The Messy Chemist: Separating a Solid Mixture

**Problem Scenario:**

A chemist working at SunCo, a solar energy start-up company in Washington, synthesized a new Fe complex as a dye for solar cells that has shown record efficiencies in laboratory tests. Unfortunately, the scientist who developed the dye was marooned on a remote tropical island due to a cruise ship mishap. Luckily a large drum of the new dye was discovered in the chemist’s workspace, but due to the experimental procedure used to synthesize N1986; NaCl, 2,2’-bipyridine, and sand are mixed with the compound. However, the lab director knows that the dye complex is an Fe2+ surrounded by three bipyridines and is the chloride salt, [Fe(bpy)3](Cl)2. You have been tasked with designing a cost effective procedure for handling N1986 separation and recovery and hazardous waste disposal.

Here are the three criteria your scheme should meet:

1. The N1986 must be isolated in the SOLID form.
2. The 2,2’-bipyridine should also be isolated as a pure SOLID.
3. Quantities of materials used in the separation process should be kept to a minimum.

**Materials and Chemicals Needed**

1. Samples of test solvents: water, hexanes, and methanol
2. Pure samples of the four substances in the mixture: sand, NaCl, 2,2’-bipyridine, and N1986
3. NH4PF6
4. 9-10 small test tubes and test tube rack
5. Marker for labeling test solutions
6. Stirring rods
7. Spatulas
8. Three 10-mL graduated cylinders (one for each solvent)
9. Two 100-mL beakers
10. Other equipment dependent upon the procedure you develop

**Safety, Handling, and Disposal**

Methanol and hexane are flammable, keep away from flames and heat sources. In addition, these two chemicals, along with 2,2’-bipyridine and N1986, are respiratory hazards and skin irritants and should be handled appropriately. Observe all laboratory safety precautions. Refer to MSDSs for chemical information.

Dispose of all waste materials in the labeled containers located in the lab hoods. DO NOT DUMP ANY CHEMICALS DOWN THE DRAIN! The waste in the containers will be disposed of according to local ordinances.

**Procedure**

Since this is an inquiry-based lab, the directions are not as specific as those you may be used to in other labs.

Part 1 – Qualitative Investigation of Solubility

A colleague of yours suggests you explore the solubility of the components in various solvents. A standard microscale procedure recommends using less than 0.15 g of solid solute and less than 3 mL of solvent for the solubility test. The procedure contains a cautionary statement that some solids may take longer than others to dissolve and may require some agitation (stirring or shaking) for complete dissolution. In addition, some materials may exhibit only partial solubility.

Conduct your analysis of the solubility of the four solutes in each of the three solvents and record your observations in a data table.

Based on your observations, answer the following questions in your notebook.

1. Suppose you want to separate the N1986 form a N1986/sand mixture. What solvent would you recommend to dissolve only one of the two solids? Will that same solvent be useful in separating a NaCl and N1986 mixture? Why or why not?
2. Based on your observations, what solvent would allow you to separate a sand/2,2’-bipyridine mixture? Would that same solvent be useful in separating a 2,2’-bipyridine and N1986 mixture? Why or why not?
3. Once you have dissolved a component of a mixture, you will then have an undissolved solid and a liquid composed of solvent and dissolved solute. What experimental technique can be used to separate the solid from the liquid phase?
4. After the solid phase has been removed from the liquid solution, how will you separate the solvent from the dissolved solute?
5. Make a 0.1 M aqueous solution of NH4PF6. Add a few drops this solution to any of the solutions of the mixture components that were soluble in water. Record any observations in your notebook.
6. Did a reaction occur with any of the components of the mixture and NH4PF6? If so, write out a chemical equation to explain the reaction (think about cation and anion solubility rules).
7. Consider your answers to the above questions and then briefly outline the steps you propose using to separate and dispose of the components of the mixture. Remember, this is initially a solid mixture of sand, NaCl, 2,2’-bipyridine, and N1986. We will compare the different separation schemes with the rest of the class at the end of the laboratory session. Show your procedure to a TA or teacher to receive Part 2.

Part 2 – Quantitating Solubility

At this point you have a basic plan for separating the substances from each other, but how much solvent is needed to dissolve each solute? You should determine the smallest quantity of solvent needed to dissolve a known quantity of solute. One approach to finding this relationship is to take a measured quantity of solid solute (about 0.5 g) and add solvent in 1 mL increments until all of the solute dissolves. This should allow you to get a fairly good estimate of the solubility of the solute in that particular solvent system (# grams solute/1 mL of solvent).

1. Which solute/solvent mixtures do you need to evaluate? It does not make sense to determine the solubility of a solute which is INSOLUBLE (or relatively insoluble) in a certain solvent.

Conduct your experiment and record your results in a data table.

You should now have enough information to design a procedure which will allow you to take a sample of the mixture and separate it into the four separate components. A model sample of the spill mixture will be available for you to evaluate the effectiveness of your method. This sample will have a total mass of about 5.0 g and contain 20% sand, 10% N1986, 65% NaCl, and 5% 2,2’-bipyridine by mass.

1. Based on the information above, and your results from Parts 1 and 2, outline a specific separation scheme for the model sample mixture. This should be your procedure from Part 1, question #5, but modified to include specific quantities of solvents. Be specific with your instructions since a fellow employee will be performing the actual separation of the mixture. Your job is method development. See a TA or teacher to proceed to Part 3.

Part 3 – Evaluation of Separation Scheme

Test the procedure you have developed by performing the separation of a 5.0 g sample of the model mixture. Collect the recovered N1986 and 2,2’-bipyridine, and use this value to calculate the % by mass of N1986 and 2,2’-bipyridine in the sample mixture. Then answer the final questions ON YOUR OWN.

1. How does your % N1986 compare to the known value for the model mixture of 10%? How effective was your procedure? If your value is different than expected, why do you think you got a value that deviated from the amount in the sample mixture?
2. How does your % 2,2’-bipyridine compare to the known value of 5%? If the value deviates from the expected value provide an explanation.
3. Provide any suggestions for modifications to the procedure which could improve the recovery of N1986.

**Trouble-Shooting Question**

1. Your procedure has been accepted and is currently being used to separate N1986. You get an urgent message from the lab technician who is performing the analysis saying that he grabbed the wrong solvent bottles and accidentally added the solvents in the wrong order. He wants to know how this error will affect the separation. What would you tell him?

**FINAL REPORT**

Prepare a Standard Operating Procedure (SOP) for technicians to follow in order to separate the mixture. See the template on writing an SOP for further instruction.