Extraction lab report

Business

Introduction Extraction involves dissolving a compound or compounds either from a solid into a solvent or from a solution into another solvent. Extraction is a method used to purify a substance by removing the impurities that was added to it. A solvent- solvent extraction is a type of extraction that is most commonly done by using two substances that are typically insoluble with each other. An acid-base extraction is a solvent-solvent extraction, in almost every case one of the solvents is water or an aqueous solution and a requirement of a solvent-solvent extraction is that the mixture of the two solvents will separate into two layers. The upper layer is that which is less dense, while the lower layer is the denser of the two.

The organic solvent must also be volatile so it can be easily removed by evaporation at the end. Extractions are extremely useful for isolating and purifying amines, carboxylic acids, phenols as well as some neutral compounds. All three of these functional groups can be interconverted from non-ionic organic-soluble forms to water-soluble ionic forms by changing the pH levels. In the experiment done in this lab, a mixture of and a neutral compound and either an acid or base impurity will be separated by an acid-base extraction. The unknown compound will then be purified by recrystallization and identified by melting points. Experimental Discussion The solubility of the basic component was manipulated to show that the basic compound was ether insoluble and water soluble.

The extraction experiment with HCl shows that the impurity in the mixture was basic; therefore the basic compound must have been 4-chloroaniline. This was determined because once the aqueous layer was drained into the test tube and NaOH was added precipitation formed. The precipitation

occurred because when base was added to the aqueous solution that contains the salt of a deprotonated organic base, the organic base is then re-protonated. When this happens it is now water insoluble which forms a precipitation in the aqueous solution. The organic layer therefore must have contained the neutral unknown; the compound remained soluble in the non-polar organic solvent throughout the extraction with HCl. At the end of the procedure the neutral compound was recovered in its solid form by evaporation of the organic solvent.

The melting point of unknown #20 was between 73-83 C? which fell within the range of the neutral compound of fluorenone that has a melting temperature of 80-83 C?. Fluorenone is a yellow flaky crystal like compound with a density of 1. 13 g/cm3 but it is highly soluble in alcohols such as ether. The properties of fluorenone supported the fact that the liquid formed a yellow flaky substance once it recrystalized from the organic layer due to evaporation. In his experiment the addition of ether dissolved both substances and the addition of HCl formed the two layers needed for the extraction. The neutral compound fluorenone separated from the amine 4-chloroaniline by the formation of the two layers.

Fluorenone stayed in the organic layer because it is very soluble in ether while the conjugate organic acid of the amine mixed with the aqueous solution. The addition of NaOH to the aqueous layer turned the solution basic and converted the deprotonated amine salt back to its neutral form. The complete reaction can be easily followed using the flow chart in figure 1.

Figure 1. Etraction flow chart for a mixture of fluorenone and 4-chloroaniline

-Add diethyl ether – Add 1M HCL Separate layers In aqueous solution In

diethyl ether layer -Add 6M NaOH -Filter precipitate -Evaporate ether Conclusion Adding 4-chloroaniline to a mixture of neutral fluorenone showed a very effective extraction. The neutral compound fluorenone separated from the amine 4-chloroaniline and formed two layers.

Fluorenone stayed in the organic layer because of its solubility with ether while the conjugate organic base of the amine stayed in the aqueous solution. The non-organic base NaOH added to the aqueous layer turned the solution basic and converted the compound back to its neutral form. This experiment showed that extraction is a useful organic tool to separate a mixture of unknown compounds and to purify organic molecules. Some mistakes that were made during the experiment was that a little of the organic solution was drained into the test tube 2 during the draining process. While the organic solution was being transferred into a small 50-mL beaker, some of the liquid was accidently dropped onto the desk and quickly evaporated. Despite all 87% of the original mixture was recovered once the experiment was concluded,