Fractional Crystallization

Purpose:
The purpose of this experiment is to quantitatively separate a mixture of sand, potassium nitrate (KNO₃), and copper sulfate pentahydrate (CuSO₄·5H₂O) based on their solubilities.

Please Read:
Review the sections on your lab page entitled “Bunsen Burner” and “Analytical Balance”.

Introduction:
One of the most important problems faced by chemists is that of obtaining usable amounts of pure compounds from mixtures. The purpose of obtaining the compound may be to determine its identity, or to use it for some purpose. Many compounds are obtained from mixtures which occur in nature. These range from gold and other minerals that occur in mixed deposits to drugs that are isolated from plants.

The techniques used to isolate one or more components of a mixture vary, but most are based in some way on the fact that different components of the mixture will have different solubilities. Some materials are essentially insoluble in any solvent, some compounds are soluble in water, and others are soluble in organic solvents such as alcohol, acetone, or toluene. Often, a compound is more soluble in hot solvent than in cold solvent.

In this experiment, you will separate a mixture of sand, potassium nitrate (KNO₃), and copper sulfate pentahydrate (CuSO₄·5H₂O). The sand is not soluble in water. As shown in the diagram below, both potassium nitrate and copper sulfate are soluble in water, with their solubilities increasing substantially as the temperature of the water increases.

The mixture contains roughly equal amounts of sand and potassium nitrate, with a small amount of copper sulfate present as an impurity. The sand is not soluble in water, so it can be removed by
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dissolving the other two components in water, and filtering out the sand. Then some of the water is boiled away, and the solution is cooled in ice water. The potassium nitrate is not very soluble in cold water, so it crystallizes. The white crystals can be filtered away from the blue copper sulfate, which remains in solution. Separation of a compound from impurities by this method is called fractional crystallization.

Safety and Waste Disposal

You will be working with a Bunsen Burner and hot liquids. Keep flammable materials away from the burner, and use caution when working with the beaker of hot solution.

The potassium nitrate and copper sulfate are somewhat toxic. Keep your hands away from your face while performing the experiment, and wash them well after you have finished, or if you spill solution on them.

Waste sand, potassium nitrate, and copper sulfate solution should be disposed of in the waste containers in the supply area.

Procedure:

1. Obtain from the supply area a Buchner funnel, a suction flask, a large rubber stopper, 2 pieces of filter paper, a watch glass, a digital thermometer, and a sample of unknown solid mixture. Put your initials on the filter papers, in pencil.

2. Weigh a 150 mL beaker and the watch glass, one item at a time, to the nearest 0.001 gram. Add your sample to the 150 mL beaker and weigh it again. Use the masses as needed in the calculations. Record the unknown number.

3. Set up a ring stand for heating with a Bunsen burner; an iron ring should be clamped to the stand. The top of the burner should be about 3 cm below the bottom of the ring. This will allow the burner flame to be at optimal height. Place a wire gauze on the ring.

4. Prepare the apparatus for suction filtration. Clamp a filtration flask to another ring stand. Attach a vacuum hose to the side-arm of the flask. Place one of your filter papers, initials down, in a Buchner funnel. Place a rubber seal in the flask, and put the Buchner funnel in the seal. Connect the vacuum hose to the aspirator (side-arm) of a water spigot.

5. Add distilled water to the 40 mL mark on the beaker containing the sample. This will be enough to dissolve the soluble solids. Touch the beaker and note the temperature change as the sample dissolves.

6. Light your Bunsen burner and adjust the flame so it is blue, quiet, and of moderate size.

Part A: Separation of Sand

1. Place the beaker on the wire gauze above the burner. Warm the mixture gently to about 50°C with stirring. Don’t let the mixture get too hot, or you won’t be able to touch the beaker. Move the burner away from the beaker if the temperature rises too much. The purpose of heating is to make the KNO₃ and CuSO₄ dissolve more quickly. Heat the mixture until the blue and white solids are all dissolved.

2. Turn on the water to your aspirator. Open the valve fully. Use a squirt bottle to moisten the filter paper in the Buchner funnel. The suction should pull the paper firmly against the bottom of the funnel.

3. Swirl the beaker to suspend the sand, and pour the contents of the beaker into the funnel. Use your spatula to scrape as much additional sand as possible from the beaker into the funnel.
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4. Using a squirt bottle, rinse the beaker into the Buchner funnel with a minimum of distilled water to transfer any remaining sand into the funnel. When the sand is as dry as suction will get it, release the vacuum by gently rocking the funnel in its collar. Then turn off the aspirator.

5. Put the watch glass over the funnel, then invert them. Use your spatula to pull the filter paper, with the sand, into the watch glass. The paper will be on top of the sand. Use your spatula to transfer any sand which remains on the sides of the funnel to the watch glass, beside the main filter cake.

6. Dry the watch glass, sand, and filter paper in the oven. Note that the filter paper serves as a label so you can retrieve your own material. When it is dry, remove the paper, keeping all the sand on the watch glass. Weigh the glass and sand once the glass is at room temperature.

7. Calculate the masses of unknown and sand, and the percentage of sand in the unknown.

Part B: Separation of KNO₃

1. While the sand is drying, clean the 150 mL beaker and transfer the blue filtrate to it. Pour the contents of the filter flask into the beaker, but don’t transfer any sand which may have come through. Rinse the flask with a minimum amount of distilled water and pour the rinse into the beaker, again keeping any sand in the flask.

2. Add about 15 drops of 6 M HNO₃ (nitric acid) to the solution.

3. Heat the solution in the beaker to the boiling point, then boil gently until the volume of the liquid is at the 20 mL mark on the beaker. Put a stirring rod into the solution, as this usually helps the solution to boil smoothly. Gentle stirring may also be helpful.

4. When the volume of the solution at the 20 mL mark on the beaker, extinguish the burner, remove the stirring rod, and allow the solution to cool to room temperature. Don’t move or stir the solution at this stage, but watch for crystals to appear and grow. When no more crystals appear, and the mixture has reached room temperature (touch the bottom of the 150 mL beaker to check this), cool the mixture further in an ice bath for about 5 minutes.

5. While the solution is cooling, assemble the filter flask, Buchner funnel, and filter paper as before. Remember to label the filter paper in pencil, then wet it and use suction to pull it tightly against the bottom of the funnel.

6. Filter the crystals of KNO₃ in the same way you did the sand, using your spatula to transfer all the solid from the beaker. Press them down evenly with the wide end of the rubber stopper. Use suction to dry the crystals as much as possible. Then break suction by rocking the funnel and turn off the aspirator.

7. Transfer the crystals and filter paper to the watch glass. Dry them in the oven, remove the filter paper, and weigh them on the watch glass.

8. Calculate the mass of KNO₃ in your unknown, and the percentage. Determine the mass and percentage of CuSO₄ · 5 H₂O by difference.

9. Have your work checked by your instructor, then discard the KNO₃ solid and the CuSO₄ solution in the proper waste containers.

10. Wash the flask, funnel (take it apart first), and watch glass with detergent and water, then give them a final rinse with distilled water. Dry the funnel, the watch glass, and the outsides of the flasks. Return the apparatus to the supply area.

11. Wash your beaker, stirring rod, and spatula, then put them in your drawer.

12. Make sure the digital thermometer is off. Rinse its stem with water and dry it with a paper towel, then put the plastic shield back on the stem and return it to the supply area.
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