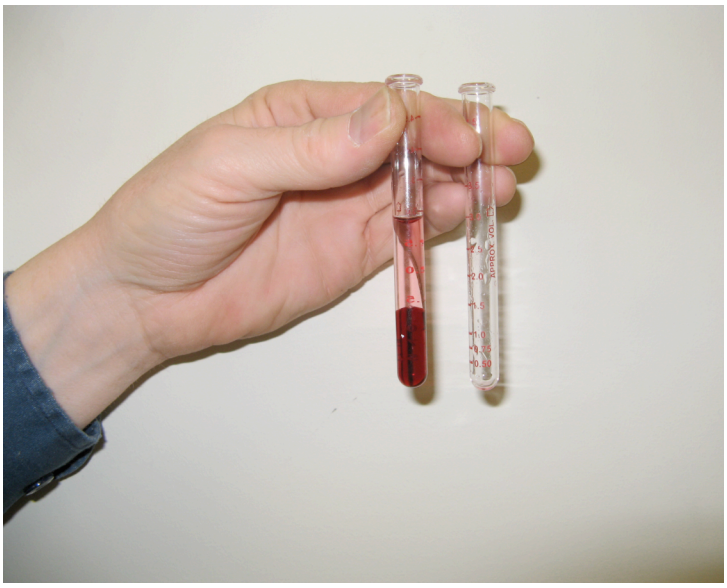
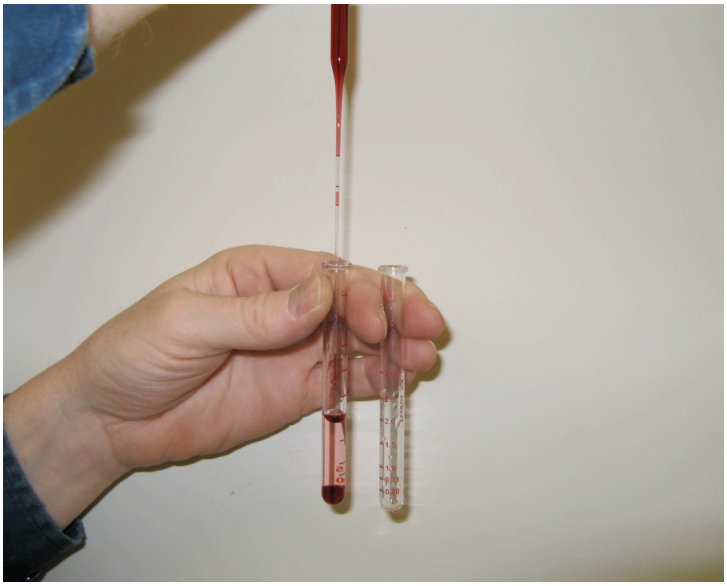
Extraction Technique in Photos.

In these photos the liquids have been dyed so the different layers (phases) show up clearly. In your extraction the layers will be nearly colorless. Here the dark lower layer represents the aqueous phase and the lighter upper layer represents the *tert*-butyl methyl ether (organic) phase. This example shows one extraction only. Usually, extraction is done two or three times, using different extracting solutions. For example, in your extraction, the ether layer is extracted with two separate portions of aqueous bicarbonate, backwashed, and then extracted with aqueous saturated salt solution.

In the first photo, the mixture of carboxylic acid and neutral compound is dissolved in *tert*-butyl methyl ether in a reaction tube. A sodium bicarbonate solution (base) is then added. In this example, the lighter-colored, dyed upper phase represents the ether phase.

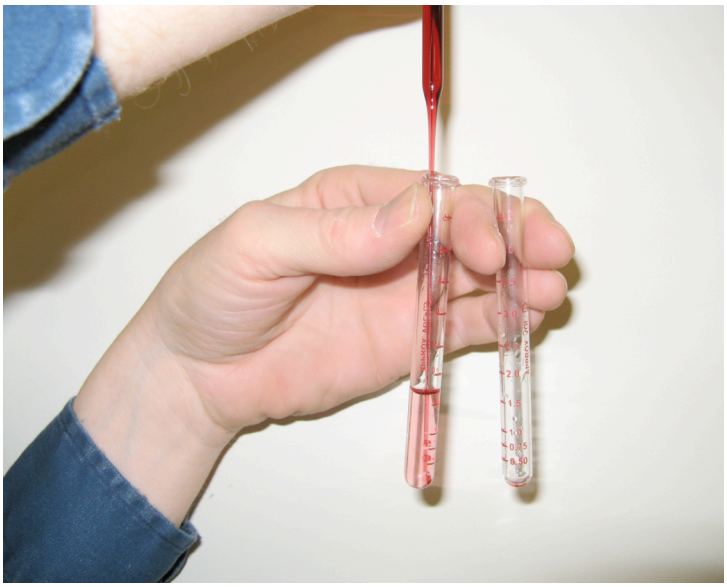


In the second photo, a portion of the lower aqueous phase is drawn up into a pipet.



In the third photo, the pipet is lifted slightly above the surface of the liquid and the contents of the pipet are squirted down into the upper, organic phase. This must be done vigorously but with care to prevent the liquid from being pushed out of the tube. Do it gently at first until you have a good feel for it. Because extraction of a compound from one layer into the other is a heterogeneous process, occurring only at the interface between the two layers, physically mixing the layers thoroughly is essential for complete extraction. This step is repeated for three minutes to ensure that equilibration has been accomplished. If the mixing is not done vigorously enough or long enough, complete separation of the neutral compound and carboxylic acid will not occur. This would lead to incomplete separation and would result in contamination of the final products.

In the fourth photo, after the layers have been allowed to settle cleanly into two distinct phases, the pipet is used to remove the lower layer. Care must be taken to make a clean separation.



Finally, in the fifth photo, the lower aqueous phase has been transferred to a second reaction tube. Thus, in the extraction process, the two layers have been mixed thoroughly, allowed to separate cleanly back into two separate layers, and separated into different tubes. Each would then be worked on separately to eventually yield the separate compounds. The ether layer would be dried, then evaporated to yield crude neutral compound which would be recrystallized to yield pure neutral compound. The aqueous layer would be neutralized with conc HCl which would cause the carboxylic acid to precipitate. A quick recrystallization followed by a more careful recrystallization would result in pure carboxylic acid.