

SEPARATION OF COPPER FROM METALS IN AN ALLOY BY LIQUID-LIQUID EXTRACTION

Background Reading: Harris, 7th ed., Chap. 23. Skoog, West, Holler and Crouch, 7th ed., Chap. 24.

Introduction

Neocuproine (2,9-dimethyl-1,10-phenanthroline), under the right conditions, is almost a specific extraction reagent for Cu(I). The complex that forms is soluble in chloroform and absorbs strongly at 457 nm. This method may be used to determine copper in various alloys such as cast iron, solder, and steel.

In this experiment, the alloy sample is dissolved and Cu(II) then reduced to Cu(I) with hydroxylamine hydrochloride (NH₂OH·HCl). Sodium citrate is used as a masking agent to prevent interference by other metals. When the Cu-neocuproine complex is formed it can be extracted into a small volume of chloroform, where the color will develop. The absorbance of the chloroform solution is read to determine the concentration of Cu in the original sample.

Procedure

This experiment consists of three major steps: (1) preparation of sample and standards, (2) extraction of the Cu-neocuproine complex into chloroform, and (3) determination of the absorbance of the chloroform/Cu-neocuproine solution. The extraction step usually takes a full lab period, as six solutions (a blank, four standards and the sample) must be extracted in the same day so that color development for all solutions is allowed to proceed for approximately the same length of time.

Sample and Standard Preparation. Weigh exactly approximately 35 mg of a dried brass sample into a 250 mL Erlenmeyer flask. Add about 20 mL of H₂O and 10 mL of concentrated HNO₃. Digest this sample until it dissolves but be careful not to let the sample go to dryness. Quantitatively transfer the dissolved sample to a 100 mL volumetric flask and dilute to the mark. The sample is still too concentrated, so a 1:20 dilution is accomplished by transferring 5 mL of this solution to another 100 mL volumetric flask and diluting to the mark. This will be the solution from which copper will be extracted and quantitated.

Weigh exactly approximately 39 mg of CuSO₄·5H₂O (not dried, formula weight 249.68) into a 100 mL volumetric flask. Dissolve in water and dilute to 100 mL. This should make a standard solution containing approximately 100 mg Cu/L. Prepare by dilution 100 mL each of copper standard solutions with concentrations approximately 5, 10, 15, and 25 mg/L.

Extraction. The extraction procedure is the same for the sample, standards and blank (water). With a volumetric pipet, transfer 10 mL aliquots of the solution to a separatory funnel. Add 5 mL of 10% hydroxylamine hydrochloride solution and 10 mL of 30% sodium citrate solution. Add NH₄OH until the pH is about 4. Add 10 mL of 0.1% neocuproine and 10 mL of chloroform, and then shake for 30 seconds. Be sure to vent the stopcock occasionally to release pressure in the funnel. **Make sure you hold the cap in place while you are shaking the separatory funnel and that the cap is pointed away from other students (and the laboratory instructor!).** Allow the layers to separate and draw off the lower CHCl₃ phase into a 25 mL volumetric flask. Repeat the extraction with another fresh 5 mL portion of CHCl₃, add this portion to the same flask that contains the original CHCl₃ phase, and then dilute to the mark with CHCl₃. Rinse the separatory funnel well between sample extractions with water. The chloroform extracts at this point will normally be cloudy because they are supersaturated with water. By the next lab period the excess water should separate into a distinct phase from the bulk of the chloroform.

Absorbance Measurements. The separated water layer can be removed from the bulk chloroform phase with a Pasteur pipet. Dilute back to the mark with chloroform. Measure the absorbance at 457 nm for the CHCl₃/Cu(I)-neocuproine solution. Refer to the **SPECTROPHOTOMETRIC**

DETERMINATION OF IRON experiment for instructions on the use of a spectrophotometer. Prepare a calibration curve (absorbance vs. Cu concentration, mg/L) using the Cu standards, and determine the percentage of Cu in the alloy. Don't forget to account for dilutions!

Report

Use Excel to plot the calibration curve and determine the equation of the best-fit line.

Report the absorbance of the sample solution and the concentration calculated from the equation of the best-fit calibration line.

Show how you calculated the %Cu in your sample and report your best estimate of the true value.