CHEM 109
Introduction to Chemistry
Revision 2.0

Laboratory Exercise: Separation of Solids

Chemistry is sometimes known as the Science of Separations as substances must be separated from mixtures before they can be properly characterized. In this exercise we will leverage one of the most common methods chemists have for purifying matter; filtration. It is our intent to separate three solids that are mixed together by exploiting the fact that each has a different solubility in Water and selectively filtering off the insoluble solid. The three solids we aim to separate are:

Sand (Silicon Dioxide)
Cupric Sulfate ($CuSO_4$)
Potassium Nitrate (KNO_3)

(Sand)

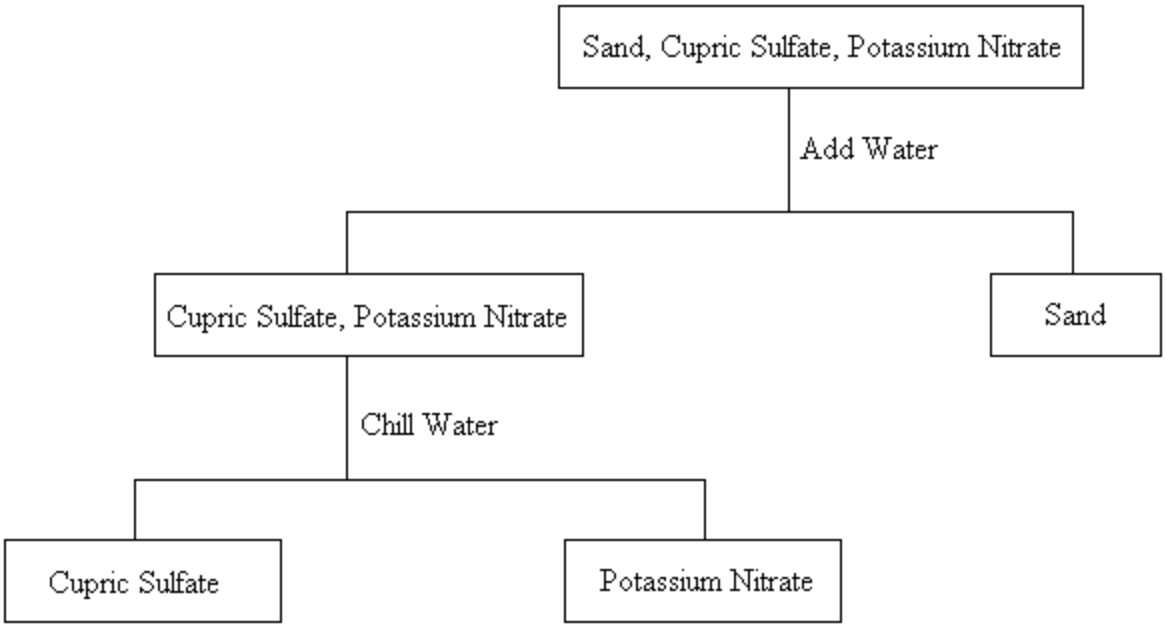
(Cupric Sulfate)

(Potassium Nitrate)

Sand, true to our common experience, is insoluble in Water, whereas both Potassium Nitrate and Cupric Sulfate are soluble in warm Water. However, when cooled to near the Ice-Point, Potassium Nitrate becomes insoluble in Water. Cupric Sulfate remains in solution, even at this cool temperature.

Thus, our separation scheme is to first add Water to the mixture of solids, dissolving the Cupric Sulfate and Potassium Nitrate solids. This leaves solid Sand, which can be filtered using a Buchner full and dried. Excess Water is then boiled off and the resulting aqueous solution is chilled, resulting in the crystallization of Potassium Nitrate. These crystals can be filtered, leaving, predominantly, Cupric Sulfate in solution. If needed, the remaining Water can be boiled off to yield solid Cupric Sulfate as a residue. We will not perform this last step.


Buchner Funnel
Side-Arm Flask
To a Vacuum


Sand, Cupric Sulfate, Potassium Nitrate
Add Water
Cupric Sulfate, Potassium Nitrate
Sand
Chill Water
Cupric Sulfate
Potassium Nitrate

Procedure

All your measurements must be made to the correct number of significant figures and must have the unit of measurement indicated.

1. Obtain ~15g of the sample mixture and make appropriate observations. Weigh this sample accurately in a 150 mL beaker.

2. Add ~30 mL distilled Water.

3. Support your beaker on a piece of wire gauze on an iron ring.

4. Gently heat the mixture to about 40^{o}C while stirring. When all of the blue and white solids have dissolved, pour the mixture through a Buchner funnel and apply suction while using your rubber policeman to transfer all the Sand to the funnel. Wash the Sand with a little distill Water. Continue the suction for a few minutes to dry the Sand. Turn off the suction and use your spatula to lift the filter paper off the funnel. Transfer the Sand, on the filter paper, to a watch glass to continue drying.

5. Add 10 drops of 6M HNO_3 to the filtate, which helps keep the Cupric Sulfate in solution. Transfer the filtrate to a 150 mL beaker.

6. Drop a toothpick or two into your solution. Heat the filtrate in the beaker to its boiling point and then boil gently until white crystals of KNO_3 are visible. (Do not heat too strongly or the system will tend to bump.) Stop heating and add 3 mL distilled Water. Stir the mixture until all the solids re-dissolve. (You may warm the solution if necessary, but do not allow the system to come to a boil.)

7. Cool the system to Room Temperature and then allow it to stand in an Ice Bath. White crystals of KNO_3 will come out of solution.

8. Stir the slurry for a few minutes at 0^{o}C. Reassemble the Buchner funnel and chill it by adding 100 mL of ice cold Water, and after about a minute, draw the Water through the funnel using suction. Filter the KNO_3 from the slurry. Use your rubber policeman to transfer the last of the KNO_3 crystals to the funnel. Continue to apply suction for about 30 seconds. Turn off the suction. Add a <u>small</u> amount of ice cold Water; just enough to wet the crystals. Allow to stand for 10 seconds and then apply the suction for about a minute. Lift the filter paper, with the crystals, out of the funnel and place on a watch glass to dry.

9. The aqueous Cupric Sulfate solution can now be discarded.

10. To recrystallize, and hence purify your KNO_3, place your crystals in a 100 mL beaker and add 8 mL boiling Water and dissolve all the crystals. Now allow the solution to come to Room Temperature and then cool in an Ice bath. Allow the KNO_3 to recrystallize for a few minutes. Filter the crystals as before.

11. Allow the crystals of KNO_3 to dry.

12. Weigh out the dried Sand.

13. Determine the percentage Sand in the original mixture.

Data Sheet

Obs. of Original Mixture:

Mass Original Mixture: _____

Obs. During Separation Procedure (Include Obs of Final Solids):

Mass Dried Sand: _____

Name: _____ Date: _____

Signature: _____

Data Analysis

Determine the Percentage Sand in the Original Mixture:

Why do we not also Determine the Percentage Potassium Nitrate and Cupric Sulfate in the Original Mixture?

Post Lab Questions

1. Is our original mixture of solids Heterogeneous or Homogeneous? Explain.

2. After the sand has been filtered off, is the remaining liquid a Heterogeneous or Homogeneous system? Explain.

3. During the 1980's a new form of Carbon, Buckminsterfullerene (C_{60}), was discovered in the sooty residue present on Carbon electrodes used in creating an electric arc. How was Buckminsterfullerene originally isolated from this soot? (An internet search is required.)