Mastership in Chemical Analysis

Part A Examination

Online

17th April 2024

1000 – 1300

Plus 10 minutes reading time

Instructions

Answer **five** questions out of eight questions.

All questions carry equal marks.

The marks allocated to each section are given in the brackets.

The answers to each section must be returned in the examination envelope provided. All examination scripts must be mailed to the RSC at the end of the examination.

There are several equations and data tables at the back of the exam script, which can be used at any point during the exam.

Non-programmable calculators are permitted.

Graph paper is required.

Question 1. Answer **ALL** parts

In evaluating a method for the determination of glyphosate in an agricultural winter bean crop the following results (µg/g) were obtained: 26.05, 21.89, 24.25, 27.65, 22.66, 26.21, 22.58, 23.81 and 21.89 when method A was used. The reported mean and standard deviation (n = 5) when an independent method B was used for the same analysis are as follows (µg/g): 19.56 and 0.96, respectively.

(a)   Calculate the mean, variance and standard deviation of the results for method A.

(9 marks)

(b)   Calculate the 95% confidence interval of the mean for method A.

(4 marks)

(c)   Are the results obtained by both methods A and B significantly different?

(5 marks)

(d)   Comment on the results of your calculations.

(2 marks)

    [Note: Statistical information is presented at the end of the examination paper.]

Question 2. Answer **ALL** parts

(a) The results of sampling for benzo(a)pyrene determined the following data:

After completing the table determine the number of samples required such that the maximum error does not exceed ± 3 µg/mL, at the 95% confidence interval.

[Note: Sampling equations are presented at the end of the examination paper.]

(5 marks)

(b) Outline a method for the preparation for analysis of dried smoked fish for a port health authority. The method should include steps from when the sample arrives in the laboratory until it is available for injection into a gas chromatograph. Include all necessary details and considerations.

(10 marks)

(Question 2 continues on the next page)

Question 3.

Question 6. Answer **ALL** parts

(a) Describe, with the aid of

Question 8. Answer **ALL** parts

(a) Solid phase extraction (SPE) is used to preconcentrate aqueous samples for chromatographic analysis. Outline the typical steps required to perform SPE.

(5 marks)

(b) An aqueous sample (50 ml) suspected of containing dieldrin is preconcentrated using SPE. The determinand is eluted in 5 ml of methanol, evaporated to dryness, and re-dissolved in 1 ml of solvent. The sample was then analysed using gas chromatography - mass spectrometry and a peak area of 520 obtained. A calibration plot was prepared which produced the following data:

Volume of a 1 µg/ml stock solution diluted to 10 ml (ml)	Internal standard corrected peak area for dieldrin
0	0
0.5	15000
1.0	29900
1.5	47500
2.0	70500

Calculate the concentration in ng/ml of dieldrin in the original sample.

(5 marks)

(c) Solid phase microextraction (SPME) is a commonly used technique to pre-concentrate analytes prior to introducing them into a gas chromatograph. Describe, with the aid of a diagram, the physical components of a SPME probe, and explain the principle of the technique.

(4 marks)

(d) Briefly describe the analytical protocol for the extraction and subsequent chromatographic analysis of **THREE** of the following. Note you cannot select the same extraction method for each answer:

    (i)   DDT and its metabolites from a contaminated milk.

    (ii)   Hexaconazole (a fungicide) from fruit and vegetables.

    (iii)   Phenols in a process water sample.

    (iv)   Dichloromethane in decaffeinated coffee.

(6 marks)

Useful Sampling Equations

$$E = \pm$$