BIRLA INSTITUTE OF TECHNOLOGY AND SCIENCE, PILANI
First SEMESTER 2023 – 2024
Environmental Sampling and Analytical Methods – Comprehensive Examination PART A (Close Book)

Course No: EEG501 **Date: 12-12-2023**
Duration: Maximum 1 hour, 2 PM-3 PM **Max. Marks: 20**

Note: All the questions are compulsory. If unit is not mentioned, marks will be deducted. Assume appropriate data if needed. Answer all parts of a question together.

1. Write the differences between gross, systematic, and random errors during sample analyses? **[3 Marks]**

2. What are the conditions when back titration is preferred over forward titration? **[3 Marks]**

3. Define potentiometric titration. Give an example of potentiometric titration for environmental sample analysis. **[3 Marks]**

4. Explain an electrochemical cell (with salt bridge) with a diagram. **[4 Marks]**

5. To separate very polar components by normal phase HPLC, would you choose a very polar or nonpolar material for the stationary phase? Which component (most polar to least polar) will be eluted first? **[2 Marks]**

6. Explain with a neat diagram how X-ray fluorescence is produced in XRF spectroscopy? **[3 Marks]**

7. Which one of the following is used to preserve samples for the analysis of total metals (excluding Cr and Hg): (a) Cool, 4°C, (b) NaOH to pH > 12, (c) H_2SO_4 to pH < 2, (d) HNO_3 to pH < 2? **[2 Marks]**

BIRLA INSTITUTE OF TECHNOLOGY AND SCIENCE, PILANI
First SEMESTER 2023 – 2024
Environmental Sampling and Analytical Methods – Comprehensive Examination Part B (Open Book)
Course No: EEG501 **Date: 12-12-2023**
Duration: 2 hours, 3 PM-5 PM **Max. Marks: 40**

Note: All the questions are compulsory. If unit is not mentioned, marks will be deducted. Assume appropriate data if needed.

1. The absorbance of a 2-cm sample cell of a 10 ppm solution is 0.43, what would be the absorbance of a 1-cm cell of 15 ppm solution of the same chemical under the same condition? **[5 Marks]**

2. A groundwater sample is analyzed for its K by FAA (Flame Atomic Absorption Spectroscopy) using the method of standard additions. Two 500 µL of this groundwater sample are added to 10.0-mL DI water. To one portion, it was added 10.0 µL of 10 mM K solution. The net emission signals in arbitrary units are 20.2 and 75.1. What is the concentration of K in this groundwater in mg/L? Molecular weight of K=39.1 g. **[6 Marks]**

3. The following data apply to a column liquid chromatography: length of packing = 24.7 cm, flow rate = 0.313 mL/min. A chromatogram of a mixture of chemical A and B provided the following data: Calculate the following and make comments regarding the separation:

	Retention time, min	Width of peak base (w), min
Nonretained solvent	3.1	–
Chemical A	13.3	1.07
Chemical B	14.1	1.16

(a) The number of plates (N) from each peak (chemical A and B)

(b) The plate height (H) for the column on the basis of the average number of plates

(c) The retention factor (k) for each peak

(d) The resolution between chemical A and B

(e) The separation factor (a) between chemical A and B

(f) The length of column necessary to give a resolution of 1.5

[7 Marks]

4. Calculation of recovery from a matrix spike QC sample. Pyrene in soil samples was extracted by sonic extraction followed by analysis using HPLC. A stock solution of pyrene (60 µL, 100 mg/L) in acetonitrile was spiked in a clean soil (0.5 g) and was extracted with 10 mL hexane. A portion of the extract (10 µL) was injected to HPLC and the concentration of the extract was determined to be 0.5 mg/L. Calculate the percentage recovery of the extraction. **[6 Marks]**

5. A new method is being developed for the analysis of a pesticide in soils. A spiked sample with a known concentration of 17.00 mg/kg was measured five times using the new method (data: 15.3, 17.1, 16.7, 15.5, 17.3 mg/kg) and the established EPA method (data: 15.4, 15.9, 16.7, 16.1, 16.2 mg/kg). The Excel outputs of descriptive statistics and one-way ANOVA are given below ($\alpha = 0.05$). On the basis of these output, (a) are these two methods significantly different at a 95% confidence level? why?
   a. Excel output of descriptive statistics

EPA method	
Mean	16.06
Standard error	0.211187121
Median	16.1
Mode	#N/A
Standard deviation	0.472228758
Sample variance	0.223
Kurtosis	1.012889863
Skewness	−0.105406085
Range	1.3
Minimum	15.4
Maximum	16.7
Sum	80.3
Count	5
Largest(1)	16.7
Smallest(1)	15.4
Confidence Level (95.0%)	0.586350662

New method	
Mean	16.38
Standard error	0.412795349
Median	16.7
Mode	#N/A
Standard deviation	0.923038461
Sample variance	0.852
Kurtosis	−2.910136878
Skewness	−0.400799173
Range	2
Minimum	15.3
Maximum	17.3
Sum	81.9
Count	5
Largest(1)	17.3
Smallest(1)	15.3
Confidence Level (95.0%)	1.146106

(b) Excel output of one-way ANOVA

Summary

Groups	*Count*	*Sum*	*Average*	*Variance*
EPA method	5	80.3	16.06	0.223
New method	5	81.9	16.38	0.852

ANOVA

Source of variation	*SS*	*df*	*MS*	*F*	*p-value*	*F crit*
Between Groups	0.256	1	0.256	0.4762791	0.5096343	5.317644991
Within Groups	4.3	8	0.5375			
Total	4.556	9				

[5 Marks]

6. Glucose is oxidized according to: $C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O$. It can be used as a standard of COD because its theoretical COD can be calculated from its known concentration.

   What is the theoretical COD value of a solution containing 0.50 g/L glucose ($C_6H_{12}O_6$; MW = 180) (Hint: 1 mole glucose consumes 6 moles of O_2)?

   [5 Marks]

7. 100 mL HCl was titrated with 16.8 mL 0.2 N NaOH solution. Calculate the normality and the percentage concentration of the HCl solution. **[6 Marks]**