UNIVERSITY OF JOHANNESBURG

DEPARTMENT OF CHEMISTRY

MODULE **CEM8X03**
ATOMIC SPECTROSCOPY

CAMPUS **APK**

SUPPLEMENTARY EXAM

DATE: JULY 2019 **SESSION:**

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EXTERNAL MODERATOR: **PROF. KL MANDIWANA**

DURATION: 3 HOURS **MARKS: 100**

NUMBER OF PAGES: 6

GENERAL INSTRUCTIONS

There are a total of 3 questions in this exam booklet. Please attempt all questions showing all steps in your calculations and giving detailed explanation wherever required. Please note that a "Yes" or "No" type answers are unacceptable

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QUESTION ONE

1.1 Analytical techniques such as ICP-MS are used for simultaneous multi-element determination. You are tasked to prepare mixed standard solutions containing four elements for ICP-MS analysis. You are given stock solutions of four elements in four different vials with the label concentrations shown in brackets (ppm): Cu (500); Mn (500); As (100); Hg (100). From these stock solutions, you are asked to describe the preparation of the following mixed standard solutions in 10 mL volumetric flask with concentration of each element shown in bracket below.

a) Standard 3 (ppm): Cu (0.5), Mn (0.5), As (0.1), Hg (0.1)
b) Standard 2 (ppb): Cu (100), Mn (100), As (20), Hg (20)
c) Standard 1 (ppb): Cu (25), Mn (25), As (5), Hg (5)

Please note that you have 10 – 100 µL and 100 – 1000 µL variable micropipettes to transfer the standard solutions. Follow the best economical approach to prepare the standard solutions. (12)

1.2 Briefly explain the following concepts in atomic spectrometry.

a) Nebulization (2)

b) Atomization (2)

1.3 Show the mechanism behind emission of radiation by hollow cathode lamp in terms of chemical reactions. (4)

1.4 Name and briefly explain the heating stages in graphite furnace atomic absorption spectrometry. (6)

1.5 Discuss the principle of flame atomic emission spectrometry. (6)

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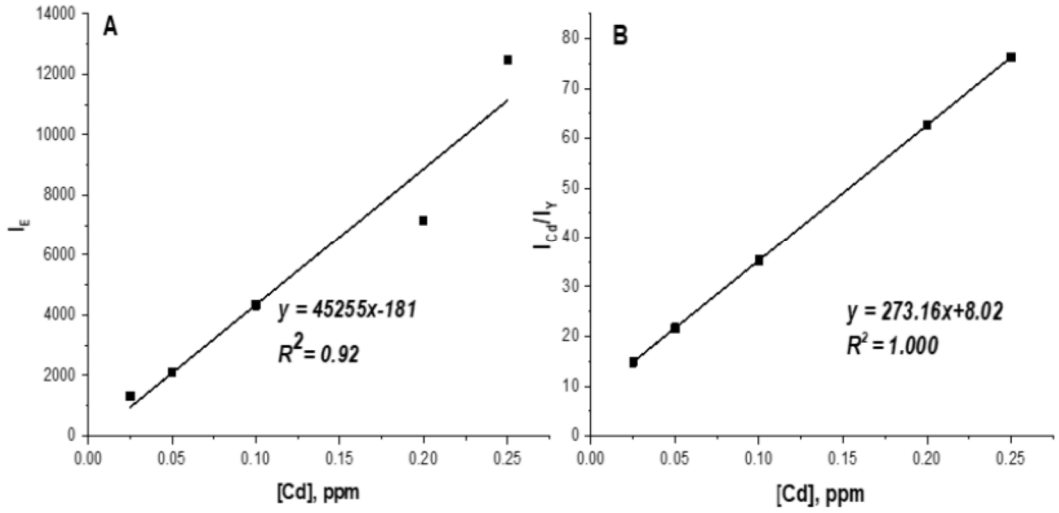
QUESTION TWO

2.1 The concentration of cadmium (Cd) in wastewater was measured using inductively coupled plasma-optical emission spectrometry (ICP-OES). A sample weighing 1.00 g was digested in 5 mL HNO_3 and 0.50 mL aliquot of 500 ppm yttrium (Y) added before it was diluted to 50.00 mL. Aliquots of Cd standard (ppm) were pipetted into four 50.00 mL volumetric flasks to ensure final concentrations which ranged between 0.02 to 0.20 ppm. Approximately 5 mL of HNO_3 and 0.50 mL of the 500 ppm yttrium standards were added to each flask before they were diluted to a final volume of 50.00 mL with de-ionized water. Emission intensity of each solution was measured at 226.5 nm (Cd) and at 371.0 nm (Y). The calibration curves were constructed and presented as shown on the graphs A and B. The measured emission intensity counts for Cd and Y were 3230 and 66, respectively.



Based on the above information answer questions (a) to (h)

a) Graph B is preferred for the quantification of Cd in the wastewater. Give a reason for your choice. (1)

b) Explain why HNO_3 was added to the Cd calibration standards. (2)

c) Why was yttrium added to the standards and the sample in the determination of Cd using ICP-OES? (2)

d) Calculate the concentration of yttrium in standard and sample solutions. (2)

e) Calculate the concentration of Cd (ppm) in wastewater using both graphs A and B. (4)

f) Choose the correct answer from (e) above and calculate the concentration, in µg/g, of Cd in the original wastewater sample. (3)

g) State an advantage of the high temperature attained in the plasma with respect to atomization and ionization of Cd. (4)

h) This analysis was carried out with the 'axial' viewing mode of the plasma. Draw a sketch to illustrate this viewing mode. State an advantage of this viewing mode. (4)

2.2 Interferences are commonly encountered in chemical analysis. Answer questions (a) through (c) based on types of interferences encountered in AAS and ICP-OES.

a) Explain why chemical interferences are less common in ICP-OES than they are in flame AAS. (4)

b) Why are ionization interferences usually not as severe in the ICP as they are in flames? (2)

c) How can ionization interference be minimized in atomic absorption spectrometry? (3)

2.3 What are the applications of isotope ratio measurements? Illustrate your answer by giving example. (5)

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QUESTION THREE

3.1 What are the functions of tangential argon gas flow in ICP-OES and ICP-MS?

(3)

3.2 How would you evaluate ICP-OES, ICP-MS and XRF techniques for the determination of Hg, Cd and Pb in wastewater at part-per-billion levels? Compare three techniques in terms of multi-elemental capability and limits of detection. (5)

3.3 Differentiate between total, fractionation and speciation analysis. (6)

3.4 Why do we use hyphenated techniques if standard techniques are available?

Illustrate your answer by providing examples. (6)

3.5 Briefly explain the difference between wavelength-dispersive X-ray fluorescence (WDXRF) and energy-dispersive X-ray fluorescence (EDXRF) techniques. Give the block diagrams of both techniques. (12)

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The Periodic Table