Name: _____   Lab Room _____   Locker _____
(cf. Honesty Declaration Statement on page 20)

Laboratory Day (circle)					Lab. Session (circle)	Lab. Section
Mon.	Tues.	Wed.	Thurs.	Fri.	AM or PM	<u>B_____</u>

Date experiment is performed _____

<div style="border:1px solid">

MARK: /6

</div>

Pre-Lab for: RECYCLING OF ALUMINUM – SYNTHESIS OF ALUM

Submit to your T.A. **BEFORE** *you begin the experiment.*
Submit your report at the end of the lab session for full credit.
Late labs will be penalized.

1.   Prepare a simplified flow diagram that would be useful to perform this experiment. You may use a copy of this diagram to carry out CExp4 lab. **Note:** if you are not familiar with this planning technique, have a look on the example given on the CHEM 1300 Laboratory Program WebCT site: "**How to get prepared!**"   (2)

2. You are expected to use a 35.0-cm^2 piece of aluminum from a used aluminum beverage can. What is the average thickness of such beverage cans if you have 1.00 gram of metal from this piece?

(2)

3. A Buchner funnel is used in both Part I and II filtrations. A Buchner funnel is normally used to recover a solid after a process. In which part (I or II) will you use the Buchner funnel to recover a solid and what should be this solid?

(1)

4. A student used an aluminum sample of 1.50 grams. What is the minimum volume of a 3.00 M KOH solution that would be required to stoichiometrically react with this mass of Al?

(1)

RECYCLING OF ALUMINUM – SYNTHESIS OF ALUM
(or "Let's review some High School chemistry: Stoichiometry")

This is your last CExp laboratory session. You will need an aluminum can (common beverage can) to use as the basic raw material for the synthesis of alum. You will work with a partner due to limitations of equipment (NO TEAMS OF THREE STUDENTS ARE ALLOWED, UNLESS APPROVED BY LAB SUPERVISOR), but you must INDIVIDUALLY complete your laboratory report within the laboratory session (report is due by the end of today's laboratory session). Therefore, you MUST be very well prepared PRIOR to coming to the laboratory. The Pre-lab exercise MUST be handed in to your T.A. when you enter the laboratory room.
CExp T.A. and PROGRAM EVALUATION WILL BE DONE AT THE END OF THIS LAB. SESSION

RECYCLING OF ALUMINUM (INTRODUCTION)

An area of growing interest is recycling of commercial products to conserve resources, minimize waste disposal and prevent pollution of the environment. Recycling of aluminum has proven to be of significant benefit. The collected waste aluminum is generally recycled by melting (aluminum melts at 660 °C), removing impurities and recasting it into new products. While aluminum is the most abundant metal in the earth's crust, it is usually found as the hydrated oxide ore, bauxite, $Al_2O_3 \bullet xH_2O$ (usually written $Al_2O_3(H_2O)_x$). Before the development of the Hall-Héroult electrolysis process in 1886, aluminum was very difficult to isolate. In fact, it was so precious that it was generally used only in jewelry. Even today, it is much cheaper to recycle aluminum than to produce it from its ore.

Alum [potassium aluminum sulfate dodecahydrate, $KAl(SO_4)_2 \bullet 12H_2O$], a compound of aluminum, is widely used in dyeing and printing fabrics, to remove suspended material from water, to clarify sugar during processing, and even in many baking powders. It is readily prepared from waste aluminum after appropriate purification.

Check Moore, Stanitski, & Jurs, *Chemistry the Molecular Science*, 2nd Edition, pages 1015-1017, for additional background information on processes and reactions involving aluminum.

In this experiment you will take advantage of the reactivity of aluminum metal to make alum. You convert the aluminum in a series of steps; the overall reaction may be represented as:

$$2\ Al(s) + 2\ KOH(aq) + 4\ H_2SO_4(aq) + 22\ H_2O \rightarrow 2\ KAl(SO_4)_2 \bullet 12H_2O + 3\ H_2(g) \qquad (1)$$
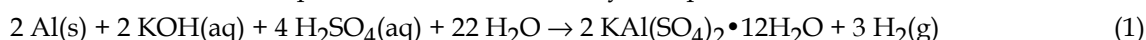
The actual reaction involves two major steps, the first being the reaction with a strong base to form the aluminate complex ion, $Al(OH)_4^-$: (Note: this reaction is described on pages 846-847 of your textbook, with accompanying pictures.)

$$2\ Al(s) + 2\ OH^-(aq) + 6\ H_2O \rightarrow\ 2\ Al(OH)_4^-(aq) + 3\ H_2(g). \qquad \text{(Process A)}$$
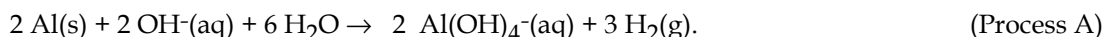
Any excess base is neutralized by the sulfuric acid, which then reacts with the soluble aluminate ion. The potassium ions in solution are electrostatically attracted to the anions, producing a hydrated salt.

$$K^+ + Al(OH)_4^-(aq) + 2\ H_2SO_4(aq) + 8\ H_2O \rightarrow\ KAl(SO_4)_2 \bullet 12H_2O \qquad \text{(Process B)}$$
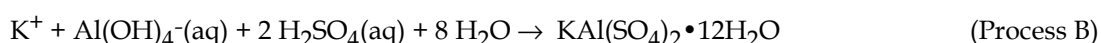
Crystallization is a widely used process for preparing and purifying solid compounds, both commercially and in the laboratory. Most compounds are more soluble in hot solvents than in cold solvents. An impure solid compound, when dissolved in the proper amount of an appropriate solvent at an elevated temperature, will re-precipitate when the solution is cooled. If the hot solution is filtered before being allowed to cool, dirt, lint, or other insoluble impurities will be removed, and the crystals that deposit in the cooled solution usually will be more nearly pure than the starting material. These crystals may then be removed from the filtrate (mother liquor) by filtration. The soluble impurities and a small amount of the desired substance will stay in solution. This process is called recrystallization and is frequently used for purifying solid compounds.

The success of the process depends on the fact that soluble impurities usually are present in smaller amounts than the desired compound, so that the cooled solution, although saturated with respect to the desired product, may not be saturated with the impurity. If this is the case, the impurity will not precipitate from the

solution. Sometimes a solution does become saturated with respect to an impurity during the cooling process. In this event, the impurity deposits along with the desired product and the recrystallization will have to be repeated, perhaps several times. In each recrystallization, a small amount of the desired compound remains behind in the mother liquor. Such losses are unavoidable if a pure product is to be obtained.

A good solvent for recrystallization has the following properties:

   i)   It dissolves a reasonable amount of the desired compound at high temperature (usually near the boiling temperature of the solvent) and very little at low temperature.

   ii) It either dissolves impurities readily at low temperature or does not dissolve them at all.

   iii) It does not react with the substance being purified.

   iv) It is readily removed from the purified product.

The process of recrystallization often may be aided, especially when the impurities are colored, by selectively adsorbing contaminants on activated charcoal. A small amount of charcoal is added to the hot solution just before the filtration step. However, charcoal will adsorb not only the impurities but also a certain amount of the desired product. You should therefore use a minimum of charcoal.

The size of crystals is affected greatly by the rate of cooling and the amount of stirring during crystallization. Rapid cooling by immersion in ice water will tend to form small crystals which may adsorb impurities from solution on their large surface area. Stirring a solution during cooling will also lead to small crystals. Slow cooling with no stirring of the solution will often result in large crystals. However, large crystals may occlude (trap) solvent and impurities within the crystals.

Sometimes crystals do not form on cooling a solution. The solution is then said to be supersaturated with respect to the solute. Crystallization can usually be induced by seeding — a small crystal of the original solid is added to the cooled solution to provide a nucleus on which crystallization can begin; crystallization then occurs quite rapidly. Scratching the inside walls of the flask in the liquid, near the surface of the solution, with a glass rod will often induce crystallization from a super-saturated solution.

Reporting yield

It is important to determine the percent (%) yield in a chemical process. The stoichiometry of the reaction and the amounts of reactants available dictate the maximum amount of product that can be obtained. The limiting reagent is the reactant which is consumed completely, even though the amount of another component could remain in excess. (See your textbook, section 4.5.) It is best to determine the molar quantities available and examine the stoichiometry to determine which reactant runs out and limits the continuation of product formation. A 100% theoretical yield assumes that the entire available limiting reagent is converted to product. (See your textbook, section 4.6.)

If you start with exactly 1.000 g of pure aluminum in your alum synthesis, and assume all other reactants are in excess, what is the maximum amount of alum that could be produced, assuming complete reaction occurred?

According to equation (1) for the overall reaction, 2 mol of Al produce 2 mol alum (*i.e.*, a 1:1 ratio),

With the respective molar masses: Al = 26.9815 g/mol   and $KAl(SO_4)_2 \cdot 12H_2O$ = 474.384 g/mol

one gram (1.000 g) of pure aluminum should produce a theoretical yield of

$$\left(\frac{1.000 \text{ g Al}}{26.982 \text{ g/mol Al}} \right) \times 474.384 \text{ g/ mol alum } = 17.58 \text{ g of alum}$$

However, it is not possible to obtain a stoichiometric yield in our laboratories. A practical way to report an individual yield is as a percent of the theoretical yield.

For example, if you recovered 8.247 g of alum from 1.000 g of aluminum starting material, what is the percent yield?

Since the theoretical yield for 1.000 g starting material is 17.58 g of alum (calculation above)

$$\text{Percent yield } = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100; \qquad \text{substituting gives } \frac{8.247 \text{ g alum}}{17.58 \text{ g alum}} \times 100 = 46.9\%$$

PROCEDURES:

All safety regulations must be strictly observed. Remember **safety glasses must be worn** *while in the laboratory.*

I.   **Synthesis of Alum**

- **Obtain** about 1 gram of aluminum by cutting a 35 cm^2 piece of metal from a used aluminum beverage container. **Scrape** off as much paint from the outside of the can and plastic coating from the inside of the can as is reasonable (the paint is just decorative advertising but the inside plastic coating prevents the soft drink from dissolving the reactive aluminum). To save lab time you may do this before coming to this experiment.

- **Find** the mass of your weighing bottle. Now **cut** the aluminum into tiny pieces, **drop** them into the weighing bottle, and **determine** the exact mass of aluminum used as starting material. Very small pieces will react faster and more completely because more surface area is exposed to the potassium hydroxide reactant.

- **Place** these aluminum clippings into a labeled 250 mL beaker and **take** it to the FUME HOOD. **Add** 20 mL of 3 M potassium hydroxide, KOH, solution. **Warm** the mixture gently on a hot plate. Once the exothermic reaction is started it should go without addition of heat. **Do not boil the solution**. **Allow** the evolving dihydrogen gas to be safely vented up the hood.

- **Prepare** a Buchner funnel assembly (Figure 1). **Make sure it is clean and securely clamped to the stand**. **Place** a circle of filter paper, pre-cut to fit exactly, into the Buchner funnel and **moisten** the paper with a few mL of water. **Place** the Buchner funnel with attached rubber stopper on the top of a suction filtering flask. **Attach** the flask by a thick-walled rubber hose, through a trap (to prevent liquid from entering the vacuum line and causing damage), to the vacuum line (Figure 2). When the reaction is complete (no more bubbles), even though the solution may still be dark and have decomposed paint or plastic, **pour** the warm solution into the Buchner funnel and **adjust** the degree of suction with the vacuum control valve. **Rinse** the beaker with about 5 mL of distilled water (**not more!**) and **add** this rinse water to the filter paper. If the filtrate is not clear, **add** a pinch of charcoal to it and **swirl** the flask. **Put** a new piece of filter paper in the Buchner funnel and **filter** the mixture again.

- **Transfer** the filtrate to a clean 250 mL beaker. **Use** about 5 mL of distilled water (**not more!**) to rinse solution from the flask walls and also **transfer** this to the beaker. **Slowly and carefully add** 20 mL of 9 M H_2SO_4 with constant stirring. As you proceed you should notice heat evolved from the neutralization of unreacted hydroxide ions and formation of a precipitate of $Al(OH)_3$. Keep going. By the time most of the sulfuric acid is added, much of the solid should have dissolved. You may stop adding acid once the solution is clear, but if solid is present after 20 mL of acid are added, keep stirring and warm the beaker on a hot plate. To check that acidification is complete, touch your wet stirring rod to a piece of blue litmus paper; it should turn red. All of the solid should be dissolved or the next crystallization step will be unsatisfactory. If all else fails, filter the solution again.
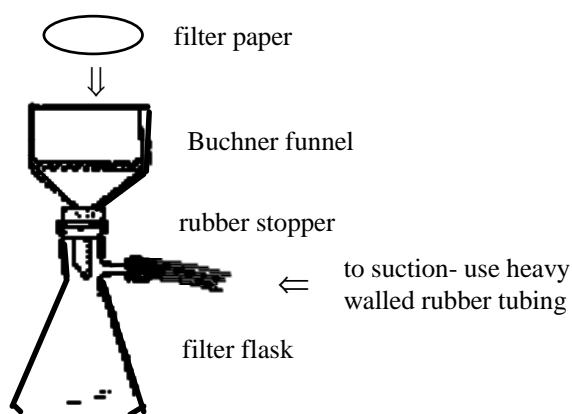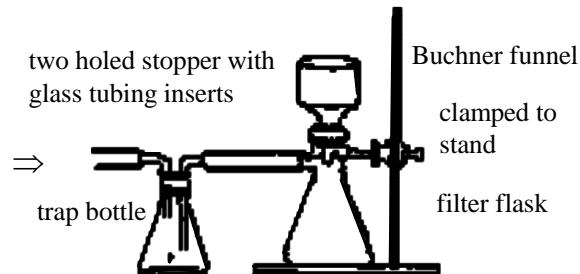
Figure 1: Buchner Funnel assembly   Figure 2: Vacuum Filtration

filter paper

Buchner funnel

rubber stopper

to suction- use heavy
walled rubber tubing

filter flask

two holed stopper with
glass tubing inserts

Buchner funnel

clamped to
stand

trap bottle

filter flask

II.   Isolation of Alum by Crystallization.

- **Prepare** an ice bath in a pan or large beaker. **Set** the 250 mL beaker containing your warm alum solution in the ice bath and **leave** it undisturbed for 10 minutes. **Observe** the solution for crystal formation. **Consult** your T.A. if no crystals begin to form within about 15 minutes. Alum readily forms supersaturated solutions and, to initiate crystallization, you may have to scratch the sides of your beaker with a glass rod.

- **Clean** and **reassemble** the filtering system. **Filter** the alum crystals from the solution. **Scrape** stuck crystals onto the paper, then **rinse** the beaker with about 5 mL of ethanol and **pour** this through your crystals trapped on the filter paper in the Buchner funnel. **Allow** air to continue to flow through the crystals for 5 minutes.

- **Remove** the filter paper containing your alum. If you turn off the suction and remove the Buchner funnel, once you use a scoopula to loosen the edges of the paper, you can simply invert the assembly onto a large filter paper and the filter paper with alum should fall out with a little tap. **Place** a second large piece of filter paper on top of the crystals. **Dry** the alum as best you can by pressing it between these papers. **Transfer** the alum to your weighing bottle or a small beaker and **determine** the mass of product.

- **Clean** up all equipment, disposing of paper in the waste basket and your solid alum product in the "Alum Recovery Container".

CLEAN YOUR BENCH TOP
BEFORE LEAVING THE LABORATORY

CExp T.A. Evaluation and Laboratory Program Survey Questionnaires are on next pages (pages 65 and 66). Answer these questions using the bubble sheet provided by laboratory supervisor.

(*cf.* **Honesty Declaration Statement on page 20**)

Laboratory Day (circle)					**Lab. Session (circle)**	**Lab. Section**
Mon.	**Tues.**	**Wed.**	**Thurs.**	**Fri.**	**AM or PM**	**B_____**

Date experiment is performed _____   **T.A. Signature**: _____

MARK:	**/16**

RECYCLING OF ALUMINUM – SYNTHESIS OF ALUM

DATA and RESULTS: Record information in Tables 1 and 2 or in the space provided.

TABLE 1.   (mark)

Mass of aluminum and weighing bottle	g
Mass of weighing bottle	g
Mass of aluminum reactant	g

(2)

Observations about starting materials and Process A.

(2)

TABLE 2.

Mass of alum and weighing bottle	g
Mass of weighing bottle	g
Mass of alum product	g

(2)

Observations about product and Process B.

(2)

Determination of your percent yield (show all calculations, including proof that the limiting reagent is aluminum):

(3)

DISCUSSION (see guidelines on page 16 of Preface)

(3)

CONCLUSION (see guidelines on page 16 of Preface)

(2)