Name:____________________

1. (20 pts; 2 pt each) State the identity of each instrument shown on the computer screen. Then describe the distinguishing features of the instrument that helped you with the identification.

ID	Instrument	Distinguishing Features
A	Single beam PDA	poly chromator linear geometry
B	double beam UV-Vis in time	ref & sample chambers one detector & chopper
C.	ICP-OES	flame source echelle mono w/ prism & grating
D	flame AA	mono after sample linear geometry
E	Atomic Fluoresence	90° Bent geometry one mono after sample
F	Molecular Fluorimeter	two monos 90° Bent geometry mono before sample
G	double beam UV-Vis in space	two detectors ref & sample chamber
H	single beam UV-Vis	mono before sample
I	FT-IR	Interferometer before sample
J	Atomic emission Spectroscopy	source = sample in a flame sample before mono

2. (15 pts) A 3.31×10^{-4} M solution of a new drug (373 g mol^{-1}) has an absorbance of 0.750 in a 1.00 cm cell. A 0.0312 g tablet containing the drug is dissolved in water to a final volume of 500.0 mL and has an absorbance of 0.654 under the same conditions. What is the mass percent of the drug in the tablet?

$A = \varepsilon b C \quad \varepsilon = \frac{A}{bC} = \frac{0.75}{1 \cdot 3.31 \times 10^{-4} M} = 2266 \frac{}{Mcm}$

$C = \frac{A}{\varepsilon b} = \frac{0.654}{2266 \cdot 1} = 2.886 \times 10^{-4} M$

$2.886 \times 10^{-4} \frac{mol}{L} \times \frac{0.5 L}{} \times \frac{373 g}{mol} = 0.05383 g$

$\frac{0.05383 g}{0.0312 g} \times 100\% = 173$ % drug by mass

Must be a problem! Standard Addition?

8 min

3. (2 pts) Describe the attributes of random error and state how it can be quantified.

- all directions & variable magnitude
- no assignable cause
- quantify using standard deviation

4. (3 pts) List at least ~~four~~ three ways you could assess a method for systematic error.

Standard reference materials
analyze blanks
multiple methods
multiple lab

5. **(4 pts) Identify an example of instrumental error and give an example of how one type of instrumental error is adjusted for in real-life measurement**

Thermal noise results from thermal agitation of electrons in resistive elements. It can be minimized by cooling detectors or measuring & adjusting for dark current

results in long term instrumental drift

& calibrate instruments regularly using standards

6. **(6 pts) List 2 factors that can affect the linearity of Beer's Law. Describe how each would be observed in the data and how it would affect the data. Also, how can each factor be avoided or corrected?**

Factor	How can it be observed in the data? How does it affect the data? How can it be corrected for?
stray light	nonlinear calibration curves measured concentrations lower than true value Paint internal instrument components black & minimize dust
too much absorption	very few photons to detect at detector when absorption is high poor S:N dilute sample into linear range

7. **(5pts) What physical portion of the instrument controls throughput and resolution? How are they related? How do you determine the setting for the physical portion of the instrument controlling them?**

slits on a monochromator control both throughput (higher w/ wider slits) and resolution (higher w/ narrower slits).

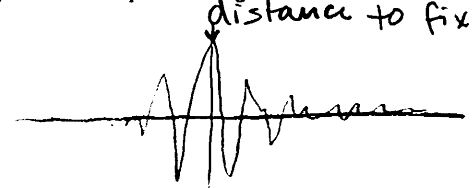
The choice of slit width depends on the analyte spectrum (are there fine features to observe?) and the goal of the experiment (quantify low levels or presence absence quickly).

smaller slits often increase time required to reach good S:N b/c of lower throughput

8. **(10 pts) Describe how FT-IR measurements are made by the instrument. Be sure to discuss the data domains that the information travels through as it moves through the instrument, interacts with the sample, and is detected.**

photons from a broad band source (frequency domain) pass through an interferometer. The interferometer consists of a moving & stationary mirror to which half of the beam travels to and back. The degree of interference of the recombine beams changes in time (data is in the time domain) based on the position of the moving mirror. The beam interacts w/ the sample and is detected by a thermal detector. The interferogram is converted back to frequency domain by fourier transform

(2 pts) Draw an example interferogram. Be sure to label the axes.



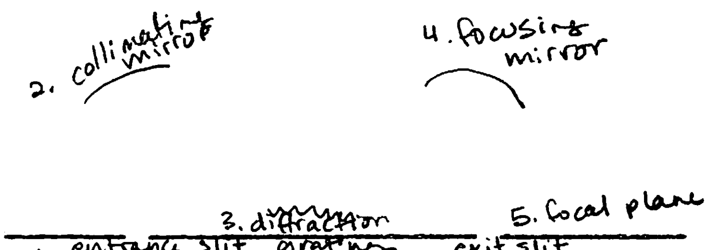
(3 pts) Draw an example output spectrum. Label the axes. Describe how the interfereogram is converted to the familiar output spectrum.

the interferogram is converted from time to frequency domain by fourier transform math, which deconvolutes signal into component sin waves

(1pt) what do regions of absorption mean?

that λ excites molecular vibrations of bonds

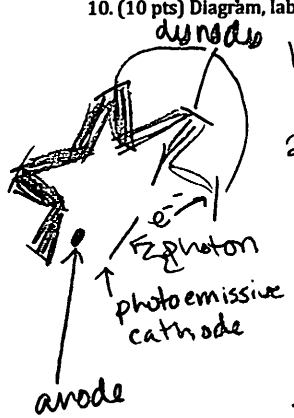
20 min



9. (10 pts) Diagram, label and describe how a monochromator works.

1. light enters the entrance slit, creating a rectangular image containing many λ.
2. the collimating mirror causes all light waves to propogate parallel to one another.
3. Diffraction or reflection grating breaks light into component λ
4. focusing mirror focuses each λ onto exit plane, recreating original rectangular image for each λ
5. light of the selected λ exits mono

10. (10 pts) Diagram, label and describe how a PMT works.



1. photon strikes photoemissive cathode, liberating electrons
2. electrons are attracted to dynode and each impact liberates several electrodes. dynodes are held at increasingly positive potentials (+90V), attracting liberated electrons
3. Cascade of electrons amplify 10^6 signal before electrons are collected at the anode
4. Anode current α photons

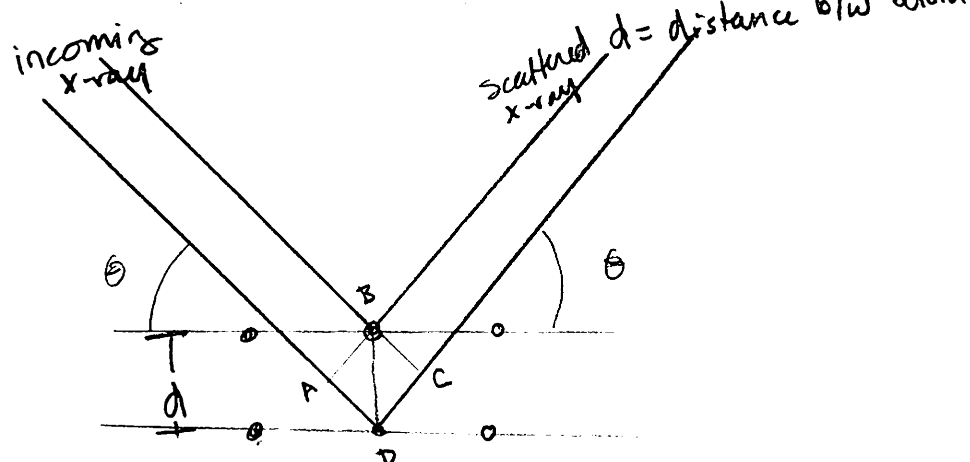
11. (10 pts) The diffraction pattern of copper metal was measured with X-ray radiation of wavelength of 1.315 Å. The first order Bragg diffraction peak was found at an angle 2θ of 50.5 °. Calculate the d-spacing between the diffracting planes in the copper metal.

$n\lambda = 2d \sin\theta$

$1 \cdot 1.315\,\text{Å} = 2d \sin 25.25°$

$d = \dfrac{1.315\,\text{Å}}{2 \sin 25.25} = 1.54\,\text{Å}$ ($\sin 25.25 \approx 0.4265$)

Extra Credit:(10 pts) Draw a schematic of how x-rays are diffracted off the crystal of sample atoms.



ray 2 travels $\overline{AD} + \overline{CD}$ farther than ray 1

$\overline{AD} + \overline{CD} = n\lambda$