Chapter 1

Methodology for Trace Elemental Analysis

Learning Objectives

- To be aware of the different types of contamination that can cause problems in trace elemental analysis.
- To be aware of a whole range of terms and definitions as used in analytical chemistry.
- To appreciate the range of units used in analytical chemistry.
- To be able to present numerical data with correct units, and be able to interchange the units as required.
- To be able to present data in the form of a table.
- To be able to present data in the form of a graph.
- To be able to determine the concentration of an element from a straight line graph using the equation $y = mx + c$.
- To be able to calculate the dilution factor for a liquid sample and a solid sample, and hence determine the concentration of the element in the original sample.
- To appreciate the concept of quality assurance in the analytical laboratory.
- To be aware of the significance of certified reference materials in elemental analysis.

1.1 Introduction

Trace elemental analysis requires more than just knowledge of the analytical technique to be used – in this case, inductively coupled plasma spectroscopy. This

Practical Inductively Coupled Plasma Spectroscopy J. R. Dean

requires knowledge of a whole range of disciplines that need to come together to create the final result. The disciplines required can be described as follows:

- sampling, sample storage and preservation, and sample preparation methodologies
- analytical technique
- data control, including calibration strategies and the use of certified reference materials for quality control
- data management, including reporting of results and their meaning

While most of these are covered to some extent in this book, the reader should also consult other resources, e.g. books. Suggestions for further study are given in Chapter 8.

The perspective that is required when faced with trace element analysis are the additional precautions required in terms of management of contamination, choice of reagents and acids, and cleanliness of the workspace. For example, the grade of chemical used to prepare calibration standards is a major concern when working at trace element analysis levels (sub-$\mu g\,ml^{-1}$). Chemicals are available in a range of grades from 'GPR – general purpose reagent' through to, for example, 'AnalaR® – analytical reagent'. However, the descriptor does not identify that the chemical is any purer (the purity is often given on the bottle, e.g. 99.8%), but identifies the amount of effort that the manufacturer has gone to in the preparation of the chemical. For 'AnalaR®' grade materials, the manufacturer has characterized the chemical by subjecting it to chemical analysis. This additional effort is then passed on to the customer in the form of a higher price. The use of sample blanks in the analytical procedure is essential to identify 'problem elements'.

The risk of contamination is a major problem in trace element analysis. Apart from the analytical reagent used to prepare standards, as discussed above, contamination can also be experienced from sample containers, e.g. volumetric flasks, pipettes, etc. For example, metal ions can adsorb onto glass containers and then leach into the solution under acidic conditions, thereby causing contamination. This can be minimized by cleaning the glassware prior to use by soaking for at least 24 h in a 10% nitric acid solution, followed by rinsing with 'clean' deionized water (three times).

DQ 1.1

What type of water would you consider to be clean?

Answer

Drinking water, commercially bottled drinking water or laboratory distilled water. The actual answer depends to some extent on the work to *be carried out. For trace element analysis, it is possible to purchase commercial systems that filter (distilled) water through a combination of ion-exchange columns to remove trace element impurities.*

The cleaned vessels should then either be stored upside down or covered with Clingfilm® to prevent dust contamination.

The individual working in the laboratory is also a major source of contamination. Therefore, as well as the normal laboratory safety associated with wearing a laboratory coat and safety glasses, it may be necessary to take additional steps such as the wearing of 'contaminant-free' gloves and a close-fitting hat.

1.2 Analytical Terms and their Definitions

The following is an alphabetical list of the most important analytical terms of use in practical inductively coupled plasma spectroscopy.

Accuracy A quantity referring to the difference between the mean of a set of results or an individual result and the value which is accepted as the true or correct value for the quantity being measured.

Acid digestion Use of acid (and often heat) to destroy the organic matrix of a sample to liberate the metal content.

Aliquot A known amount of a homogenous material assumed to be taken with negligible sampling error.

Analyte The component of a sample which is ultimately determined directly or indirectly.

Bias Characterizes the systematic error in a given analytical procedure and is the (positive or negative) deviation of the mean analytical result from the (known or assumed) true value.

Calibration The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system and the corresponding known values of the measurand.

Calibration curve Graphical representation of a measuring signal as a function of quantity of analyte.

Certified Reference Material (CRM) Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the units in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

Complexing agent The chemical species (an ion or a compound) which will bond to a metal ion using lone pairs of electrons.

Confidence interval Range of values that contains the true value at a given level of probability. The latter is known as the *confidence level.*

Confidence limit The extreme values or ‘end-values’ in a confidence interval.

Contamination In trace analysis this is the unintentional introduction of analyte(s) or other species which are not present in the original sample and which may cause an error in the determination. This can occur at any stage in the analysis. Quality assurance procedures, such as analyses of blanks or of reference materials, are used to check for contamination problems.

Control of Substances Hazardous to Health (COSHH) Regulations that impose specific legal requirements for risk assessment wherever hazardous chemicals or biological agents are used.

Co-precipitation The inclusion of otherwise soluble ions during the precipitation of lower-solubility species.

Dilution factor The mathematical factor applied to the determined value (data obtained from a calibration graph) which allows the concentration in the original sample to be determined. Frequently, for solid samples, this will involve a sample weight and a volume to which the digested/extracted sample is made up to prior to analysis. For liquid samples, this will involve an initial sample volume and a volume to which the digested/extracted sample is made up to prior to analysis.

Dissolved Material that will pass through a 0.45 μm membrane filter assembly prior to sample acidification.

Dry ashing Use of heat to destroy the organic matrix of a sample to liberate the metal content.

Error The error of an analytical result is the difference between the result and a ’true’ value:

Random error Result of a measurement minus the mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions.

Systematic error Mean that would result from an infinite number of measurements of the same measurand carried out under repeatability conditions, minus the true value of the measurand.

Extraction The removal of a soluble material from a solid mixture by means of a solvent or the removal of one or more components from a liquid mixture by use of a solvent with which the liquid is immiscible or nearly so.

Figure of merit A parameter that describes the quality of performance of an instrument or an analytical procedure.

‘Fitness for purpose’ The degree to which data produced by a measurement process enables a user to make technically and administratively correct decisions for a stated purpose.

Heterogeneity The degree to which a property or a constituent is randomly distributed throughout a quantity of material. The degree of heterogeneity is the determining factor of sampling error.

Homogeneity The degree to which a property or a constituent is uniformly distributed throughout a quantity of material. A material may be homogenous with respect to one analyte but heterogeneous with respect to another.

Interferent Any component of the sample affecting the final measurement.

Limit of detection The detection limit of an individual analytical procedure is the lowest amount of an analyte in a sample which can be detected but not necessarily quantified as an exact value. The limit of detection, expressed as either the concentration c_L or the quantity q_L, is derived from the smallest measure, x_L, that can be detected with reasonable certainty for a given procedure. The value x_L is given by the following equation:

$$x_L = x_{bl} + ks_{bl}$$

where x_{bl} is the mean of the blank measures, s_{bl} is the standard deviation of the blank measures and k is a numerical factor chosen according to the confidence level required. For many purposes, the limit of detection is taken to be $3s_{bl}$ or 3 × 'the signal-to-noise ratio', assuming a zero blank.

Limit of quantitation For an individual analytical procedure, this is the lowest amount of an analyte in a sample which can be quantitatively determined with suitable uncertainty. It may also be referred to as the *limit of determination*. The limit of quantitation can be taken as 10 × 'the signal-to-noise ratio', assuming a zero blank.

Linear dynamic range (LDR) The concentration range over which the analytical working calibration curve remains linear.

Linearity This defines the ability of the method to obtain test results proportional to the concentration of analyte.

Liquid–liquid extraction A method of extracting a desired component from a liquid mixture by bringing the solution into contact with a second liquid, the solvent, in which the component is also soluble, and which is immiscible with the first liquid or nearly so.

Matrix The carrier of the test component (analyte), all of the constituents of the material except the analyte, or the material with as low a concentration of the analyte as it is possible to obtain.

Measurand A particular quantity subject to measurement.

Method The overall, systematic procedure required to undertaken an analysis. This includes all stages of the analysis, and not just the (instrumental) end determination.

Microwave digestion A method of digesting an organic matrix to liberate metal content by using an acid at elevated temperature (and pressure) based on microwave radiation. Can be carried out in either open or sealed vessels.

Organometallic An organic compound in which a metal is covalently bonded to carbon.

Outlier This may be defined as an observation in a set of data that appears to be inconsistent with the remainder of that set.

Precision The closeness of agreement between independent test results obtained under stipulated conditions.

Qualitative analysis Chemical analysis designed to identify the components of a substance or mixture.

Quality assurance All those planned and systematic actions necessary to provide adequate confidence that a product or services will satisfy given requirements for quality.

Quality control The operational techniques and activities that are used to fulfill requirements of quality.

Quality control chart A graphical record of the monitoring of control samples which helps to determine the reliability of the results.

Quantitative analysis This is normally taken to mean the numerical measurement of one or more analytes to the required level of confidence.

Reagent A test substance that is added to a system in order to bring about a reaction or to see whether a reaction occurs (e.g. an analytical reagent).

Reagent blank A solution obtained by carrying out all steps of the analytical procedure in the absence of a sample.

Recovery The fraction of the total quantity of a substance recoverable following a chemical procedure.

Reference material This is a material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Repeatability Precision under repeatability conditions, i.e. conditions where independent test results are obtained with the same method on identical test items in the same laboratory, by the same operator, using the same equipment within short intervals of time.

Reproducibility Precision under reproducibility conditions, i.e. conditions where test results are obtained with the same method on identical test items in different laboratories, with different operators, using different equipment.

Robustness For an analytical procedure, this is a measure of its capacity to remain unaffected by small, but deliberate variations in method parameters, and provides an indication of its reliability during normal usage. It is sometimes referred to as *ruggedness*.

Sample A portion of material selected from a larger quantity of material. The term needs to be qualified, e.g. representative sample, sub-sample, etc.

Selectivity (in analysis) Qualitative – the extent to which other substances interfere with the determination of a substance according to a given procedure. Quantitative – a term used in conjunction with another substantive (e.g. constant, coefficient, index, factor, number, etc.) for the quantitative characterization of interferences.

Signal-to-noise ratio A measure of the relative influence of noise on a control signal. Usually taken as the magnitude of the signal divided by the standard deviation of the background signal.

Solvent extraction The removal of a soluble material from a solid mixture by means of a solvent or the removal of one or more components from a liquid mixture by use of a solvent with which the liquid is immiscible or nearly so.

Speciation The process of identifying and quantifying the different defined species, forms or phases present in a material or the description of the amounts and types of these species, forms or phases present.

Standard (all types) A standard is an entity established by consensus and approved by a recognized body. It may refer to a material or solution (e.g. an organic compound of known purity or an aqueous solution of a metal of agreed concentration) or a document (e.g. a methodology for an analysis or a quality system). The relevant terms are as follows:

Analytical standard (*also known as* ***Standard solution***) A solution or matrix containing the analyte which will be used to check the performance of the method/instrument.

Calibration standard The solution or matrix containing the analyte (measurand) at a known value with which to establish a corresponding response from the method/instrument.

External standard A measurand, usually identical with the analyte, analysed separately from the sample.

Internal standard A measurand, similar to but not identical with the analyte, which is combined with the sample.

Standard method A procedure for carrying out a chemical analysis which has been documented and approved by a recognized body.

Standard addition The addition of a known amount of analyte to the sample in order to determine the relative response of the detector to an analyte within the sample matrix. The relative response is then used to assess the sample analyte concentration.

Stock solution This is generally a standard or reagent solution of known accepted stability, which has been prepared in relatively large amounts of which portions are used as required. Frequently, such portions are used following further dilution.

Sub-sample This may be either (a) a portion of the sample obtained by selection or division, (b) an individual unit of the lot taken as part of the sample, or (c) the final unit of multi-stage sampling.

True value A value consistent with the definition of a given particular quantity.

Uncertainty A parameter associated with the result of a measurement which characterizes the dispersion of the values that could reasonably be attributed to the measurand.

1.3 Units

The Systeme International d'Unites (SI) uses a series of base units (Table 1.1) from which other terms have been derived. Some of the most commonly used SI derived units are shown in Table 1.2. When using units, it is standard practice to keep numbers between 0.1 and 1000 by the use of a set of prefixes, based on multiples of 10^3 (Table 1.3). It is an extremely useful skill to be able to interchange these units and prefixes. For example, 1 mol l^{-1} can also be expressed as 1000 μmol ml^{-1}, 1000 mmol l^{-1} or 1000 nmol μl^{-1}.

SAQ 1.1

The prefixes shown below in Table 1.3 are frequently used in analytical science to represent large or small quantities. Reassign the following quantities with the suggested prefixes.

Quantity 3×10^{-7} m	m	μm	nm
Quantity 6.9×10^{-3} mol l^{-1}	mol l^{-1}	mmol l^{-1}	μmol l^{-1}
Quantity 2.80 ppm	μg ml^{-1}	mg l^{-1}	ng μl^{-1}

1.4 Calibration Strategies

Quantitative analysis in plasma spectroscopy requires the preparation of a series of calibration standards from a stock solution. These standards are prepared in

Table 1.1 Commonly used base SI Units. From Dean, J. R., *Methods for Environmental Trace Analysis*, AnTS Series. Copyright 2003. © John Wiley and Sons, Limited. Reproduced with permission

Measured quantity	Name of unit	Symbol
Length	Metre	m
Mass	Kilogram	kg
Amount of substance	Mole	mol
Time	Second	s
Thermodynamic temperature	Kelvin	K
Luminous intensity	Candela	cd

Table 1.2 Commonly used SI derived units. From Dean, J. R., *Methods for Environmental Trace Analysis*, AnTS Series. Copyright 2003. © John Wiley and Sons, Limited. Reproduced with permission

Measured quantity	Name of unit	Symbol	Definition in base units	Alternative in derived units
Energy	Joule	J	m^2 kg s^{-2}	N m
Force	Newton	N	m kg s^{-2}	J m^{-1}
Pressure	Pascal	Pa	kg m^{-1} s^{-2}	N m^{-2}
Electric charge	Coulomb	C	A s	J V^{-1}
Frequency	Hertz	Hz	s^{-1}	—

Table 1.3 Commonly used prefixes. From Dean, J. R., *Methods for Environmental Trace Analysis*, AnTS Series. Copyright 2003. © John Wiley and Sons, Limited. Reproduced with permission

Multiple	Prefix	Symbol
10^{18}	exa	E
10^{15}	peta	P
10^{12}	tera	T
10^{9}	giga	G
10^{6}	mega	M
10^{3}	kilo	k
10^{-3}	milli	m
10^{-6}	micro	μ
10^{-9}	nano	n
10^{-12}	pico	p
10^{-15}	femto	f
10^{-18}	atto	a

volumetric flasks. Calibration solutions are usually prepared in terms of their molar concentrations, i.e. mol l^{-1}, or mass concentrations, i.e. g l^{-1}, with both referring to an amount per unit volume, i.e. concentration = amount/volume. It is important to use the highest (purity) grade of chemicals (liquids or solids) for the preparation of the stock solution, e.g. AnalaR® or AristaR®.

DQ 1.2

How would you prepare a 1000 ppm solution of lead from lead nitrate?

Answer

The molecular weight of lead nitrate, $Pb(NO_3)_2$*, is 331.20 and the atomic weight of lead is 207.19. Therefore, simply dividing the molecular weight* *by the atomic weight will give you the exact amount of lead nitrate to be dissolved in 1 litre to produce a 1000 ppm solution of lead.*

$$331.20/207.19 = 1.5985 \text{ g of } Pb(NO_3)_2 \text{ in 1 litre}$$

Frequently, however, a litre of stock solution is not required.

DQ 1.3

What mass of lead nitrate would need to be accurately weighed to prepare 100 ml of the 1000 ppm stock solution?

Answer

With reference to DQ 1.2, the mass is simply obtained by dividing the amount in 1 litre by 10, i.e. 0.1599 g.

1.5 Presentation of Data: Tables

It is often convenient when carrying out quantitative laboratory work to record data in tabular form. This is often best done by creating two columns into which the data can be entered. It is essential, however, for future consultation of these data, that the columns are given the appropriate headings, e.g. concentration ($\mu g\,l^{-1}$) and signal (mV), to prevent errors occurring later. It is also important to record details of any sample dilutions that have taken place (see also Chapter 8). A typical table of data for an experiment to determine the concentration of lead is shown in Table 1.4. It is perfectly acceptable to record information, by using a pen, in a laboratory notebook. Any modifications should then be carried out by simply crossing out the erroneous data and entering the correct information.

Table 1.4 An example of how to record quantitative data for an inductively coupled plasma–mass spectrometry (ICP–MS) experiment

Concentration ($\mu g\ l^{-1}$)	^{208}Pb intensity (counts s^{-1})
0	565
10	19 887
20	45 356
30	59 876
40	78 543
50	99 654

1.6 Presentation of Data: Graphs

Most graphs are now plotted by using computer-based graphics packages, e.g. Microsoft Excel™, rather than by hand on graph paper. You should remember, however, that you still need the skill to plot a graph by hand on graph paper as this is still the most common method used in examinations. Irrespective of the mode of preparing the graph, it is important to ensure that the graph is correctly labelled and presented. All graphs should have a numerical descriptor and title, e.g. 'Figure 3.2 Influence of time (hours) on the recovery of metal species from a sediment sample'.

Graphs are normally used to describe a relationship between two variables, e.g. x and y. It is normal practice to identify the x-axis as the horizontal axis (absicca) and to use this for the independent variable, e.g. concentration (with its appropriate units). The vertical axis or ordinate (y-axis) is used to plot the dependent variable, e.g. signal response (with units, if appropriate). The mathematical relationship most commonly used for straight-line graphs is as follows:

$$y = mx + c$$

where y is the signal response, e.g. signal (mV), x is the concentration of the working solution (in appropriate units, e.g. μg ml^{-1} or ppm), m is the slope of the graph, and c is the intercept on the x-axis.

A typical graphical representation of data obtained from an experiment to determine the level of lead in a sample using inductively coupled plasma–mass spectrometry (ICP–MS) is shown in Figure 1.1 (from the data tabulated in Table 1.4).†

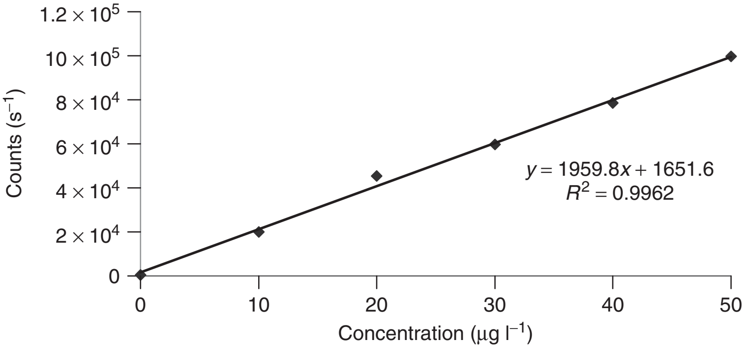

Figure 1.1 Typical calibration graph used to determine lead (^{208}Pb) in a sample (cf. Table 1.4).

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† R, in this figure (and also in Figure 1.2), is known as the *correlation coefficient*, and provides a measure of the quality of calibration. In practice, R^2 (the *coefficient of determination*) is used because it is more sensitive to changes. This varies between -1 and $+1$, with values very close to -1 and $+1$ pointing to a very tight 'fit' of the calibration curve.

An alternative approach to undergoing a *direct calibration*, as described above, is the use of the method of *standard additions*. This may be particularly useful if the sample is known to contain a significant portion of a potentially interfering matrix. In standard additions, the calibration plot no longer passes through zero (on both the x- and y-axes). As the concept of standard additions is to eliminate any matrix effects present in the sample, it should be implicit that the working standard solutions all will contain the same volume of the sample, i.e. the same volume of the sample solution is introduced into a succession of working calibration solutions. Each of these solutions, containing the same volume of the sample, is then introduced into the inductively coupled plasma and the response recorded. However, plotting the signal response (e.g. signal (mV)) against analyte concentration produces a graph that no longer passes through zero on either axis, but if correctly drawn, the graph can be extended towards the x-axis (extrapolated) until it intercepts it. By maintaining a constant concentration x-axis, the unknown sample concentration can be determined (Figure 1.2). It is essential that this graph is linear over its entire length or otherwise considerable error can be introduced.

In both the direct calibration graph and the method of additions graph, the result obtained from the sample is normally not the final answer of how much of the metal was in the original sample. This is because the sample has normally undergone some form of sample preparation. In the case of a solid sample, this might have involved acid digestion (see Section 2.3.1), while in the case of a liquid sample, liquid–liquid extraction (see Section 2.2.1). What is required in both cases is a correction (or dilution factor) that takes into account the sample

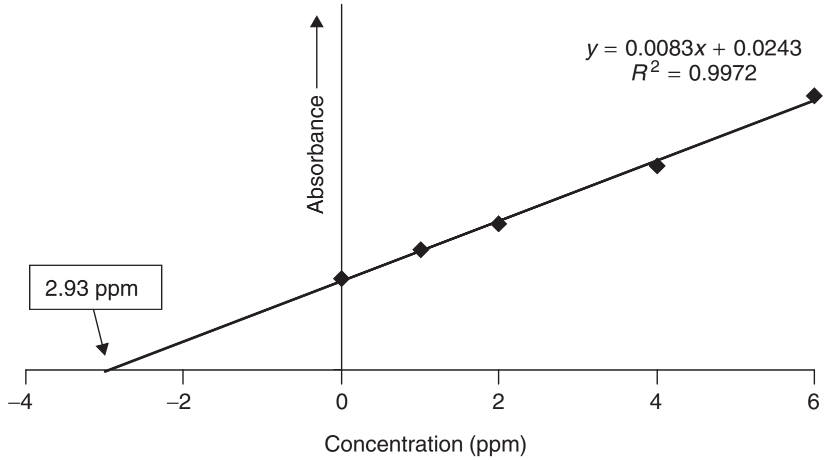


Figure 1.2 Determination of lead in soil: standard additions method. From Dean, J. R., *Methods for Environmental Trace Analysis*, AnTS Series. Copyright 2003. © John Wiley & Sons, Limited. Reproduced with permission.

preparation procedure. The following provides examples of the general forms of calculations that are necessary in the case of (1) a liquid sample that has been extracted using ammonium pyrrolidine dithiocarbamate (APDC)–methylisobutyl ketone (MIBK), and (2) a solid sample that has been acid-digested (see Figures 2.1 and 2.3, respectively, for further details of the procedures).

1.7 Calculations: Dilution Factors

Case study 1. Calculate the concentration ($\mu g\ ml^{-1}$) of copper in a waste water sample obtained from the local waste treatment plant. A waste water sample (150 ml) was extracted with APDC–diethylammonium diethyldithiocarbamate (DDDC) into MIBK (20 ml) using liquid–liquid extraction. The extract was then quantitatively transferred to a 25 ml volumetric flask and made up to the mark with MIBK. What is the dilution factor?

$$25\ \mathrm{ml}/150\ \mathrm{ml} = 0.167\ \mathrm{ml\ ml^{-1}}$$

If the solution was then analysed and found to be within the linear portion of the graph (see Figure 1.1), the value for the dilution factor should then be multiplied by the concentration from the graph, hence producing a final value indicating the concentration of copper in the waste water sample.

Case study 2. Calculate the concentration ($\mu g\ g^{-1}$) of lead in a soil sample obtained from a contaminated land site. An accurately weighed (5.2456 g) soil sample is acid-digested (see Figure 2.3) using nitric acid and hydrogen peroxide, cooled and then quantitatively transferred to a 100 ml volumetric flask and made up to the mark with distilled water. This solution is then diluted by taking 10 ml and transferring to a further 100 ml volumetric flask where it is made up to the mark with high-purity water. What is the dilution factor?

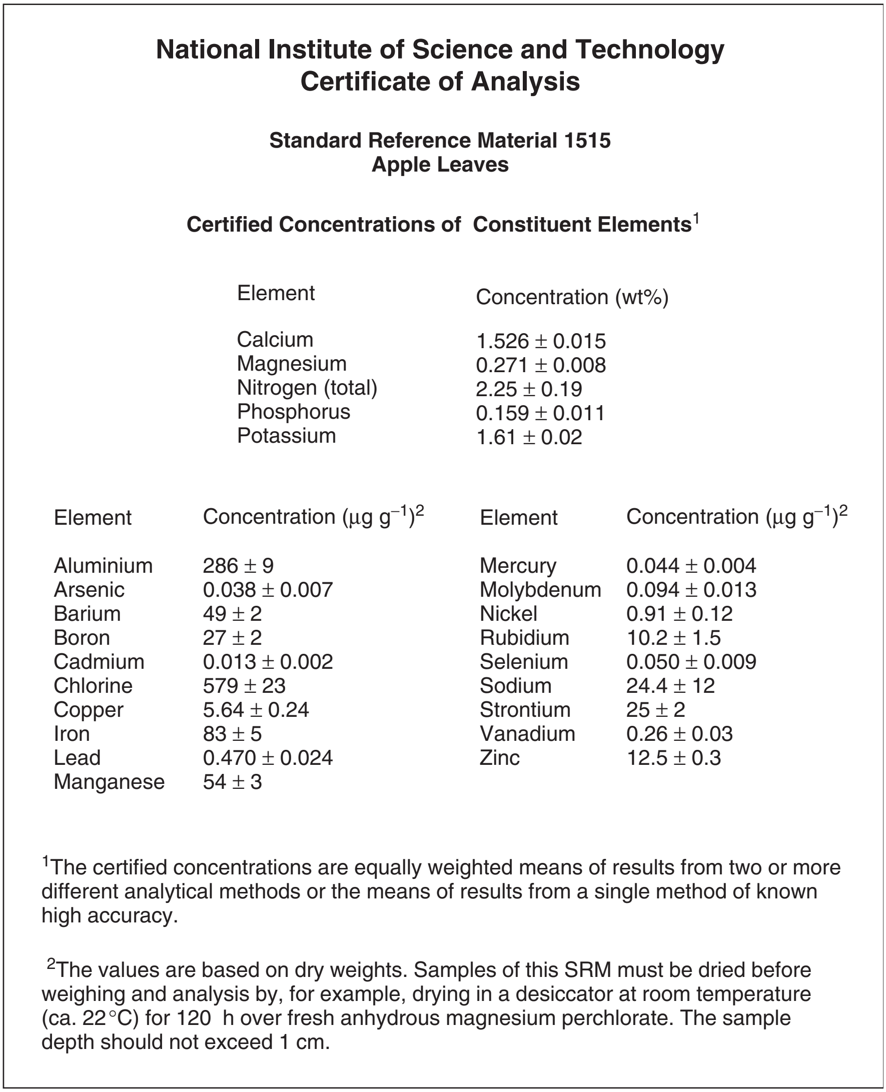
$$[(100\ \mathrm{ml})/(5.2456\ \mathrm{g}) \times (100\ \mathrm{ml}/10\ \mathrm{ml})] = 190.64\ \mathrm{ml\ g^{-1}}$$

If the solution was then analysed and found to be within the linear portion of the graph (see Figure 1.1), the value for the dilution factor should then be multiplied by the concentration from the graph, so producing a final value indicating the concentration of lead in the contaminated soil sample.

1.8 Quality Assurance and the Use of Certified Reference Materials

Quality assurance is all about getting the correct result. The main objectives of a quality assurance scheme are as follows:

- to select and validate appropriate methods of sample preparation



National Institute of Science and Technology
Certificate of Analysis

Standard Reference Material 1515
Apple Leaves

Certified Concentrations of Constituent Elements[1]

Element	Concentration (wt%)
Calcium	1.526 ± 0.015
Magnesium	0.271 ± 0.008
Nitrogen (total)	2.25 ± 0.19
Phosphorus	0.159 ± 0.011
Potassium	1.61 ± 0.02

Element	Concentration ($\mu g\ g^{-1}$)[2]	Element	Concentration ($\mu g\ g^{-1}$)[2]
Aluminium	286 ± 9	Mercury	0.044 ± 0.004
Arsenic	0.038 ± 0.007	Molybdenum	0.094 ± 0.013
Barium	49 ± 2	Nickel	0.91 ± 0.12
Boron	27 ± 2	Rubidium	10.2 ± 1.5
Cadmium	0.013 ± 0.002	Selenium	0.050 ± 0.009
Chlorine	579 ± 23	Sodium	24.4 ± 12
Copper	5.64 ± 0.24	Strontium	25 ± 2
Iron	83 ± 5	Vanadium	0.26 ± 0.03
Lead	0.470 ± 0.024	Zinc	12.5 ± 0.3
Manganese	54 ± 3		

[1] The certified concentrations are equally weighted means of results from two or more different analytical methods or the means of results from a single method of known high accuracy.

[2] The values are based on dry weights. Samples of this SRM must be dried before weighing and analysis by, for example, drying in a desiccator at room temperature (ca. 22 °C) for 120 h over fresh anhydrous magnesium perchlorate. The sample depth should not exceed 1 cm.

Figure 1.3 An example of a certificate of analysis for elements in apple leaves. Reprinted from Certificate of Analysis, *Standard Reference Material 1515, Apple Leaves*, National Institute of Standards and Technology. Not copyrightable in the United States.

- to select and validate appropriate methods of analysis
- to maintain and upgrade analytical instruments
- to ensure good record-keeping of methods and results

- to ensure quality of the data produced
- to maintain a high quality of laboratory performance

In implementing a good quality control programme it is necessary to analyse *certified reference materials*. By definition, a certified reference material is a substance for which one or more elements have known values and estimates of their uncertainties, produced by a technically valid procedure, accompanied with a traceable certificate and issued by a certifying body. Typical examples of certifying bodies are the National Institute for Standards and Technology (NIST), based in Washington, DC, USA, the Community Bureau of Reference (BCR), Brussels, Belgium, and the Laboratory of the Government Chemist (LGC), London, UK. The accompanying certificate, in addition to providing details of the certified elemental concentration and their uncertainties in the sample, also provides details of the minimum sample weights to be used, storage conditions, moisture content, etc. An example of a typical certificate is shown in Figure 1.3.

Summary

The methodology for trace elemental analysis requires an understanding of a whole range of inter-related issues centred around the sample, sample preparation, analysis, data interpretation/presentation and quality assurance. This chapter has highlighted some of the most important aspects. In addition, the main strategies for calibration are discussed, including the preparation of a standard solution. Two examples of how to calculate a dilution factor are given for aqueous and solid materials.