on Canvas. The first day of lab will be used to make all the solutions and adjust the pH. This will allow everyone time to ask questions about Veusz and coding. On the second day of lab we will immediately mix the EDTA and chromium solutions and start obtaining the UV-Vis spectra.

Students will also build writing skills this week by writing an abstract and introduction formatted in the same way as the ACS (American Chemical Society) journal *Inorganic Chemistry*. Rather than writing lab reports, you will learn how to write a research paper that is suitable for such journals. Each week you will practice writing a different section of the research paper. This will build your skills and prepare you to write a complete research paper during Week 7 for Lab 9. Please read through https://publish.acs.org/publish/author_guidelines?coden=inocaj#manuscript_types to learn how to format your work. Also use the resources on Canvas to help guide your writing of this section.

## II. Chemical Data Table (CDT):

In your lab notebook, construct a Chemical Data Table for this experiment. Fill in the table with all the compounds you will be working with, including products, byproducts, and intermediates. You must list the name, structure, molecular weight, melting/decomposition point (for solids), boiling point (for liquids), density, quantity (mLs/mgs), mmols, hazards, and comments (color, smell, extra hazards). Reference the SDSs to help you fill in the chart. An example can be found below. Red X's mean that you don't need to fill in that part of the chart. You can print this page or scan it and attach it to your lab notebook.

| Name | Structure | MW | MP/BP | Density | Quantity | mmol | Hazards | Comments |
|---|---|---|---|---|---|---|---|---|
| Chromium(III) Nitrate Nonahydrate (7789-02-8) | | | | X | | | | |
| EDTA Disodium Salt (6381-92-6) | | | | X | | | | |
| Sodium Hydroxide (1310-73-2) | | | | | X | X | | |
| Hydrochloric Acid (7647-01-0) | | | | | X | X | | |
| $[CrEDTA]^-$ | | | X | X | X | X | X | X |

 

## III. Synthetic Approach:

Write the procedure for each experiment in your lab notebook before entering the lab. This procedure should be concise but thorough enough so that you can set up and run the experiment without bringing your textbook. Drawing schematics is encouraged, especially for techniques you are not familiar with. The procedure should be written in passive past tense.

For more information on how to correctly set up a lab notebook read pages 31 - 34 in *MIC*.

## IV. Prelab Questions:

1. Write the **linear equations** for zero, first, and second order kinetics. Write the corresponding equations that will be used for plotting your data. You will determine which of these fits your data the best.

2. Speculate on the reaction mechanism that transforms $[Cr(H_2O)_6]^{3+}$ to $[CrEDTA]^-$. Identify the slow and fast steps. This reaction will have multiple steps, so a literature search is **highly** recommended. If any literature is used, please provide the DOI. (HINT: Use literature)

3. The rate determining step for this reaction was speculated on in the literature to be an associative interchange mechanism. Explain this mechanism in general terms. What does this suggest about the formation of intermediates?

4. Is $Cr^{3+}$ kinetically inert or labile? How does this impact a kinetics study? Compare $Cr^{3+}$ to $Cr^{2+}$. Assume each metal is the central atom in an octahedral orientation with water ligands. Use CFSE to justify your answer.

5. On Canvas locate the Veusz sample data set. Plot the data using Veusz. Follow the instructions found in the Canvas module and upload the png or jpg here. If you are experiencing difficulties downloading the dataset try using Chrome.

6. Explain the chelate effect. How does this apply to the system you will be synthesizing? How do chelating ligands impact the thermodynamics of a reaction? (Hint: Think about entropy!)

## V. Calculations:

Since we are monitoring the formation of a complex at varying pH's in this lab, theoretical yield does not need to be calculated. However, to reactivate your prior knowledge of dimensional analysis calculate the mmoles of your reagents.

1. Calculate the mmols of chromium(III) nitrate nonahydrate. Please show all work with units.

2. Calculate the mmols of the EDTA disodium salt. Please show all work with units.

## VI. Characterization:

This experiment uses UV-Vis spectroscopy to monitor the reaction. We will track the intensity of the lambda max peak for $[CrEDTA]^-$. Using your prior knowledge of color, answer the following questions.

1. The $[CrEDTA]^-$ complex is a vivid purple color. Since we will be monitoring this reaction with visible spectroscopy, predict at what wavelength range this complex will absorb. Justify your answer.

2. Hypothesize what will happen to the intensity of lambda max as pH is increased. Additionally, justify your hypothesis with a brief discussion on why this may happen. Use your speculated mechanism to support your answer.

## VII. Observations:

As you perform any experiment in lab, write your observations and your data in your lab notebook. Attach data tables and plots to empty notebook pages. Have your notebook signed by your instructor at the end of each week.

You can add your discussion and analysis of the results after the lab period is done. Do not answer the MIC Questions at the end of the procedures unless noted in the prelab. Some labs will have a separate Results assignment.

Be sure to include the following in your notebook, along with anything else that is important:

- Changes from the textbook procedure
- Unexpected results
- Color changes
- Gas evolution and precipitate formation
- Amount of time elapsed
- Temperatures, if known
- Tables of data obtained
- Mass of isolated products and calculation of percent yield
- Characterization, including melting point, IR, NMR, UV-Vis data, etc.
- Physical or chemical tests performed on reagents and products
- Graphs or plots of data
- Discussion and explanation of data or data analysis

## VIII. Data Analysis:

Once the experiment is completed you will submit a brief Data Analysis Report to Canvas. This will include all your data worked up (plotted and analyzed) and a brief write-up summing up what your results indicate. The point is to clearly communicate the major results of the experiment through use of data visualization and concise writing.

When preparing this please use the outline found on Canvas. Some tips can be found below.

1. Slide titles should be declarations of the main point. It is not acceptable to write:
   - IR Data of X Compound
     Instead try,
   - Increase in IR Shift Indicate Linear Binding Mode of Ligand to Metal
2. Use bullet points. Complete sentences are okay but try to avoid lengthy paragraphs.
3. This is not a presentation, it is meant to teach you an effective way to communicate results quickly to your future PI, Boss, etc. Presentations will have different tips but will also use PowerPoint/Google Slides.
4. Keep figure heights at or below 3 inches.
5. Only use a MAX of 5 slides.