A) Resolution of a Solid Mixture

- Obtain small samples of benzoic acid and charcoal (approximately 1 g of each). Combine the benzoic acid and charcoal on a watch glass, and mix with a stirring rod until the mixture is fairly homogeneous. Charcoal is not soluble in water and will be used as a "contaminant" for the benzoic acid. Transfer the benzoic acid/charcoal sample to a clean 150-ml beaker.

- Set up a short-stem gravity funnel in a clay triangle on a small metal ring clamped to a ringstand. Fit the filter funnel with a piece of filter paper folded in quarters to make a cone.

- Moisten the filter paper slightly so that it will remain in the funnel.

- Set up a 250-ml beaker about half filled with distilled water on a wire gauze over a metal ring. Heat the water to boiling.

- When the water is boiling, pour about two-thirds of the water into the beaker containing the benzoic acid sample. Use a towel to protect your hands from the heat.

- Pour the remainder of the boiling water through the gravity funnel to heat it. If the funnel is not preheated, the benzoic acid may crystallize in the stem of the funnel rather than passing through it. Discard the water that is used to heat the funnel.

- Transfer the beaker containing the benzoic acid mixture to the burner, and reheat it gently until the mixture just begins to boil again. Stir the mixture to make sure that the benzoic acid dissolves to the greatest extent possible.

- Using a towel to protect your hands, pour the benzoic acid mixture through the preheated funnel. Catch the filtrate in a clean beaker.

- Allow the benzoic acid filtrate to cool to room temperature.

- When the benzoic acid solution has cooled to room temperature, filter the crystals to remove water. Wash the crystals with two 10-ml portions of ice-cold water.

- Transfer the liquid filtrate from which the crystals have been removed to an ice bath to see whether additional crystals form at the lower temperature. Examine, but do not isolate, this second crop of crystals.

- Transfer the filter paper containing the benzoic acid crystals to a watch glass, and dry the crystals under a heat lamp or over a 400ml beaker of boiling water. You can monitor the drying of the crystals by watching

for the filter paper to dry out as it is heated. If a heat lamp is used, do not let the paper char or the crystals melt.

- When the benzoic acid has been dried, determine the melting point of the recrystallized material using the method discussed in Experiment 5. Compare the melting point of your benzoic acid with that indicated in the handbook. If the melting point you obtain is significantly lower than that reported in the handbook, dry the crystals for an additional period under the heat lamp or over the hot-water bath. (N/A)

B) Simple Distillation

- Simple distillation can be used when the components of a mixture have very different boiling points. In this experiment, a partial distillation of a solution of sodium chloride in water will be performed (to save time, the distillation is not carried to completion). This is an extreme example, because the boiling points of water and sodium chloride differ by over 1000degC, but the technique will be clearly demonstrated by experiment.

- Your instructor has set up a simple distillation apparatus for you. He or she will explain the various portions of the apparatus and will demonstrate the correct procedure for using the apparatus. The source of heat used for the distillation may be a simple burner flame, or an electrical heating device (heating mantle) may be provided. Generally, electrical heating elements are preferred for distillation, because often the substances being distilled are flammable.

- Obtain about 50ml of 1% sodium chloride solution. Place 1ml of this solution in a small test tube, and transfer the remainder of the solution to the distilling flask.

- Place a clean, dry beaker under the mouth of the condenser of the distillation apparatus to collect the water as it distills from the salt solution.

- Begin heating the sodium chloride solution as directed by the instructor, and continue distillation until approximately 20ml of water has been collected. Transfer approximately 1ml of the distilled water to a clean small test tube.

- To demonstrate that the distilled water is now free of sodium chloride, test the sample of original 1% sodium chloride solution that was reserved before the distillation, as well as the 1ml sample of water that has been distilled, with a few drops of 0.1 M silver nitrate solution. Silver ion forms a precipitate of insoluble AgCl when added to a chloride ion solution. No precipitate should form in the water that has been distilled.

C) Fractional Distillation

- Fractional distillation may be used to separate mixtures of volatile substances that differ by at least several degrees in their boiling points. The vapor of the liquid being boiled passes into a fractionating column, which provides a great deal of surface area and the equivalent of many separate simple distillations.

- Your instructor has set up a fractional distillation apparatus in one of the exhaust hoods. Compare the fractional distillation apparatus with the simple distillation apparatus used in Part B, and note the differences. Your instructor will explain the operation of the fractional distillation apparatus. The apparatus is set up in the hood, because the mixture you will distill is very volatile and may be flammable.

- Obtain an unknown mixture for fractional distillation and record its identification number. Use a graduated cylinder to transfer 40ml of the unknown mixture to the distillation flask. Record the exact volume of the mixture used. During the distillation, carefully watch the thermometer that is part of the apparatus. The temperature is used to monitor the distillation, because the temperature will increase very suddenly as one component finishes distilling and another component begins to distill.

- Place a clean, dry flask under the mouth of the condenser to collect the first component of the mixture as it distills. Have ready a second clean, dry flask for collection of the second component. Have ready corks or rubber stoppers that fit snugly in the two collection flasks.

- Have your instructor approve the apparatus, and then begin heating the distillation flask with very low heat until vapor begins to rise into the fractionating column.

- Allow the vapor to rise to the level of the thermometer bulb, and adjust the heat so that the thermometer will remain bathed in droplets of liquid as the mixture distills. Record the temperature indicated by the thermometer as the first component of the mixture begins to distill. Collect the distillate coming from the condenser.

- Continue heating the distillation flask, using the smallest amount of heat that will maintain distillation. Monitor the temperature constantly. At the point at which the first component of the mixture has finished distilling, the temperature will rise suddenly and abruptly by several degrees. At this point, remove the flask used to collect the first component of the mixture, and replace it with the second flask. Stopper the flask containing the first component to prevent its evaporation.

- Record the temperature indicated by the thermometer as the second component of the mixture begins to distill. Continue the distillation until approximately 5 ml of liquid remains in the distillation flask. Remove the source of heat, but do not remove the collection flask until distillation stops.

- Do not heat the distillation flask to complete dryness, or it may break from the heat. When distillation is

complete, stopper the flask containing the second component of the mixture.

- With a graduated cylinder, determine the respective volumes of each of the two components of the mixture. Calculate the approximate composition of the original mixture, in the terms of the percentages of low-boiling and high-boiling components. This percentage is only approximate, because some of the vapor being distilled may have been lost, and not all of the high-boiling component may have been isolated.

- Report the approximate composition of your mixture to the instructor, along with the boiling temperatures of the two components.

- Turn in the two flasks of distillate to the instructor for proper disposal.