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Spectrophotometric Analysis of Aspirin

T.S.Rajendra Kumar
Lecturer in Chemistry
S.N.S.R Degree College, Velgode, Kurnool (Dt)

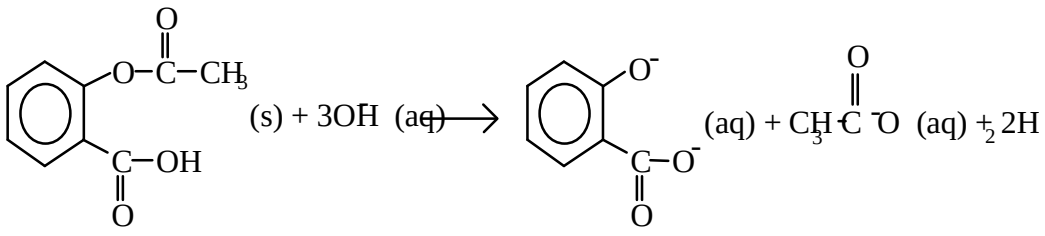
Abstract: A colored complex is formed between aspirin and the iron (III) ion. The intensity of the color is directly related to the concentration of aspirin present; therefore, spectrophotometric analysis can be used. A series of solutions with different aspirin concentrations will be prepared and complexed. The absorbance of each solution will be measured and a calibration curve will be constructed. Using the standard curve, the amount of aspirin in a commercial aspirin product can be determined.

Key Words: Electromagnetic Radiation, Absorbance, Beer-Lambert Law

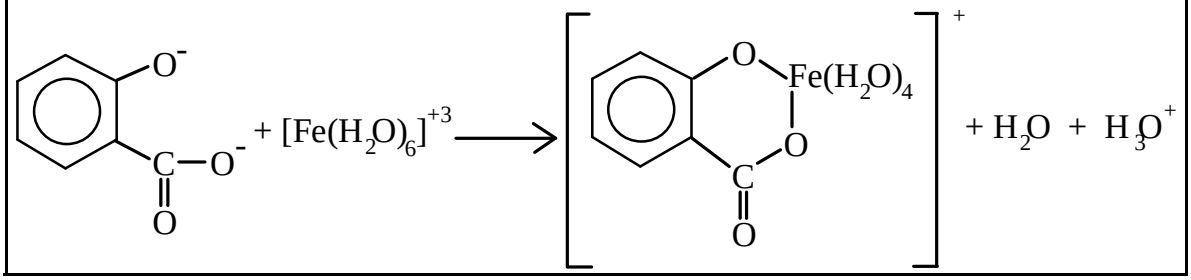
Introduction

Light is a form of electromagnetic radiation. We are most familiar with the visible portion of the electromagnetic spectrum because this is the region of light to which our eyes are sensitive. Visible light, however, is only a small segment of the entire electromagnetic spectrum (see Silberberg, pg.218). Electromagnetic radiation is a form of energy that may be represented as a wave or as particle. The wave model for light is more useful for predicting the behavior of light in our day to day activities, but at a anatomic scale light is better described by particles called photons.

The complex is formed by reacting the aspirin with sodium hydroxide to form the salicylate dianion.



The addition of acidified iron (III) ion produces the violet tetraaquosalicylatroiron (III) complex.



The purpose of this lab is to determine the amount of aspirin in a commercial aspirin product. This lab may also be used to determine the purity of the aspirin produced in the Microscale Synthesis of Acetylsalicylic Acid lab. The wave model for light describes electromagnetic energy in terms of wave length, frequency and intensity (see Figure 1). One wavelength is represented by the time from one peak to the next in any wave front (also, one trough to the next). As the wavelength of the radiation becomes shorter, a larger number of waves per unit of time (i.e., the frequency), becomes greater. Wavelength is often expressed in meters, or nanometers for visible light, and frequency is usually expressed in hertz (Hz). One Hz is one cycle per second. The energy of light is calculated from frequency using equation 1: $E = h\upsilon$ equation1 where E is the energy in joules, h is Planck's constant (6.626 x 10^{-34} J·s), and υ is the frequency in Hz.

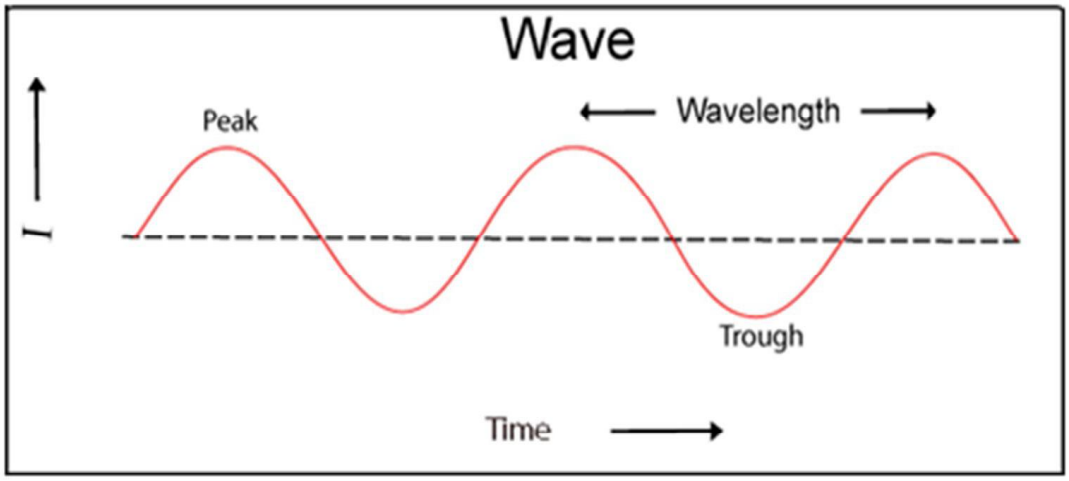


Figure1. The wave model for light

The symbol for wave length is the Greek letter lambda, λ, and the symbol for frequency is the Greek letter nu, υ. The speed of light in a vacuum is 3.0 x 10^{8} m/s. Since the speed of light is a constant, we can use it to calculate wavelength if we know the frequency of radiation (see sample Problem 7.1 in Silberberg). The relationship is shown in equation2:

$\lambda\upsilon = c$ equation2

where λ is the wavelength of radiation, υ is the frequency of radiation, and c is the speed of light (3.0 x 10^8 m/s). White light is composed of all the wavelengths within the visible region of the spectrum (see Figure 7.3 in Silberberg). When white light falls on an object, some of the incoming radiation may be absorbed. Wavelengths that are not adsorbed will be transmitted from the object. In the example below (Figure 2), the white egg does not adsorb any of the incoming radiation, and therefore appears white. The apple adsorbs blue and green radiation from white light, transmitting red light. Therefore, the apple appears red.

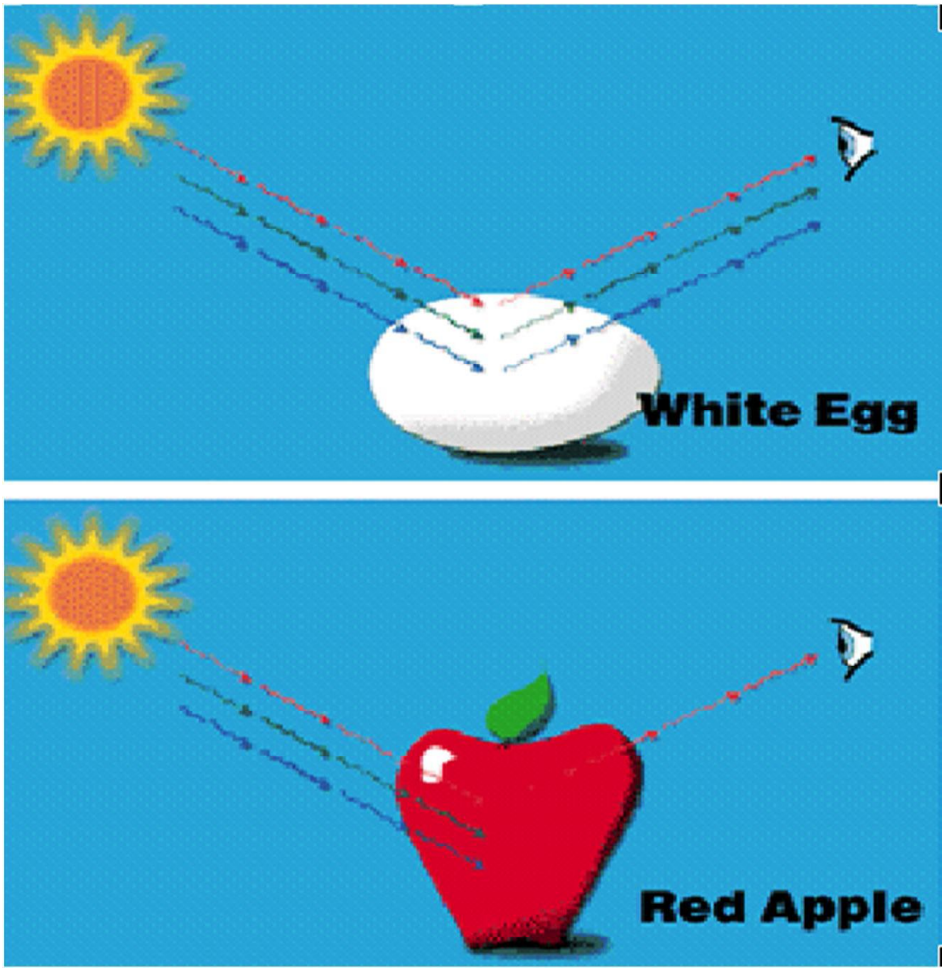
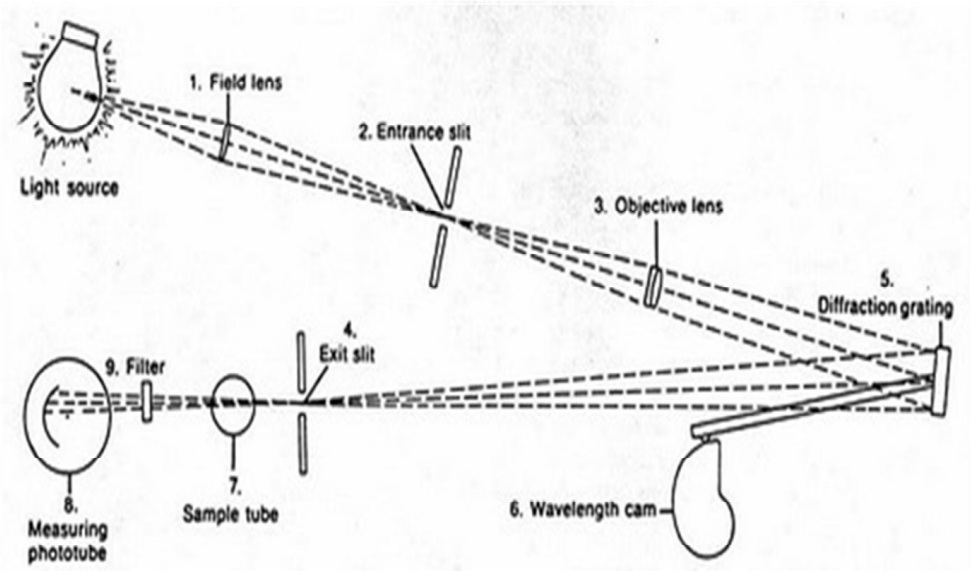


Figure 2. The white egg in this illustration does not absorb red, green or blue light, and appears white when illuminated with white light. The apple absorbs green and blue light, and transmits red light. The apple therefore appears red. Colors are often used to identify objects. The spectrum of an object is a graph of the absorbance of that object plotted against the wavelength of light. At the molecular

level, spectra are used to identify unknown molecules. The peaks in the spectrum can be used to identify unique components within a molecular structure. Notice that the chlorophyll spectrum has intense adsorption peaks in the blue and the red regions of the spectrum. Why do you think chlorophyll is green?

Spectrophotometers are used to measure the amount of light absorbed or transmitted by a sample. The instrument disperses white light into its component wavelengths by passing light through either a prism or a grating. The intensity of light at any wavelength can be measured with a detector, such as a photocell, a photomultiplier tube, or a solid-state device called a charge-coupled device (CCD). The optical path of the Spectronic 200 used in today's experiment is shown below in Figure 3:



Absorbance is the ratio of the negative logarithm of light intensity transmitted from a sample divided by the intensity of incoming light. This is expressed mathematically in equation 3:

$$-\log I/I_o = A \quad \text{equation-3}$$

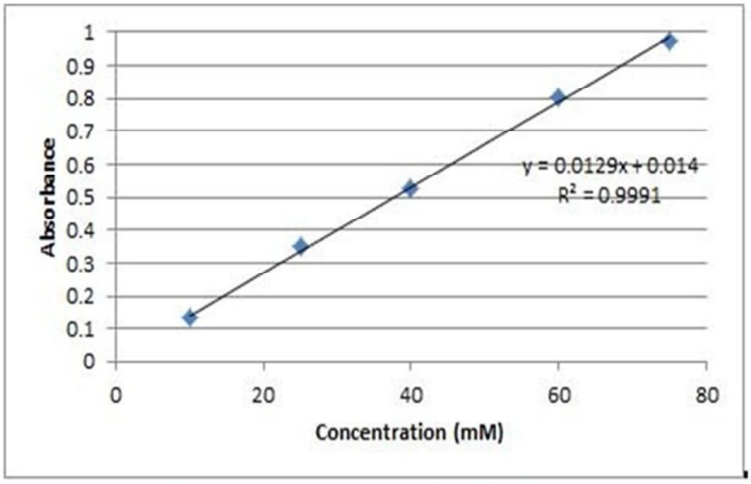
This relationship can be used to measure the amount of material present by measuring the intensity of the absorption peak at a specific wavelength. In today's experiment we will measure the intensity of the colored complex that forms when iron (III) is mixed with aspirin to determine the amount of pure aspirin (acetylsalicylic acid) in commercial aspirin tablets.

The Beer-Lambert Law (often shortened to Beer's Law) relates the absorbance of a sample to the concentration of a species in solution and is the relationship used when making quantitative measurements. Mathematically, Beer's law is expressed as shown in equation4:

$$A = \epsilon l c \quad \text{equation4}$$

Where, A is the measured

absorbance of the solution, $\epsilon$is the molar absorptivity of the substance, *l* is the path width for the cell, and c is the concentration. By measuring the absorbance of several solutions of known concentration, we are able to prepare a graph that can be used to determine concentrations of unknowns. A graph of absorbance vs concentration is called a Beer's Law curve in honor of the chemist who first discovered the relationship between absorbance and concentration. Figure 4 is Beer's Law curve for the absorbance of an iron-salicylate complex (the substance prepared in today's experiment) plotted against different concentrations.



Absorbance vs Concentration of Fe(III)-salicylate complex.

Figure 4. Beer's Law plot of Fe(III)-salicylate complex. *Note: The graph you generate in lab may not look exactly like the example above.*

Acetylsalicylic acid, commonly known as aspirin, absorbs light in the UV region of the electromagnetic spectrum. The Spectronic 200 operates in the visible region. Therefore, we must perform a series of chemical reactions to convert acetylsalicylic acid to a colored complex. In reaction 1, a base (e.g., sodium hydroxide) hydrolyzes acetylsalicylic acid to yield salicylate dianion. In reaction 2, acidification converts the dianion to a monoanion, which complexes with iron(III) in reaction 3 to produce a violet-colored complex.

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