THE PAPUA NEW GUINEA UNIVERSITY OF TECHNOLOGY

FIRST SEMESTER EXAMINATION

CH331 – INSTRUMENTAL ANALYSIS II

TUESDAY 23rd JUNE 2020 - 12:50 PM

TIME ALLOWED: 2 HOURS

INFORMATION FOR CANDIDATES:

1. You will have 10 minutes to read the question paper. You **MUST NOT** begin writing in the answer book during this time.
2. **ANSWER ALL QUESTIONS.**
3. All answers **MUST** be written on the answer book provided.
4. Calculators are permitted in the examination room. Lecture notes, notebooks, plain papers, and textbooks are **NOT** allowed.
5. Mobile phones are not allowed. **SWITCH OFF THE MOBILE PHONES.**
6. Show all working and calculations in the answer book.
7. **DRAW any FIGURES** clearly and visibly.
8. **DO NOT** over write
9. Write your name and number clearly on the front page of the answer book. **DO IT NOW.**

MARKING SCHEME: [TOTAL 60 MARKS]

1. (a) Name the THREE main types of hollow radiation sources (lamps) used in Atomic Absorption Spectrometry (AAS).

[3 marks]

(b)

(i) What type of radiations are produced by deuterium lamp, and tungsten lamp, respectively?

(ii) Why are deuterium and tungsten lamps used in a modern AAS instruments?

[6 marks]

(TOTAL: 9 MARKS)

2. (a) Mention any FOUR types of interferences encountered in Flame Atomic Absorption and Emission Spectrometry.

[4 marks]

(b) For each interference (in (2a) above), mention ONE way the interference can be taken care of.

[4 marks]

(c) Why would chemical interference be common in flame emission spectrometry, but not in plasma emission spectrometry?

[2 marks]

(TOTAL: 10 MARKS)

3. (a) What are the advantages of ICP Emission spectrometry over flame emission spectrometry?

[6 marks]

(b) Practically, how would you determine if matrix interference exists in a quantitative spectrometric determination?

[4 marks]

(TOTAL: 10 MARKS)

4. In modern flame atomic absorption spectrometry, the continuum source background correction is preferable over the two-line background correction.
   (a) How is the continuum source background correction done in modern flame AAS instruments?

   [6 marks]

   (b) Mention any TWO disadvantages of the continuum source background correction technique?

   [2 marks]

   (c) How would the instrument "know" which continuum source to select for background correction?

   [2 marks]

   (TOTAL: 10 MARKS)

5. Tin (Sn), is leached into canned foods from tin-plated steel cans. For Quality Assurance purposes, Sn must be analyzed in foods sold in such steel cans. For analysis by inductively-coupled plasma atomic emission, the food is digested by microwave heating in a Teflon bomb in three steps with HNO_3, H_2O_2, and HCl.

   (a) CsCl is added to the final solution at a concentration above 500 ppm. What is the purpose of the CsCl addition?

   [2 marks]

   (b) Calibration data are given in the table below. Using the data, develop the calibration plot on the graph sheet provided.

Sn (µg/L)	Emission at 189.9 nm
0	4.0
10.0	9.0
20.0	20.0
30.0	24.0
40.0	31.0
60.0	42.0
100.0	79.0
200.0	159.0

   [5 marks]

(c) From the graph in (5b) above, find the slope and intercept, and write the linear equation for the calibration using the slope and intercept.

[4 marks]

(d) Interference by high concentration of other elements was assessed at TWO emission lines of Sn. Foods containing little tin were digested and spiked with tin at about 100.0 µg/L. Then, other elements were deliberately added. The Table below show the selected results.

Element added at 50 mg/L	Sn found (µg/L) with 189.9 nm emission line	Sn found (µg/L) with 235.5 nm emission line
None	100.0	100.0
Cu	100.9	116.2
Fe	103.3	Emission too high
Mn	99.5	126.3
Zn	105.3	112.8
Cr	102.8	76.4

Using the data provided in this Table in (5d), answer the following questions.

(i) Which elements interfere in the assay, and at what wavelengths?
(ii) From your decisions in (5d) (i) above, which wavelength must be preferred for the analysis?
(iii) What type of interference could be happening with the presence of Cr at 235.5 nm, and Fe at 235.5 nm?

[6 marks]

(TOTAL: 17 MARKS)

6. (a) Mention TWO reference electrodes used in potentiometric measurements.

[2 marks]

(b) Mention TWO sources of errors encountered using pH measurements.

[2 marks]

(TOTAL: 4 MARKS)

----------**THE END**----------