

CHEMISTRY I-H SYNTHESIS OF ASPIRIN

Place approximately 1g of salicylic acid in a clean, dry 250cm³ flask. From a 50cm³ buret (see Fig. 1), located on the side shelf in the laboratory, add 2cm³ of acetic anhydride, and swirl the flask for a minute or two.

Now add 2cm³ of glacial acetic acid from a 50cm³ buret, also located on a side shelf. From a small dropping bottle add 1-2 drops of concentrated sulfuric acid. Swirl the contents of the flask for 3-4 minutes.

When the liquid is clear (or nearly so -- when there is no visible solid present), empty the contents of the flask into a clean 250cm³ beaker containing 20cm³ of water. Place the beaker in an ice bath (see Fig. 2) until thoroughly chilled. If crystallization has not already begun, scratch the side of the beaker beneath the surface of the solution with a stirring rod. Allow crystallization to become complete.

Set up a funnel fitted with filter paper (see Fig. 3). Filter the product from the beaker, and wash it three times with 10cm³ portions of water. Remove the filter paper from the funnel, unfold it, and place it on a paper towel. Place the marked samples in an area designated by your teacher. Leave the samples in this area for drying.

SUGGESTED TESTS:

1. Place a small sample of your aspirin on a small can lid. About 2-3cm away on the lid place an equal-size sample of commercial aspirin (see Fig. 4). Place the can lid on the ring of a ring stand. With a low Bunsen flame, heat the lid from below at a point between the two samples. Compare the melting points of the two samples.

2. Test the solubility of your sample. Add a few crystals (about the size of a match head) of your sample to 3cm³ of toluene in a beaker. Repeat, using hot water instead of toluene.

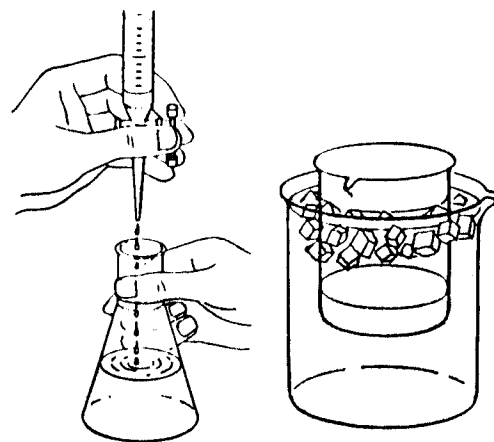


FIGURE 1

FIGURE 2

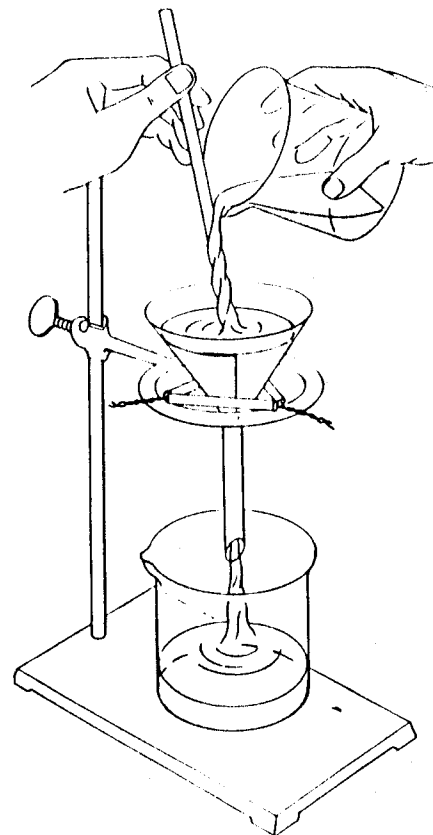


FIGURE 3

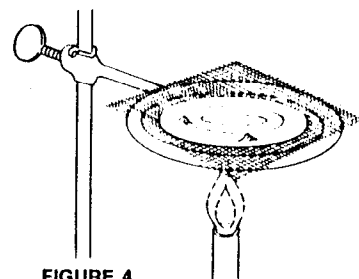


FIGURE 4

3. Try dissolving a few crystals of your aspirin sample in 5cm^3 of cold 0.5M sodium bicarbonate solution. Does it dissolve? Add 0.5M HCl to the solution until it becomes acid. Use litmus paper to test the acidity. Chill in an ice bath. What do you conclude about the solubility of aspirin in an acidic solution? In a basic solution?

4. To a few milligrams of salicylic acid in a clean, dry test tube, add 1cm^3 of methanol and 1 drop of 0.1M FeCl_3 . Observe the color. Repeat, using milligram samples of your aspirin and commercial aspirin. Do you observe the same color? If so, what could this mean?