

ANALYTICAL CHEMISTRY

Tutorial 2: Calibration and methods of analysis

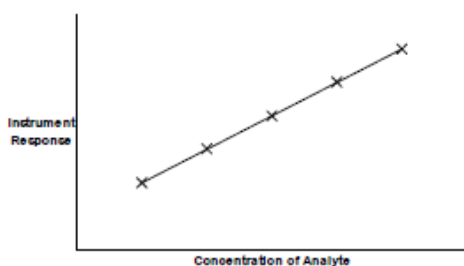
Question 1

You have a number of water samples that you would like to have analysed for Pb by ICP-OES. You are required to make up a set of standard solutions for the analysis. Show how you would make 500.0 mL of a 300 ppm Pb stock solution from the $\text{Pb}(\text{NO}_3)_2$ salt.

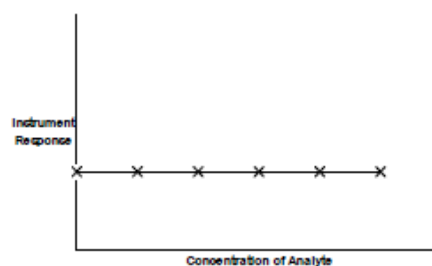
Question 2

Explain what is wrong (if anything) with each of the following calibration graphs?

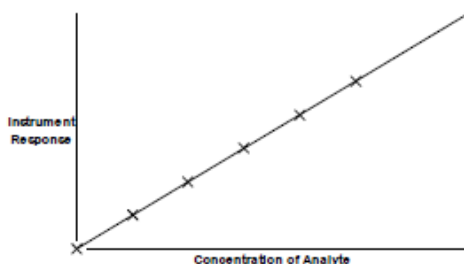
a)



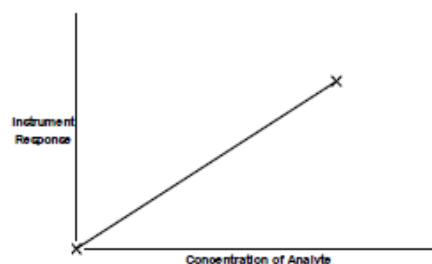
b)



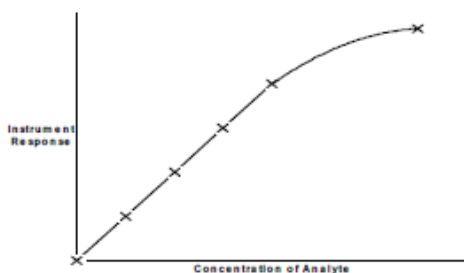
c)



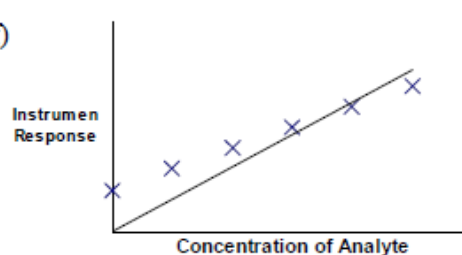
d)



e)



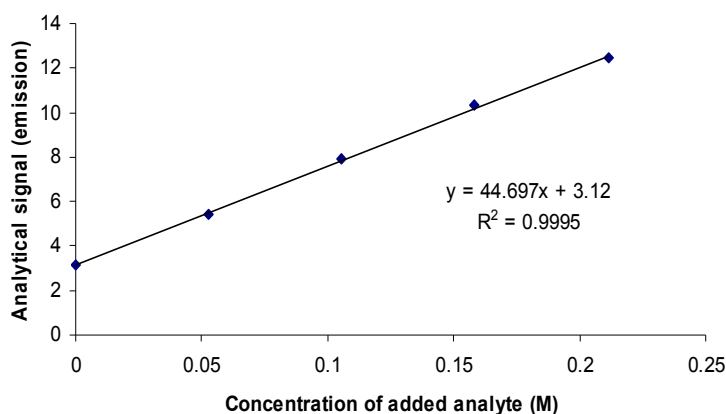
f)



Question 3

The Na^+ concentration in a blood serum sample was determined using the method of standard addition and atomic spectroscopy. 25.00 mL aliquots of serum were pipetted into five 50.00 mL volumetric flasks into which 2.640 M NaCl standard was added as shown in the table below. After dilution to volume, the emission of each solution was measured and used to plot a calibration graph.

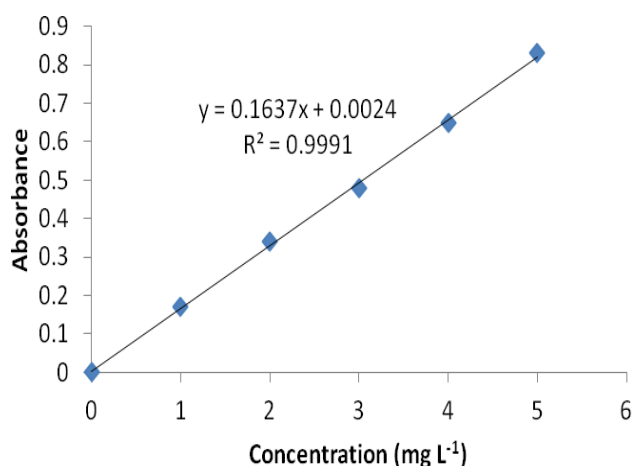
Flask	Vol. of standard (mL)	Emission signal
1	0	3.13
2	1.00	5.40
3	2.00	7.89
4	3.00	10.30
5	4.00	12.48



- Using the plotted calibration graph, calculate the Na^+ concentration in the blood serum sample.
- Provide a possible reason why the serum sample might have been analysed using this analytical procedure.

Question 4

Atomic Absorption Spectroscopy (AAS) was used to determine the lead content in a soil sample collected from the side of a road. A 0.4500 g sample was digested in concentrated HCl, filtered, and then transferred into a 50 mL volumetric flask for analysis. A set of five standards were prepared and the absorbance of these solutions were measured and used to plot the calibration graph shown. The absorbance of the sample was found to be 0.58. Calculate the % lead in the original soil sample.



Question 5

10 mL aliquots of a natural water sample were pipette into 50.00 mL volumetric flasks. Exactly 0.00, 5.00, 10.00, 15.00 and 20.00 mL of a standard solution containing 11.1 ppm of Fe were added to each, followed by an excess of thiocyanate ion to give the red complex $\text{Fe}(\text{SCN})^{2+}$. After dilution to volume, absorbances for the five solutions were measured using a photometer and were found to be 0.240, 0.437, 0.621, 0.809, and 1.009, respectively.

Plot the standard addition graph in *excel* and using the equation of the line, calculate the concentration of Fe in the water sample.