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Spectrophotometric Determination

10/15/2019

Objective

This experiment is conducted to determine the concentration of aspirin in the analgesic tablet by using the calibration curve of known standard aspirin solution's concentration. This is done by using the spectrophotometer in LabQuest 2 to determine the light absorption of each sample.

Introduction

As stated in the objective, the spectrophotometer in LabQuest 2 will be used as a device to measure the absorbance of light for every measured sample in order to plot the calibration curve of "absorption versus concentration" graph. The unknown ~~sample~~ concentration sample is done the same as the previous samples and are ensured to fall within the calibration curve in order to determine the concentration of aspirin in the tablet. The samples are mixed with appropriate reactants in order to produce the products that we are interested in (to analyze).

Procedure

A) Measurement of Absorption Spectrum.

*important point 1:

- all solutions will be transferred with transfer pipets, not serological pipettes
- make sure to rinse the pipet with a small volume aliquots of tranferred solution before making the actual transfer.

*important point 2:

- measure the absorbance at the entire spectrum, rather than just at the λ max (~~max~~ wavelength with max. absorbance)

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Concentration of standard solution of aspirin (stock solution) (ppm) → ~~202.4 ppm~~ 202.4 ± 0.8 ppm

Initial volume (mL) → 5 mL

Final volume (mL) → 25 mL

Final concentration (ppm) → $\frac{5}{25}(202.4) = 40.48$ ppm.

Tablet data	
Absorbance of tablet solution	0.2262
[aspirin] after third dilution (ppm)	29.011
Third dilution factor	25
[aspirin] after second dilution (ppm)	725.27
Second dilution factor	2
[aspirin] after first dilution (ppm)	1450.53
[aspirin] in the tablet (mg)	362.6
Weight % aspirin in tablet (%)	98.70

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1. Pipette carefully 5 mL of standard aspirin solution into a 50- or 100-mL beaker. Record the actual concentration in ppm.

2. Add ~~5~~ 2.0 ± 0.2 mL of 0.5-M NaOH to the beaker and allow the solution to stand at least 20 minutes. (the aspirin is quantitatively converted to salicylate and acetate ions by the action of hydroxide, at this time).

3. Make a blank solution: pipette ~~5~~ 2.0 ± 0.2 mL of 0.5-M NaOH into a 25-mL volumetric flask. Add 4 mL of 0.1-M Fe^{3+} with swirling. Dilute to the mark with DI water. (if enough cuvettes, keep and reuse it)

4. Quantitatively transfer the converted aspirin solution from before into a 25-mL volumetric flask. Rinse the beaker with distilled water, making sure to empty the insate into the flask.

5. Add 4 mL of 0.1-M Fe^{3+} solution to the above flask with swirling. (precipitation may happen but it can be dissolved by mixing, due to excess of OH^- precipitate $Fe(OH)_3$).

6. Dilute to the mark with distilled water and mix the flask well by repeatedly inverting the flask. (the iron-salicylate complex now have been prepared).

7. Make the first absorbance reading at λ_{max} (should be near 530 nm, but use my own optimized value from spectrum).

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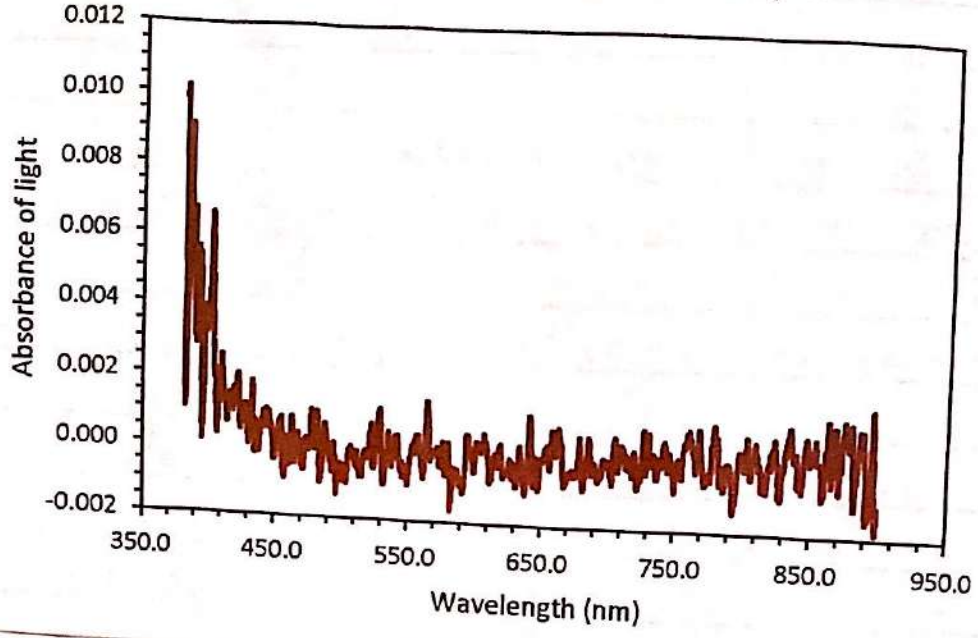
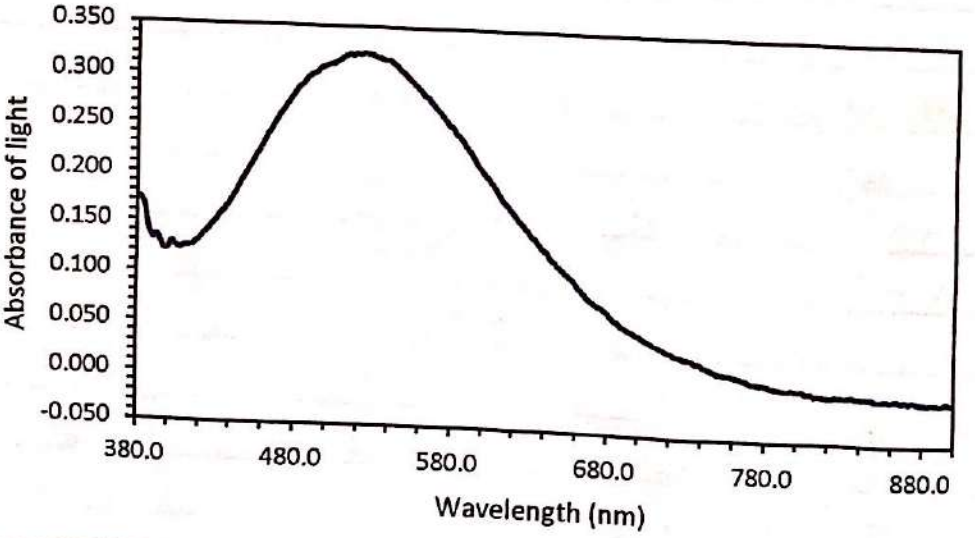
Absorption spectrum of blank sample



Absorption spectrum for 40.48 ppm aspirin solution



8. Insert the blank solution's cuvette and calibrate cuvette. Be sure the reading is stable. Make sure orientation of cuvette stays the same.

9. Remove the cuvette containing the blank and replace it with the cuvette containing the iron–salicylate complex. Collect the absorbance at λ_{max} at a minimum, or, collect the entire spectrum.

B) Generation of a Calibration Curve for the iron salicylate complex.

1. Choose the wavelength that is suitable for the measurements (530 nm is close to the ~~blank solution~~ wavelength of max. absorbance for complex, but not so low that the blank solution absorbs excessively.

2. Repeat the steps in part A), but pipette 1 mL of standard aspirin solution. We can continue to use the same blank solution prepared previously. The iron-salicylate complex is not extremely stable so it is important to measure the absorbance within a known & reproducible time after preparation. (5 minutes is recommended — don't guess, use stopwatch).

3. Repeat with 2 mL, 3mL & 5mL of standard aspirin

4. The repetition of 5-mL quantity is done to ~~measure~~ ensure that the time elapsed before measurement of absorbance is the same for all standard solutions.

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Weight of aspirin tablet (g) → 0.3674g

At wavelength 523.1 nm (Standard aspirin)							
Volume of aspirin solution (mL)	Concentration (ppm)	Concentration (mg/mL)	Replicate measurement of absorbance			Average	Standard deviation
			1	2	3		
1.00	8.10	0.0081	0.0536	0.0528	0.0526	0.0530	0.0005
2.00	16.19	0.0162	0.1169	0.1160	0.1150	0.1160	0.0010
3.00	24.29	0.0243	0.1809	0.1867	0.1872	0.1850	0.0035
5.00	40.48	0.0405	0.3235	0.3246	0.3238	0.3240	0.0006
-	29.01	0.0290	0.2262	0.2262	0.2261	0.2262	0.0001

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5. Repeat the process for the second & third time & use the average value with a standard deviation as the error bars in the calibration plot. (because the time after mixing is critical, it is not possible to simply re-measure each solution a second & third time)

6. Make a plot of absorbance at the chosen wavelength versus aspirin concentration (ppm) in the flasks for the 1-, 2-, 3-, & 5-mL aspirin standards. Remember to account for the dilution of the aspirin in the flask. Ignore the effects of hydrolysis reaction on the aspirin present because it has been quantitatively converted the aspirin to the iron-salicylate complex → concentrations of aspirin = caplex's.

7. Use method of least-squares to determine the best straight line for the working calibration curve generated above.

C) Determination of Aspirin in Analgesic Tablet

1. Weigh an aspirin tablet to the nearest 0.1mg.

2. Place it in a clean 50-mL beaker and add 10mL of 0.5-M NaOH with graduated cylinder.

3. Crush carefully the tablet with a stirring rod.

4. Allow the NaOH to act on the tablet to soften it for a few minutes. Dissolve as much of tablet as possible, then let stand at least 10 more minutes.

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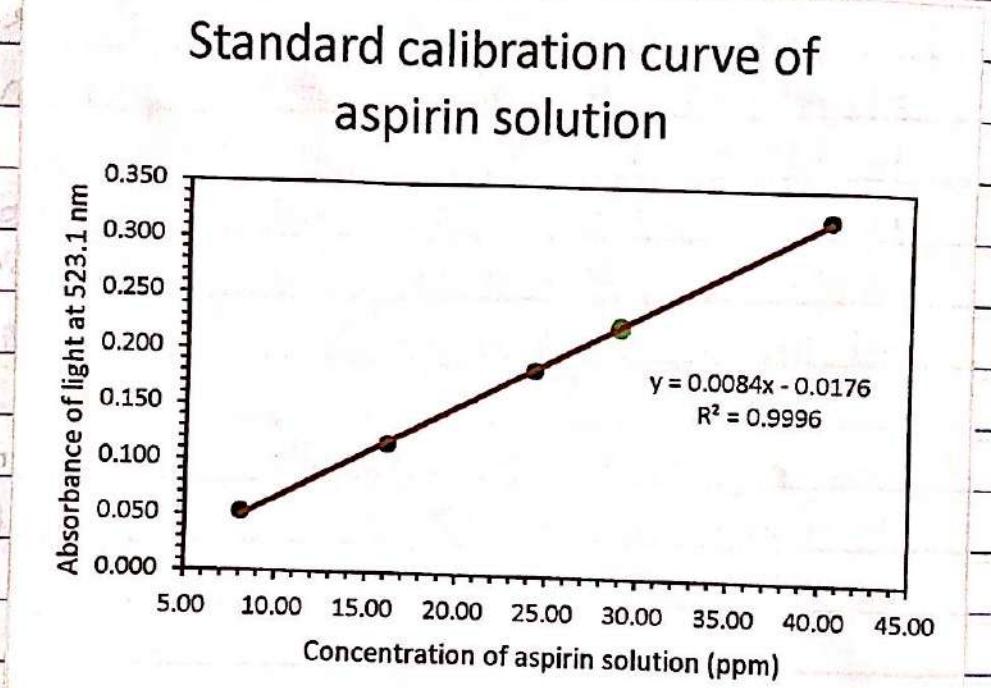
LINEST output			
m (per ppm)	0.008404	-0.01762656	b
um (per ppm)	0.000123	0.003099104	ub
R^2	0.999575	0.002935877	sy
Fisher value	4699.434	2	d.f
regression SS	0.040506	1.72388E-05	residual SOS
Equation of the line		y = mx + b	



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5. Quantitatively transfer the solution to a 250-mL volumetric flask, being careful to collect all solution and rinsings in the flask. Dilute to the mark and mix thoroughly.

6. There may be a faint cloudiness due to undissolved binder from the tablet.

7. Take 50mL of the diluted solution & transfer to a 100-mL volumetric flask and dilute to the mark with DI water. (use 25-mL pipet; 2 times transfer)

8. Make a blank solution as before

9. Pipet exactly 1 mL of the final unknown analyte solution into a 25-mL volumetric flask. Add ~~5~~ 2 mL of 0.5-M NaOH plus 4 mL of 0.1-M Fe3+ solution, the latter with swirling. Dilute ~~the~~ to the mark with distilled water. Mix well. Be consistent in the way of making all the standard & unknown solutions.

10. Set the zero on the instrument using the blank. Measure the absorbance of unknown analyte solution at the chosen analytical wavelength. If the absorbance is not in the appropriate range for an accurate determination, repeat the above steps with a different volume of unknown solution in order to be within the standards' concentrations.

11. Use time efficiently to perform at least 3 replicate measurements on the unknown.

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Analysis and Results

The analysis and calculations were ~~made~~ written and made in the summary report & excel file respectively. The calibration curve was plotted and the unknown concentration of aspirin in the tablet was ensured to fall within the calibration curve. The green ~~curve~~ point on the curve is the tablet's aspirin absorbance measurement.

Conclusion

The calibration curve is used to determine the concentration of aspirin in the analgesic ~~tablet~~. The concentration is found to be 362.6 mg of aspirin in the tablet and contains 98.70% in weight of aspirin in the tablet.