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**Exp. 4 Paper Chromatography**

**Purpose:**

* To become familiar with chromatography, a technique for separating the components of a mixture
* To separate a mixture of transition metal cations by paper chromatography

**Materials and Reactions (if any):**

* 2 mL of three unknown solutions
* 2 mL of five known solutions(Mn2+, Fe3+, Co2+, Ni2+, and Cu2+)
* Test tubes ×8
* Capillary tubes× 8
* Chromatographic paper × 1
* Plastic wrap
* 1000-mL beaker × 1
* 600-mL beaker × 1
* 30-mL beaker × 1
* 5 mL of conc NH3
* 10 mL of an eluting solution that consists of 9 mL of acetone and 1 mL of 6 M HCl

**Procedure:**

* **Safety precautions**
  + Acetone is flammable.
  + HCl is corrosive.
  + Do not inhale the ammonia fumes.
  + The heat lamp or hair dryer is hot.

1. **Preparation of Chromatography Apparatus**
   1. **Developing chamber.** Obtain a 600-mL beaker and enough plastic wrap for a cover. In the fume hood, prepare 10 mL of an eluting solution that consists of 9 mL of acetone and 1 mL of 6 M HCl. Pour this eluent into the middle of the beaker using a stirring rod; be careful not to wet the sides of the beaker. The depth of the eluent in the beaker should be 0.75–1.25 cm but less than 1.5 cm. Cover the beaker with the plastic wrap for about 10 minutes. Hereafter, this apparatus is called the developing chamber.
   2. **Ammonia chamber.** Obtain a dry, 1,000-mL beaker and place it in the fume hood. Pour 5 mL of conc NH3 into a 30-mL beaker and position it at the center of the 1,000-mL beaker. Cover the top of the 1,000-mL beaker with plastic wrap.
   3. **Capillary tube.** When a glass capillary tube touches a solution, the solution should be drawn into the tip. When the tip is then touched to a piece of filter paper, it should deliver a micro drop of solution. Try “spotting” a piece of filter paper with a known solution or water until the diameter of the drop is only 2–4 mm.
   4. **Stationary phase.**
      1. Obtain one piece of chromatographic paper. Handle the paper only along its top 20-cm edge and lay it flat on a clean piece of paper, not directly on the lab bench. The chromatographic paper should remain dry and free of skin contact (avoiding skin oil) at all times.
      2. Draw a pencil line 1.5 cm from the bottom 20-cm edge of the paper. Starting 2 cm from the 10-cm edge and along the 1.5-cm line, make eight X’s with a 2-cm separation. Use a pencil to label each X below the 1.5-cm line with the five cations being investigated and unknowns U1, U2, and U3.
2. **Preparation of the Chromatogram**
   1. **Spot the stationary phase with the knowns and unknowns.** Using the capillary tubes “spot” the chromatographic paper at the marked X’s with the five known solutions containing the cations and the three unknown solutions. The micro drop should be 2–4 mm in diameter. Allow the spots to dry. A heat lamp or hair dryer may be used to hasten the drying—do not touch the paper along this bottom edge. Repeat the spotting and drying procedure two more times in order to increase the amount of metal ion at the spot on the chromatographic paper. Be sure to dry the sample between applications.
   2. **Prepare the stationary phase for elution.** Form the chromatographic paper into a cylinder and, near the top, attach the ends with tape, a staple, or small paper clip. Do not allow the two ends of the paper to touch. Be sure the spots are dry and will not come into direct contact with the eluting solution in the developing chamber!
   3. **Develop the chromatogram.** Place the paper cylinder into the developing chamber. The entire “bottom” of the cylindrical chromatographic paper must sit on the bottom of the developing chamber. Do not allow the paper to touch the wall. Make certain that the eluent is below the 1.5-cm line. Replace the plastic wrap. Do not disturb the developing chamber once the paper has been placed inside. When the eluent front has moved to within 1.5 cm of the top of the chromatographic paper, remove the plastic wrap. Measure and record the Deluent on the Report Sheet.
3. **Analysis of the Chromatogram**
   1. **Detection of bands.** Remove the paper from the developing chamber and quickly mark the position of the eluent front with a pencil. Allow the chromatogram to air-dry or gently dry with a heat gun set at a low temperature. While the chromatogram is drying, cover the developing chamber with the plastic wrap. Analyze the paper and circle (with a pencil) any colored bands, those from the solutions containing the known cations and those from the unknown solutions. Record the color for each metal ion on the Report Sheet.
   2. **Enhancement of the chromatogram.** To enhance the appearance and locations of the bands, move the chromatogram to the fume hood. Position the paper in the ammonia chamber and cover the 1,000-mL beaker with the plastic wrap. After the deep blue color of Cu2+ is evident, remove the chromatogram and circle any new transition metal ion bands that appear. Mark the center of each band with a pencil. Allow the chromatogram to dry. Record the color of each metal ion in the ammonia chamber.
   3. **Band enhancement.** The “exact” band positions for the known cations and those of the mixture may be better defined using a second “spot solution.” As necessary, use a capillary tube to spot the center of each band with the corresponding spot solution identified in Table 4.1.
   4. **Analysis of your chromatogram.** Mark the center of each band with a pencil. Measure and record the distance between the origin and the spot for each ion, Dion. Calculate and record the Rf values for each transition metal ion.
   5. **Composition of the unknowns.** Record the Rf value for each band, ions present, etc., in each unknown on the Report Sheet.

**Calculations (if any):**