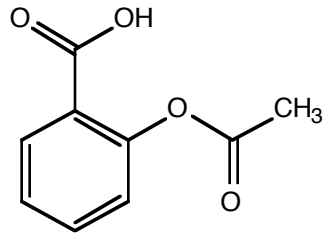
Experiment 19 - Spectrophotometric Determination of Acetylsalicylic Acid in an Aspirin Tablet

Most aspirin tablets are said to contain 5 grains of the active ingredient acetylsalicylic acid. (The grain is a unit of mass.) How reliable is this figure? This experiment will enable you to check up on the manufacturer.

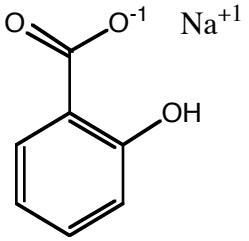
The method of analysis that we will use is called "spectrometry" or "spectrophotometry". It depends on the fact that molecules can absorb electromagnetic radiation of certain wavelengths, using the energy of the radiation to "excite" electrons in their atoms. (Thus they have absorption spectra, just as isolated gas atoms do.) The greater the concentration of a particular molecule present in a sample, the more light of a particular wavelength the sample will be able to absorb. The absorbance of light by the sample increases in direct proportion to the concentration of the molecule present. We will use electromagnetic radiation ("light") of wavelength (λ) 297 nm, which falls in the ultraviolet range. In the experiment, 297 nm light shines through the sample, and the amount of light absorbed by the sample is measured.

The chemical name of the active ingredient in aspirin is acetylsalicylic acid. You will convert it chemically to the salt sodium salicylate before measuring the absorbance. Notice that 1.00 mole of sodium salicylate is produced for every 1.00 mole of acetylsalicylic acid used up:



+ 2 $NaOH_{(aq)}$ $\longrightarrow$

acetylsalicylic acid
(aspirin)



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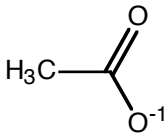

Na^{+1} + H_2O

sodium salicylate

sodium acetate

Need for Calibration

The UV (ultraviolet) Spectrophotometer will show the absorbance due to sodium salicylate (from aspirin) present in the sample. This number will be directly proportional to the concentration of sodium salicylate in the sample, but will not tell us the actual concentration in any given sample. In order to be able to convert an instrument reading to an actual concentration of sodium salicylate, we must first calibrate the instrument using solutions of known sodium salicylate concentration.

Procedure for Calibration

The instructor will explain and demonstrate the correct use of the UV Spectrophotometer. You will then measure and record the absorbance of five solutions of known concentration of sodium salicylate: 7.25×10^{-5} M, 14.5×10^{-5} M, 21.8×10^{-5} M, 29.0×10^{-5} M, and 36.3×10^{-5} M. Measure the absorbance with the wavelength selector set at 297 nanometers. Use distilled water as the reference liquid - that is, set the instrument for zero absorbance when the light path is passing through distilled water. Each student should construct his or her own calibration "curve" (really a straight line) on graph paper, showing absorbance as a function of molar concentration of sodium salicylate.

Procedure for Preparation of Aspirin Sample

Crush one aspirin tablet in a clean porcelain mortar. Add 20 mL of 0.1 M $NaOH_{(aq)}$ to the powder and stir for several minutes. This will dissolve the aspirin but not the "filler" or "binder" in the tablet. Prepare a folded filter paper in a funnel, and stand the funnel in the neck of a clean 250.0-mL volumetric flask. (The flask must be clean but does not have to be dry.) Make sure no particles of dust, paper, or other impurities get into the flask. Pour the aspirin-containing liquid from the mortar onto the filter paper in the funnel. The clear solution that comes through the filter contains your sample; the solid that is left on the filter paper will eventually be discarded. To remove all traces of aspirin from the mortar, rinse the mortar out with six successive (one after the other) 10-mL portions of 0.1 M $NaOH_{(aq)}$. Pour these rinsings onto the filter funnel also. (Each 10-mL volume need only be measured approximately.)

When the sample has been filtered, remove the funnel, discard the paper and contents, and add more 0.1 M $NaOH_{(aq)}$ to the sample in the flask. Add enough to bring the solution level exactly to the 250.0-mL line on the neck of the flask. **Beware:** it is very easy to overshoot the line on the flask! Use extreme caution when you are getting close to the line, and make sure you use a dropper.

Mix the sample thoroughly by carefully inverting the stoppered flask repeatedly for about five minutes. (Hold the glass or rubber stopper tightly in place with your thumb. Make sure no liquid leaks out before the solution is completely mixed.) Check to verify that the solution is clear and colorless and does not contain any floating specks.

The solution at this point is too concentrated to be used. Prepare a dilution of it by pipetting exactly 2.00 mL of it into a clean 100.0-mL volumetric flask and diluting with distilled water. Carefully add water to bring the solution level up to the 100.0-mL mark on the flask. Mix thoroughly as before by inverting repeatedly.

Measurement of sample concentration

Measure the absorbance of the diluted solution in exactly the same way you measured absorbances of the "knowns" when you calibrated the instrument. Use the same cuvette (sample holder) you used before, and read at the same wavelength (297 nm). Distilled water is still the reference. Before measuring the absorbance of your unknown, be sure to set the instrument for zero absorbance with distilled water in the light path. Record the absorbance of your "unknown", then read off from the calibration curve (the graph you made) the molar concentration of sodium salicylate that must be present to give this absorbance. This concentration in moles/liter is the concentration after you diluted the original aspirin solution. Calculate from it what the concentration of that original solution must have been. (Make sure your answer makes sense!)

From this original concentration and the original volume (250.0 mL), calculate the total number of moles of sodium salicylate in the original solution. For every one mole of sodium salicylate in the original solution, how many moles of "aspirin" (acetylsalicylic acid) were there in the tablet? Convert the number of moles of acetylsalicylic acid ($C_9H_8O_4$) in the tablet to grams and then to grains (15.43 grains = 1.000 gram). How does this value compare with the value given on the label of the aspirin bottle? Calculate the percent error. Write your result on the chalkboard, along with your name. In your lab report, thoroughly discuss possible reasons for any discrepancies.

Using student results that have been listed on the chalkboard, calculate the standard deviation in the number of grains of aspirin per tablet for the class. You may either: a. Calculate the standard deviation, showing all of your work, for 5 student results, or b. Calculate the standard deviation for the results of the entire class on your calculator.

Questions:

1. Why is it necessary to zero the spectrophotometer before using it?
2. What is the purpose of the calibration curve?
3. Once you have calculated the standard deviation of the student results for the class, explain its significance. (We have 95% confidence that the true value of the number of grains in an aspirin tablet lies between _____ and _____ , assuming only random errors.)

Use your calibration curve to answer the following questions:

4. If a solution of sodium salicylate has an absorbance of 0.207 at 297 nm, what is the concentration of sodium salicylate in the solution?
5. An aspirin tablet is crushed, dissolved in sodium hydroxide, and filtered. This solution is diluted to a total volume of 500.0 mL and mixed thoroughly. A 5.00-mL sample of this solution is transferred using a pipet to a 200.0-mL volumetric flask, water is added to the mark, and the solution is mixed. If this solution has an absorbance of 0.143 at 297 nm, how many milligrams of aspirin were in the tablet?
6. An aspirin tablet containing 500 mg of aspirin is prepared as in the procedure we used in this experiment. Predict the absorbance of the final solution.