Chemical Analysis of Brass

Alloys and their composition

FL53098

Introduction

Alloys

An alloy is a substance made by mixing two or more metallic elements into a single crystalline structure. Alloys are often advantageous to produce because they retain properties similar to those they exhibit in their pure state while also producing additional desirable properties due to the controlled nature of the mixed elements. Since alloys are mixtures, their composition can vary widely. One example of an alloy is brass, which is composed primarily of copper (Cu) and zinc (Zn). Some brasses also have small amounts of other types of metals, such as tin (Sn). Brass with a high copper content is often used to produce musical instruments due to its malleability, workability, and resistance to corrosion. Naval brass is a particular mixture of copper, zinc, and tin and is commonly used for various hardware components. The added tin imparts an extra degree of corrosion resistance. Other common applications of brass include plumbing fittings, electrical applications, and architectural or design use where a golden finish is desired. In each case, the composition of the brass is tailored to give the best physical properties for the application.

Oxidation of metals

Since the copper and zinc in brass exist in their elemental forms, they need to be oxidized to their ionic forms to get them into solution. For active metals including zinc, this can be done by adding an acid (H^+) where the following reaction takes place:

$$Zn_{(s)} + 2H^+_{(aq)} \rightarrow Zn^{2+}_{(aq)} + H_{2\,(g)}$$

Less active metals like copper require a stronger oxidizing agent. Concentrated nitric acid (HNO_3; ~15 M) at room temperature or hot 7.0 M nitric acid are each strong enough to oxidize copper by the following reaction:

$$Cu_{(s)} + 4HNO_{3\,(aq)} \rightarrow Cu(NO_3)_{2\,(aq)} + 2NO_{2\,(g)} + 2H_2O_{(l)}$$

Not all metal cations are soluble in acidic aqueous solution. One example would be tin which is a common component of brass. Tin(II) ions (Sn^{2+}) are formed by the oxidation of metallic tin in nitric acid and are soluble in aqueous solution. However, they slowly undergo additional oxidation to form tin(IV) oxide, which is insoluble in water. In this experiment, you can assume that your brass samples are composed of only copper and zinc.

Metal coordination chemistry

Since metal ions carry a positive charge, negatively charged species in a solution, like anions or negatively charged atoms of complex ions, will be attracted to them.

If the metal is a *d*-block element (transition metal) this means that the electron clouds of the ligands overlap with the d-orbitals in the metal to different extents. The d-orbitals split, with the d_{xy}, d_{xz} and d_{yz} forming one group and the $d_{x^2-y^2}$ and d_{z^2} forming a second group at a different energy level. The energy gap between these energy levels is very often in the range of a photon of visible light. If a photon with the right energy strikes an electron in the lower energy orbital, that photon can be absorbed and the electron is promoted to the higher orbital. This absorbance gives the metal ion solution its color and allows us to measure the concentration by colorimetry. By changing the metal or the ligand, we change the energy gap which affects both the color/wavelength of light absorbed and how strongly it is absorbed.

Experimental

Purpose

In this experiment, you will perform an analysis of three brass samples to determine the copper content of each by dissolving brass in a solution and measuring the absorbance at a particular wavelength after workup.

Procedure

Making a measurement with the Thermo Scientific™ SPECTRONIC™ 200 Visible (Vis) Spectrophotometer*

1. Turn on the instrument and allow it to complete its startup sequence. Let the instrument warm up and stabilize for at least 30 minutes. Set up the experiment you want to perform in the spectrophotometer software. Obtain a square plastic cuvette or glass test tube to use in your experiments. If using a test tube cuvette, use a pen to place a mark near the top if the cuvette is not already marked with a white line. The mark allows you to ensure consistent placement into the instrument.

2. Add liquid to the cuvette until there is ~3 cm of liquid in the bottom (4 cm for test tubes). If plastic transfer pipettes are available, use one. The exact liquid level in the cuvette is not critical for good measurements as long as it is above 3 cm. Do not waste solution or risk spills by over-filling the cuvette.

3. Place the cuvette in the sample stage of the

SPECTRONIC 200 Visible Spectrophotometer. If using a plastic cuvette, the clear sides should be on the right and left. If using a test tube cuvette, place it so that the mark faces to the right.

4. After the warm-up period, follow steps 2 and 3 using water or the appropriate "blank" solvent. Zero the instrument by pressing the autozero button.

5. For each subsequent measurement, empty and rinse your cuvette, shaking out as much of the rinse solvent as possible. When preparing samples, never return excess solution to the stock bottle. Pour all waste or excess into the appropriate waste receptacle. Follow steps 2 and 3 using your sample.

SPECTRONIC 200 Visible Spectrophotometer

*SPECTRONIC 200 Spectrophotometers are available on loan from Thermo Fisher Scientific™ at no cost. We will ship it to you, and you ship it back after one week. If you are interested in this program, please visit: www.thermofisher.com/spec200freetrial

Reagents

- Brass Samples

- 7.0 M Nitric acid (HNO_3)

- 0.1 M Copper(II) sulfate ($CuSO_4$)

- 2.0 M Ammonia (NH_3)

Part 1. Getting brass samples into solution

1. You will start with one of the samples derived from three different brass sources. Label a 100 mL beaker with the identity of the brass sample you will analyze in it. Weigh ~0.5 g of the brass directly into the labeled beaker. Record the identity and exact mass of the sample in Data Table 1 of your Lab Report.

2. Carefully pipette 10.0 mL of 7.0 M HNO_3 into the beaker.

3. Perform this step in a fume hood or in a well ventilated area. Cover the beaker with a watch glass and set it on a hot-plate set to low. Observe and record what happens.

   Warning: Do not inhale the fumes that rise from the beakers: they are corrosive.

4. While the brass is reacting, proceed to Part 2.

Part 2. Preparation of standard $[Cu(NH_3)_4]^{2+}$ solutions

1. Pipette 3 mL of a 0.1 M $CuSO_4$ solution into a clean cuvette and record its absorbance spectrum from 400 nm to 900 nm using the SPECTRONIC 200 Spectrophotometer. Use the cursor to determine the wavelength of maximum absorbance, λ_{max} and record the data in the Lab Report.

2. For each solution, use a pipette to add the amount of 0.1 M $CuSO_4$ indicated in the table below to a 25 mL volumetric flask.

Standard solution	Volume of 0.1 M $CuSO_4$
1	1.0 mL
2	2.0 mL
3	4.0 mL
4	5.0 mL

3. Using 2.0 M NH_3, dilute the solutions to the mark on the volumetric flask. The final deep blue color indicates formation of the $[Cu(NH_3)_4]^{2+}$ complex. Place the stopper in the flask and mix the solutions well by inverting and swirling.

4. Transfer the prepared solutions to clean, dry, labeled 50 mL beakers and save for analysis.

5. Measure your standards using the SPECTRONIC 200 Spectrophotometer.

   a. Using Standard Solution 4, perform a scan from 400 nm to 900 nm in absorbance mode.

   b. Record the wavelength of maximum absorbance (λ_{max}) for $[Cu(NH_3)_4]^{2+}$.

   c. Record the measured absorbance at this wavelength for your solution in Data Table 2.

   d. Either in Scan mode, or in Live Display mode, measure the absorbance of the other three standard solutions at λ_{max} and record the data in Data Table 2.

6. Using your concentration and absorbance readings, construct a Beer's Law plot. Use a spreadsheet program or a graphing calculator to plot your data and determine a best-fit line to calculate the slope of your line. Record the slope of the line in the Lab Report.

Part 3. Analysis of the brass samples

1. After all the brass has reacted away, pipette ~3 mL of the solution into a clean, dry cuvette and record its absorbance spectrum from 400 nm to 900 nm. Use the cursor to determine the wavelength of maximum absorbance, λ_{max} and record the result in Data Table 3 of the Lab Report. Discard the sample.

2. With the remaining solution:

   a. Pipette 2.0 mL of the solution into a 100 mL volumetric flask. Be as careful as you can to deliver exactly 2.0 mL.

   b. Add 20 mL of 2.0 M NH_3 solution to the volumetric flask and swirl the contents. Record your observations in detail.

   c. Add additional 2.0 M NH_3 solution to make the volume up to the mark. Stopper the flask and invert it several times to mix the contents thoroughly.

   d. Pour the contents of the flask into a labeled beaker.

   e. Pour or pipette ~3 mL of this solution into a clean, dry cuvette. Record the absorbance of the solution at the same wavelength that you used for the $[Cu(NH_3)_4]^{2+}$ standards in Data Table 4 of the Lab Report.

   f. Complete Part 3 for all three brass samples.

3. Report the values in your Lab Report:

   a. Identity of the brass sample

   b. Mass of brass used in your experiment

   c. Absorbance of your solution

   d. λ_{max} used to make the measurement

Disposal of chemicals:
Check with your instructor before discarding any solutions. All solutions can be poured down the sink and rinsed with lots of water to dilute. Discard solids in the trash.

Lab Report

Chemical Analysis of Brass

Name: _____

Date: _____

Section No. or Lab Period: _____

Part 1. Getting brass samples into solution

Questions

1. Record the identity of the brass and the exact mass of the brass you weighed out in your beaker:

Data Table 1

Sample number	Identity of brass	Mass of brass (g)
1		
2		
3		

2. What happens when the brass reacts with the nitric acid solution?

Part 2. Preparation of standard $[Cu(NH_3)_4]^{2+}$ solutions

1. Record the wavelength of maximum absorbance (λ_{max}) for the 0.1 M $CuSO_4$ solution:

λ_{max} ($CuSO_4$ solution): _____ nm

2. Is color of the $[Cu(NH_3)_4]^{2+}$ solution the same as the color of the 0.1 M $CuSO_4$ solution?

☐ Yes   ☐ No

3. Record the wavelength of maximum absorbance (λ_{max}) for $[Cu(NH_3)_4]^{2+}$.

λ_{max} ($[Cu(NH_3)_4]^{2+}$ solution): _____ nm

4. Calculate the concentration of Cu in mol/L of each of the standard solutions and record the results:

Data Table 2

Standard nolution	Measured absorbance	Concentration of Cu solution (mol/L)
1		
2		
3		
4		

5. Record the slope of the best-fit line:

_____

6. Write the full equation ($y = mx + b$ format) for the best fit line on the graph you just created using the slope.

7. Use the slope of the line to determine the molar absorptivity (ε) of the $[Cu(NH_3)_4]^{2+}$ complex. Use the equation for Beer's Law ($A = \varepsilon bc$) to derive and include the units. Note that absorbance has no units. Show your calculation here:

8. Is the peak in the spectrum of the 0.1 M $CuSO_4$ solution the same peak that you saw in the $Cu(NH_3)_4]^{2+}$ standards?

☐ Yes ☐ No

Part 3. Analysis of the brass samples

1. Record the wavelength of maximum absorbance (λ_{max}) for the dissolved brass samples before adding ammonia.

Data Table 3

Sample mumber	λ_{max} before ammonia (nm)
1	
2	
3	

Were these wavelengths the same as the wavelength of maximum absorbance (λ_{max}) for $[Cu(NH_3)_4]^{2+}$?

☐ Yes ☐ No

2. Record the absorbance for the dissolved brass samples after adding ammonia using the same wavelength that you used for the $[Cu(NH_3)_4]^{2+}$ standards.

Data Table 4

Sample number	Measured absorbance
1	
2	
3	

3. Using this absorbance values and the previously prepared calibration curve, determine the concentration of copper in mol/L in the brass solutions. Show your calculations here:

4. Using this concentration, determine the number of moles of copper in your 100 mL unknown brass solutions. Show your calculations here:

5. Using the moles of copper in the 100 mL unknown brass solutions, determine the mass of copper found in the original solid brass samples. (Hint: you used 1/5 of the 10 mL dissolved brass solution to prepare the 100 mL unknown brass solution.) Show your calculations here:

6. Calculate the mass percentages of copper and of zinc in your original brass samples using the mass of each original brass sample and the calculated mass of copper in each brass sample. Show your calculations here:

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