Experiment 12: Make-Up Experiment
Copper Analysis by Complexometric Titration

A quantitative analysis of copper in a soluble copper salt will by performed by complexometric titration. The complexing agent will be ethylenediaminetetraacetic acid (EDTA) in the form of its disodium dihydrate salt ($Na_2C_{10}H_{18}N_2O_{10}$), with a molar mass of 372.24 g mol^{-1}. Since EDTA forms complexes with many metal ions, this particular method can only be used in the absence of such ions as Ca^{2+}, Ni^{2+}, etc. The reaction of complexation is:

$$Cu^{2+}_{(aq)} + (EDTA)^{2-}_{(aq)} \rightarrow Cu(EDTA)^{2-}_{(aq)} + 2H^{+}_{(aq)}$$

The stoichiometry is one metal cation to one EDTA anion. However, for Cu^{2+} (since it has lost 2 electrons), the equivalent mass is 63.546/2 = 31.773 g eq^{-1}, and since $(EDTA)^{2-}$ is a dianion its equivalent mass is 372.24/2 = 186.12 g eq^{-1} (for the disodium dihydrate salt). The equation above represents two equivalents reacting with two equivalents. The complex dianion is formed with the release of two moles of H^+ from EDTA, with the indicator being released from the copper ion.

The complex dianion has the structure shown in Figure 13.1. Note that the anion completely surrounds the cation, forming six coordinate covalent bonds to copper and a very stable complex. The bonding to the copper ion is nearly octahedral.

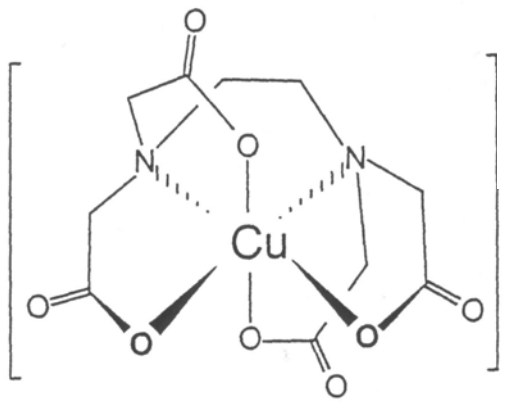

Figure 13.1
Structure of Cu^{2+}— EDTA dianion

The indicator used for the titration is called murexide. This indicator is highly colored and will complex with the copper ion to give a different colored species. During the titration, the $EDTA^{2-}$ forms a more stable complex and frees the indicator, which then displays its original color. The appearance of the free indicator means that all metal ions have been complexed by $EDTA^{2-}$, which signals the end point. At the end point, the following equation applies:

$$N_{EDTA}V_{EDTA} = N_{Cu(II)}V_{Cu(II)} = \#eq\ Cu(II), \text{ if V is given in L}$$
$$= \#meq\ Cu(II), \text{ if V is given in mL}$$

The mass of Cu equals (#eq Cu(II)) × (equivalent mass of Cu(II)), and

$$\frac{\text{mass Cu(II)}}{\text{mass Cu(II) salt}} \times 100 = \%\ Cu$$

12A Experiment

1. Rinse your burette and fill it with standardized $Na_2EDTA•2H_2O$ solution
2. (7.445 g $Na_2EDTA \cdot 2\ H_2O$ per liter of water).
3. Weigh accurately three approximately 0.1 g samples of the copper salt, reported to three decimal places. Your instructor may specify that each sample be > 0.08 g and < 0.1 g. If so, remember that this will limit your final results to two significant figures.
4. Dissolve each sample in 50 mL of de-ionized water.
5. Add *exactly* the same amount of indicator to each sample, three to five drops to start off with. If the indicator solution is not strong enough, add more but always the same for all samples. (The indicator's concentration should be 100 mg/100 mL H_2O)
6. Titrate each sample with the standardized EDTA. The light yellow solution turns green near the end point, then suddenly purplish blue at the end point. This end point is fairly hard to see, so put a white sheet of paper under your beaker and watch carefully. The distinctly purplish hue, due to free murexide, is the key to observing the end point. Look for the complete absence of green.
7. For each titration, calculate the number of equivalents or (milliequivalents) of Cu(II) found.
8. For each titrated sample, calculate the mass of copper in that sample.
9. For each titration, calculate the % copper content in the sample, then average them.

	Sample 1	**Sample 2**	**Sample 3**	
(a) Normality of EDTA				
(b) grams of Cu(II) sample				
(c) mL of EDTA solution				**Average % copper content**
(d) eq (or meq) of Cu(II)				
(e) mass of copper				
(f) % copper content				

12B Exercise

Copper(II) sulfate forms a hydrate which contains 36.1% by mass water. Since the only component (other than H_2O and Cu^{2+}) is the sulfate ion, SO_4^{2-}, we can now determine the complete formula of the hydrated copper(II) sulfate. Do this determination.

NOTE Solutions preparation:
Either weigh the EDTA analytically or standardize the solution. Label the bottles with the normality of EDTA. Use deionized water. About 1 liter will be used by 20 students, 10 groups. Make these solutions up fresh, including the murexide solution.

Report Form 12: Copper Analysis by Complexometric Titration

Name:_____ Partner:_____

12A Experiment

	Sample 1	Sample 2	Sample 3	
(g) Normality of EDTA				
(h) grams of Cu(II) sample				
(i) mL of EDTA solution				**Average % copper content**
(j) eq (or meq) of Cu(II)				
(k) mass of copper				
(l) % copper content				

12B Exercise

Complete formula of hydrated copper(II) sulfate. _____