**Homework #2 : Calibration Curves & Intro to UV-VIS methods 20 pts total**

*Chem 6614 Chemical Instrumentation*

*Due Wed 8 Feb 2017*

*Your name: \_\_\_\_\_\_\_\_\_\_answers\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_*

***2.1. Lambda 4b classic double beam questions***

1. The Alfred double beam Lambda 4b UV-VIS generates the UV end of the spectrum with what kind of lamp? ***\_\_\_\_\_deuterium\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_***
2. What mechanical device allows the Alfred double beam Lambda 4B instrument to switch between the reference and sample cuvette?

 ***\_\_\_\_\_optical chopper\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_***

3) Higher wavelength resolution is correlated with smaller monochromator slit width, which is always desirable. Why then does the Alfred double beam Lambda 4B instrument instructions suggest setting larger slit widths as scan rates are increased?

 ***Higher rates of scan lower the net amount of light seen by the sample and so decreases sensitivity. This is compensated for by increasing slit widths but with a corresponding decrease in resolution since higher slit widths admit more wavelength range onto the sample***

1. Your text notes that while double beam instruments are the currently preferred because they allow convenient subtraction of backgrounds, scanning single beam instruments are still made. What fundamental advantage in signal processing does a single beam instrument have over a double beam instrument?

***More light throughput with just a single beam***

1. What is the name of the monochromator pathway used by the Lambda 4b ?

***Czerny-Turner***

***2.2. Lambda 25 state-of-the art UV-VIS questions***

6) What 2 features of the holographic, concave monochromator grating make it superior

 to the traditional echellette style grating ? (2 pts)

***a)concave surface allows focusing of light with monochromator(eliminating need for chopper)***

***b)higher blaze angle and rounded grating edges lowers light scattering***

***c) grating copies ~ same as master d) less spherical aberration***

7) What is the transducer in the Lambda 25 ?

**photodiodes**

8) What’s` missing’ from the modern Lambda 25 that was critical in the Lambda 4b ?

***Optical chopper***

9) A photodiode detector operated in reverse bias has what two advantages for detection? (2 pts)

***a)Output is linear with impinging light***

***b)Lowered capacitance provides faster response***

10) What component has essentially the same design in both the Lambda 4b and Lambda 25 ?

***a) sources (still D2 and tungsten halogen); b) cells***

***2.3. UV-Vis Practicalities***

11) What is the physical reason the `cut’-off of either UV-vis instrument is around 180 nm?

*quartzuv lamp housing start to absorb strongly below 180 nm*

12) If you require higher wavelength resolution, what two scan conditions should you

 change, and in which direction?

1. Slow down rate of scan

b) narrow the slit width

13) In practice, it is not possible to obtain a purely monochromatic, single wavelength output from a tungsten or deuterium lamp since both are continuous, polychromatic sources. When you analyze at a fixed wavelength-call it λnominal – you are actually using a narrow band of wavelengths clustered around λnominal . Based on what you can deduce by reading pp 340-341:

1. what part of compound Cu(II)’s spectrum shown would yield the most reliable Beer’s law plot? (circle it)



UV-VIS Absorbance Spectrum of Cu(II) in THF

1. Why ? Want a reasonably large A at

λmax which doesn’t change substantially in value

 if spectral resolution creates a bandwidth that

 samples wavelengths near λmax

14) If the uncertainty in T, sT, varies linearly with T, e.g

 ST ~ kT, then to minimize relative uncertainty in a

 determined sample concentration, it is best to:

1. Pick an analysis wavelength where sample has high A
2. Pick an analysis wavelength where sample has low T
3. Pick an analysis wavelength where sample has low A
4. Pick an analysis wavelength where the spectrum

 changes slowly with λ (hint: read pp 343-346, especially case III)

* 1. Use linear regression to find the concentration of Ni2+ in **M** which produces an absorbance =0.183 at 459 nm given the following calibration data for known concentrations of Ni2+:

**Absorbance at 459 nm for known concentrations of Ni2**+

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **[Ni2+ (M)]** |  **0.20** | **0.35** | **0.5** | **0.7** |
| **Observed A** | **0.13** |  **0.24** | **0.33** | **0.47** |

 ­unknown [Ni2+(M)] = \_\_\_\_\_\_\_\_0.27\_\_\_\_\_\_\_\_\_ *2 pts*

***Fit of data to form: A=m\*[Ni2+(M)] + b => A= 0.6707\* [Ni2+(M)] -0.00074 r2=0.9994 (included A=0,Ni=0) Substituting A=0.183 into the fitted equation:***

***0.183 = A= 0.6707\* [Ni2+(M)] -0.00074 => (0.183 + 0.00074)/0.6707=[Ni2+(M)] = 0.274 M🡪 0.27 (2 sig fig in data only)***

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