## **Experiment 39**

# SYNTHESIS OF COPPER(II) GLYCINATE

#### **Text Topics**

Geometric isomers, isomerization, organometallic compounds.

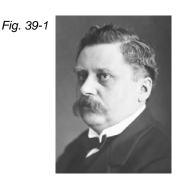
#### Discussion

While your body is relatively inactive during sleep, some argue that the brain continues to work on ongoing issues and problems. Two famous stories of dream initiated creative thinking involve famous chemists and their important insights. The claim has been made that concepts of spatial aspects of carbon bonding and the structure of benzene originated in a dream by August Kekulé in the late part of the 19<sup>th</sup> century. In 1892, French-born Swiss chemist Alfred Werner reportedly awoke with the concepts of coordination chemistry. Since coordination compounds such as heme and chlorophyll are vital in life processes, the concepts that resulted from Werner's dream were extremely important. In coordination compounds, a metal ion is bonded to and generally surrounded by an organic molecule or molecules. Copper(II) is one of the ions that forms coordination complexes and a number of its complexes are found in nature. Although copper(II) ion is toxic in appreciable amounts, it is necessary as a trace element in human nutrition. Copper(II) is instrumental in the function of enzymes that are involved in mitochondrial energy production and formation of melanin, elastin, bone and nervous tissue. Copper(II) ion is absorbed in the stomach and upper small intestine by the formation of complexes with amino acids and peptides. The experiment today involves the synthesis of copper(II) glycinate monohydrate.

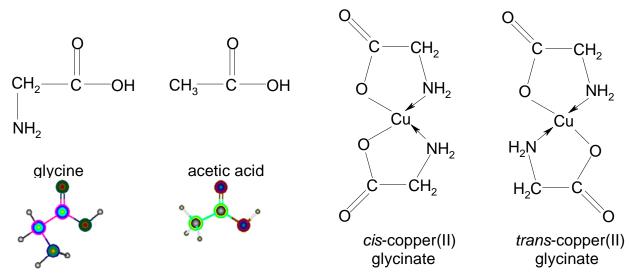
The synthesis of copper(II) glycinate monohydrate can be viewed as a double replacement reaction between copper(II) acetate and the simplest of the amino acids, glycine.

 $Cu(C_2H_3O_2)_2(aq) + 2HC_2H_4NO_2(aq) = Cu(C_2H_4NO_2)_2(s) + 2HC_2H_3O_2(aq)$ 

Glycine is one of the 20 amino acids that can link together to form proteins. It is actually related very closely to the acetic acid it replaces in the reaction. In the coordination compound that results, the copper is literally surrounded by the two glycines and one water.



Alfred Werner (1866-1919) was awarded the Nobel prize in chemistry in 1913. http://nobelprize.org/nobel\_prizes/chemistry/ laureates/1913/werner-bio.html



Notice that there is a square planar configuration of the four atoms around the copper and there are two geometrically different ways of positioning the glycines. Compounds which have the same formula but different structures are called isomers and the special type of isomerism present here is called geometric isomerism. Your synthesis could yield one or the other or a mixture of both. Experimental evidence indicates that in this synthesis the product is exclusively the *cis* isomer. The *cis* isomer can be converted to the *trans* isomer by heating..

#### Procedure

Weigh about 1.6 grams of copper(II) acetate monohydrate to at least the nearest 0.01 g into a 250 mL beaker and add 15 mL of deionized water. Weigh out about 1.3 g of glycine into a 150 mL beaker and add 10 mL of deionized water. Place both beakers on a hot plate and heat to the boiling point. Stir occasionally while heating until both solids dissolve. After the solids dissolve and the solutions are near the boiling point, **carefully** remove the beakers (use beaker tongs) from the hot plate and add the glycine solution to the copper(II) acetate solution and stir. Allow the solution to cool for several minutes and place the beaker in an ice bath. After crystals begin to form, add 20 mL of 1-propanol with continuous stirring. Continue to cool for several minutes and vacuum filter (use a Büchner funnel and a filter flask) to collect the product. Wet the filter paper first to insure a good seal. Rinse the beaker to make sure all solid is transferred to the funnel with acetone from a wash bottle (keep away from flames) and add the wash liquid to the funnel. Wash the precipitate with acetone twice and continue to pull air through the sample for a few minutes. Then transfer the solid to a weighed piece of filter paper. Divide the presumed *cis*-copper(II) glycinate monohydrate in half and allow one part to dry for a few days and then weigh it to determine the experimental and percent yields.

Transfer the remaining *cis*-copper(II) glycinate monohydrate to a 25 mL round bottom flask mounted in a heating mantle with a reflux condenser. Add 10 mL of water and an amount of glycine equal to about 2/3 the amount of the *cis*-copper(II) glycinate monohydrate already in the flask. Reflux gently for 1 hour. Filter the resulting mixture and collect and air dry the solid. After drying, weigh the presumed *trans*copper(II) glycinate monohydrate and determine the percent yield of the isomerization.

An alternative method for the isomerization reaction involves placing a small amount of the *cis* isomer in an oven at 170°C for about an hour.

Analysis. Run and compare the ir spectra of the *cis* and *trans* isomers in nujol mulls or KBr.

#### References

O'Brien, P. J. Chem. Educ., **1982**, *59*, 1052-1053. Delf, B. W.; Gillard, R. D.; O'Brien, P., J., Chem. Soc. Dalton, **1979**, 1301 Tomita, K. Bull. Chem. Soc. Japan, **1961**, *34*, 280, 286. Murov, S.; Stedjee, B. Experiments and Exercises in Basic Chemistry, 7<sup>th</sup> ed., Wiley, **2009**, 233. Murov, S. Experiments in General Chemistry, 5<sup>th</sup> ed., Brooks/Cole, **2007**, 365. http://www.rsc.org/ej/CP/1999/a903018b.pdf http://icn2.umeche.maine.edu/genchemlabs/Acid\_Base\_Titration/Amino\_Acids/aminoacids1.htm http://webs.wofford.edu/hilljb/Chem%20323/CopperGlycine.pdf http://wwwchem.uwimona.edu.jm:1104/lab\_manuals/c31lex1.html http://faculty.concordia.ca/bird/c241/wet\_labs/expt3.pdf Note: A recent publication describes the synthesis of a thermochromic copper complex that could be performed instead of this experiment. However, consider the hazards before choosing this interesting alternative. Cui, Ai-Li; Chen, X.; Sun, L.; wei, Jing-Z.; Yang, J; Kou, Hui-Z. J. J. Chem. Educ., **2011**, 88, 311-312.

## **Prelaboratory Preparation - Experiment 39**

First, be sure to list all the goals of the experiment. Prepare a table for insertion of useful and observed data such as molecular mass, mass, moles, melting points and percent yields and recoveries. Considering the symmetries of the *cis* and *trans* isomers, which isomer should have more active bands in the infrared?

## **Observations**

Report all relevant observations including physical properties and infrared spectra of both products.

## Conclusions

This section should include the following:

- 1. Were the goals of the experiment achieved? Explain your answer.
- 2. Discuss any available information that suggests your syntheses were successful.
- 3. How could the percent yield and recoveries have been improved?
- 4. According to the web site: http://www.freepatentsonline.com/y2007/0099886.html, "Copper Glycinate, for comparison, has strong ir bands at 1610, 1367, 1330, 1139, 1048, 742, and 647cm<sup>-1</sup>." The web site did not specify whether the IR was for the *cis* or *trans* isomer. Are these absorptions consistent with the infrared spectrum of either of your products? Explain your answer.
- 5. Did the infrared spectra agree with your predictions made in the answer to the question in the *Prelaboratory Preparation*? Do you have confidence that you can distinguish between the two isomers using ir?