Results---

Discussion:

The percent yield of the 4-nitrobenzoic acid precipitate was 80.357%. Although much of the precipitate was recovered, the percent yield was not close enough due to a few factors including human error. Starting at the beginning of the procedure, while separating the two layers from the separatory funnel, I let out less than I should have, fearing I could contaminate the aqueous solution extracted. Later on, while separating out the reprotonated solution through vacuum filtration, my solution in the filter flask was not clear, as it had some white powder. I can only assume that my filter at the beginning of this process did not fully cover the Buchner funnel, leading to the loss of some of the precipitate. Finally, while going to weigh my precipitate and filter paper, some of the precipitate was still clumped to the walls of the Buchner funnel, and a miniscule amount fell off my filter paper as I walked near an open door. While the percent yield for the 4-nitrobenzoic acid precipitate was acceptable, the value for the ethyl-4-nitrobenzoate was ridiculously low. As stated before, I did not fully separate out the two layers at the starting procedure, which could have led to a higher percentage yield due to the fact I had left some of the aqueous solution in the separatory funnel. My biggest mistake in this lab occurred while trying to gravity filter my organic solution combined with 1.501 g of MgSO4 . My stemless funnel was not resting on the 100 mL round bottle flask, but rather on the stand. This caused a major amount of the liquid to drop out of the funnel and onto the bottom of my fume hood. With the remaining liquids I had, I quickly substituted the medium round bottle flask with the smaller round bottom flask and lowered the funnel into the flask. After the Rotary Evaporator and some nitrogen crystalized my ethyl-4-nitrobenzoate, I ended up with a recovery yield of a mere 37.089%.

Conclusion:

Through this experiment, we used the extraction technique to separate 4-nitrobenzoic acid from ethyl-4-nitrobenzoate. Our first step was to dissolve one gram of the mixed compounds into an organic solvent of ethyl acetate, along with sodium hydroxide. This deprotonated the 4-nitrobenzoic acid, making it more water soluble. After mixing in a separatory funnel, an organic and aqueous layer were created as they settled. After extracting the lower-settling aqueous solution three time (through repeated practice of adding sodium hydroxide), it was reprotonated through the addition of hydrochloric acid. This forced the acid to precipitate, which was separated using vacuum filtration and then finally weighed. The organic layer that was remaining in the separatory funnel was dried with anhydrous MgSO4 ,filtered, placed in a rotary evaporator, and finally dried with nitrogen if needed. The recovery of ethyl-4-nitrobenzoate was unsuccessful with a percent recovery of 37.089%, however, the recovery of the 4-nitrobenzoic acid was successful with a percent recovery of 80.359%.