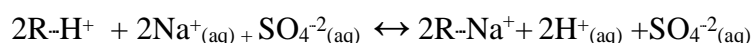


# Determination of the Exchange Capacity of a Cation Ion-Exchange Resin

## Introduction

Ion exchange is a process in which ions attached to a high molecular weight polymer are exchanged for other ions in solution. The high molecular weight polymer is normally in the form of small, round spheres called beads. Ion exchange resins are used to demineralize water and to separate mixtures of ions. One important characteristic of ion exchange resin is its capacity, expressed in terms of milliequivalents of exchangeable ion per gram of resin.

Exchange capacity can be measured by exchanging sodium ion ( $\text{Na}^+$ ) for hydrogen form ( $\text{H}^+$  initially attached to the resin). The hydrogen ion is then titrated with standard sodium hydroxide solution. The exchange reaction can be represented as:



In the equation, R represents the ion exchange resin. The reaction is an equilibrium process (shown by the double-headed arrow,  $\leftrightarrow$ ) and can be forced to completion by using “concentrated” sodium sulfate solution.

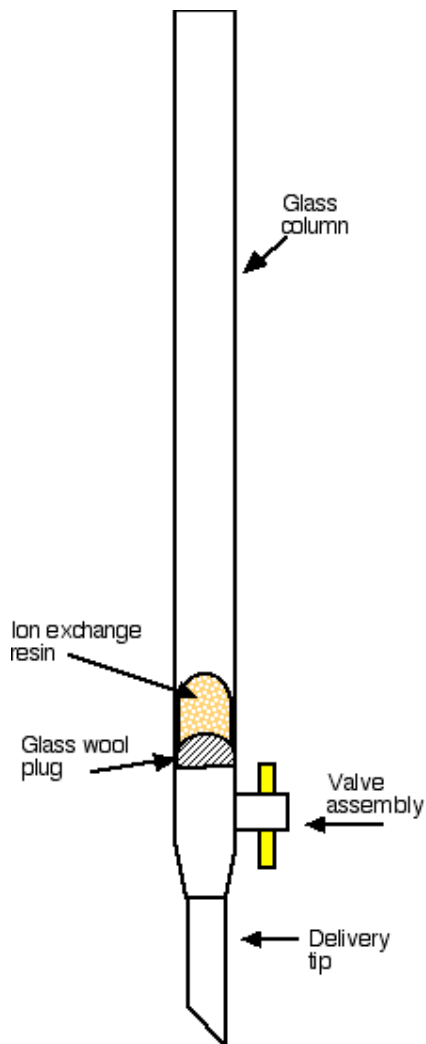
Solutions needed for this experiment:

0.5 M sodium sulfate	Phenolphthalein indicator
0.1000 M sodium hydroxide (standardized)	

Instructions for preparing and standardizing sodium hydroxide solution are found at the end of this experiment.

### Specialized equipment needed:

You will be using a chromatography column (Figure 1). The column is assembled from three pieces; a relatively long glass column, a valve assembly, and a delivery tip. Before using the column, test it for leaks by filling it with water. If it leaks, tell your instructor so that the problem can be corrected before you start the experiment. Place a small plug of glass wool at the bottom of the chromatography column. The glass wool prevents beads of ion exchange resin from plugging the valve assembly.



**Figure 1. Chromatography column assembled and loaded with ion exchange resin.**

## **Experimental Procedure**

### **Air-drying the resin**

1. Transfer ~1 gram of cation exchange resin to a 150 or 250 mL beaker. Cover the beaker with a watch glass, and store it in your equipment locker overnight.

### **2. DO NOT DRY THE RESIN IN THE OVEN!**

### **Loading the chromatography column**

1. Fill the column ~ 2/3 full with deionized water. Remove any air bubbles from the column by gently tapping the sides with a glass stirring rod.
2. Accurately weigh a sample of the air dried resin (~0.9 – 1.0 grams) on the analytical balance, and transfer to a 50 mL beaker.
3. Add about 25 mL of deionized water to the resin, and transfer the resin slurry to the column. Use a wash bottle containing DI water to completely transfer all resin to the column.
4. If necessary, a piece of rubber tubing can be attached to the delivery tip, applying intermittent pressure to the tubing causing the water level in the column to rise and fall, expelling air bubbles. (You will of course have to open the valve for this to work).
5. Once the column has been prepared, adjust the water level so that it is ~ 1 cm above the top of the resin bed. Do NOT allow the water level to fall below the top of the resin bed.

### **Ion exchange**

1. Prepare ~300 mL of 0.5 M sodium sulfate solution by dissolving 21.3 grams of sodium sulfate in ~280 mL DI water.
2. Add ~5 mL of the sodium sulfate solution to the column, and adjust the valve so that the solution passes through the column at a rate of 2-3 mL/minute.
3. Add fresh sodium sulfate solution to the column, in 5 mL portions, just often enough to keep the liquid level from falling below the top of the resin.
4. Collect the effluent (the liquid draining from the bottom of the column) in a 500-mL Erlenmeyer flask. When all 300 mL of sodium sulfate has been passed through the column, pass 50 mL of deionized water through the column, collecting the deionized water in the same flask as the effluent.

## Titration

1. Using standardized 0.1000 M sodium hydroxide solution, titrate the entire contents of the 500-mL Erlenmeyer flask to the phenolphthalein endpoint. NOTE: you get to perform this titration ONCE, so be extremely careful!
2. The exchange capacity, in milliequivalents/gram of resin, is calculated using the formula:

$$Capacity = \frac{mL_{NaOH} \times M_{NaOH}}{Mass_{resin}}$$

## Preparation and standardization of sodium hydroxide solution

1. Dry ~2 grams of potassium hydrogen phthalate (KHP) in the oven for 2 hours at 110 °C. Remove from the oven and store in a desiccator.
2. Prepare 1 liter of 0.1N sodium hydroxide by adding ~500 mL of deionized water to a clean 1 liter plastic bottle. Add either 4.0 grams of solid sodium hydroxide, or 8.0 grams (~5.3 mL) of 50% sodium hydroxide solution (NOT BOTH). Mix the contents until the sodium hydroxide is completely dissolved, and then fill the bottle with deionized water. Cap the bottle and mix by shaking.
3. Using an analytical balance, dispense three 0.5 gram samples of KHP into separate beakers or flasks. Make sure you record the mass of KHP used to four decimal points. Add about 25 mL of water to each sample. The KHP should completely dissolve, but if it doesn't DON'T WORRY ABOUT IT.
4. To each KHP sample, add 2-3 drops of phenolphthalein solution.
5. Titrate each KHP sample with your sodium hydroxide solution to the first, pale pink endpoint that is stable for at least 10 seconds (Figure 1). The entire contents of the beaker/flask must be pale pink, and the palest endpoint possible is the most desirable. Record the volume of sodium hydroxide solution used.
6. Sodium hydroxide concentration is found from the equation:

$$N_{NaOH} = \frac{\frac{grams_{KHP}}{MW_{KHP}}}{mL_{NaOH}}$$

where  $mL_{NaOH}$  are the milliliters of sodium hydroxide needed to reach the phenolphthalein endpoint. The molecular weight of KHP ( $KHC_8H_4O_4$ ) is 204.2212 g/mole. Label your sodium hydroxide solution with the average concentration from three titrations.

### Standardization of NaOH solution

Mass, KHP (grams)			
mL NaOH			
Normality, NaOH			
Average Normality, NaOH			

Exchange capacity data:

Mass of resin used (grams, air dried):

mL NaOH used to titrate:

Normality NaOH:

Exchange capacity:

Calculation:

Exchange capacity =  $(\text{mL}_{\text{NaOH}} \times N_{\text{NaOH}}) / \text{mass}_{\text{Resin}}$

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