Experiment-2

Spectrophotometric determination of Aspirin in tablets By standard addition method

Outcomes:-

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

1. Prepare standard solutions of sodium salicylate.
2. Construct standard addition method based on Beer's Law.
3. Camper between calibration curve and standard addition methods.

Introduction:-

Direct calibration curve method can be applied for analyzing unknown sample only and only if the standard_solutions and the unknown solution are prepared and measured under exactly the same conditions, however, there are frequently what are known as **matrix effects**, where substances other than the analyte affect the absorbance reading of the sample solution at the wavelength chosen for the analysis, we can avoid the complication of matching the matrix of the standards to the matrix of the sample by conducting the standard addition method, in this method, the matrix, that present in the sample, will affect the absorbance readings for both the analyte in the standard and the sample in the same way at the same time, thus, it is possible the analyze the analyte accurately even in the presence of matrix.

In certain circumstances the matrix, defined as everything except the analyte, contributes significantly to the absorbance of a sample and is also highly variable, one method that can be used to improve results is the method of standard additions, the basic idea is to add standard to the analyte sample so that the standard is subjected to the same matrix effects as the analyte, this method assumes that the system obeys the Beer's Law, the ASA-Fe^{3+} complex formed in the analysis of aspirin absorbs wavelengths in the blue, green and yellow regions of the spectrum, as shown in figure1

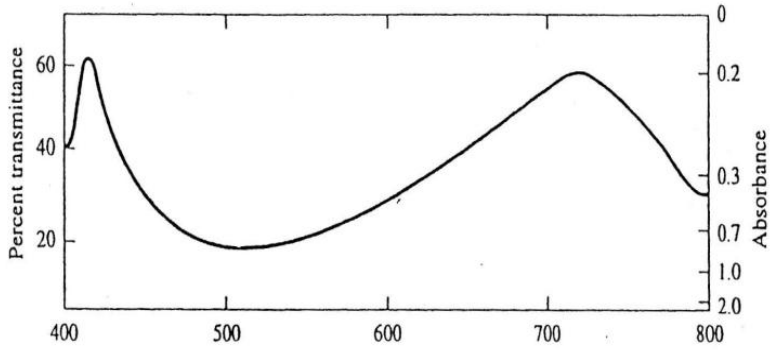
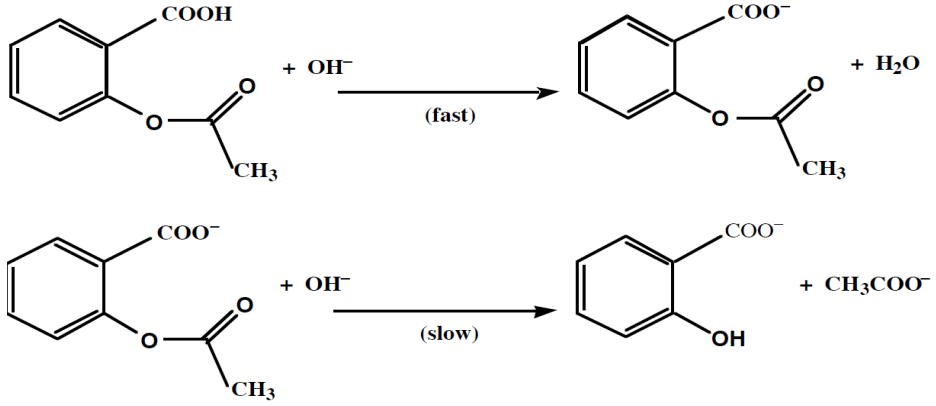


Figure 1: The absorption spectrum of the Fe^{3+}-salicylate complex.

The fast aspirin + NaOH (acid-base reaction) consumes one mole of hydroxide per mole of aspirin, the slow aspirin + NaOH hydrolysis reaction also consumes one mole of hydroxide per mole of aspirin, and so for a complete reaction **we will need to use a total of twice the amount of NaOH that you have already used, plus we will add some excess NaOH:-**



For good analysis the sample must have these properties:
(1) Stability in solution. (2) Adherence to Beer's law. (3) Large molar absorptive.
(4) Sufficient separation of the desired analyte absorbance wavelength from interfering substances.
If Not, the substance is usually converted into a new species suitable for quantitative spectroscopy.

Sample + Chromogenic Reagent → Uv-Vis Absorbing Product

Materials and Equipment:-

UV/VIS spectrophotometer and polystyrene cuvettes or quartz cuvettes (Absorption cells), pipette, 10mL graduated cylinders, 250, 100, 50& 25mL volumetric flasks**,** beakers, Aspirin tablets, Ferric nitrate, Nitric acid, ethanol, sodium salicylate.

Procedure:-

Preparing the stock solution and standard solutions and sample solution:-

1. **Stock solution of sodium salicylate:** Weight a 1.16 g of sodium salicylate in beaker, dissolve the solid by the addition of D.W., than transfer to volumetric flask (1L), and continue adding D.W. to the mark on the volumetric flask.
2. **Ferric Nitrate:** Dissolve 1.0 g ferric nitrate in 99 mL of water to make a 1% solution of ferric nitrate. (Total volume = 100 mL of 1% ferric nitrate).
3. **Nitric Acid I:** Prepare 100 mL of 0.07 M nitric acid.
4. **Aspirin sample:** Accurately record the weight of a group of **ten** aspirin tablets so that you can determine an average tablet weight, use a mortar and pestle to crush enough tablets to produce **an average** (g) tablet powder, using a clean dry weighing

bottle (beaker or conical flask), add 20 mL of ethanol (measure by graduated cylinder), swirl gently to dissolve. **(Aspirin is not very soluble in water, the ethanol helps the aspirin dissolve, note that an aspirin tablet contains other compounds in addition to aspirin, some of these are not very soluble, and the solution will be cloudy due to insoluble components of the tablet),** add 75mL of NaOH (0.1N) than heat in a water bath to speed up the hydrolysis reaction**, avoid boiling, because the sample may decompose**, while heating, swirl the beaker occasionally, after 15 minutes, remove sample from the water bath and cool for 5 minutes, than filtered the solution.

Student Preparations:

1. **Standard solution:** Transfer 5.00 mL of stock solution to a volumetric flask (100mL), than dilute with D.W. to mark (Its concentration is …… mg/L).
2. **Dilute Ferric Nitrate:** Mix 5 mL of 1% ferric nitrate with 4 mL of 0.07 M HNO3 (nitric acid I) and label the container "dilute ferric nitrate."
3. **Unknown solution:** Transfer 5.00 mL of Aspirin sample solution to a volumetric flask (10mL), than dilute with D.W.

Procedure for spectrophotometer instrument in the visible range:-

1. Turn on the instrument and allow it to warm up for about 10 min.
2. Set the wavelength to λ_{max} nm.
3. Depress the "mode" control key and set the mode to "Absorbance."
4. Fill seven volumetric flask (5.0mL) with varying amounts of solution and adjust the volumes of each to 5.0 mL by D.W. as shown in Table 1 below:-

Sample	Vol. of Standard (…..mg/L)	Vol. of Dilute Ferric Nitrate	Unknown	Absorbance	Concentration mg/L
Blank	**0.0**	**1.0**	**0.0**	**0.0**	**0.0**
# 1	**0.0**	**1.0**	**1.0**		**0.0**
# 2	**0.2**	**1.0**	**1.0**		
# 3	**0.4**	**1.0**	**1.0**		
# 4	**0.6**	**1.0**	**1.0**		
# 5	**0.8**	**1.0**	**1.0**		
# 6	**1.0**	**1.0**	**1.0**		

5. The zero absorbance will be determined with the blank solution cuvette.
6. Replace the blank cuvette with each of the numbered samples and determine absorbance for each solution, carefully record the **A** as you go into your lab notebook vs **concentration**.
7. Draw a plot having X-axis as concentration (mg/L) and Y-axis as Absorbance at $\boldsymbol{\lambda_{max}}$ (*Plotting* Use the program Excel to plot the calibration curve**).**

8. Use Beer's law to calculate **ε** for sodium salicylate, given the cell width (path length l cm).
9. Use the curve to calculate concentration of unknown solution.