2411/305
INSTRUMENTAL METHODS
OF ANALYSIS
June/July 2020
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** 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 as 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.

Turn over

SECTION A (40 marks)

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

1. State **four** spectral interferences which cause deviation from Beer-Lambert's law in AAS. (4 marks)

2. (a) Define 'memory effects' as used in flame photometry. (1 mark)

   (b) Explain how 'memory effects' are minimised during analysis of a sample by flame photometry. (3 marks)

3. The compound butane is IR active due to the carbonyl group. Draw the structural formula of butane and on it indicate harmonics, overtones and the fundamental. (4 marks)

4. Explain how separation takes place in each of the following types of chromatography:

   (a) partition; (2 marks)

   (b) adsorption. (2 marks)

5. State **two** characteristics of each of the following:

   (a) mobile phase; (2 marks)

   (b) solid support in GLC. (2 marks)

6. (a) Calculate the number of IR absorption peaks for carbon dioxide. (3 marks)

   (b) State why the observed peaks in the IR spectrum of CO_2 is different from those calculated in (a). (1 mark)

7. Describe the basic principle of head-space sampling in GLC. (4 marks)

8. (a) Explain why samples for analysis by HPLC are purged of dissolved air. (3 marks)

   (b) State how the samples in (a) above are purged of dissolved air. (1 mark)

9. Calculate the approximate wave number of the carbonyl group in acetone.
   ($K = 1.0 \times 10^5\ Nm^{-1}, C = 12, O = 16, N = 6 \times 10^{23}$) (4 marks)

10. State **four** advantages of the Lovibond colour comparator as used in colorimetry. (4 marks)

SECTION B (60 marks)

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

11. (a) Describe ech of the following:

(i) Beer's law; (2 marks)

(ii) Lambert's law. (2 marks)

(b) Derive the mathematical expression for Beer-Lambert's law. (16 marks)

12. A photometer with linear response to radiation gave a reading of 685 mV with a blank in the path of radiation and 179 mV when the blank was replaced by an absorbing sample of 200 ppm of $K_2Cr_2O_7$. Given that K = 39, Cr = 52, O = 16, calculate the:

(a) absorbance of the sample; (5 marks)

(b) percent transmittance of the sample; (4 marks)

(c) molar absorptivity of the solution. (11 marks)

13. (a) (i) Define vibrational coupling as used in IR spectroscopy. (1 mark)

(ii) State **four** factors which reduce the number of observed peaks of an IR active compound. (4 marks)

(iii) State **seven** factors which affect bond vibrations of an IR active molecule. (7 marks)

(b) (i) Write FT - IR in full. (1 mark)

(ii) State **five** advantages of FT - IR over conventional IR instruments. (5 marks)

(c) State **two** causes of peak broadening in IR spectroscopy. (2 marks)

14. (a) Describe with the use of a labelled block diagram a flame photometer. (11 marks)

(b) Outline the steps that lead to production of analytical signal in flame photometry. (9 marks)

15. (a) Describe how substances are quantified in GLC. (8 marks)

(b) The data shown in table I was obtained during analysis of tri-halomethanes in drinking water by GLC.

Table I

Tri-halomethane	Concentration in ppb	Peak area
$CHCl_3$	1.30	1.35×10^4
$CHCl_2Br$	0.90	6.12×10^4
$CHClBr_2$	4.00	1.71×10^4
$CHBr_3$	1.20	1.52×10^4

(i) Explain the type of detector used in this GLC instrument. (3 marks)

(ii) Estimate the concentration of dibromochloromethane in a water sample if its peak area is 1.20×10^4. (9 marks)

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