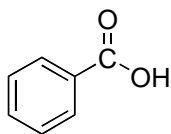
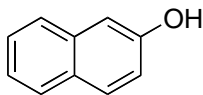


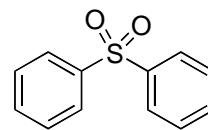
Separation of a Three-Component Mixture by Extraction



$C_7H_6O_2$
M. Wt.=122.12
M. pt.=120-121°C



$C_{10}H_8O$
M. Wt.=144.16
M. pt.=122-123°C



$C_{12}H_{10}O_2S$
M. Wt.=218.27
M. pt.=126-128°C

A 1.5-gram sample of a 1:1:1 (by weight) mixture of benzoic acid, 2-naphthol and diphenyl sulfone is added to a 125 mL separatory funnel. Isopropyl acetate (20 mL) is added to the separatory funnel and the mixture swirled until all solids had dissolved. The solution is sequentially extracted with saturated aqueous $NaHCO_3$ solution (3 X 6mL), then with water (1 X 5 mL); with the aqueous extracts from each extraction being added to a 50 mL beaker (flask 1). *[The separatory funnel should be shaken for at least 2 minutes during each of the three $NaHCO_3$ extractions. Venting of the funnel should be done every 20-25 seconds during each extraction. When draining off the aqueous layer in these and all subsequent extractions leave a two mm tall layer of aqueous solution in the separatory funnel. This greatly reduces the amount of organic layer that contaminates the aqueous extracts.]* Next the organic layer is extracted sequentially with 5% aqueous NaOH solution (4 X 5 mL) then with water (1 X 5 mL). Each aqueous extract is collected in a 50 mL beaker (flask 2). *[The separatory funnel should be shaken for at least 1.5 minutes during each NaOH extraction. The layer separation after the first two NaOH extractions will take about three minutes. Subsequent layer separations are faster.]* The remaining organic layer is extracted with saturated aqueous NaCl solution (5 mL) and the aqueous layer is discarded *[Do not leave any water in the funnel after this extraction]*. The organic layer is transferred to a 50 mL Erlenmeyer flask (flask 3). The separatory funnel is washed with isopropyl acetate (2 X 2 mL) with each washing being added to flask 3. The organic solution in flask 3 is dried (Na_2SO_4) then decanted into a tared 50 mL Erlenmeyer flask (flask 4). The drying agent is washed with isopropyl acetate (2 X 1 mL) with each rinse being added to flask 4. The solvent in flask 4 is evaporated to give crude diphenyl sulfone as a white solid. *[Evaporate the solvent using a sand bath heated to 110°C. If the sand is much hotter, the phenyl sulfone will be isolated as a melt. The only way to actually know when all of the solvent has been removed is to obtain the weight the flask at the point of "presumed dryness", then reheat for 1-2 minutes and re-weigh the flask. If the weight does not change, the product is solvent free. An alternative procedure is to evaporate the isopropyl acetate until the phenyl sulfone begins to precipitate, cool, add hexane (10 mL), chill in an ice bath then filter. Both procedures work well.]*

The aqueous solution in flask 1 is acidified (acid to litmus paper) by the addition of 6 N HCl solution to give a white precipitate. *[Caution! Foaming will occur due to liberation of CO_2]* After cooling the resulting mixture in an ice bath for 10 minutes, the solid is isolated by vacuum filtration, washed with ice water (3 X 1 mL), and air dried to give crude benzoic acid as a white solid.

The aqueous solution in flask 2 is acidified (acid to litmus paper) by the addition of 6 N HCl to give an off-white precipitate. The resulting aqueous mixture is heated to boiling, then immediately cooled in an ice bath for 20 minutes. *[The boiling simply causes the very fine, but virtually unfilterable, particles to coalesce into larger crystals so that they can be easily filtered. It is not in any way meant to be a recrystallization]*. The resulting tan solid is isolated by vacuum filtration, washed with ice water (3 X 1 mL), and air dried to give the crude 2-naphthol.

Figure 1. Summary of Extraction Process.

