Microscale crystallization of sulfanilamide essay

The purpose of the experiment was to use the crystallization technique to purify the solute sulfanilamide using 95% ethanol as the solvent, to calculate the amount of sulfanilamide that was recovered, and to determine the purity of the final product. Introduction In this experiment, recrystallization will be the method used to purify the solute sulfanilamide using ethanol as the solvent.

Based on the solubility curve on the solubility versus temperature graph, the ideal solvent will allow the solute to be oluble at higher temperatures and Insoluble at lower temperatures, thus making ethanol an excellent solvent for crystallizing sulfanilamide. Sulfanilamide is first dissolved in the smallest amount of ethanol Just enough to dissolve the sulfanilamide and then left undisturbed to cool.

It is important that cooling be slow In order to achieve the best results.

The solute will become less and less soluble as the temperature decreases and will start to separate from the solution and form crystals. Only molecules with the right shape will fit into the crystals structure as the ther molecules that make the substance impure will stay in the solution. Thus, the process of recrystallization is slow and selective. After recrystallization. the purity of the final product will be determined by observing its crystals and finding Its melting point.

The melting point Is usually expressed as a range.

The first number is the temperature at which melting is first observed and the second number Is the temperature at which the solid is completely

melted. Pure substances will have a higher melting point and a narrower melting point range closer to the literature value ersus impure substances which will have a lower melting point and a larger range. The literature melting point of pure sulfanilamide Is 164.

5 – 166. 50c. By using the recrystallization technique and observing its appearance and finding Its melting point, a purer form of sulfanilamide would be obtained. OF3 rhe ethanol is heated to completely dissolve the sulfanilamide. The solution is slowly cooled to form crystals, which is sulfanilamide in a more pure form.

The crystals can then be weighed and the percent recovery determined. The purity of the final solid Nould be determined by performing a melting point and comparing the color of the purified sulfanilamide to that of the original sample. Procedure 50ml of tap water was added to a dry 100ml beaker and a thermometer placed inside it. The hot plate was set to 165 degrees Celsius.

Exactly, approximately 0. 10g of impure sulfanilamide was measured and put in a test tube. About 6ml of 95% ethanol was collected in another beaker and 3ml of that was pipetted into a test tube with two small boiling stones in it. The test tube was then placed in the beaker f water and heated until the ethanol started boiling at about 77. 3 degrees Celsius. Seven drops of the hot solvent ethanol was carefully added to the test tube of sulfanilamide, Just enough to cover the sulfanilamide, and placed it in a water bath along with a stirring rod.

It took a total of 24 minutes of intermittent stirring, adding 3-8 drops of the ethanol, and heating the mixture to fully dissolve while trying to keep the

same amount of solvent that was originally started with. The hot plate was also increased from 165 degrees Celsius to 185 degrees Celsius to aid in the dissolution f the sulfanilamide. When all of the sulfanilamide crystals finally dissolved, the test tube was transferred to a test tube rack to slowly cool to room temperature. The test tube was placed in a tap water bath for further crystallization.

Observations were then recorded. Once crystallization was complete, the test tube was centrifuged and decanted two times.

The first time it was centrifuged for two and a half minutes and the second time it centrifuged for less than a minute. A filter paper was used to help remove the last bit of water that could not have been removed by decantation. The ry crystals were transferred with a spatula toa watch glass and a filter paper was placed on top to continue to dry overnight. Then it was weighed and the melting point was determined. Findings were recorded.

It was observed that the appearance of the sulfanilamide prior to recrystallization Nas off-white in color compared to a shinier and brighter color of white that was seen after recrystallization. The sample also appeared like fibers and round as the crystals were growing, which might indicate the proper fitting of the molecules in the crystal structure. Additionally, the melting point of the experimented sample of ulfanilamide was observed at 159” 163. 50C about five degrees lower than the literature melting point.

This might indicate that there are still other unwanted compounds in the purified sample.

If the melting point of impure sulfanilamide before recrystallization was determined, then a more concrete conclusion about its purity can be fgured. However, based on appearance alone, it can be concluded that more pure form of the sample was obtained. By obtaining pure sulfanilamide, antibiotics can be made and are used to treat diseases such as leprosy, tuberculosis, and malaria.