Experiment-7

Determination of Caffeine and Acetylsalicylic Acid in an Analgesic Tablet by UV-Vis. Spectroscopy

Outcomes:-

After completing this experiment, the student should be able to:

1. The calculations of determination of Caffeine and Acetylsalicylic Acid in an Analgesic Tablet by UV. Vis. Absorption Spectroscopy.

Introduction

In this experiment, you will determine the amount of caffeine and acetylsalicylic acid in an analgesic tablet by UV-Vis. Spectroscopy; many molecules absorb ultraviolet or visible light, when an atom or molecule absorbs energy; electrons are promoted from their ground state to an excited state.

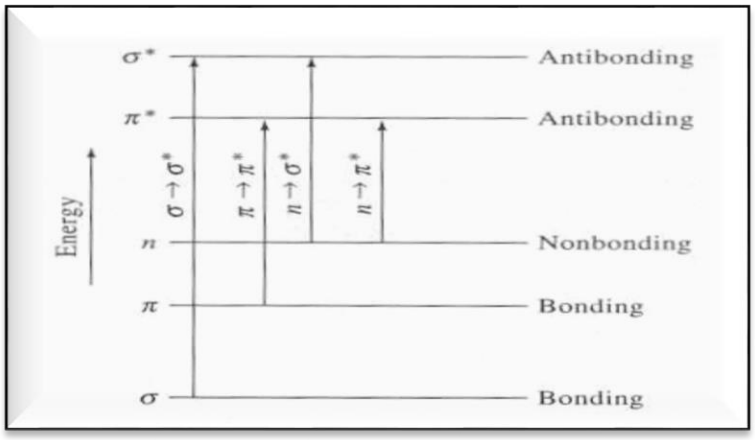

Figure1:- Molecular energy levels

Absorption spectrometry involves measuring the fraction of light of a given wavelength that passes through a sample, when a monochromatic light beam passes through a layer of solution with a thickness *b* and a concentration *c* of an absorbing species, as the consequence of interactions between the photons and absorbing particles, the power of the beam is attenuated from P_o to *P*. The absorbance *A* of a solution is defined by the equation:

$$A = -\log (P / P_o) = abc$$

This is Beer's law; where *a* is a proportionality constant called **absorptivity** and *b* is the path length of the light beam through the absorbing medium. When the *c* is expressed in M (moles per liter), and *b* in cm, *a* is called the **molar absorptivity** and is given the special symbol ε, with the units of L cm^{-1} mol^{-1}, Thus**,**

$$A = \varepsilon bc$$

Absorbance is an additive parameter, that is, the total absorbance, A_t of a sample containing more than one component that can absorb the same wavelength is the sum of the individual absorbance, A_i:

$$A_t = \Sigma A_i$$

The additive characteristic of *A* enables absorption measurement to be applicable to multi-component analysis without separation of the components using simultaneous equations to determine the concentrations, in this case it is necessary that the absorptivity of the substances be different at various wavelengths but not necessary zero for one of the components.

In this experiment, there are two components caffeine and acetylsalicylic acid in the analgesic tablet; you will first determine the molar absorptivity ε of each component by constructing a calibration curve (absorbance vs. concentration) with standard solutions; then by measuring absorbance of the tablet solution at maximum absorption wavelength of both components, you will be able to figure out the amount of each component in the tablet.

Reagent:-

Methanol, reagent grade; Caffeine; Acetylsalicylic acid; and Analgesic tablet

Preparation of solutions

1. *Caffeine stock solution* – Dissolve 0.024 g of caffeine in 50 mL volumetric flask with methanol and dilute to the mark.
2. *Acetylsalicylic acid* (ASA) stock solution – Dissolve 0.024 g of ASA in 50 mL volumetric flask with methanol and dilute to the mark.
3. *Analgesic sample solution* – Dissolve 0.06~0.09 g of an analgesic tablet or the content of an analgesic capsule with 20 mL of methanol in 50 mL volumetric flask with methanol and dilute to the mark. Make three sample solutions out of three tablets.

Procedure

1. Prepare working standards and sample from the stock solutions in the following manner.
2. Run the spectra for ASA5 and Caffeine5; determine the wavelength of maximum absorbance (λ_{max}) for each chemical.
3. For each solution in the table, measure the absorbance at the two selected wavelengths.

Standard no.	Caffeine stock added (mL)	ASA stock added (mL)	Total volume (mL) Diluted w/ methanol
ASA1	-	0.5	50
ASA2	-	1.0	50
ASA3	-	1.5	50
ASA4	-	2.0	50
ASA5	-	2.5	50
Caffeine1	0.5	-	50
Caffeine2	1.0	-	50
Caffeine3	1.5	-	50
Caffeine4	2.0	-	50
Caffeine5	2.5	-	50
Test	0.5	1.0	50
Sample1	2.5mL of sample solution 1		50
Sample2	2.5mL of sample solution 2		50
Sample3	2.5mL of sample solution 3		50

Data Treatment

1. Tabulate the absorbance at the two selected wavelengths and concentration for the ASA and caffeine standard solutions.
2. Plot absorbance vs. concentration (M) for both caffeine and ASA at the two selected wavelengths. Determine molar absorptivity for each component at those two wavelengths from the calibration curve.
3. Calculate the expected absorbance for test at each wavelength and compare to that of the experimental values.
4. Determine the weight percent of each component in the original analgesic tablets and report the standard deviation.
5. Compare your result with the amount labeled on the bottle and report the precision of your results.

Questions

1. Would benzene be a satisfactory solvent for the analysis?
2. If a pharmaceutical mixture has three components with different maximum absorbance, comment the possibility of determining all three components simultaneously.
3. Would it be more desirable to use spectra grade methanol for the blank solution? Justify your answer.

Note

1. If at any time any sampling variable changes including integration time, averaging, boxcar smoothing, distance from light source to sample, etc. you must store a new reference and dark spectrum.
2. Turn the light source power on. The switch is located at the back on the cuvette holder. Allow half an hour for the lamp to warm up before the experiment starts.