

# Analytical Chemistry Cumulative Exam

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Topics related to practical aspects of optical spectra techniques

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Focus on those problems you can answer. Some correct, well thought out responses are better than poorly framed attempts at all questions.

*Note, since I am not in Chicago, grading will be delayed*

**Answer enough questions to total = 100 -- I will stop grading at ~ 100.**

*Optical components are central to being able to make spectroscopic measurements such as IR, Raman, UV, fluorescence, microscopy, etc. The key to choosing the best device to use is to first understand the basic principles behind its operation and to focus on which component has the greatest advantage for your experiment. This would include selecting optimal light sources and detectors for various measurements, it would include analysis of optics for imaging, and would include discussion and analysis of wavelength selective devices, which might include filters, monochromators and/or interferometers.*

**(24) - 1. For three of the following incoherent light sources**, explain the mechanism of operation, sketch a typical example and label components, give typical (relative) power and spectral region of optimal operation and then briefly describe an analytical experiment and/or spectroscopic instrument which typically uses that source, and explain why it is the best choice:

- |                           |                                  |
|---------------------------|----------------------------------|
| a. Tungsten-halogen lamp  | d. Hollow cathode lamp           |
| b. Xe-arc lamp (DC or cw) | e. D <sub>2</sub> discharge lamp |
| c. Glowbar (SiC)          | f. Xe flash lamp (pulsed)        |

**(24) - 2. For three of the following laser light sources or associated devices**, explain the mechanism of operation, identify the critical components by use of a sketch for a typical example, give typical power (or at least relative) and spectral region of optimal operation and then briefly describe an experiment where you would use that laser and explain why it is preferred.

- |  |  |
|--|--|
| a. Ar ion laser                        | f. Nd-YAG laser                                  |
| b. Ti Sapphire laser                   | g. Semiconductor diode visible laser (e.g. GaAs) |
| c. OPO - optical parametric oscillator | h. Lead salt (e.g. PbSnTe) IR diode laser        |
| d. FRED – frequency doubled Kr laser   | i. QCL - quantum cascade laser                   |
| e. Frequency doubler                   | j. Raman shifter (e.g. H <sub>2</sub> )          |

**(18) - 3.** For three of the following light detectors, explain the mechanism of operation, identify the critical components by use of a sketch of a typical example, note typical spectral region of optimal operation and then briefly describe an experiment and/or instrument where you would use that detector.

- |                      |  |
|----------------------|--|
| a. Photomultiplier   | e. Ge avalanche photodiode                     |
| b. DTGS pyroelectric | f. CCD – charge coupled device                 |
| c. InSb photovoltaic | g. MCT focal plane array detector              |
| d. Si photodiode     | h. doped Ge photoconductor detector, eg. Au:Ge |

**(36) 4.** Often it is important in analytical measurements to discriminate among wavelengths (frequencies) of the light used for measurement. One approach is a dispersive monochromator or spectrograph. Answer the following:

- a) Czerny-Turner monochromators are commonly used as free standing scanning monochromators (components) and even as small spectrographs in uv-vis experiments, but also can be used in the IR. Please describe, with a drawing, what is a Czerny-Turner design, label the important parts, and state what advantages (or disadvantages) it has as compared to other designs (for example, Fastie-Ebert, Seya-Namioka, Littrow mounts). Give an example of a spectroscopic experiment where it would be used.
- b) I have a Czerny-Turner style monochromator with the following specifications: focal length 0.3 m, take-off angle of  $9^\circ$ , 150 g/mm grating of 50 mm x 50 mm size blazed at  $\theta_B = 5000$  nm, please calculate:
- The effective numerical aperture of the monochromator in f/#.
  - The grating angle needed in this instrument to obtain  $\theta = 5000$  nm (5 p) at the exit slit.
  - The angular and linear dispersion at 5000 nm, in first order ( $m=1$ ).
  - The resolution in nm and  $\text{cm}^{-1}$  at 5000 nm if both slits are open to 2 mm width,  $m=1$ .
- c) I previously used a Czerny-Turner monochromator like that above for Raman spectrometer, but have recently changed to a different design. The new design has much faster optics, f/1.8, and no mirrors. Explain the advantage of these two changes for Raman spectroscopy. Please propose a design (optical layout) for such a monochromator/ spectrograph, indicating the necessary optical elements and their purpose. [There are two commercial instruments that I know of now available.]
- d) Most monochromators use gratings for wavelength dispersion. Why are prisms no longer used, i.e. what are disadvantages of prisms, advantages of gratings? Propose an experiment where a prism might have an advantage and explain why.

**(16) 5.** *To use a monochromator efficiently, it is necessary to optimally “fill” its optics.* Assume you have available 3 spherical mirrors, each 50 mm x 50 mm square but of different focal lengths, 0.20 m, 0.40 m, and 0.50 m. You also have a number of plane mirrors available for redirecting the light path as needed and have magnetic mounts and an optical table so that you can place the mirrors wherever they should be to optimize light collection for the monochromator in Problem 4 (above). [Drawings will help here.]

a) If you have an SiC source whose cross sectional projection on the optical path is roughly 3 mm x 3 mm, describe the optical arrangement (calculate components and positions) you would choose to optimally fill the monochromator and to obtain the maximum flux at the exit slit for the 2 mm slit in Problem 4, part b above.

b) For the new Raman experiment, with the fast spectrograph in part c of Problem 4, the illuminated spot is about 10 m in diameter and the light is collected as backscatter. Describe (only qualitatively) a different set of optics that would be used to optimally maximize collection of Raman scatter and fill the f/1.8 spectrograph. Defend your choice by explaining the advantages/improvement of the changes.

**(15) 6.** Lenses and mirrors can both be used to focus light beams and move them around to get to a sample or a detection system.

- a. Briefly explain the advantages of lenses over mirrors, and vice versa, of mirrors over lenses.
- b. Briefly explain why most microscopes use compound lens systems
- c. Briefly explain why mirror optics are almost universally used in the IR.
- d. Briefly explain what is chromatic aberration and in what kind of optical system are you likely to find it.
- e. Briefly explain what is astigmatism and in what kind of optical system it is found.

**(12) 7.** The use of polarizers gives another dimension to spectroscopic measurements.

- a) compare wire-grid and Polaroid polarizers and describe their similar and different properties, how they work, i.e. in what frequency (wavelength) region used, power limitations, possible polarization ratio and angular aperture. Describe different experiments in which they would be used and explain why.
- b) compare Glan-Taylor and Rochon (or an equivalent design) prism polarizers, and briefly explain how they work, and why one might be favored over the other.