2411/305
INSTRUMENTAL METHODS
OF ANALYSIS
Oct./Nov. 2018
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 7 printed pages.

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

Turn over

SECTION A (40 marks)

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

1. (a) Define the following terms as used in instrumental methods of analysis:

   (i) instrument optimization; (1 mark)

   (ii) zero-adjustment. (1 mark)

   (b) Explain why analytical instruments are:

   (i) optimised; (1 mark)

   (ii) zero-adjusted. (1 mark)

2. (a) State **two** monochromation methods which are used in flame photometry. (2 marks)

   (b) Describe the qualitative aspects of flame photometry. (2 marks)

3. (a) Calculate the expected number of IR absorption peaks for:

   $CH_2 = CH_2$ (2 marks)

   (b) Give the reasons for the difference between the expected number of peak and the observed number of peaks in the I.R. spectrum of

   $CH_2 = CH_2$ (2 marks)

4. (a) Describe the pressed pellet technique of preparing samples for analysis with IR spectrophotometer. (3 marks)

   (b) Explain **one** disadvantage of the pressed pellet technique. (1 mark)

5. The hollow Cathode lamp in Atomic Absorption Spectrophotometer (AAS) produces 100% monochromatic radiation. Explain the function of a monochromator in AAS. (4 marks)

6. Calculate the range of the absorbance values in AAS. (4 marks)

7. (a) Define the term instrumental sensitivity. (1 mark)

   (b) Outline how the instrumental sensitivity for analyte is determined. (1 mark)

   (c) The absorbance of a 200 ppm solution of $KMnO_4$ (Rfm = 158) is 0.65. Determine the instrumental sensitivity of $KMnO_4$. (2 marks)

8. During a colorimetric estimation of phosphates in a water sample by standard addition method, sample standards were prepared. The water sample was then run before samples were prepared and then sample standards added.

(a) Explain why the sample was run before the addition of the standards. (2 marks)

(b) Explain **two** advantages of the standard addition method of calibrating analytical instruments. (2 marks)

9. Describe the "head space" method of preparing samples for analysis by GLC. (4 marks)

10. (a) Write the Van Deemter equation. (1 mark)

(b) What is the derivative of the Van Deemter equation? (1 mark)

(c) Explain the application of the derivative in (b) above in HPLC. (2 marks)

SECTION B (60 marks)

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

11. (a) Using an illustration derive the wave number equation as used in IR Spectroscopy. (10 marks)

(b) Calculate the wave number of the C—C bond in benzene.

[C = 12, N = 6.0×10^{23}, K = 7.6×10^{5} dynes/cm.] (5 marks)

(c) The IR spectrum shown in figure 1 below is that of a liquid with a molecular formula $C_9H_{10}O$. Use the IR correlation data in table I provided to identify the compound. (5 marks)

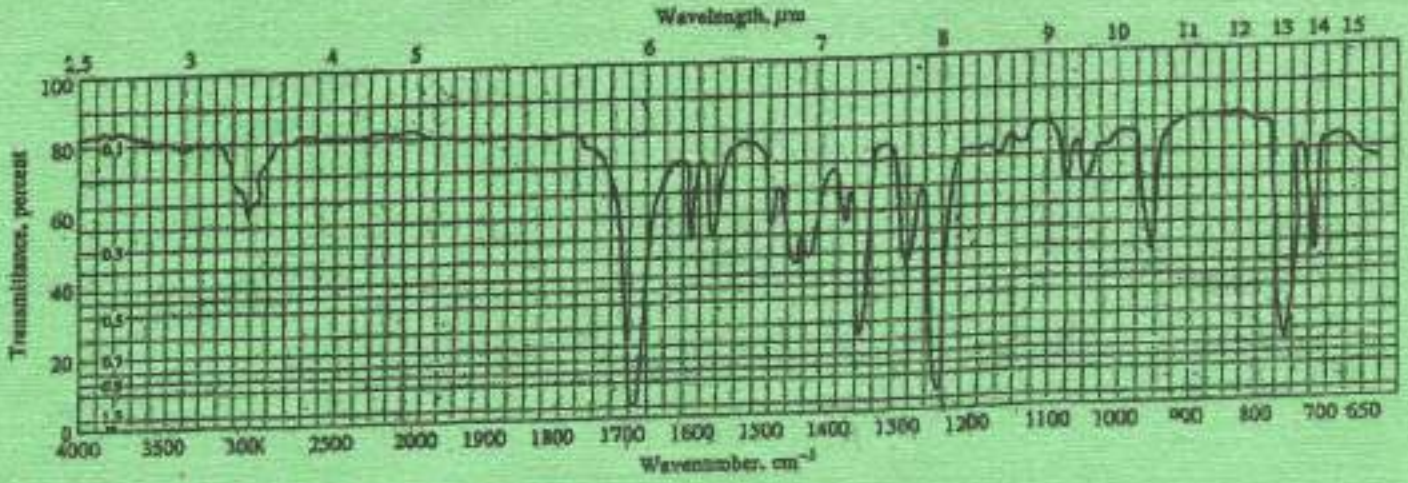


Fig. 1

Table I

Abbreviated Table of Group Frequencies for Organic Groups

Bond	Type of Compound	Frequency Range, cm^{-1}	Intensity
C—H	Alkanes	2850–2970	Strong
		1340–1470	Strong
C—H	Alkenes (>C=C<, H)	3010–3095	Medium
		675–995	Strong
C—H	Alkynes (—C≡C—H)	3300	Strong
C—H	Aromatic rings	3010–3100	Medium
		690–900	Strong
O—H	Monomeric alcohols, phenols	3590–3650	Variable
	Hydrogen-bonded alcohols, phenols	3200–3600	Variable, sometimes broad
	Monomeric carboxylic acids	3500–3650	Medium
	Hydrogen-bonded carboxylic acids	2500–2700	Broad
N—H	Amines, amides	3300–3500	Medium
C=C	Alkenes	1610–1680	Variable
C=C	Aromatic rings	1500–1600	Variable
C≡C	Alkynes	2100–2260	Variable
C—N	Amines, amides	1180–1360	Strong
C≡N	Nitriles	2210–2280	Strong
C—O	Alcohols, ethers, carboxylic acids, esters	1050–1300	Strong
C=O	Aldehydes, ketones, carboxylic acids, esters	1690–1760	Strong
NO_2	Nitro compounds	1500–1570	Strong
		1300–1370	Strong

12. (a) With the aid of labelled diagrams explain the effect in AAS of:

(i) rotating the burner head at 90°; (7 marks)

(ii) changing the burner height. (5 marks)

(b) State the:

(i) advantages; (6 marks)

(ii) disadvantages
of the lamina flow burner as a sample atomizer in AAS. (2 marks)

13. (a) Draw a labelled diagram of the flame photometer. (5 marks)

(b) Outline the steps that lead to the production of analytical signal in flame photometry. (4 marks)

(c) The results in table II were obtained in an experiment to calibrate a flame photometer by the standard series method.

Table II

Concentration of standards in ppm	0	200	300	400	500
Instrumental response	10	50	69.5	91	110

(i) State the criteria used in selecting the concentration range of the standards. (1 mark)

(ii) Plot the appropriate calibration graph. (8 marks)

(iii) Use the graph in (ii) above to estimate the analyte concentration of a sample that produced an instrumental response 83 units after tenfold preconcentration. (2 marks)

14. (a) Define the term "detector" as used in UV-visible spectrophotometry . (1 mark)

(b) (i) Draw a labelled diagram of the photomultiplier tube photodetector. (8 marks)

(ii) Describe how the photomultiplier tube photo detector functions. (5 marks)

(c) (i) Give a reason why monochromatic radiation is used in UV-visible spectrophotometry. (1 mark)

(ii) List **three** interferences in UV-visible spectrophotometry. (3 marks)

(iii) State **two** advantages of UV-visible spectrophotometry over AAS in the analysis of metals. (2 marks)

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

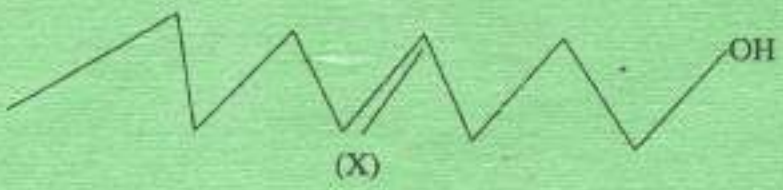
(i) void volume; (1 mark)

(ii) column resolution; (1 mark)

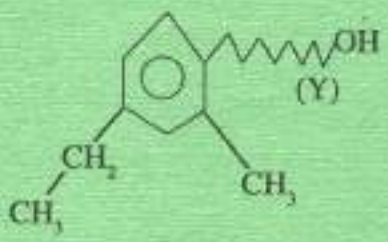
(iii) plate height; (1 mark)

(iv) theoritical plate. (1 mark)

(b) The retention times of two solutes X and Y in a 90 cm packed column were 22.78 and 18.33 minutes. The un retained species passed through the column in 1.95 minutes. The base width were 2.18 and 3.84 minutes.



(X)



(Y)

(i) With reasons, state the retention times of X and Y. (5 marks)

(ii) Calculate the:

(I) mobile phase flow rate; (2 marks)

(II) column resolution; (2 marks)

(III) column efficiency; (3 marks)

(IV) plate height; (2 marks)

(V) selectivity factor. (2 marks)

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