

CHEM 221

Instrumental Analysis

FINAL EXAM

May 9, 2003

Name: _____

INSTRUCTIONS: Read through the entire exam before you begin. For questions involving calculations, show **all** of your work -- **HOW** you arrived at a particular answer is **MORE** important than the answer itself! Similarly, you must *explain* how you arrived at an answer to a question if you are to receive full credit for your answer.

All questions are worth 25 pts each – answer **any 16** of the 20 questions (for a total of 400 points). You can receive extra credit by answering additional questions (up to 5 extra credit points per question) – if you do so, you must **CLEARLY** identify those questions which are to be graded as extra credit by writing "EC" next to the question number. If no questions are marked "EC" and more than 16 questions are answered, then I will grade the first 16 answers at full credit value (25 points each) and any remaining questions at extra credit value (5 points). Attached are a periodic table and a formula sheet jam-packed with useful stuff!

Good Luck!

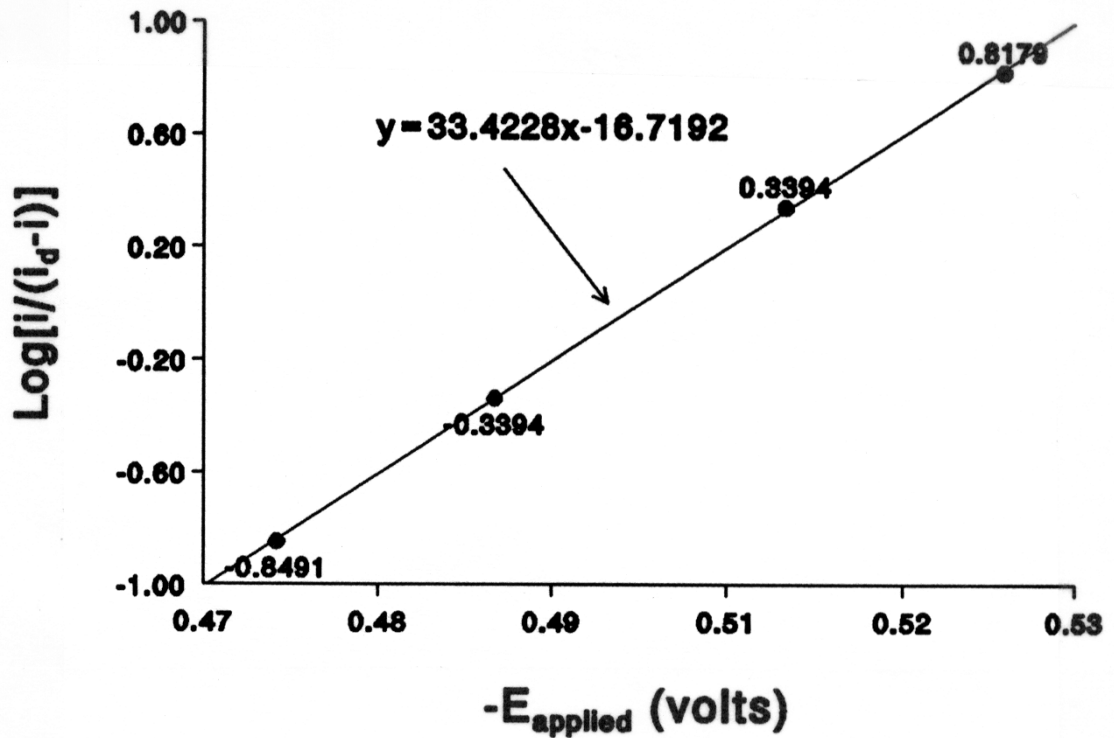


Professor E. F. Gizmo and some of his many inventions

A current-sampled dc polarogram of an unknown solution containing either Cd or Cr was acquired and found to have a limiting current of 5.00 μA . From the rising portion of the polarographic wave, the following data were obtained:

<u>E(applied), volts</u>	<u>Current, μA</u>
-0.475	0.62
-0.490	1.57
-0.510	3.43
-0.525	4.38

Further processing of these data produced the following plot, with the included linear least squares fit to the data:



3. Explain why differential pulsed polarography (DPP) gives superior detection limits to sampled-dc polarography.

4. Give two reasons why the dropping mercury electrode (DME) is the electrode of choice for routine voltammetry.

5. Describe the two main steps involved in an analysis using anodic stripping voltammetry (ASV) and indicate why it has detection limits superior to the other Echem methods we discussed.

6. How would the chemical ionization mass spectrum for a compound differ from its corresponding electron impact ionization mass spectrum?

7. The jet separator serves what function in a GC-Mass Spectrometer? Briefly describe how it works (a simple diagram would help!).

8. Elemental analysis at ppb levels can be performed using ICP atomic emission spectrometry (ICP-AES), graphite furnace atomic absorption spectrometry (GFAAS), and anodic stripping voltammetry (ASV). Which one of these methods would be best for the daily determination of carcinogenic Cr(VI) in water discharged from a manufacturing facility? Briefly justify your choice.

9. Elemental analysis at ppb levels can be performed using ICP atomic emission spectrometry (ICP-AES), graphite furnace atomic absorption spectrometry (GFAAS), and anodic stripping voltammetry (ASV). Which one of these methods would be best for daily screening 10 toxic elements in cooling water effluent from a local manufacturing facility? Briefly justify your choice.

10. Quantitative analysis of environmental samples for trace levels of halogenated pesticides is often accomplished using GC-Mass Spec. If you were to replace the mass spec detector with one of the three common detectors used with GC (i.e., TCD, FID, ECD), which one would best be able to satisfactorily accomplish the analysis task? Briefly justify your choice.

11. In the electron impact mass spectrum for Cl_2 , there are *three* peaks observed for the molecular ion – Explain.

12. Calculate the frequency of electromagnetic radiation (EMR) in the IR spectral region at a wavelength (in a vacuum) of $8.136 \mu\text{m}$. Compare this with the frequency of EMR commonly encountered in NMR spectroscopy and explain why an interferometer is only needed for FT-IR but not FT-NMR.

13. Thanks to the rapid scanning capabilities of FT-IR, you've been busy watching the FT-IR signal-average a noisy spectrum for an hour. You are interested in determining whether or not a feature appearing at 1620 cm^{-1} is due to noise or a real absorption band. Suppose that the S/N for this feature after 100 scans is equal to 1.2 – assuming you can average 100 scans each hour, how much longer (hours) must you scan before this feature can be positively differentiated from noise (i.e., has a S/N equal to 3.0)?

14. **TRUE or FALSE (and WHY)?** A common tungsten filament continuum source is ideal for atomic absorption spectrometry.

15. **TRUE or FALSE (and WHY)?** Broad absorption bands are typically observed in UV/Vis molecular spectroscopy due to the limited resolution of the instruments used.

16. **TRUE or FALSE (and WHY)?** Lasers are intense sources of optical EMR, but are not good sources for absorption spectrophotometry because they are too large and bulky.

17. Why do open tubular columns produce gas chromatograms with significantly better resolution than do packed GC columns?

18. **TRUE or FALSE (and WHY)?** Charge transfer bands arise from $d \rightarrow d$ electronic transitions of the metal ion in metal-ligand complexes.

19. Arrange the following stretches in order of *increasing* vibration frequency: C-H, C-C, C=O, C-D. Briefly justify/explain your answer.

20. **TRUE or FALSE (and WHY)?** With a blank measurement of 0.001 ± 0.0005 (average \pm standard deviation) and a sample measurement of 0.002 ± 0.0005 , it was determined from the calibration curve that Pb was present in the drinking water sample at a level of 500 ppb.