2411/305
INSTRUMENTAL METHODS
OF ANALYSIS
Oct./Nov. 2019
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:
A scientific calculator (non-programmable);
Answer booklet.
*This paper consists of **TWO** sections; **A** and **B**.*
*Answer **ALL** the questions in Section **A** and any **THREE** questions from Section **B** in the answer booklet provided.*
*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 4 printed pages.

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

SECTION A (40 marks)

*Answer **ALL** the questions in this section.*

1. Identify any **four** factors that influence flame temperature in flame photometry. (4 marks)

2. Describe the preparation of 500 cm^3 of a solution of potassium sulfate whose concentration is 250 ppm with respect to potassium, by the method of direct weighing.
(K = 35, S = 32, O = 16) (4 marks)

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

   (b) The absorbance of a solution of Mn^{2+} is 0.80 in the absence of stray radiation. Calculate the absorbance of the solution in the presence of 20% stray radiation. (3 marks)

4. State **four** advantages of grating monochromators over other types of monochromators used in AAS. (4 marks)

5. (a) Define molar absorptivity as used in UV-visible spectrophotometry. (1 mark)

   (b) (i) A 1.5×10^{-5} M $KMnO_4$ solution had an absorbance of 0.8. Calculate molar absorptivity of $KMuO_4$. ($KMnO_4$ = 158) (1 marks)

   (ii) A 25 cm^3 aliquot of the solution in b(i) above was pipetted into a 100 cm^3 volumentric flask and topped upto the mark with water. Calculate the absorbance of the diluted solution. (2 marks)

6. List any **four** advantages of colorimetry as a method of analysis. (4 marks)

7. Calculate the expected number of IR absorption peaks for the hydrazine molecule. (4 marks)

8. Explain **two** advantages of double beam IR spectorphotometers over the single beam IR spectrophotometers. (4 marks)

9. (a) State **two** methods that are used to vapourise samples of low volatility so that they can be analysed by GLC. (2 marks)

   (b) State any **two** advantages of capillary columns used in GLC instruments. (2 marks)

10. The retention times of two solutes **M** and **N** were 17.25 and 9.38 minutes respectively in a 50 m porous column. The carrier gas migrated through the column in 2.95 minutes. The base widths for **M** and **N** were 3.08 and 1.94 minutes respectively.

    (a) Identify the void volume in this analysis. (1 mark)

    (b) Determine the column efficiency in this analysis. (3 marks)

SECTION B (60 marks)

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

11. (a) Define the following terms as used in IR spectroslopy:

(i) hydrogen bonding; (1 mark)

(ii) overtones; (1 mark)

(iii) field effects; (1 mark)

(iv) frequency. (1 mark)

(b) Explain how the factors in (a) above affect molecular vibrations in IR spectroscopy. (8 marks)

(c) Give **four** reasons as to why the wave number of an IR active group is not a constant. (8 marks)

12. (a) (i) State any **three** spectral differences between AAS and FES. (3 marks)

(ii) Name any **three** causes of spectral interferences in AAS. (3 marks)

(iii) Explain how the spectral interferences in a(ii) above are minimised. (6 marks)

(b) Explain why the instrumental response in AAS is not affected by slight increments in flame temperature. (8 marks)

13. (a) State:

(i) Beer's law; (1 mark)

(ii) Lambert's law; (1 mark)

(iii) Beer-Lambert's law. (1 mark)

(b) (i) Identify all the terms in Beer-Lamberts law and state their units. (3 marks)

(ii) Differentiate between absorption coefficient and molar absorptivity. (2 marks)

(iii) The RMM of an organic compound is 398 and its absorption coefficient is 252 in a standard cuvette. Calculate the molar absorptivity of this compound if its absorbance is 1.15. (6 marks)

(c) (i) State **two** techniques of calibrating UV-visible spectrophotometers by applications of the standard additions method. (2 marks)

(ii) A metal napthalate, ashed and diluted to a fixed volume gave an absorbance reading of 29 units. Solution B containing the analyte plus 50 Mg of barium gave an absorbance reducing of 78 units. Calculate the amount of barium in the sample. (4 marks)

14. (a) Give any **four** factors which are considered in choosing the method of analysis for a given sample. (4 marks)

(b) Draw a labelled block diagram that shows the steps followed in solving an analytical problem. (14 marks)

(c) Name **two** classifications of analytical methods. (2 marks)

15. (a) Define the following terms as used in GLC - HPLC:

(i) resolution. (1 mark)

(ii) selectivity. (1 mark)

(iii) partition ratio. (1 mark)

(iv) column efficiency. (1 mark)

(b) Two solutes **A** and **B** were analysed by HPLC and their retention times were 7.2 and 11.3 minutes respectively. The pure solvent passed through the column in 1.4 minutes. The base widths for the resolution curves of **A** and **B** were 2.5 and 3.1 minutes respectively.
Calculate column:

(i) resolution; (3 marks)

(ii) selectivity; (3 marks)

(iii) plate height if mobile phase flow rate was 25 cm^3/minute. (6 marks)

(c) Give **four** advantages of HPLC over GLC. (4 marks)

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