

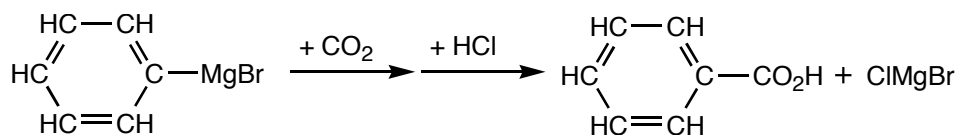
Recrystallization of Benzoic Acid

Objective

To purify benzoic acid by recrystallization and gain experience with a basic organic laboratory techniques.

Background

Products of chemical reactions are often contaminated with impurities. One method for purifying chemicals, recrystallization, takes advantage of the differences in the solubilities of the desired products and the impurities and the tendency for the slow formation of crystals to exclude impurities from the crystalline solid.



Benzoic acid, the chemical that we will be purifying, can be made by reacting phenylmagnesium bromide with carbon dioxide. When the product of that reaction is treated with hydrochloric acid, benzoic acid is formed. From the balanced chemical equation, it is clear that the benzoic acid will not be pure since the byproduct MgClBr , an ionic solid, forms at the same time the desired product forms.

Benzoic acid can be separated from ionic solids because the materials have different solubilities in water. At room temperature, benzoic acid is not particularly soluble in water, whereas ionic solids like MgClBr are. Thus, adding water to the crude reaction products will dissolve the ionic solid, and nearly all of the benzoic acid will remain undissolved. If the suspension were filtered, much of the impurity would be washed away; however, the resulting product wouldn't be as pure as a recrystallized product.

To take advantage of the extra purity made possible by recrystallization, all of the benzoic acid must be dissolved. On the other hand, the benzoic acid needs to come back out of solution to crystallize. Simply adding room-temperature water cannot accomplish this goal; once the benzoic acid is dissolved there would be no reason for it to crystallize. Since benzoic acid is much more soluble in hot water as compared to cold, small amounts of *hot* water are added to dissolve the benzoic acid. If the hot, saturated, aqueous solution of benzoic acid is cooled, the solution becomes supersaturated, and from the supersaturated solution benzoic acid crystallizes.

Procedure

1. Obtain a sample of benzoic acid.

2. Add approximately 0.5 g (record the actual mass to four decimal places) of the contaminated benzoic acid and 5 mL of water to a 25-mL Erlenmeyer flask.
3. Warm the flask and its contents on a hot plate and slowly add near-boiling water to dissolve the solids.
4. Continue to warm the flask and add near-boiling water until all of the solids have dissolved
5. Remove the flask from the hot plate and allow it to cool slowly on the bench top. Covering the flask with a beaker can help slow the rate at which the solution cools. After approximately twenty minutes, crystals will have formed. Cool the suspension further using an ice bath.

If crystals have not formed after 20 minutes, there are a few techniques that can be used to coax the crystals out of solution. Often scratching the glass container with a glass rod will induce crystallization. Alternatively, a seed crystal can be obtained by evaporating a drop of the solution on the tip of a stirring rod or spatula. Returning this seed crystal to the super saturated solution will cause crystals to form. If none of these techniques work, it could be that too much water has been added.

6. Transfer the crystals to a Hirsch funnel and filter the benzoic acid–water suspension by vacuum filtration.
7. While continuing to use the vacuum filtration apparatus, wash the benzoic acid crystals with ice-cold water by pouring ice-cold water over the crystals.
8. Dry the crystals on the Hirsch funnel; that is, allow the vacuum filtration system to draw air through the crystals until the mass of the crystals remains relatively constant. Place the benzoic acid in your drawer and allow it to dry completely.
9. After allowing the benzoic acid crystals to dry for a week, determine the mass and the melting point of the benzoic acid.

Experimental Report

In your typed report:

1. Report the amount (mass) of benzoic acid that you started with, the amount (mass and moles) that you recovered, the percent recovery, its appearance, and the observed melting point range. For example:

From x.xxxx g of impure benzoic acid, x.xxxx g (x.xxx mol) of a (describe the appearance) crystalline solid that had an observed melting point range of xx.x – xx.x °C were isolated with a percent recovery of xx.x%.

2. In a few sentences describe why the solvent must be kept hot during the dissolving phase and what would happen to the percent recovery if the solvent is not kept as hot during the dissolving phase.