



Camborne School of Mines
Analytical laboratories

Induction manual

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Introduction

This document is designed to give an introduction to work in the analytical laboratories at Camborne School of Mines. It contains important procedures for health and safety, a section on the principles and recommended practices for analysis and data management, and at the end a statement of receipt of induction that must be signed and returned to the laboratory manager before work can commence.

1. Health and safety in the laboratories

This section provides an introduction to health and safety in the analytical laboratories at Camborne School of Mines.

Access

Access to the laboratories is controlled with swipe cards and keys. Upon entering the laboratories, ensure to locate escape routes, telephones, fire and first aid equipment, and contact information for first aiders. Read and follow the safety and security notices for the laboratories you will be working in. All work undertaken in the laboratories must be agreed by the laboratory manager. Work by students and external clients must only be carried out during times when competent laboratory staff members are available. Instructions from staff should always be followed.

Code of conduct for laboratory work

You must read and understand the “Code of Conduct in the CSM Research Laboratories” (included in this manual) before any laboratory work is undertaken. This Code of Conduct provides you with information relating to general behaviour in the laboratories.

Risk assessments

Before work is carried out, you must complete the adequate risk assessments relating to the work. The aim is to identify the appropriate health issues specific to a project, as well as to ensure that the requirements for insurance cover are met. A risk assessment will include all or some of the following parts:

1. Management of health and safety at work.

This is a general assessment of the risks involved with work carried out in the laboratories and must always be completed.

2. Manual handling.

This assessment must be completed where work involves manual handling (lifting / crushing / grinding / mineral processing).

3. Control of Substances Hazardous to Health (COSHH).

This includes work with substances that have known health effects. Material safety datasheets are available for most chemical substances, but an overall assessment must be carried out on the total risks involved in the work undertaken.



CODE OF CONDUCT IN THE CSM RESEARCH LABORATORIES

To keep the laboratories running smoothly and safely, it is vital that your work complies with the general laboratory practice. Safety must be your primary concern in the laboratories, and therefore we ask you to read the following information carefully. In the laboratories, we are working with potentially damaging and hazardous equipment and materials, so it is vital that you never disregard the following procedures.

1. The laboratories are generally available for your use on work days between 9.00am and 5.30pm. If you need to work outside these times you will need to arrange it with the laboratory staff.
2. All laboratory work should be booked and costed in advance following the approved laboratory management system and guidelines (costing not applicable to scheduled teaching activities). Where samples need to be submitted to technicians (for example for mechanical or electrical testing, or for analytical chemistry), you will be given an indication of when you may expect the results. If your project has a deadline, please allow for plenty of time for the technicians to carry out the work. Things can go wrong, and some pieces of equipment are time consuming to repair. Please leave an email or mobile number and you will be contacted when your results are available. Please don't hassle the technicians, you won't get your results any quicker that way.
3. Before starting practical work, make sure that the appropriate Health, Safety, and Risk assessments have been carried out (COSHH, manual handling, radiation, electrical; details are available from the technicians). Lecturers must ensure that these assessments are carried out for the safe conduct of teaching activities.
4. Never work alone in the laboratory without the permission of the laboratory staff. Keep the laboratory staff informed about what you are doing so that they can identify and potential hazards.
5. Before commencing work, make sure you are aware of the safety procedures; know the emergency escape routes, the location and availability of the nearest first aiders, and the location of the following emergency safety equipment: Eye wash stations, Emergency showers, Fire blankets and Fire extinguishers, First aid boxes, Telephones.
6. Wear sensible clothing that protects you against the potential hazards in the labs. Always comply with the signage, and follow guidance and recommendations by laboratory staff. For work involving manual handling and mechanical equipment, you may be required to wear a hard hat, ear protection, protective eyewear, dust masks, steel-toe capped footwear, and clothes that cover most of your skin. For work involving chemicals, long trousers should be worn at all times and you may consider purchasing a lab coat. Open shoes, sandals, bare feet, and shorts are not permitted. Long hair must be tied back.
7. Wear protective glasses for all chemical procedures and whenever there is a danger of exposure to air-borne particles or explosions (rock crushing and grinding, use of rock saws, coring equipment and pressure rig). Ensure that the proper extracts (dust, fumes, heat) are always used.
8. Smoking, food, drinks, and snacks are not allowed in the laboratories.
9. Never work in the laboratory if you are ill, tired, or under the influence of drugs or alcohol.
10. Please respect the decisions of the technical staff, they are working to help your classes and projects run as safely and effectively as possible. Any form of abuse to lab staff is unacceptable and will result in lab exclusion.
11. Always keep safe distances from crushers, rock saws, pressure rigs, furnaces, ovens, hot plates, fume cupboards in use, and other equipment or instruments that could be of danger.
12. Never leave chemicals, glassware, or instruments in places where they can easily be knocked over. Never leave bottles or jars open - even during short procedures! Take care not to mix up the lids to different bottles and beakers - Sloppy behaviour leads to contamination and could result in dangerous chemical reactions.

13. Always follow instructions carefully and make sure you understand the procedures completely. If in doubt, ask before doing anything that could lead to hazards! Equipment should not be operated without permission and proper instruction from technical staff.
14. When working in the fume cupboards, ensure that the front cover is pulled down far enough to cover your face. If this is not possible, wear protective glasses and clothing. Close the front cover when leaving the fume cupboards, even if it is only for a short time.
15. Ensure that there is minimal risk for spills (and consequent electrical shocks) when working with electrical equipment and liquids.
16. Never leave gases flowing, uncovered electrical installations connected (even if switched off), or open burners burning unattended for any period of time!
17. If leaving items in the labs; make sure they are not in the way for others. Make sure the laboratory staff know the purpose of the setup and are aware of what is involved. Ensure the items are properly labelled with your name, course, tutor responsible, time left, time to be removed, equipment and substances involved (toxicity, acidity, alkalinity, radioactivity, any biohazard) and procedures to follow in case of an emergency.
18. Never take laboratory inventory (tools, glassware, instruments, pH meters, etc.) outside the labs without permission. Never "borrow" things – no matter how small and insignificant – from the laboratories without permission.
19. Glassware, tools, and equipment are very expensive. Accidents do happen, please report any breakages, but be aware that items broken due to careless conduct may be charged to the individual responsible.
20. Never move equipment without permission. Some pieces of equipment (such as balances) are very sensitive and can be easily damaged during movement.
21. Keep work surfaces clean and dry. If you encounter liquid droplets or powders on the work surfaces, treat them with suspicion. Even though they may be harmless water or dust, they could easily be of toxic or acid substances.
22. Clean up after your work, and do not occupy bench space unnecessarily. If you need bench space, please arrange it well in advance with the laboratory staff.
23. Never leave anything that can obstruct the escape routes in case of an emergency.
24. Never joke with safety! False alarms are dangerous. Always take alarms seriously and act accordingly.
25. Never "play around" in the laboratories.
26. Report people to the technician or lecturer in charge if you think they create a hazard to themselves or others.
27. These rules apply to everybody working in the laboratory – including staff.
28. You must ensure that you keep informed of updates to these terms and conditions (updates will be on different coloured sheets).

These rules are made for your own and other peoples safety. Failure to comply with them may lead to hazards and may result in your temporary or permanent exclusion from the laboratories.

For the College of Engineering, Mathematics and Physical Sciences

21/11/2012 G.K. Rollinson, F. Wall

2. Basic principles of laboratory analysis

This section gives an introduction to laboratory analysis at Camborne School of Mines. The intention is to introduce a best practice for laboratory work, to ensure that users are aware of the general methodologies and procedures, and to develop an awareness of the importance of quality control for analytical data.

INORGANIC MATERIALS IN NATURE: ELEMENTS, MINERALS AND ROCKS

It is common to view nature in the light of chemistry. This is the background for environmental legislation, where soils, for example, are classified according to environmental guideline values for individual elements. Unfortunately this is a crude and unjustified simplification. Liquids can (with some justification) be considered as substances with dissolved atoms of the chemical elements. In solids, however, the chemical elements are not simply present in their atomic form. In most solid materials, minerals represent the physical form that the chemical elements take, and these minerals govern how the elements behave in the environment. In contrast to liquids, where elements can diffuse and react freely, minerals have the elements locked into crystal lattices in fixed proportions (according to size and a balance of electrical charges). Where a liquid is mostly homogeneous, a rock is typically made up of several different minerals that react differently to physical and chemical processes. A mineral can be separated from a rock; a chemical element can only be separated by the breakdown of its host mineral.

SAMPLE COLLECTION

Your analyses are never better than your samples! Poor samples give useless analyses and are a waste of time and money. It is generally considered that more than 50% of analytical errors are introduced at the sampling stage – either because samples are of poor quality, too few, or too small.

A sample is a selected part of a larger bulk extracted for a particular purpose. There are essentially two types of samples:

1. **Representative samples** are supposed to be representative of a larger, homogeneous bulk. The minimum number and size of a sample depends on the homogeneity of the bulk (e.g., grain size of a rock) as well as the abundance and distribution of the constituents being investigated.
2. **Targeted samples** are typically aimed at exploring specific variations within an inhomogeneous bulk. The sample size is restricted only by the amount required for analysis.

As an integral part of a study, it is necessary to build a sampling strategy that ties in with the specific aims of a project and is targeted towards a particular type of analysis. This strategy must address issues such as sample size, number of samples, the expected variability of the study area, and the specific question that is being investigated. It is also important to know how these aims are affected by other processes that may have influenced the samples (weathering, alteration, mineralized vein systems, metamorphism, etc.).

SAMPLE SELECTION FOR PREPARATION AND ANALYSIS

As a general rule, it takes much longer to carry out laboratory work than expected. It also takes a lot longer to interpret and analyse the results. We can all suffer from a lack of data before a deadline because of conflicting laboratory work, instrument downtime or simple poor time management.

The following guidelines should be used when submitting large numbers of samples:

1. Discuss with laboratory staff at the earliest possibility the scope of your study, how many analyses you require, and the timeframe involved.
2. Select samples so that they at all times cover the entire region of interest. If you select samples from one end of an area and continue systematically through to the other, you risk having an incomplete project by your deadline. Instead, start by picking every fourth or second sample for analysis, and then progressively fill in the gaps until you have a complete dataset.

SAMPLE PREPARATION

Once a sample has been collected, it will need to be reduced to a format suitable for analysis. This typically involves a reduction in size, powdering and potentially dissolution or melting. A representative sample must remain representative through all of the processes involved in the preparation, and extreme care must be taken to ensure this. There are many things that can go wrong. Sample preparation is boring, time consuming and dirty, and you can't expect anybody else to be as meticulous and careful as yourself. Common sources of error include contamination, sample degradation, and improper sample division.

Contamination

The incorporation of foreign material can significantly affect the composition of a sample. Contamination can occur both by the incorporation of unwanted material during sample collection, improper handling, or the use of improper equipment during sample storage or preparation.

Common sources of contamination include:

1. Failure to remove inhomogeneities (weathering crust, mineral veins)
2. Sampling while wearing rings, bracelets or other jewellery.
3. Use of improper sampling equipment or containers (e.g., Coke or milk bottles for water samples)
4. Improper cleaning of crushers and grinding pots
5. Improper tracking of samples through preparation
6. Exposure to dust

To minimize contamination:

1. Remove all impurities before crushing and grinding of samples (weathering crust, veins, fractures, inclusions etc.)
2. Keep sample bags or containers open for a minimum of time.
3. Keep only a minimum number of bags or containers open at any time.
4. Keep work surfaces clean. Cover surfaces with cling film when working.
5. Clean all equipment carefully before use and between each of your samples.

6. If using the mills, discuss with the laboratory staff which barrel is best suited for your samples. Clean the barrel with quartz sand and prime the barrel by grinding a small amount of sample (to be discarded) before grinding each sample.
7. If leaving samples uncovered to dry, leave them uncovered for a minimum time only. Ensure that they are kept in a clean area, isolated and not interfered with. Most samples can dry in closed paper bags.
8. Follow work manuals and instructions from lab staff rigorously.
9. If in doubt, ask staff and opt for the cleanest option.

Sample degradation

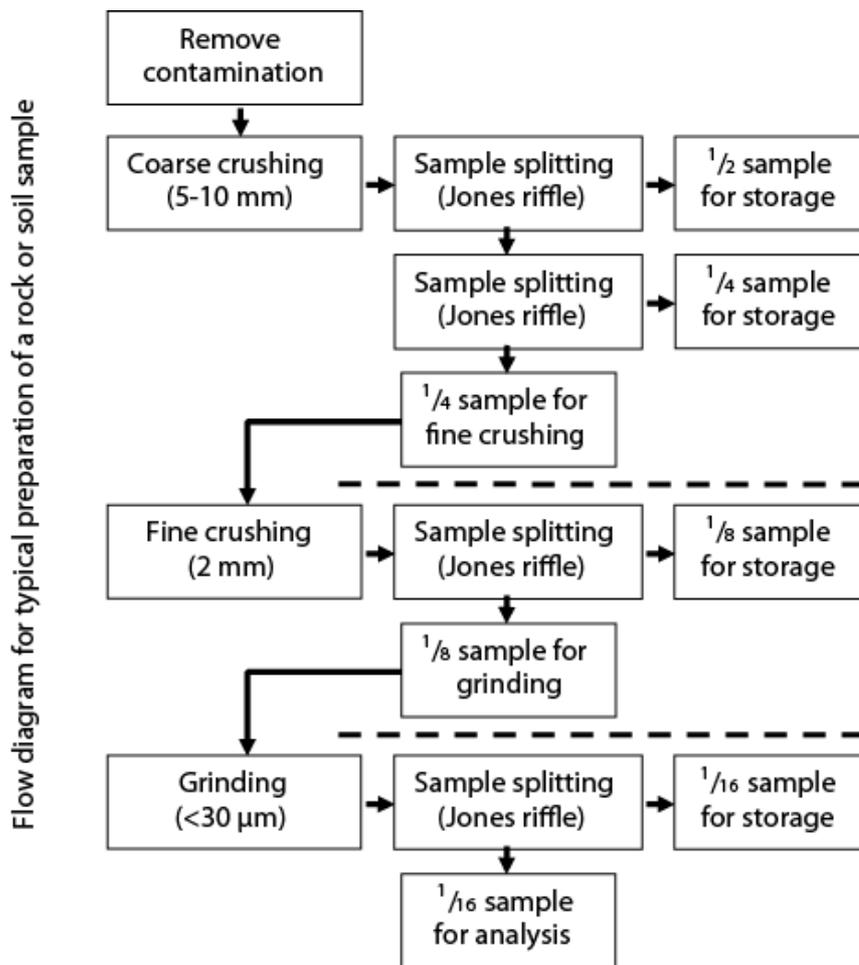
Certain materials may change in composition over time. Powdered samples tend to segregate mineral grains according to their different specific gravities. Sulphide grains oxidize by exposure to air, salts may recrystallise, and moisture may be gained or lost. Liquid samples may lose dissolved gases, precipitate minerals, or alter due to biological activity. Dissolved elements may become adsorbed into the container material.

Common ways to minimize sample degradation include:

1. Filtrate, acidify, and potentially freeze natural liquids as early as possible after collection.
2. Store sample powders in sealed plastic or glass containers.
3. Analyse samples within a strict time limit.

Sample division

Solids: The collection of part of a particulate sample is not a trivial task, as material can fractionate within a stored powder and through the process of sub sampling. For example, if a powder is poured from a container, individual particles will fall differently according to differences in their size and specific gravity. An appropriate method of sample division should be integrated with the crushing and grinding procedures. Proper sample division should be carried out with a series of Jones riffles appropriate for the particle size of the sample. The sample reduction in weight and particle size must be done during the same process. If large samples are prepared, sample splitting can be repeated after both the coarse and fine crushing.



Liquids: When sub sampling liquids; it is important to check for precipitation or other sample degradation which could affect the composition of a sample. Unaltered liquids can usually be adequately homogenized (by swirling of the container) before collection of a subsample.

QUALITATIVE AND QUANTITATIVE ANALYSIS

Qualitative analysis is a confirmation of the presence or absence of a target substance at concentrations above a detection limit. Quantitative analysis additionally provides information on how much of the target substance is present. Semi-quantitative analysis gives a rough indication of how much is available, but with some inaccuracy.

Quantitative analytical methods include the measurement of a signal that is correlated in size with a certain amount of the target substance. This signal is typically superimposed on a background (which includes noise amongst other effects). A reliable method must be capable of accurately quantifying the magnitude of both, as well as any signals that may interfere with these measurements. It also involves a correct method of translating the signal into an accurate measure of quantity. For modern analytical methods, all of this is commonly carried out by computers

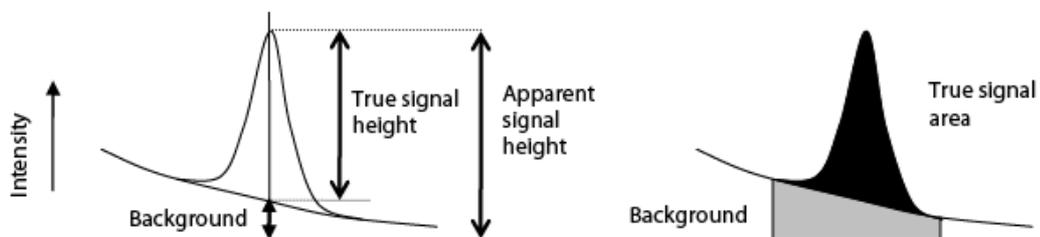
Measurement of a signal

There are two major ways to measure a signal - either to record the maximum intensity of the signal or the integrated area of the signal. The preferred method depends on the analytical technique. Measuring the maximum intensity has an advantage for spectra that do not change over time (such as x-rays) as more time can be dedicated to the maximum signal, whereas measuring the area of a signal is usually better for spectra that are variable over time (such as in liquid chromatography) as more of the target substance will be measured.

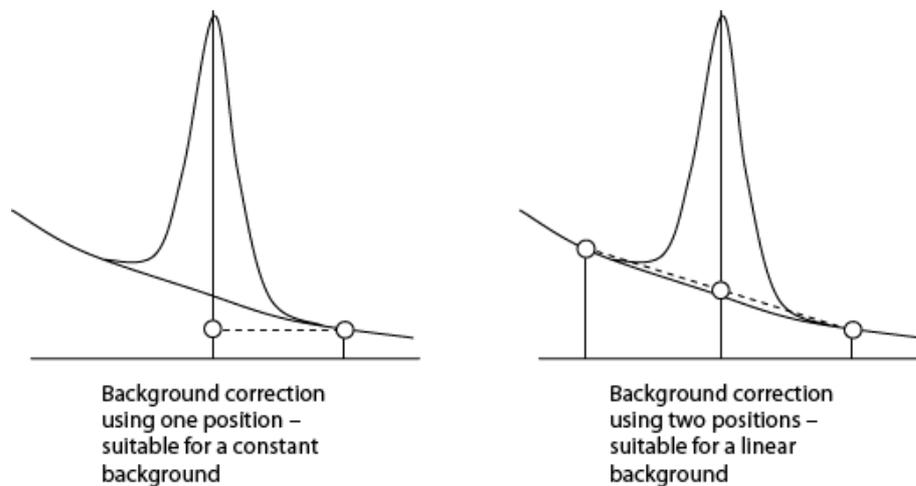


Background subtraction

The background is measured and subtracted from the measured signal to give the true signal. However, as the true background is hidden beneath the signal, this requires a judgment of the most appropriate method. The common practice is to either measure a "blank" (a sample with no signal) or an approximation of the background from measurements outside the region of the signal. The preferred method will be based on a judgment of the factors that influence a specific type of analysis. Blanks are usually used for mass spectrometry but not for x-ray analysis.

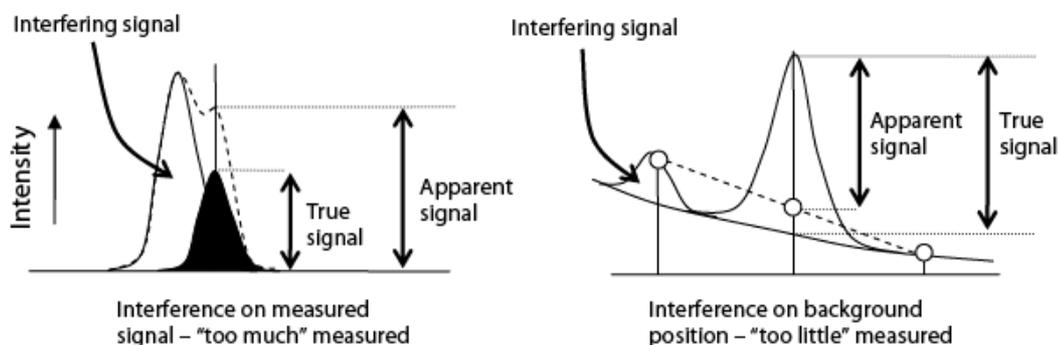


The background subtraction is particularly important for small signals (i.e., analyses near the detection limit). Common ways to correct for background are by the use of one or two background positions. For most analyses, this is adequate. However, for curved background profiles, the background should ideally be modelled by measurement of three or more positions.



Interferences

When analyzing complex materials, a common problem is to have interfering signals from different substances than the actual target. Interferences can lead to misidentification or incorrect quantification of the target substance. It is important to carefully assess any potential interference before a result is interpreted.



Interferences can severely limit the usefulness of analytical measurements, and it is commonly necessary to consider and test for the effect of potential interferences before analysis.

Common ways to reduce interferences:

1. If the target substance produces more than one signal, find a signal without interferences.
2. Interferences in liquids can commonly be reduced by chemical preparation
3. Optimise the signal resolution.
4. Small, well characterized interferences can in some instances be resolved numerically.

Calibration

Most analytical methods require calibration to ensure that the instruments work in a similar way at different times. The calibration commonly includes a check and adjustment of signals to their theoretical positions, but also commonly tests to ensure the instrument is working correctly. There may be systematic variations in signals before and after an instrument calibration.

Quantification of signal

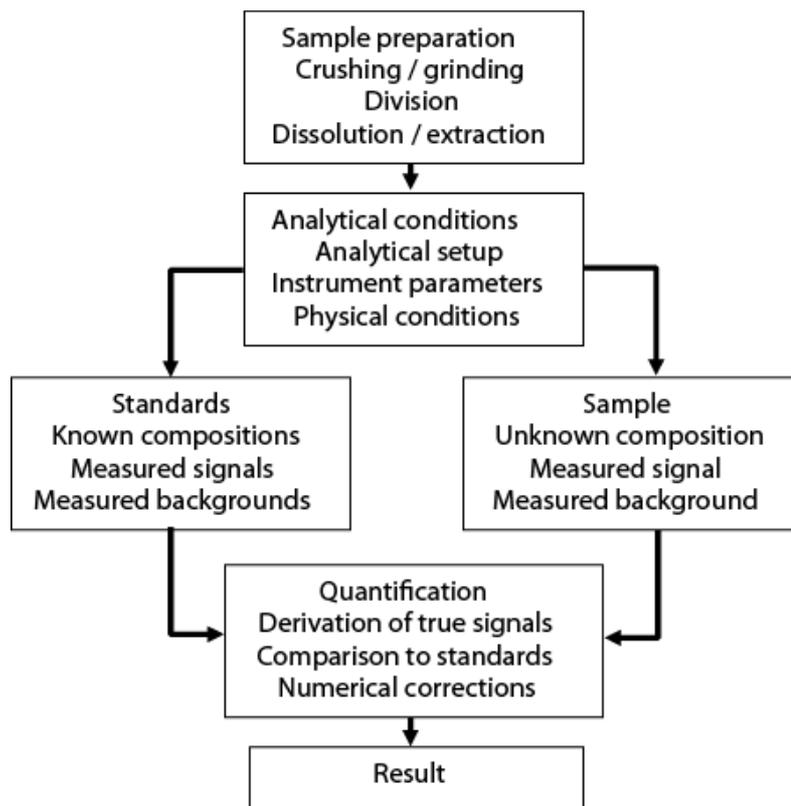
The conversion of the true signal into a quantity is commonly done with reference to a set of universally accepted certified reference materials. The measurement of standards ensures that the results obtained are independent of the instrument used. The reference materials must be homogeneous, well characterised, and not degrade over time.

Depending on the type of analysis, it may also be important that the standards match some of the characteristics of the samples to be measured. For x-ray techniques, for example, the background spectrum is strongly influenced by the bulk composition, so it is best to have approximately matching compositions of standards and samples. For electron beam techniques, it is additionally an advantage to match the electrical and thermal conductivities.

To minimise the variables during analysis, it is essential, as far as possible, to use matching preparation and analytical conditions for the standards and samples.

The following issues should be considered:

1. During sample preparation, it is important to know if all of the target substance is being made available for measurement. Any loss due to incomplete dissolution, incomplete oxidation or other relevant effect will result in errors.
2. During analysis, it is important to know that the sample measurement is comparable to that of the standards. Differences in instrument setup (e.g., electron beam current on the electron microprobe, flow rate on the liquid chromatograph) can cause systematic differences in the results achieved. The safest method is to run samples and standards subsequently using the exact same setup.



QUALITY CONTROL MEASURES

Quality control is essential to ensure reliable analysis. The level at which data (and data variations) can be confidently interpreted informs and limits the geological conclusions that can be drawn. When, for example, the environmental guideline value for arsenic in soil is 20 ppm, there is an enormous difference between a reported analysis with 15 ± 2 ppm and 15 ± 10 ppm.

Duplicate samples

The analysis of duplicate samples is the accepted way to test the repeatability of analysis. As a general rule, 10% of samples should be prepared and analysed in duplicate. Duplicate samples must be anonymised and independently go through the exactly same and complete sample preparation procedures. It is not enough to simply analyse the same sample twice.

Blanks

If you can't measure anything, try to measure nothing. The analysis of blanks is a good way to test for interferences and the appropriateness of the chosen background correction method. It also serves as a good test of the detection limit. Matrix matching is important if interferences and backgrounds are dependant on the matrix composition or structure. As for duplicate analysis, blanks should be taken through the exactly same and complete sample preparation procedures as other samples.

Standards and reference materials

Most methods are calibrated to internal standards and certified reference materials that are used directly in the quantification process. However, it is common also to monitor analyses by the inclusion of standards in an analytical run (“secondary” standards). The use of secondary standards serves as a check on the accuracy and repeatability of analysis as well as comparability of data between different laboratories. This is an important link to establish when submitting samples to external laboratories as well as when comparing data collected to the literature or geochemical databases. A systematic deviation from the published value is a sign of poor quality control. For many analytical techniques, it is recommended to include one standard per ten or twenty analyses.

Two types of standards are common:

1. **Certified reference materials** – materials that have been tested and certified by a large number of international laboratories. These standards are expensive but essential to justify that results are comparable to results obtained in other laboratories. It is important that certified reference materials are reported along with any results
2. **In-house standards** – materials developed and used internally within a laboratory to test for reproducibility of results. These standards are cheaper to use than certified reference materials. However, it is important that they are homogeneous, do not degrade over time, and are well documented and calibrated to certified reference materials. In-house standards can be used to ensure comparability within a dataset but not to validate results externally.

Recovery

If the quantification involves dissolution or extraction of a substance from a matrix, it is appropriate to test how much of the material has been recovered. Any loss (or indeed gain) of material through the processes could result in systematic errors. Tests of recovery involve the use of an independent monitor that itself is not affected by the processes tested.

Other quality control measures

Laboratories commonly include other quality measures that individual users may not get involved in. This can include the analysis of spiked samples, comparison of analyses by different operators, and the participation in inter-laboratory test schemes (such as GeoPT and G-probe schemes). Laboratory accreditation schemes (such as UKAS and NAMAS) serve to enhance quality control, and are commonly required for commercial work. However, research laboratories rarely find the time and funding required for going through accreditation.

UNCERTAINTY

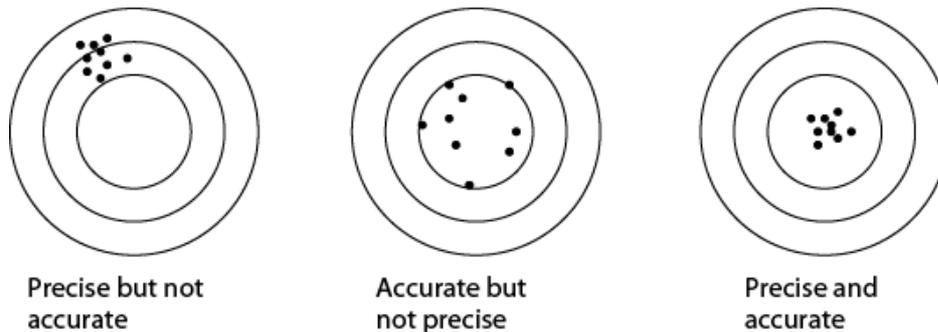
Uncertainty is a measure of how reliable the data are. The certainty to which a substance can be quantified depends on all aspects of sampling, sample preparation and analysis. Dealing with uncertainty is an integral part of analytical work – and one of the major issues to be addressed by quality control. It governs the extent to which data can be interpreted, and ultimately the conclusions that can be drawn from the data.

Precision and accuracy

Many factors can contribute to uncertainty. The most common factors include:

1. Samples may not be truly representative of the bulk
2. Samples may contain unidentified inhomogeneities
3. Samples have fractionated during handling and storage
4. Instruments may be calibrated to standards that do not provide an exact match to the physical makeup of the samples
5. Backgrounds may not be exactly matched

Measurements of signals and background are subject to noise and statistical uncertainty. Errors are commonly considered to include two parts: Precision and accuracy. A poor precision leads to systematic errors (analytical bias), whereas a poor accuracy yields unsystematic errors. A quality laboratory strives to improve both the precision and accuracy. However, a poor laboratory will be more likely to try to hide problems than reveal them to you. As a user, it is consequently your responsibility to test the precision and accuracy. An absence of data for duplicates, standards, and other quality control measures ultimately means that you can't prove your data to be valid.

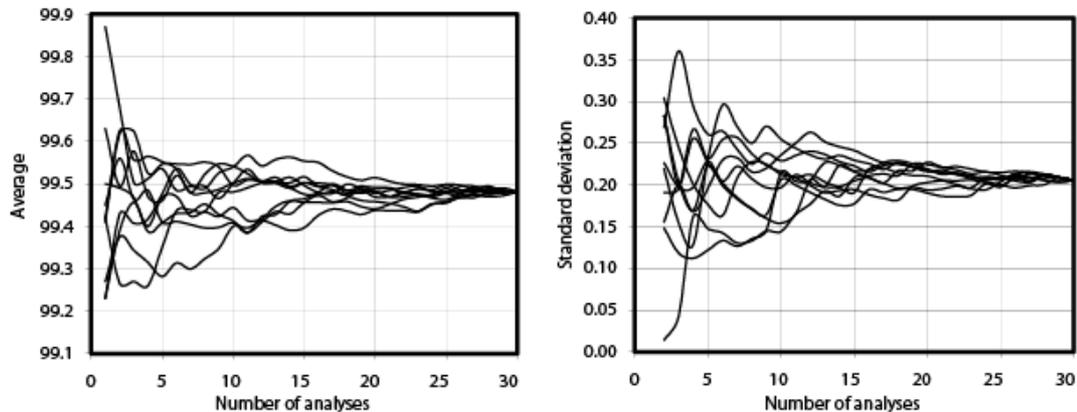


Detection limit and limit of reporting

The detection limit is the lowest limit at which a signal can be detected with any certainty. This is usually taken to be 3 x the background (although for some techniques, it is acceptable to use 3 x the standard deviation on the background). At the detection limit, however, the uncertainty will typically be 100% and results are consequently only qualitative. The limit of reporting for quantitative analyses is defined at 10 x the background (or 3 x the detection limit).

Average and standard deviation

A common way of reporting data is to present an average and standard deviation. This practice most commonly assumes a normal distribution of data. Unfortunately the values are often calculated without any regard to any natural variations within a dataset or an assessment of the relevant statistical distribution. The analytical uncertainty is usually best evaluated from a compilation of standard analyses. The uncertainty can usually be assumed to follow a normal distribution, apart from near the detection limit, where the distribution is log-normal. An important question is how many analyses are required to give a meaningful average and standard deviation?



The above graphs display the changes in the successive averages and standard deviations for the same 30 analyses (analyses added in random order). Note that both the average and standard deviation are highly unreliable for less than 15-20 analyses.

Reporting of data

Data should be reported along with documentation of the analytical method, setup and procedures (the analytical conditions), analyses of certified reference materials and other quality control results. This ensures that data can be verified and compared to data obtained in other laboratories. It is important to report the correct number of significant digits that can be justified from the uncertainty of the methods used (0.1 wt% is not the same as 0.100 wt%). Uncertainties should be quoted as 2 x standard deviation (which is approximately similar to a 95% level of confidence).

Acknowledgment of the laboratory

It is important to give proper acknowledgment to the laboratory that has been used. The most obvious reason is to credit the laboratory that has worked for you, but it is also a way of sharing responsibility for the data. Where analyses have been produced for free or at less than full commercial rates, payment is commonly expected by other means – the most common being services offered in return or co-authorship on the publication of data.

DATA QUALITY AND USE

Quality control

Cheap data can be expensive! You can't base a million pound project or a quality geological study on data produced without the appropriate quality control. You can spend a fortune on analyses, but you can't rely on data unless you personally ensure their quality. Ultimately, if your data are inaccurate (or do not support your conclusions), you could face astronomical insurance claims or a tarnished academic reputation.

Use of other data

It is often necessary to use data collected by different researchers or companies, either for a direct comparison of variations, or to explore their data for specific variations. Obviously using other people's data is based on a level of trust in their analytical procedures.

When using other people's data, the most important question is to check that the data are comparable. Without careful testing, systematic deviations can go undiscovered or, in a worst case scenario, generate apparent variations that do not actually exist. Data can be checked when they are published with adequate quality control (certified reference materials, duplicate analyses, etc.). Data that are not provided with such support will need to be carefully screened to ensure that variations are genuine and comparable.

ACKNOWLEDGMENT OF RECEIPT OF INDUCTION

Name _____

Address _____

Telephone _____

Please tick

- I have received induction in health and safety for work in the laboratories
- I have read and understood the "Code of Conduct in the CSM research laboratories"
I will carry out the necessary risk assessments for my work
- I accept that documents provided in aid of laboratory work are confidential and must not be removed from the laboratories without the written consent of the laboratory manager.
- I will not deviate from the laboratory procedures as instructed by laboratory staff and written manuals

Method specific induction undertaken (please tick):

Public relations

- Competence to show laboratories to visiting groups
- Competence to use laboratories for media relations (including photography, video, and interviews)

Preparation

- Crushing / grinding of rocks
- QEMSCAN preparation
- Use of carbon coater
- Preparation for x-ray diffraction
- Wet chemistry preparation (e.g. ICP-MS)

Operation of instruments

- Use of Scanning Electron Microscope
- Assisted use of Electron Microprobe
- Use of x-ray diffraction spectrometer
- QEMSCAN interactive
- Use of ICP-MS

Data interpretation

- Interpretation of x-ray diffraction spectra
- QEMSCAN offline data processing

Date

*Signature
Client*

Signature

Lab manager / Asst. Lab Manager