

1. What is meant by a pulse NMR experiment? 7%
2. (A). State the radiation sources of the following instruments: 10%
- Ultraviolet Spectroscopy,
  - Infrared Spectroscopy,
  - X-ray Diffractometer,
  - Atomic absorption Spectroscopy,
  - Nuclear Magnetic Resonance Spectroscopy.
- (B). State the most frequently used detectors of the following instruments: 10%
- Infrared Spectroscopy,
  - Mass Spectroscopy,
  - Atomic Absorption Spectroscopy,
  - Ultraviolet Spectroscopy,
  - X-ray absorption, emission and fluorescence.
3. Determine the resolution of an instrument that is capable of separating two adjacent peaks centered at 573.5 and 569.8 nm. The distance of separation between the two peaks on the focal plane of the instrument is 2.1 mm. Calculate the dispersion and the linear reciprocal dispersion of the instrument. 6%
4. What are the principal interferences encountered while performing analyses with atomic absorption spectroscopy? 7%
5. A water sample initially contains unknown amounts of M(III) and M(II). The M(III) species can be electrochemically reduced to M(II) at proper potential.



To this sample solution, several portions of M(III) were added and the the cell potentials were measured after each addition at 25 °C. The results are summarized in the following table:

Addition #	Total amount of M(III) added to the initial solution, (M)	Cell Potential (V)
0	0	-0.424
1	$5.0 \times 10^{-4}$	-0.413
2	$1.0 \times 10^{-3}$	-0.405
3	$1.5 \times 10^{-3}$	-0.399
4	$2.0 \times 10^{-3}$	-0.396

Please calculate the concentrations of both M(III) and M(II) tha were present in the original sample solution. Use all of these data. 15%

6. The differential polarographic reduction current due to the reduction of Mo(V) to Mo(III) has been applied for the determination of the molybdate content in water sample. It was noted that if  $\text{NO}_3^-$  or  $\text{ClO}_4^-$  was added to the supporting electrolyte, the differential reduction current of Mo(V) was greatly enhanced and such enhancement in the reduction current increases as the concentrations of  $\text{NO}_3^-$  or  $\text{ClO}_4^-$  increase. Please explain this phenomena. 5%
7. Define the ultramicroelectrode, and describe the analytical applications as well as the advantages and disadvantages of it. 5%
8. A student need to analyze a series of samples containing substantially different amount of  $\text{Ca}^{++}$ . He has two analytical methods to choose, and following table gives the primary test results for each of four test samples:

Sample #	Method 1	Method 2
1	93 (ppb)	91 (ppb)
2	71	71
3	49	52
4	21	23
5	12	9

- Do the two methods give values of the mean sample concentration which differ significantly? 5%
9. A 500 cc. water sample was acidified by adding of 1N HCl solution first, then extracted by 100 cc. benzene. The pH value of this sample was then adjusted to basic(pH=10) and extracted with  $\text{CH}_2\text{Cl}_2$ . The  $\text{CH}_2\text{Cl}_2$  layer was dried, concentrated and finally was analyzed by GC method. If we want to have the best GC sensitivity, what detector will you choose? Please give the explanation. 5%
10. Why mass spectroscopy must operated under high vacume? 5%
11. Please describ what is the "solvent effect" and give the reasons why and how we can use it when a splitless injection mode was used with a capillary column GC? 10%
12. What are the advantages and disadvantages of using supercritical fluids as solvents in extration and chromatographic separation? 10%