Analytical Chemistry: A Practical Approach

Exercises and Short Problems

1. Define what is meant by the terms, “homogeneous sample” and “random sample”. Give a suitable example in each case, in order to demonstrate what these mean. This question links in again with Problem 1.1 from chapter 1 but more importantly focuses on the terms used specifically in chapter 2

2. Explain, giving reasons, which of the analytical techniques, given in Table 1.1, you would use for the following analyte/sample combinations after suitable sample preparation:

   (a) High levels of Mn in a food-grade stainless steel sample
   (b) Fluoride content of toothpaste
   (c) Individual PCBs in contaminated soil from below an old electrical sub-station
   (d) Low levels of Se in vegetables
   (e) Total quinine content in tonic water and bitter lemon drinks

Table 1.1 Analytical techniques

High performance liquid chromatography with UV-vis detection	Gas chromatography – mass spectrometry	ICP-AES
ICP-MS	Ion selective electrode	UV-vis spectrophotometry
Molecular fluorescence	CHN analyser	Atomic fluorescence

NB: It may be possible to use more than one technique after sample preparation
This question relates to chapter 4.

3. The mean of 5 measurements for the determination of Se in CRM1566b Oyster Tissue is 2.2 μg g^{-1} with a standard deviation of 0.24 μg g^{-1}. The certified value is 2.06 μg g^{-1}. Use a t-test to determine whether there is any evidence of systematic error in the mean of the measurements compared with the certified value. This question relates to chapter 9.

4. You have used atomic absorption spectroscopy to determine Cu in sediment from a contaminated river by the standard additions method, and obtained the data given in Table 1.2. Calculate the blank-corrected concentration of Cu in the digested sample. This question relates to chapter 5.

Table 1.2 Results for the determination of Cu in sediment by the method of standard additions

	Sample mass / g	Slope, *b* / absorbance per μg cm^{-3}	Intercept, *a* / absorbance
Procedural Blank	0	0.0184	0.0501
Digested sediment	0.5086	0.0186	0.3218

5. You have just started a new job as quality control manager of a tuna cannery. As one of your first tasks, you have been asked to produce a Shewhart moving range chart using the data given in Table 1.3 for the determination of mercury in a raw tuna quality control sample. Assume that the first 10 samples are in control and use them to calculate your target value and average moving range. Plot the chart for all twenty samples and make any appropriate comments. This question relates to chapter 9.

Table 1.3 Quality control data for mercury in a quality control sample

Date	QC sample number	QC result/ mg kg^{-1}	Moving range
18/06/2015	1	4.08	
19/06/2015	2	3.74	0.34
20/06/2015	3	4.05	0.31
21/06/2015	4	4.37	0.32
22/06/2015	5	4.43	0.06
23/06/2015	6	4.20	0.23
24/06/2015	7	4.66	0.46
25/06/2015	8	4.56	0.10
26/06/2015	9	3.76	0.80
27/06/2015	10	3.74	0.02
28/06/2015	11	4.09	0.35
29/06/2015	12	3.73	0.36
30/06/2015	13	4.30	0.57
01/07/2015	14	4.03	0.27
02/07/2015	15	4.28	0.25
03/07/2015	16	5.60	1.32
04/07/2015	17	4.28	1.32
05/07/2015	18	3.72	0.56
06/07/2015	19	3.68	0.04
07/07/2015	20	3.81	0.13