EXPERIMENT 12: RECYCLING ALUMINUM

INTRODUCTION

In this experiment, you will prepare a compound called alum (potassium aluminum sulfate) from an aluminum can. The equations for the formation of alum are as follows:

$$2\text{Al}(s) + 2\text{K}^{+}(aq) + 2\text{OH}^{-}(aq) + 6\text{H}_{2}\text{O}(I) \rightarrow 2\text{K}^{+}(aq) + 2\text{Al}(\text{OH})_{4}^{-}(aq) + 3\text{H}_{2}(g)$$

$$2\text{K}^{+}(aq) + 2\text{Al}(\text{OH})_{4}^{-}(aq) + 4\text{H}_{2}\text{SO}_{4}(aq) \rightarrow 2\text{K}^{+}(aq) + 2\text{Al}^{3+}(aq) + 4\text{SO}_{4}^{2-}(aq) + 8\text{H}_{2}\text{O}(I)$$

$$\text{K}^{+}(aq) + \text{Al}^{3+}(aq) + 2\text{SO}_{4}^{2-}(aq) + 12\text{H}_{2}\text{O}(I) \rightarrow \text{KAl}(\text{SO}_{4})_{2} \cdot 12\text{H}_{2}\text{O}(s)$$

$$(alum)$$

The alum you make will then be used to fix an organic dye to a piece of cloth. There are several types of dyes used in the textile industry, and the type of dye used in a particular situation will depend on many factors. The dye color may need to match that used on a previous batch; it will need to be color fast or permanent when it is washed. Whether the cloth is wool, cotton, or a synthetic will be important (in this experiment, the cloth will be cotton). Some dyes will react permanently and directly with cloth, to which anyone who has spilled red wine or grape juice on a white cotton shirt will attest. These dyes, which attach by hydrogen bonding to the cellulose molecules of cotton, are called *direct dyes*. Some dyes, however, cannot bond directly to the cellulose, but can bond to an intermediary molecule called a *mordant*. Alum is a common mordant, and you will use the alum you produce in this experiment to mordant a natural dye.

The dye you will use is *alizarin* which, until this century, was extracted from a plant called madder. Today it is synthesized, a much cheaper process. However, a common Oklahoma plant called cleavers or bedstraw is in the same genus as madder, and the roots of the perennial bedstraw can be used to extract alizarin.

PRE-LABORATORY QUESTIONS

1a.	Identify the gas produced when aluminum metal is added to 2 M KOH.
b.	list some physical properties of the element in 1a. (Include color, phase at room temperature and boiling point.)
c.	list one chemical property of this element.
2.	Determine which reactant is in excess when 0.200 g of Al is mixed with 10.0 mL of 2.0 M KOH.
3.	Calculate the maximum amount of $KAl(SO_4)_2 \not\equiv 12H_2O$ that will be formed when 0.195 g Al completely reacts according to the series of reactions listed on page 1.

4.	Consider each of the following experimental variations, and indicated whether the amount of $KAI(SO_4)_2 \not\equiv 12H_2O$ will be higher, lower or not effected? Provide a brief explanation supporting your answer.				
	a)	in step 3) a student cools the Al/KOH mixture before all the Al has reacted;			
	b)	in step 5) the student uses 200 mLs of ice in the 600 mL beaker;			
	с)	in step 6) the student drys the crystals after washing with two 5-mL			
		aliquots of ethanol for 30 secs.			

PRE-LABORATORY DISCUSSION

Your instructor will discuss the following with you:

- a. alum
- b. bauxite
- c. amphoteric
- d. dye
- e. mordant

EQUIPMENT NEEDED

FROM YOUR LOCKER

100 mL beaker 10 mL graduated cylinder 2 - 600 mL beakers wire gauze 250 mL beaker funnel watch glass forceps

FROM THE STOREROOM

B chner funnel 250 mL filter flask and vacuum hose

FROM THE CABINET

- 2 ring stands and rings
- 2 Bunsen burners and hoses

PROCEDURE

SAFETY: You will be using 2 M potassium hydroxide and 6 M sulfuric acid in this lab. **Both are caustic!** If either of these is spilled on your skin, rinse well with running cold water and inform your instructor.

CLEANUP: All solutions can go down the drain with water. If you have any solid alum left, dissolve it in water, and wash it down the drain. Wash your hands before leaving lab.

- 1. Cut very small pieces off the aluminum can, and weigh out about 0.2 g of aluminum. Place the Al pieces in a 100-mL beaker. Add 10 mL of the KOH solution. There should be several hot plates available in the hood. Mark your beaker with your initials and place it in the hood on a hot plate. Go on to step 2, since it will take the Al about 10-15 minutes to react. Periodically check on your Al reaction.
- 2. While you are heating the mixture, obtain and set up the B chner funnel for step 6. Then set up the following 2 solutions (dye and mordant).

Dye solution: Put about 300 mL of <u>tap</u> water (DO NOT use distilled water) and 0.4 g of alizarin in a 600-mL beaker. Set up a ring stand and burner, bring the water and dye to a boil, and boil it for 5 minutes.

Mordant solution: While the dye is boiling, set up another ring stand and Bunsen burner. Put 300 mL of tap water (DO NOT use distilled water) in another 600-mL beaker, and place 2 peas-worth of calcium oxide, CaO, in the water. Heat to boiling. Turn off both Bunsen burners and continue the alum preparation. (CaO keeps the pH of the solution constant when the alum is added below.)

- 3. When the Al reaction has stopped, cool the solution by setting the 100-mL beaker in a larger beaker of cold water. There may be small amounts of non-Al material floating around. While the solution is cooling, set up a conical funnel with a piece of coarse filter paper. When the beaker is cool enough to touch, carefully pour the warm solution through the filter paper to trap the undissolved solids. What are they?
- 4. Slowly add 6 mL of 6 M H₂SO₄ to the Al solution from step 3 with stirring. What is happening? When you have added all the acid, use blue litmus paper to test the Al solution. It should be slightly acid. Heat the mixture very gently over the Bunsen burner for about 10 minutes. What happens? While the mixture is heating, set up the ice bath in step 5. Cool the beaker for a few minutes after heating.
- 5. Put about 100 mL of ice in a 600-mL beaker. Add water to the level of the ice. Place the beaker of solution from step 4 on the ice for about 15 minutes. While

- you are waiting for the alum crystals to form, set up a B chner funnel with a filter paper.
- 6. Transfer the crystals to the B chner funnel, scraping the last crystals from the beaker with a rubber policeman. Wash with 5-mL portions of 50% ethanol solution several times to remove all soluble ions and to begin drying the alum. When the last rinse is done, let the crystals dry for several minutes while the vacuum is running to make them easier to remove from the funnel.
- 7. Break the vacuum, and carefully remove the filter paper and crystals together, and place them on a watch glass to use in the dyeing process. Neutralize the filtrate with sodium bicarbonate, NaHCO₃, from the hood, and pour the solution down the drain.
- 8. Relight the Bunsen burners under the *dye* and *mordant* solutions in step 2 above. Use a low flame in each case to boil the two solutions *gently*. To the *mordant* solution (not the dye!) add about 2 pea-sized lumps of the alum you have produced. (It doesn t matter if the alum is still damp.) Continue to boil the solution gently.

Each student should do the following so that both can have samples for their reports.

- 9. Obtain 2 pieces of cloth 1 inch x 1 inch square. Place one of the small pieces of cloth in the alizarin dye solution. Boil for 30 sec. With forceps, remove the cloth, and rinse it under running water. What happens?
- 10. Now place the other piece of 1 inch x 1 inch cloth in the mordant solution, and boil it for 30 sec. With forceps, remove the cloth from the mordant and transfer it to the alizarin dye solution. Boil it for 30 sec, remove it with forceps and rinse with running water. When the cloth squares are dry attach them to your report. What do you observe?

Name	Section
RESULTS	
Attach the pieces of cloth to your report h	ere.

Sign-out: All equipment is put away, and the student bench is clean and dry.

TA_______Date______

POST LABORATORY QUESTIONS

1.	How is alum used in water treatment plants? Use your text or an encyclopedia for help.
2.	Look at the structural formula for alizarin. What functional groups does it have?
3.	What is the purpose of a <i>mordant</i> in dyeing?