2411/305
INSTRUMENTAL METHODS OF ANALYSIS
Oct./Nov. 2017
Time: 3 hours

THE KENYA NATIONAL EXAMINATIONS COUNCIL

DIPLOMA IN ANALYTICAL CHEMISTRY

INSTRUMENTAL METHODS OF ANALYSIS

3 hours

INSTRUCTIONS TO CANDIDATES

You should have the following for this examination:
Answer booklet;
Non-programmable scientific calculator.
*This paper consists of **TWO** sections; **A** and **B**.*
*Answer **ALL** the questions in section **A** and any **THREE** questions from section **B**.*
*Each question in section **A** carries **4** marks while each question in section **B** carries **20** marks.*
Maximum marks for each part of a question are indicated.
Candidates should answer the questions in English.

This paper consists of 5 printed pages.

Candidates must check the question paper to ascertain that all the pages are printed and that no questions are missing.

SECTION A (40 marks)

Answer ALL the questions in this section.

1. Explain why copper cannot be analysed by atomic emission spectroscopy (AES). (4 marks)

2. Explain why AAS does not suffer from severe spectral interferences. (4 marks)

3. A solution of $K_2Cr_2O_7$ was prepared by pipeting 50 cm^3 of a 0.1 M solution into a 5 litre volumetric flask and topping up to the mark with pure water. The 0.1 M solution had an absorbance of 1.2; 25 cm^3 of the diluted solution was pipetted into a 100 cm^3 volumetric flask and topped up to the mark with pure water.

   (a) Write an expression for Beer-Lamberts Law. (2 marks)

   (b) Calculate the absorbance of the final diluted solution. (2 marks)

4. Describe the preparation of 250 cm^3 of a solution of sodium phosphate of concentration 200 ppm with respect to sodium. (Na = 23, P = 31, O = 16) (4 marks)

5. (a) Define the following terms as used in IR spectroscopy:

   (i) fundamental; (1 mark)
   (ii) overtone. (1 mark)

   (b) The frequency of the fourth overtone of an IR active bond is 3050 cm^{-1}. Calculate the frequency of the third harmonic. (2 marks)

6. (a) (i) Define resolution as used in chromatographic methods of analysis. (1 mark)

   (ii) Write down the expression used in estimating column resolution in both GL and HPLC. (1 mark)

   (b) State the tone method of increasing column resolution in:

   (i) GLC; (1 mark)
   (ii) HPLC. (1 mark)

7. (a) State **two** methods of monochromation used in flame spectroscopy. (2 marks)

   (b) List **two** differences between a flame photometer and atomic absorption spectrophotometer. (2 marks)

8. (a) State the conditions for analysis of a sample by GLC. (1 marks)

   (b) Outline the principle of separation in a HPLC column by a normal phase chromatographic technique. (3 marks)

9. (a) Name **two** types of detectors used in IR spectrophotometer. (1 mark)

(b) IR radiation does not cause electron excitation like UV - visible radiation because of low energy. Explain how a phototube incorporated in one of the detectors in (a) above functions. (3 marks)

10. Describe how the lovibond colour comparator is used in colorimetric analysis. (4 marks)

SECTION B (60 marks)

*Answer any **THREE** questions from this section.*

11. (a) Calculate the energy of one mole of photons of red light:

(i) whose frequency is 4×10^{14} *Hz*; (3 marks)

(ii) wavelength of the radiation in nm.
($h = 6.63 \times 10^{-34} JS$) (2 marks)

(b) At a particular location and time, sunlight is measured on a one meter square solar collector with an intensity of 1000 W. The peak intensity of this sunlight has a wavelength of 560 nm. Calculate the rate at which the photons hit the solar collector per second. (5 marks)

(i) List any **three** properties of electromagnetic radiations. (3 marks)

(ii) Explain how each of the properties listed in (c)(i) above is used in the manufacture of spectroscopic instruments of analysis. (7 marks)

12. (a) Explain why a solution of $KMnO_4$ which is purple in colour is analysed by passing green radiation of $\lambda = 545$ nm and not purple radiation of $\lambda = 480$ nm. (5 marks)

(b) (i) List **five** colorimetric methods of analysis. (5 marks)

(ii) State **one** limitation of colorimetry. (1 mark)

(iii) List any **four** advantages of colorimetry. (4 marks)

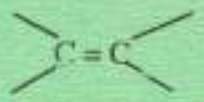
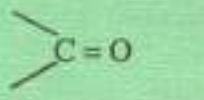
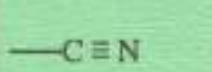
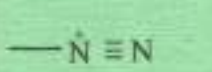
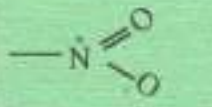
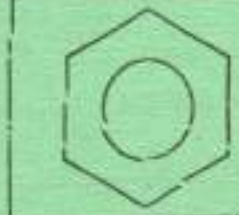
(c) In a colorimetric estimation of proteins in a urine sample, the following results were obtained. A 2500 cm^3 sample had an absorbance of 0.814. The sample was than spiked with a 1.00 cm^3 standard containing 5 mg of pure protein and the absorbance was 0.915. Calculate the concentration of the proteins in the urine sample in ppm. (5 marks)

13. (a) Define the following terms as used in UV-visible spectrophotometry:

(i) chromophore; (1 mark)
(ii) auxochrome. (1 mark)

(b) The table I below shows some chromophores and their absorbance maxima in nm.

Table I

Chromophore	λ_{max} in nm.
	190
	190 and 250
	160
	350
	270
	

An organic compound showed absorption bands at λ_{max} = 160 nm and at λ_{max} = 190 nm. The composition by mass of the compound showed C= 67.7%, H= 5.75 and N = 26.4%. The empirical and molecular formulae are the same. Use this information to determine a possible structure for this compound. (18 marks)

14. (a) (i) Define the term hydrogen bonding. (1 mark)

(ii) State **four** effects of hydrogen bonding on the IR spectrum of a pure compound. (4 marks)

(b) State **five** advantages of the pressed pellet techniques of preparing sample in IR spectroscopy. (5 marks)

(c) The frequency of the -OH stretching vibration in CH_3OH is 3300 cm^{-1}. Estimate the frequency of the -OD stretching vibration in CH_3OD. (10 marks)

15. (a) (i) Define stray radiation as used in AAS. (1 mark)

(ii) Identify **two** sources of stray radiation in AAS. (2 marks)

(iii) Stray radiation is spectral interference in AAS. Name **five** other causes of spectral interference in AAS. (5 marks)

(b) A sample was found to have a transmittance of 80% when analysed by AAS in the absence of stray radiation:

(i) calculate the absorbance of the sample in presence of 15% stray radiation. (11 marks)

(ii) State the effect of stray radiation on absorbance. (1 mark)

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