NAMIBIA UNIVERSITY
OF SCIENCE AND TECHNOLOGY

FACULTY OF HEALTH, APPLIED SCIENCES AND NATURAL RESOURCES

DEPARTMENT OF NATURAL AND APPLIED SCIENCES

QUALIFICATION: BACHELOR OF SCIENCE HONOURS	
QUALIFICATION CODE: 08BOSH	**LEVEL:** 8
COURSE CODE: AAC811S	**COURSE NAME:** ADVANCED ANALYTICAL METHOD AND CHEMOMETRICS
SESSION: JULY 2022	**PAPER:** THEORY
DURATION: 3 HOURS	**MARKS:** 100

SUPPLEMENTARY/SECOND OPPORTUNITY EXAMINATION QUESTION PAPER	
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MODERATOR:	PROF JAMES ABAH

INSTRUCTIONS
1. Answer ALL the questions in the answer book provided. 2. Write and number your answers clearly. 3. All written works MUST be done in blue or black ink.

PERMISSIBLE MATERIALS

Non-programmable Calculators

ATTACHMENTS

List of Useful Tables and formulas

THIS QUESTION PAPER CONSISTS OF 6 PAGES (Including this front page and attachments)

Question 1 [20]

1.1 Name the different parts of the analytical strategy also known as the experimental design. (5)

1.2 A standard sample of pooled human blood serum contains 42.0 g of albumin/L. A laboratory performs replicate determinations of the albumin concentration on the same standard sample and obtains the following results (in g L^{-1}): 42.2; 41.6; 42.0; 41.8; 42.6 and 39.0.

(a) Use appropriate statistics tests to assess whether the used method was affected by systematic error (P = 0.05). (5)

(b) It is suspected that the last replicate measurement is an outlier. Use the recommended ISO test to confirm whether this is the case at P = 0.05. (3)

(c) How does your finding in (b) affect the precision and accuracy of the results? (3)

(d) What would be a more robust way of estimating the central tendency of the obtained replicate measurements? Explain your choice. (4)

Question 2 [30]

2.1 Sample containers used for the collection of solutions (liquids) samples are made from glass or plastic.

(a) What are the common issues associated to glass containers? (3)

(b) Clearly explain (with reaction if necessary) why glass containers are not that recommended to collect solutions used for trace metals analysis. (4)

(c) If glass bottles are the only available containers to collect liquid samples for heavy metals analysis, how would you proceed during sampling to prevent the underlined issue in (b) to happen? (2)

2.2 Why is it important to reduce the size of solid particles present in a sample? (4)

2.3 A quantitative analysis gives a mean concentration of 12.6 ppm for an analyte. The method's standard deviation (S_{meth}) is 1.1 ppm and the standard deviation for sampling (S_{samp}) is 2.1 ppm.

(a) What is the overall variance, S^2, for the analysis? (1)

(b) By what percentage does the overall variance change if we improve S_{meth} by 10% to 0.99 ppm? (2)

(c) By what percentage does the overall variance change if we improve S_{samp} by 10% to 1.89 ppm? (2)

(d) Briefly discuss the meaning/implication of your findings from (a) to (c). (2)

2.4 Provide the main purpose of each of the following acids during a wet digestion process

(a) Nitric acid (1)

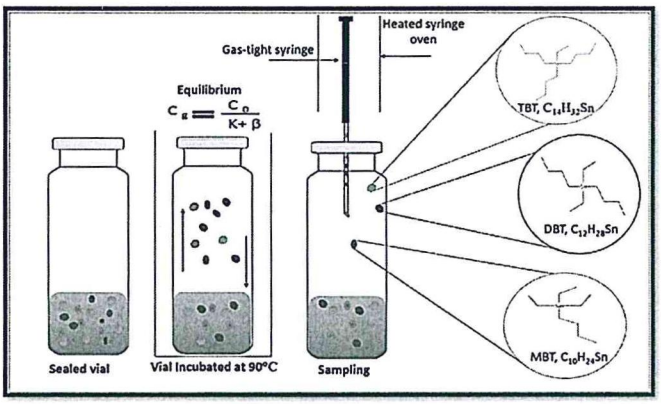
(b) Hydrochloric acid (1)

(c) Aqua Regia (1)

(d) Hydrofluoric acid (1)

(e) Perchloric acid (1)

2.5 The following figure represents a technique that is widely used for the extraction of gaseous analytes in liquid samples



(a) What is the name of the technique represented in the above figure? (2)

(b) Briefly explain the principle of the represented technique (3)

Question 3 [20]

3.1 One method for the analysis of Fe^{3+}, which can be used with a variety of sample matrices, is to form the highly coloured Fe^{3+}–thioglycolic acid complex. The complex absorbs strongly at 535 nm. Standardizing the method is accomplished using external standards. A 10.0 ppm Fe^{3+} working standard is prepared by transferring a 10-mL aliquot of a 100.0 ppm stock solution of Fe^{3+} to a 100-mL

volumetric flask and diluting to volume. Calibration standards of 1.0, 2.0, 3.0, 4.0, and 5.0 ppm are prepared by transferring appropriate amounts of the 10.0 ppm working solution into separate 50-mL volumetric flasks, each containing 5 mL of thioglycolic acid, 2 mL of 20% w/v ammonium citrate, and 5 mL of 0.22 M NH_3. After diluting to volume and mixing, the absorbances of the external standards are measured against an appropriate blank. Samples are prepared for analysis by taking a portion known to contain approximately 0.1 g of Fe^{3+}, dissolving in a minimum amount of HNO_3 and diluting to volume in a 1-L volumetric flask. A 1.00-mL aliquot of this solution is transferred to a 50-mL volumetric flask, along with 5 mL of thioglycolic acid, 2 mL of 20% w/v ammonium citrate, and 5 mL of 0.22 M NH_3 and diluted to volume. The absorbance of this solution is used to determine the concentration of Fe^{3+} in the sample.

(a) What is an appropriate blank for this procedure? (2)

(b) Ammonium citrate is added to prevent the precipitation of Al^{3+}. What is the effect on the reported concentration of iron in the sample if there is a trace impurity of Fe^{3+} in the ammonium citrate? (2)

(c) Why does the procedure specify that the sample contains about 0.1 g of Fe^{3+}? (3)

3.2 (a) Define an internal standard. (1)

(b) What is the basic principle of internal standardisation? (2)

(c) When do you use an internal standard? (3)

3.3 Many of the analytical methods used to determine the concentration of fibrinogen in plasma are based on light scattering following its precipitation. Light scattering is measured nephelometrically at a wavelength of 340 nm. Analysis of a set of external calibration standards gives the following calibration equation

$$I_S = -4.66 + 9907.63 \times C$$

where Is is the intensity of scattered light and C is the concentration of fibrinogen in g/L. A 9.0-mL sample of plasma was collected from a patient and mixed with 1.0 mL of an anticoagulating agent. A 1.0-mL aliquot of this solution was then diluted to 250 mL in a volumetric flask. Analysis of the resulting solution gave a scattering intensity of 44.70. What is the concentration of fibrinogen, in gram per liter, in the plasma sample? (4)

3.4 Give three disadvantages of the isotope dilution method. (3)

Question 4 [30]

4.1 The following diagram describes different spectrometric techniques labelled A to D.