

CHEM 450 STUDY GUIDE - FINAL EXAM

Part I. Cumulative – Instrumental methods

- Given the type of information needed and the analysis required (see example below), decide which *instrumental method* would be best suited for the analysis. Be able to justify your answer with a brief explanation of why it is the best method for the given analysis. (Note that in some cases two complementary methods might be needed to accomplish the analysis. You have to explain why you need both methods in such cases.)

Instrumental methods to choose from:

Molecular UV/Vis or Fluorescence	ICP/MS or Atomic MS	GC/MS or GC/FID
Atomic (UV/Vis) - Flame or GFAA	Molecular MS	HPLC
Atomic Emission (ICP-AES)	IR spectrometry	NMR

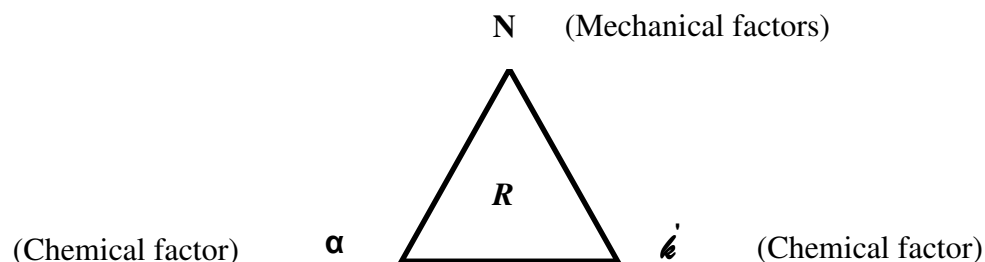
Examples of required analysis and information:

- Quantitation of C-13 and N-15 isotopes in bone collagen (this provides a method for determining the dietary practices of humans and animals hundreds to tens of thousands of years ago).
- Determining the structure of a liquid product of an organic synthesis whose molecular formula is C₉H₁₃N.

Part II. Chromatography (GC and HPLC)

- Know the specific applications [i.e. what is it used for?] and limitations of: (a) GC and (b) HPLC.
- Explain the difference between *isothermal* and *programmed temperature* in GC. Describe the applicability of each technique.
- Explain the difference between *isocratic* and *gradient elution* in HPLC. Describe the applicability of each technique.
- Discuss the effect on peak or band width, and consequently separation efficiency as indicated by plate height, H), of each of the following: (a) stationary film thickness, (b) carrier gas flow rate and (c) packing material in GC.
- Predict the order of elution in GC or HPLC given the components being separated, the polarity of column stationary phase and other pertinent chromatographic parameters (such as oven T in GC or solvent composition in HPLC). NOTE: You need a good knowledge of relative polarities of organic compounds --- ethers, esters, carbonyls, carboxylic acids, amines, alcohols, hydrocarbons, aromatics
 - See lecture notes for specific chromatograms of named components (such as the “Effect of changing the polarity of stationary phase in GC” slide)
- Given a chromatogram (detector response vs. retention time) and column parameters, propose a way by which resolution can be achieved while minimizing analysis time.
- Using data provided, such as a chromatogram, calculate for two components the:
 - Retention factor, k' , for each
 - Selectivity factor, α
 - Number of theoretical plates, N (Individual, followed by average N)
 - Plate height, H
- Predict the effect of changing chromatographic conditions (such as increasing oven T, increasing the proportion of IPA vs. water in HPLC) on retention time, t_R , of components.

9. Differentiate between normal phase and reversed phase liquid chromatography in terms of the nature of stationary phase material.
10. Explain how the eluent strength can be increased (like in gradient elution) in (a) *normal phase* and (b) *reversed phase* HPLC.
11. Explain the effect of chain length (ex. C_8 vs. C_{18}) on t_R in reverse phase HPLC.
12. Given a HPLC chromatogram and chromatographic conditions (ex. solvent composition, nature of column), predict the polarity of components and propose a way by which the analysis time can be reduced. (i.e. What chromatographic parameter will you change and why?)
13. There are three factors that affect resolution in any chromatographic separation as shown in the diagram below: Capacity or retention factor k' , selectivity factor α , and number of theoretical plates, N (mechanical factor).



- (a) What parameter can be changed in GC to improve k' and α (and therefore R)? Explain.
- (b) What parameter can be changed in HPLC to improve k' and α (and therefore R)? Explain.
- (c) What is the role of N in improving resolution?

14. READ your lecture notes