CHEM 226 Analytical Spectroscopy EXAM #2

April 18, 2000

Name:_____

Date:_____

Start Time:_____

End Time:_____

INSTRUCTIONS: Answer **all** of the questions that follow in this exam. You must show **all** of your work in order to receive full credit. You might find the following physical constants and conversion factors useful:

 $k = 1.381 \times 10^{-16} \text{ erg/K}$

 $1 \text{ eV} = 8067.5 \text{ cm}^{-1} = 1.60 \text{ x} 10^{-12} \text{ erg}$

h = 6.626×10^{-27} erg-sec = 6.626×10^{-34} Joule-sec

 $c = 3.00 \text{ x } 10^{10} \text{ cm/sec}$





1. (30 pts) One of the dispersive devices in my laboratory is a 0.64-meter Czerny-Turner monochromator equipped with a 2400-gr/mm holographically ruled grating. The grating measures 110 mm x 110 mm and the optical speed of the instrument is f/5.2. Calculate the angle of incidence (α).

2. *(40 pts)* Calculate the slitwidth need in order to *baseline resolve* the Ni doublet (3101.88 Å and 3101.55 Å) using the monochromator described in the previous problem. Assume operation in the first order.

3. *(30 pts)* There are a number of ways in which one can improve the resolution of a grating-based dispersive device, but all have their disadvantages. Cross-dispersed echelle grating spectrometers and Rowland circle configuration concave-grating spectrometers represent two approaches that result in increased resolution. Describe these two spectrometer configurations and identify how they attain the desired increased resolution.

4. *(30 pts)* Describe in words why the radiant power throughput of a monochromator shows a different dependence on slit width for a continuum source than for a line source. How, then, is the line-to-background ratio (L/B) for an atomic emission line affected by a decrease in slit width?

5. *(40 pts)* For performing elemental analyses of solution samples, the ICP is the undisputed atomic emission source of choice. Describe the operating properties of the ICP that make it truly unique as a plasma atomic emission source. You should touch upon the following issues: sample introduction, atomization, and excitation.

6. *(30 pts)* Aspirating a 500 ppb Ni solution into an ICP results in the generation of a photocurrent of 1.5×10^{-6} amps from a PMT detecting emission from the 3101.55 Å Ni resonance line. The PMT has an average dark current of 1.0×10^{-9} amps. Calculate the S/N if a $10,000-\Omega$ load resistor is used to make the current measurement (using a current-follower operational amplifier, of course!) and identify the predominant noise source. Assume room temperature operation (20°C) with a measurement bandwidth of 10 MHz.

7. **<u>QUICKIES!</u>** – One or two sentences only! – 10 pts each

a. Under what conditions would cooling a PMT detector result in an improvement in S/N?

b. Why are mirrors used instead of lenses for imaging optics with a monochromator dispersive instrument?

c. What advantages are there to the use of Fourier Transform multiplex techniques for atomic emission spectroscopy in the UV/Vis spectral region?

d. Which charge transfer device (CTD) detector (CCD or CID) is best suited for making low light level measurements?

e. How does one increase the resolution attainable with a Michelson interferometer-based Fourier transform spectrometer?