Assiut University
Faculty of Science
Chemistry Department

Date: 6/9/ 2015
Time: 3 hours
Instructor: Dr. M. S. Ibrahim

Spectroscopic Analysis Methods (C-531)
(Final Examination, Analytical Chemistry Diploma)

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Answer the following questions:

(A) Choose the correct answer: (2 points each)

(1) A molecule is known to absorb radiation at a wavelength of 600 nm. The instrument one would probably use to study this absorption band would be
- (a) an infrared spectrophotometer.
- (b) a vacuum ultraviolet spectrometer .
- (c) a visible spectrometer.
- (d) a gamma ray spectrometer.

(2) Chemical interferences due to formation of species of low volatility in AAS can be eliminated by using of,
- (a) Higher temperature.
- (b) Releasing agents.
- (c) Protective agents.
- (d) All of the above.

(3) The principle use of the hollow cathode lamp in AAS is that:
- (a) It generates atomic emission lines using the element which is being analysed.
- (b) It generates atomic emission lines using any element.
- (c) It generates atomic emission lines using laser source.
- (d) All of the above.

(4) Which of the following technique is applied in atomic absorption spectroscopy?
- (a) Thermal conductivity.
- (b) Electroreduction process.
- (c) Flame ionization.
- (d) Flame atomization.

(5) The presence of EDTA has been shown to eliminate the AAS interference of PO_4^{3-} in,
- (a) The determination of Ca^{2+}.
- (b) The determination of Ca^{2+} and Cu^{2+}.
- (c) The determination of Ca^{2+} and Al^{3+}.
- (d) All of these.

(6) For ionization interferences in AAS, the effects of shifts in ionization equilibrium can be eliminated by:

$$M(g) \leftrightarrow M^{+}(g) + \bar{e}$$

- (a) Increasing the temperature of flame.
- (b) Using oxygen or nitrous oxide as oxidant.
- (c) Addition of potassium salt.
- (d) Addition of releasing agent.

(7) What is the advantage of graphite furnace, in compared to flame atomization in AAS?

(a) Small sample size. (b) More efficient atomization.
(c) Leading to lower detection limits. (d) All of these.

(8) XRF (X-ray Fluorescence)can be produced by:

(a) X-rays
(b) Alpha particles (APXS)
(c) Protons (PIXE)
(d) Electron beams (SEM electron microprobe)

(9) The advantages of X-ray spectrometric methods are:

(a) It is not very sensitive to molecular effects or chemical state, or excitation conditions.
(b) Atomization is not necessary for elemental analysis.
(c) Surface-sensitive (penetration of 100 μm at most).
(d) All of these.

(10) Which statement is **FALSE** about Sample atomization techniques?

(a) Flame ionization.
(b) Electrothermal atomization
(c) Hydride atomization
(d) Cold-Vapor atomization

(11) Which of the following are the most common transitions observed in organic molecular UV-Vis, observed in compounds with lone pairs and multiple bonds with λ_{max} = 200-600 nm?

(a) $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ (b) $\sigma \rightarrow \sigma^*$ and $\sigma \rightarrow \pi^*$
(c) $n \rightarrow \sigma^*$ and $\pi \rightarrow \sigma^*$ (d) All of these

(12) Which technique generally has the most complete atomization in AAS?

(a) Flame (b) graphite furnace
(c) Inductively coupled plasma (d) All of these

(13) Which is the correct order of events in the process of atomization?

(a) Nebulization - Desolvation - Volatilization
(b) Volatilization - Desolvation - Nebulization
(c) Desolvation - Volatilization - Nebulization
(d) Nebulization–Volatilization -Desolvation

(14) The energy and wavelength of x-rays from an x-ray tube depends on the energy of the -

(a) Electron beam colliding with the target element.
(b) Laser beam used to excite the sample.
(c) Cathode ray tube.
(d) Flame.

(15) The K- and L-lines in an atomic x-ray spectrum correspond to --

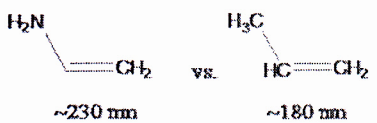
(a) Transitions from lower to higher energy levels.
(b) electron-electron collisions.
(c) Absorption and emission of high-energy photons.
(d) Transitions from upper energy levels to the innermost shell of the atom.

(B) Enter in the appropriate box the expressions you would select to define each of the phenomena Labeled **A-J** inclusive. **(15 points)**

Expression	Phenomena
Atomization	A. High-energy transitions, accessible in vacuum UV (λmax <150 nm). Not usually observed in molecular UV-Vis.
Beer' Law	B. UV-Visible absorbing functional group.
Chromophore	C. The process by which a sample is vaporized and decomposed into atoms usually by heat.
hollow cathode lamp	D. Produced by the deceleration of high-energy electrons and then electronic transition of electrons in the inner orbitals of atoms.
X-Rays (emission)	E. Absorbance is linearly related to the concentration of the absorbing species and the pathlength of the absorbing medium.
Moseley's law	F. Predicts the basic relationship of atom number and the frequency of the characteristic x-ray lines.
Releasing agent	G. Substituent on a chromophore that causes a red shift.
$\sigma\rightarrow\sigma^*$ and $\sigma\rightarrow\pi^*$ transitions	H. Refers to the absorption of emitted radiation by unexcited atoms in the gas phase of a hollow-cathode lamp, flame, or plasma.
Auxochrome	I. Cation that preferentially reacts with a species that would otherwise react with the analyte to cause a chemical interference in AAS.
Self-absorption	J. The mostcommon lightsource for atomic absorption measurements has a tungsten anode and a cylindrical-shaped cathode containing the element of interest.

A	
B	
C	
D	
E	
F	
G	
H	
I	
J	

(C) Are the following statements correct (✔) or false (✖)? (2 points each)

1. The X-ray spectrum is *not* very sensitive to molecular effects or chemical state, or excitation conditionsfor elemental analysis.	( )
2. One of the applications of XRF (X-ray Fluorescence) is the determination of elemental composition and impurities of ceramics.	( )
3. The technique applied in UV-Visible spectroscopy is the flame atomization.	( )
4. UV-Visible spectra can be interpreted to help determine molecular structure, but this is presently confined to the analysis of electron behavior in known compounds.	( )
5. Bathochromic shift is the shift of absorbance to shorter wavelengths(blue shift).	( )
6. UV-Vis spectra of lanthanides/actinides are particularly sharp, due to screening of the 4f and 5f orbitals by lower shells.	( )
7. Charge transfer (CT) occurs when electron-donor and electron-acceptor properties are in the same complex, electron transfer occurs as an "excitation step"	( )
8. A plasma is a conducting gas that contains a large concentration of ions and/ or electrons.	( )
9. The presence of some elements such as Sr and La lead to spectral interference in AAS due to scattering by oxides product.	( )
10. The radiation emitted by the hollow cathode lamp is of exactly the correct wavelength to be absorbed by the analyte, therefore No need for a monochromator ahead of the analyte.	( )
11. The spectral interference in AAS occurs when an absorption or emission peak of an element in the sample matrix overlaps that of the analyte.	( )
12. The electrothermal atomizer is a more efficient atomizer. It requires much less sample and keeps the atomic vapor in the beam for a longer time than does a flame.	( )
13. A chemical interference is the result of any chemical process which decreases or increases the absorption or emission of the analyte.	( )
14. Because atomic spectra are lines, the absorbing and emitting wavelengths are identical … contrast molecular spectra, for which λ(emission) > λ (excitation)	
15. The conjugation of a lone pair on anenamineshifts the λ_{max} from 190 nm (isolated alkene) to 230 nm. The nitrogen has an auxochromic effect. 	( )

Part II: Problems and Questions

(1) A 2.83×10^{-4} M solution of $KMnO_4$ has an absorbance of 0.510 whenmeasured in a 0.982 cm cell at 520 nm. Calculate **(6 points)**

(a) The molar absorptivity for $KMnO_4$(158 g/mol) at this wavelength.

(b) The absorptivity when the concentration is expressed in ppm.

(c) The molar concentration of $KMnO_4$ in a solution that has an absorbance of 0.747 when measured in a 1.5 cm cell at 520 nm.

(d) The transmittance of the solution in (C).

(e) The absorbance of a solution that has twice the transmittance of the solution in (c).

(2) Two substances M and N have the ultraviolet absorption characteristic shown below;

		Absorbance, A, (1.00 Cm Cells)	
substance	C, mol/L	A at λ_1	A at λ_2
M	8.50×10^{-5}	0.129	0.764
N	4.65×10^{-5}	0.567	0.000

A solution containing both of these substances gives an absorbance of 0.502 at λ_1 in a 1.25 cm cell and an absorbance of 0.912 at λ_2 in a 1.50 cm cell. What is the concentration of M and N in mol/L. **(7 points)**

(3) The drug tolbutamine (270 g/mol) has a molar absorptivity of 703 at 262 nm. Five tablets are dissolved in water and diluted to a volume of 2 L. If the solution exhibits an absorbance in the UV region at 262 nm equal to 0.687 in a 1.0 cm cell. How many mg tolbutamine are contained per tablet? **(6 points)**

(4) Draw the schematic cross section of ***hollow cathode lamp*** which the most common source for atomic absorption measurements.

(3 points)

(5) The interferences by formation of compounds of low volatility in AAS can be eliminated by using of :

(3 points)

1-

2-

3-

The End

Assiut Universiry
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Chemistry Department

September:2015

Electroanalytical Chemistry and Electrochemistry(C-534)Examination for Diploma Students.

Part I : Electroanalytical Chemistry.

Answer the following questions:

1-a-i-Ilkovic equation and its application in chemical analysis.

ii-The diffusion coefficient (D)for Tl^+ is $2.00x10^{-5}cm^2/sec$.whereas D for Cd^{2+} is $0.72x10^{-5}$ cm^2/sec,if a $1.00x10^{-3}$ M solution of Cd^{2+} gives a diffusion current of 8.15μ A,predict the diffusion current for $1.00x10^{-3}$ M Tl^+ under the same conditions.

b-i-Explain the use of cyclic voltammetry for estimating the values of E° and n(number of electrons)for a reversible system.

ii-What is the relation between half-peak potential,$E_{p1/2}$ and the polarographic half-wave potential,$E_{1/2}$?

c-Write briefly on differential pulse voltammetry.

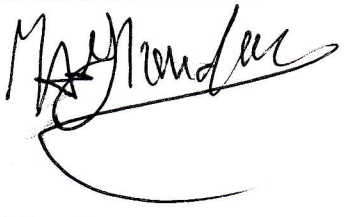
2-Write short notes on **Only Two** of the following:

a-Square-wave voltammetry.

b-Anodic stripping voltammetry.

c-Adsorptive stripping voltammetry.

Examiner:Prof.Dr.Mahmoud A.Ghandour



فضلاً أنظر الجزء الثانى للأمتحان فى الخلف

Part 2 Electrochemistry and Corrosion

Answer the following questions

Q1) a) Give a short note for E-pH diagram of iron-water system and explain vertical, horizontal and sloping lines in this diagram. Indicate how can escape out from a corrosion zoon.

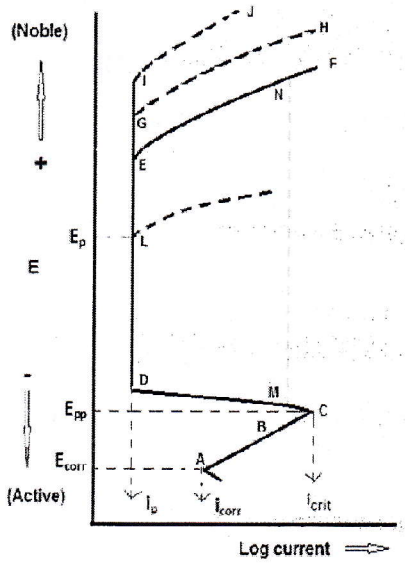
b) Explain two methods for protection of metals against corrosion.

Q2) a) you are provided with the following anodic polarization diagram for a metal in a given corrosive medium:

i) **Explain the points and lines represented by characters A, B, C,....**

ii) **Define the potentials and currents appeared in the diagram.**

b) According to parameters of the anodic polarization, indicate the best selection of materials to resist the corrosion.



الدكتور مصطفى وهدان

Assiut University
Faculty of Science

Sep.2015
Time allowed 1.5 hr

Analyical Chemistry Diploma

Applied Organic Analytical Chemistry

Answer the following questions:-

1)Write an account of each of the following

(a)State the testes needed for distinctuin between the following:

(i)Natural and synthetic wool fibers

(ii)Primery alcohol and secondary alcohol

(iii)Esters of fates and ester of waxes

(b)Micro determination of:

(i)Carboxylic acid, amide , esters

(ii)Peroxide compounds, epoxide(oxirane)compounds

(iii)Iso cynate , isothiocyanate

II)Discuss tge theoretical bases for detection of the following:

(i)Aromatic substances with chloranil test

(ii)Detecation of pyridine

(iii)Carbon disulphide in benzene and carbon tetrachloride

(iv)Rancidity of fats and oils

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ii-What is the relation between half-peak potential,$E_{p1/2}$ and the polarographic half-wave potential,$E_{1/2}$?

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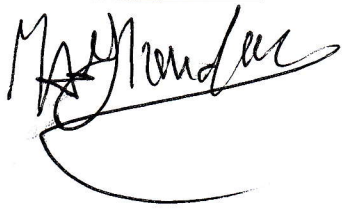
Examiner:Prof.Dr.Mahmoud A.Ghandour



فضلاً أنظر الجزء الثاني للأمتحان في الخلف

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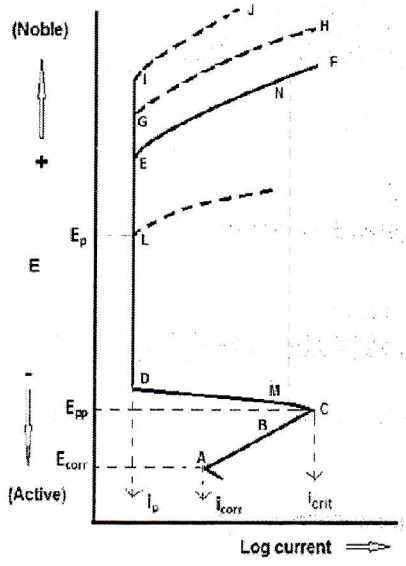

الدكتور مصطفى وهدان

Assiut University
Faculty of Science
Chemistry Department

Sept. 2015
Time 1.5 hrs

Bio.Anal.Chem.Exam.(533-C) For Anal.Chem.Diploma Students

Answer the following questions:

1)a-Give the theoretical bases of :

i-Zero-Crossing technique.

ii-Derivative ratio spectra.

b-Write briefly on .

i-Coagulation.

ii-Cl_2 as a disinfecting agents.

2)Answer **Three** Only:

a-Explain the solubility of CO_2 in water.

b-Contrast ground water with surface water.

c-Discuss only one method used for water softening.

d-Given that the atmosphere contains 0.21 atm.O_2 , calculate the amount of O_2 dissolved in water. (K_H=1.3*10^{-3})

Good luck.

Prof.Dr.Hassan Sedaira

Assiut University Sept2015

Faculty Of Science Time:3 hrs

Chemistry Department

Analytical Chemistry For Diploma(532C)

Analytical Chemistry

Answer the following questions

I-Write on the followimg

a)the important poinls in choosing the suirable solvent for column chromatography.

b-the essential parts of HPLC with special reference to precation must be taken during using HPLC.

c-(i)visualization methods for separation by thin layer or paper chromatography.

(ii)the RF value.

d-(i)the important points in choosing any adsorbent for chromatography.

(ii)coating materials in TLC.

II-a)Describe the principles of the following gas liquid chromatography detectors.

i-Arogon ionization ii-Double flame iii-Electron capture

b)Write Van Deemeter equation discuss the factors affecting the spreeding of gas molecules through GLC column.

c-Discuss GLC applications in qualitative and quantitative analysis.

d-i-Explain the principles of non aqueous titrations.

ii-On the basis of non aqueous titrations,describe the analysis of the following mixtures.

a)perchloric acid+HCL

b)perchloric acid+HCL+salicylic acid+acetic acid+phenol.

c)Aniline and ethylamine

Good luck,,, أ.د زهره عبد العزيز ؛أ.د ماجد محمد سليم