THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in Chemistry
Level 5 – Assignment test 1 -2010 / 2011

CHU 3129/CHE 5129 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One and half hours
Date and time: 28TH February, 2011 from 4.00 p.m. to 5.30 p.m.

Reg. No.....

Question number	marks
1	
2	
3	
Total	

Instructions to students

Answer all questions in the spaces given. Additional sheets will not be marked.

1. The absorbance of a 2.0 x 10^{-3} M $K_2Cr_2O_7$ solution and the blank solution were 0.314 and 0.104 respectively in a 1.00 cm cell at 460 nm. (all the answers should be given to the correct number of significant figures.)

   (a) Why do you think the absorbance was measured at 460 nm?

   (b) What is the molar absorptivity coefficient value at 460 nm?

   (c) Calculate the percentage of transmittance.

(d) Draw and label a schematic diagram of the instrument used in this analysis.

(e) Give three major differences of this instrument compared to the Atomic Absorption Spectrophotometer.

(f) Give two advantages of this method compared to classical methods.

(g) Why a blank solution has been used?

(h) What do you mean by the chemical deviation of absorption spectroscopy?

(50 marks)

2. Explain the following terms in brief.

(a) Base peak in Mass Spectroscopy.

(b) Sensitivity of an instrument.

(c) Rayleigh scattering in Raman Spectroscopy.

(d) Singlet excited state.

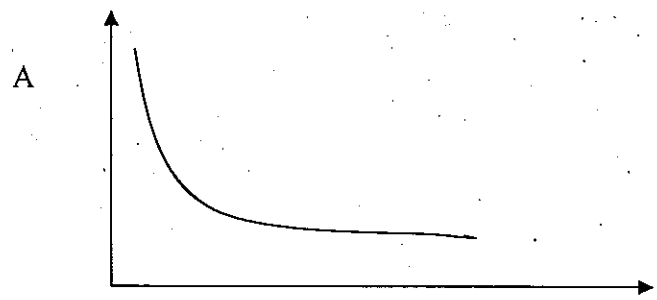
(e) Phosphorescence

(25 marks)

3. (a) Why chemical ionization is more advantageous compared to Electron impact in Mass Spectroscopy?

(b) Give two differences in the instrumentation of IR spectrophotometer and UV/ Visible Spectrophotometer.

(c) What can you say about the light absorbing properties of the titrant, analyte and the product which resulted the titration curve given below?



Titrant –
Analyte-
Product-

(d) Give two advantages of photometric titrations over volumetric titrations.

(25 marks)

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Answer Guide – Assignment Test 1

(01) (a) It may be the λ at which the absorbance is maximum (or λ_{max}) or λ at which a significant absorbance could be obtained without any interference.

(b) $A_{correct} = A_{measured} - A_{blank}$

$= 0.314 - 0.104 = 0.210$

$A = \varepsilon Cl$

$0.210 = \varepsilon \times 2.0 \times 10^{-3}\ mol\ dm^{-3} \times 1.00\ cm$

$\varepsilon = 10.5\ dm^3\ mol^{-1}\ cm^{-1}$

10×10^1 or $11 \times 10^1\ dm^3\ mol^{-1}\ cm^{-1}$

{There is a rule in mathematics as follows: In the case where the first decimal point is 0.5 ,and the number left to 0.5 is odd , one number will be added to the number left to 0.5.(eg: 21.5 = 22). If the number left to 0.5 is even, one will not be added (eg : 10.5 = 10). Therefore , 10×10^1 was also accepted as the answer)

(c) $A = \log\left(\frac{I_0}{I}\right)$ $T = \left(\frac{I}{I_0}\right)$ Therefore, $A = \log\frac{1}{T}$

T $= 0.616$ %T $= 61.6\%$

(d) Fig. 3.3 of Book I

(e)

UV/ Visible	AAS
1. The light source is either the deuterium or tungsten lamp	Hollow cathode lamp
2. A monochromator between the source and the sample is required.	Not required.
3. The sample is in a cell.	Sample is nebulized to the flame.
4. An energy source like a flame is not required.	An energy source like a flame is required to form atoms.
5. A chopper is not required.	A chopper is required.
6. A nebulizer is not required.	A nebulizer is required.

(f) Advantages-

1. More sensitive.
2. Minimum detection limit is low.
3. More accurate.
4. More précised.
5. Less time consuming.
6. Sample amount is less.
7. Operation is easy.

(g) A blank is used to eliminate the errors introduced by the reagents added to the sample.

(h) At high concentrations, the absorbing molecules react with each other or dissociate. Therefore, Beer – Lambert law will be no longer obeyed. This situation is called chemical deviation.

02. (a) Base peak – The peak which shows the highest relative abundance which is resulted by the most abundant fragment in mass spectroscopy.

(b) Sensitivity- The difference in signal or the response resulted by a unit difference in the amount of analyte.

(c) Rayleigh Scattering - The scattered light having the same frequency as the incident light

(d) Singlet excited state - When an electron of a filled orbital of a molecule in the ground state occupies an empty orbital in the excited state without changing its spin as a result of absorbing energy.

(e) Phosphorescence - Energy released as a result of relaxation of a molecule from the triplet excited state to the singlet ground state.

03. (a) In chemical ionization, the molecule is ionized without colliding with a fast moving electron. The amount of energy transferred is less minimizing the fragmentation. Therefore, the possibility of observing the molecular ion peak is high.

(b)

IR spectroscopy	UV/Visible spectroscopy
1. Source of radiation- Nernst glowers/ globars/ metal wires	Tungsten / hydrogen lamp
2. Cell is made out of- inorganic salts	Glass or quartz
3. Energy transducers are -heat sensitive	Light sensitive

(c) Titrant – non absorbing
Analyte – absorbing
Product – non absorbing

(d) The end point is located by the intersection of two straight lines. Therefore,

1. Accuracy is high.
2. Measurements near the end point are not required.
3. High sensitivity.
4. High reproducibility