Experiment A

Synthesis of Aspirin

Preparation and isolation of crude aspirin:

Pre-heat a water bath in a 600 mL beaker on the steam cone to a temperature of about 50 °C.

You will be given approximately 3.0 g of salicylic acid. Set aside a small sample (aprx. 20 mg) in a corked and labeled 13x100 mm test tube for later ferric chloride analysis and weigh the rest into a clean, dry 125 mL Erlenmeyer flask. In the fume hood, add 6.0 mL acetic anhydride via pipet. To this white slurry, add 3-4 drops concentrated sulfuric acid. Heat the flask in the hot water bath with periodic swirling for a period of about seven minutes, then allow it to cool to room temperature on the benchtop.

Note: Your mixture may or may not go completely clear, depending on how fast the product begins to crystallize out from solution (which in turn may depend on how smooth or scratched the inside of your flask is.) If your reaction does not look like your neighbor's, this is not a cause for concern.
If a white solid has not formed upon cooling, induce crystallization by scratching the inside of the flask with a glass rod, or by seeding with a small sample from a neighbor.

Once a significant amount of solid has formed, add 30 mL chilled water and stir with a glass rod to break up lumps that may have formed. Place the flask in an ice bath for 10-15 minutes and begin to set up for vacuum filtration.

The crude aspirin is isolated by vacuum filtration on a Buchner funnel, and washed with two small portions of cold water. (Do not use a large volume of wash water, or a significant amount of product will dissolve - more than will be left behind with minimal washing.) Continue to pull a vacuum for five minutes to air dry the solid before placing it in a jar desiccator until the next period.

Purification:

Weigh the crude aspirin. Reserve approximately 20 mg for a ferric chloride test as above, and place the remainder in a 250 mL beaker. Stir in 25 mL of 5% aqueous sodium bicarbonate, and break up as much of the solid as possible. When the reaction appears to be complete (fizzing has stopped), remove the remaining solid impurities by gravity filtration, washing with two small portions of (room temperature) water. Add, in portions with stirring,
approximately 8 mL of 3 M HCl. Test with pH paper to verify the pH is below 2, if not add more acid until it is sufficiently acidic. Chill the mixture in an ice bath for 10-15 minutes while setting up for vacuum filtration.

Isolate the purified aspirin by vacuum filtration on a Buchner funnel and wash with two small portions of chilled water. Air dry with vacuum for five minutes and store in a tared vial in a jar desiccator for further drying. Dry to constant weight. Set aside a small (apprx. 20 mg) sample for the ferric chloride test and weigh the remainder, which will be turned in with your report. Base your yield on the amount actually submitted.

Analysis:

Ferric chloride reagent is used as a colorimetric test for phenols: phenolic compounds tend to give deeply colored solutions. As with most tests, it is not perfect and some phenols do not test positive. Conversely, some compounds such as highly enolizable carbonyl species will give dark colors. But ordinary alcohols, ethers, esters, ketones, carboxylic acids, and hydrocarbons test negative.

Dissolve approximately 20 mg of the substance of interest in 1-2 mL denatured (95%) ethanol in a test tube. Add 2 drops of 1% FeCl3 solution, shake, and record your observations. (The sample need not be completely dry in order to perform this test.)

Perform four such tests: salicylic acid; crude aspirin; purified aspirin; blank.
Present your results in a table in the observations section of the notebook.