

## Separation by ion-exchange chromatography

### 1) Performing the exercise

#### 1.1. Simple separation.

- 2) 5 - 10 drops of 0.1 M  $\text{Cr}(\text{NO}_3)_3$  solution pour into the test tube, add approx.  $2 \text{ cm}^3$  of water and a little cation exchanger by the tip of the spatula, shake several times and wait a few minutes - observe the color changes of the solution and the resin. After separation of two phases, decant the aqueous phase and pour the resin by 2 M  $\text{H}_2\text{SO}_4$  (only available in the fume cupboard - put on the glasses!) and shake again. Observe and note the changes.

#### 1.2. Separation by changing the charge of the ion through complex formation.

2. Prepare a mixture of a little cation exchanger and 5 drops of 0.1 M  $\text{CuSO}_4$  solution. Shake it and wait for a few minutes; then add about 10 drops of 2 M  $\text{NH}_3$  aq (available only in the fume cupboard - put on the glasses!) and mix thoroughly. Observe the cation exchanger and the solution at all times.

**ATTENTION!** After decanting, transfer the ion exchanger to the regenerating vessels.

#### 1.3. Deionization of water.

- By using a measuring cylinder, measure  $50 \text{ cm}^3$  of tap water and pour it into a beaker with a prepared magnetic stirrer. Then add approx.  $5 \text{ cm}^3$  of the buffer solution with  $\text{pH} = 10$  and approx. 0.1 g of the Eriochrome Black indicator - stir the solution slightly by turning on the magnetic stirrer to obtain a homogeneous color. Start the titration by adding 0.005 M EDTA solution from the burette until the color changes from violet to blue. Read the volume of EDTA solution used to titrate the water sample.
- Calculate the hardness of the water sample (formula for water hardness in German degrees  $T = V \times 0.56$  - where V is the volume of 0.005 M EDTA used to titrate  $50 \text{ cm}^3$  of a given water sample).
- **The same test (titration) and water hardness calculations should be made for boiled water and for tap water passed through the ion-exchange stationary phase.**
- Pour  $5 \text{ cm}^3$  of tap water into the first test tube. Into the second test tube pour  $5 \text{ cm}^3$  of tap water passed through ion-exchange resin. To each of the samples, add 2 drops of 2

M HCl and 0.5 M BaCl<sub>2</sub>. Wait and observe the turbidity of the solution which indicate the presence of sulphates.

- Similar to p. 1.3.2. perform a chloride test with a few drops of 0.1 M AgNO<sub>3</sub>. Prepare the report the observations and the results.

## 2. Preparation of the results

Describe the color changes of the resin and solution above the resin, justify them with chemical equations for exercises 1.1. and 1.2.

## 3. Conclusions

## 4. The scope of the material

- The occurrence of the ion-exchange reaction.
- Technique of using ion exchangers.
- Application of ion exchangers in chemical analysis.
- Water hardness - types, units, removal etc.

## 5. Literature

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