Exp. No. 2	Experiment/Subject CHEM 438 (NDL)	Date 9/20/2021	
Name Abdul Fayeed	Lab Partner Giana Alepa	Locker/ Desk No.	Course & Section No. 021L

Pre-Lab Assignment

upload to canvas!

1. Upper limit of A ~3 ~~(or 2.5, a bit lower)~~

Wavelength = 290 or 330 nm. (use λ = 340nm)

For λ = 290nm, wavenumber = 34,482.76 cm^{-1}

" λ = 330nm, " = 30,303.03 cm^{-1}

molar extinction coefficient [=] $M^{-1} cm^{-1}$

For λ = 290 nm, ε ≅ 2750 $M^{-1}cm^{-1}$

" λ = 330nm, ε ≅ 7900 $M^{-1}cm^{-1}$

λ = 340nm ?

Upper Limit Absorbance	3		2.5		0.56
λ (nm)	290	330	290	330	340
ε ($M^{-1}cm^{-1}$)	2750	7900	2750	7900	9750
b (cm)	1.00	1.00	1.00	1.00	1
Max. Conc. (M)	1.091E-03	3.797E-04	9.091E-04	3.165E-04	5.744E-5
Conc. 1 (M)	5.455E-04	1.899E-04	4.545E-04	1.582E-04	2.872E-5
Conc. 2 (M)	2.727E-04	9.494E-05	2.273E-04	7.911E-05	1.436E-5
Conc. 3 (M)	1.364E-04	4.747E-05	1.136E-04	3.956E-05	7.179E-6
Conc. 4 (M)	6.818E-05	2.373E-05	5.682E-05	1.978E-05	3.590E-6
Conc. 5 (M)	3.409E-05	1.187E-05	2.841E-05	9.889E-06	1.795E-6
Min. Lowest Conc (M)	4.364E-05	1.519E-05	3.636E-05	1.266E-05	2.297E-6

⇒ some possible concentrations of Quinine -

conc. 1 = ~~5.455~~ 5.673 x 10^{-4} M

conc. 2 = ~~2.727~~ 2.618 x 10^{-4} M

conc. 3 = 1.309 x 10^{-4} M

conc. 4 = 8.727 x 10^{-5} M

conc. 5 = 4.364 x 10^{-5} M

⇒ Final concentration made!!

ε = 9750 $M^{-1}cm^{-1}$ Final ε used!

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PRE-LAB

2. No need to make new solutions. Just repeat the blank many times. ~~(not sure why)~~ To get the avg, std. dev. then LOD.

3.

Molar mass (g/mol)	782.96			
Stock soln. volume (mL)	50			
Stock soln. Conc (M)	1.091E-03	3.797E-04	9.091E-04	3.165E-04
Mass of salt for stock (g)	0.0427	0.0149	0.0356	0.0124
Total vol. after dilution (mL)	25	25	25	25
Vol. of stock soln. added (mL)				
1	12.500	12.500	12.500	12.500
2	6.250	6.250	6.250	6.250
3	3.125	3.125	3.125	3.125
4	1.563	1.563	1.563	1.563
5	0.781	0.781	0.781	0.781
Total volume added (mL)	24.219	24.219	24.219	24.219

Final Stock solution used! (annotation pointing to 1.091E-03 / 0.0427)

Handwritten beside rows 1–5: 13, 6, 3, 2, 1

Notes: Consider which instrument to use, and jot it down!

~~For 3LOF → mass = 0.00225 g For stock of 5.744 E-5 M~~

Final volume added
13, 6, 3, 2, 1 mL
in order of conc. mentioned from prev. page

⇒ Possible mass of quinine sulfate to be added.

4. If P_0 is drifting, it will hurt the LOD, because it impacts sensitivity. Noise is from the fluctuations of lamp intensity, which are random on short time scales, but slowly drifting over long time scales. Recording absorbance over time and applying the slope of the drift to correct the measurements can detect the drift. Take measurement with a blank for a shorter amount of time. ~~to ca~~

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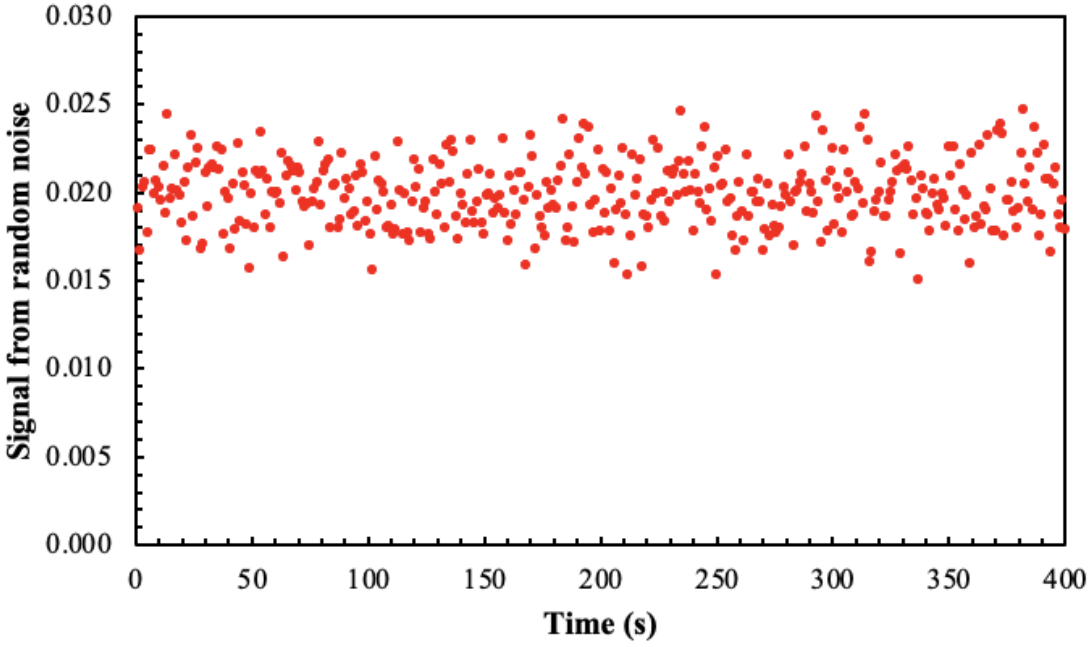

Figure 1. Signal from the random noise fluctuates with time.

Standard deviation for random noise data = 0.0019

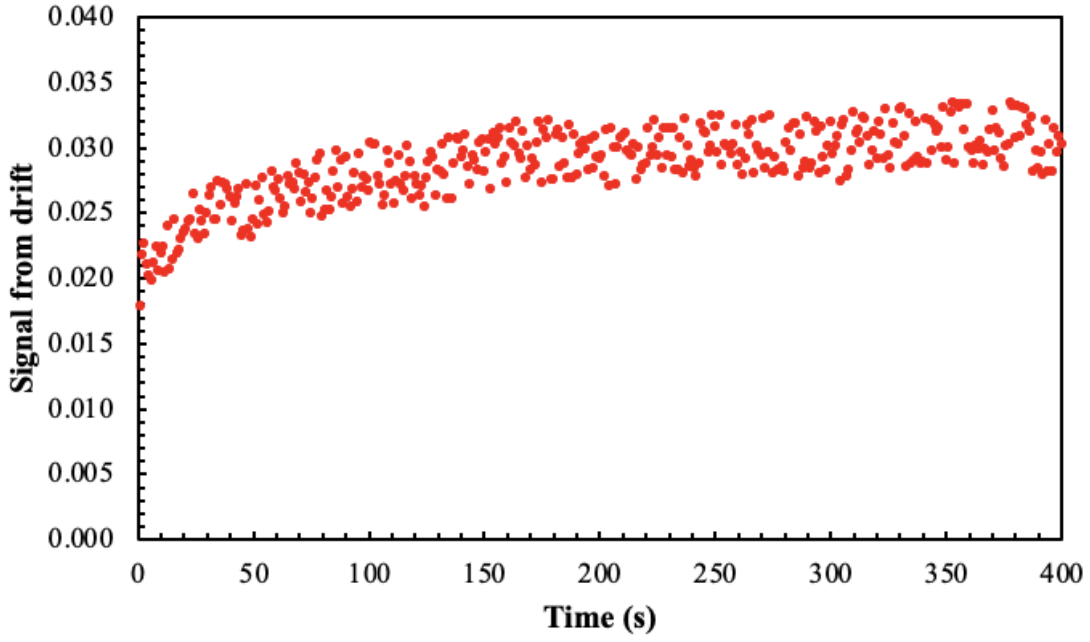

Figure 2. Signals from the drift fluctuate with time.

Standard deviation for drift data = 0.0028

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Objective

Purpose of this experiment is to explore the limitations of Beer's Law, by using the UV-Vis instrument to collect the data. Student will be exposed of experience in observing the behavior of noise in data and the estimation of detection limits

Introduction

Beer's Law has 2 limitations: a) Accuracy limit at high absorbance, and b) Detection limit from the effect of noise due to light source. For a), there is a maximum absorbance where the absorbance still follows Beer's Law, before it curves down and disobey linearity. Signal due to background overpower the signal due to sample, which creates this inaccuracy at high absorbance. For b), added random noise fluctuates the data at both the high and low-concentration region. This will affect the detection limit of the sample and gives inaccurate data.

Procedure

PART 1

1. Prepare 5 solutions from the concentration table from pre-lab.
2. Dilute the solution with 0.05 M H_2SO_4
3. Record which instrument is used for each dilution.

All data saved in txt. file in a drive, nothing is recorded in this page.

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PART 2

1. Measure absorbance and record the data on a USB stick. TA will guide with the instrument.
2. In the instrument settings, use a cycle time equal to integration time.
3. Study the noise on the blank. Use "kinetics mode" in the software. Use only blank!
4. Choose >= 340nm, integration of 1s.
5. Record blank measurement, then record signal for the same blank sample for 64s.
6. Save the data, using last name as file name.
7. Record a blank measurement for integration time of 1s, but acquire for 448s. (will give same # data points)
8. Repeat step 7 using integration time of 15s, but with a 960-s acquisition time.
9. Dispose of all chemicals properly.

Conclusions

Absorbances are collected at 340nm for all 5 concentrations. The data are saved in a .txt. file to be analyzed in the final report. Same goes to the blank measurement collected at 3 different integration times.

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