CHEM 215 Lab #4 - Determination of Cationic Concentration (rev 10/2005)
Principals of Ion Exchange Chromatography

Reagents provided:

- 1.0 - 1.5M HCl: about 100 mL per student (provided by the instructor)
- 0.1XXX M NaOH (Prepared and Standardized by the student)
- 0.3xxx M NaCl: (Prepared by the student).

Introduction:

In this experiment a cation-exchange resin in the acid form is used to determine the total milliequivalents of a **cation** in a sample. When a salt solution is passed through a column of this resin, cations in solution exchange with hydrogen ions on the resin to replace the ions initially present with H^+. (The cations have a greater affinity for the resin than the H^+.)The H^+ ions are then washed from the column (eluted), collected quantitatively, and titrated with standard sodium hydroxide. The amount of NaOH required is a direct measure of the amount of all cations present in the original sample.

1. The resin must be converted initially to the acid form. This conversion process, called <u>regeneration</u>, is accomplished by passing a high concentration of hydrochloric acid through the column. The reaction is:

$$1M^{n+}(R^-)_n + nH^+ \text{ ---> } nH^+R^- + M^{n+}$$

where R^- is an exchange site on the resin. Although the resin has a greater affinity for the metal ion than for hydrogen ions, the use of a large volume of moderately concentrated acid permits quantitative conversion of the resin to the resin in the acidic form.

2. Before a sample is placed on a column, the excess acid must be washed from the column to prevent its being titrated along with the H^+ that is released when the sample is passes through the column.

Procedure:

Obtain a plastic chromatography column. Fill it with DI H_2O and test for leaks. Drain off all the water except for about 1 mL. "Pack" the chromatography column using a slurry consisting of 10 - 11 grams of resin with about 10 mL of DI H_2O. Add this slurry to the column. If it cannot all be added at once, allow the resin to settle, then drain off the excess H_2O and add the remaining slurry. Regenerate the column by pouring 100 mL of 1.5 M HCl through the column. <u>Do not allow the level of solution to fall below the surface of the resin</u>. After adding all of the 100 mL of HCl, <u>pass deionized water</u> through the column until the effluent is neutral. (To test for neutrality, collect about 20 mL of effluent (the liquid which is currently dripping out the column), add 1 drop of phenolphthalein indicator then 1 drop of 0.10 M NaOH. If the effluent is neutral 1 drop of NaOH is sufficient to change the indicator to its alkaline color. The process usually takes about 150 mL of distilled water to to reach neutrality.)

The column can store H^+ in two places, attached to the beads (not in solution) or between the beads (in solution). Once all the sites for H^+ are full, you must rinse all of the excess H^+ (that which is between the beads) from the column.

Once a **cationic sample** is added to the column, each H^+ ion that comes off the column is released because a metal ion (eg. Na^+ or Mg^{2+}) was exchanged for its place on a bead. The total number of H^+ ions collected is directly related to the total number of cations (and their charge) in the sample--3 H^+ for each M^{3+}, 2 H^+ for each M^{2+}, etc.

Part I. Standardization of NaOH

Standardize your ~0.1 M NaOH with 4 samples of KHP and determine its molarity to 4 significant figures following procedures from the two previous experiments. Record data in your notebook.

Part II. Preparation of a 0.300 M NaCl solution

Calculate the approximate amount of NaCl needed to prepare a 0.3XXX M solution of NaCl in a 100 mL volumetric flask. Using a weighing dish, carefully weigh the amount of NaCl needed and record its mass to the nearest ± 0.1 mg. Transfer the NaCl quantitatively to a 100 mL volumetric flask. Dissolve with distilled water and then add more water until the meniscus is at the mark on the flask. Seal with parafilm or a stopper and invert (about 30 times) to homogenize.

Part III. Determining the Sodium Concentration by Ion Exchange

Fill a buret with standardized NaOH solution and record your initial volume. Next, pipet 5.00 mL of your solution (0.3 M NaCl) onto your (previously regenerated) column. **Collect all of the effluent in a clean, 250 mL flask!**

Allow the solution to drain to just above the top of the resin. Wash the sample "onto" the column with 5 mL to 10 mL of distilled water. Continue washing with water at an effluent flow rate of about 1 drop per second. Follow the sample with distilled water until you have collected about 75 mL of effluent in the 250 mL flask. Afterwards, test your effluent by collecting a 20 mL portion in a small beaker, adding one drop of phenolphthalein and and one drop of NaOH as before.**(This time take the drop of NaOH from your buret for which you have taken an initial volume measurement!)**

If the test portion is still acidic, pour it into the collection flask. You do not want to lose excess H^+! Continue washing the column with 10 to 25 mL of water and test again. When the effluent is neutral, add several drops of phenolphthalein indicator to the total effluent **(including all test portions)**, and titrate with standard 0.1xxx M NaOH solution.

Repeat the procedure above for three more samples of the NaCl solution. (There is no need to regenerate the column between trials.)

Questions for thought:

1. If a 5.00 mL aliquot of a Ca^{2+} solution were passed through an ion exchange column, and the eluted solution required 25.24 mL of 0.0500 M NaOH to titrate to the endpoint, what would be the concentration of the original Ca^{2+} solution?

2. A solution determined to be 0.125 M Mg^{2+} was accidently contaminated with Na^{+} ions. If a 5.00 mL aliquot of this contaminated solution were passed through an ion exchange column, and the eluted solution required 20.02 mL of 0.0801 M NaOH to titrate to the endpoint, what would be the concentration of the Na^{+} in the contaminated solution? (Assume the contamination did not change the volume of the original solution.)

3. What ions would be in the eluted solution resulting from passing 3.00 mL of 0.300 M NaOH through the ion exchange column?