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Manaaki Whenua
Landcare Research

## South Pacific Agricultural Chemistry Laboratory Network (SPACNET)

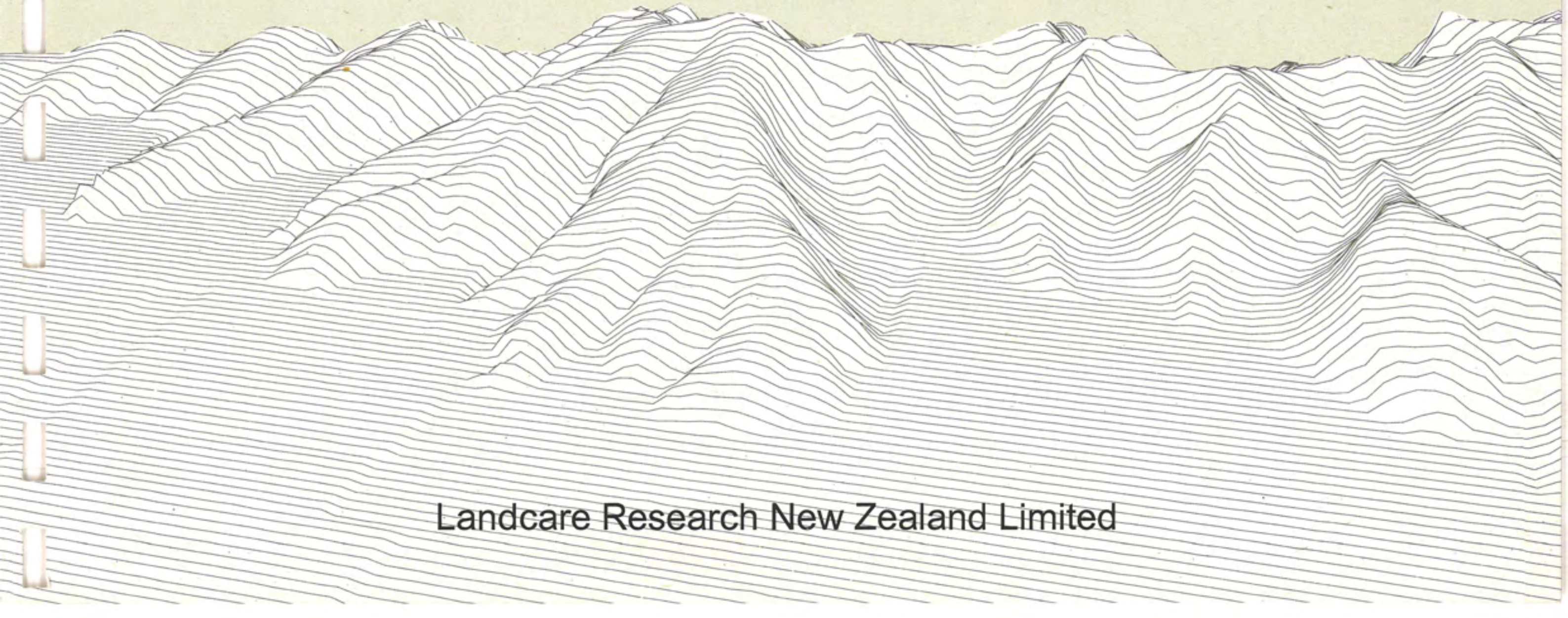
### Second Revised Edition

# Generic Quality Assurance Manual for Soil and Plant Laboratories

*(Needs to be customised for an individual laboratory)*

compiled by

Brian Daly & Linda Hill



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# Table of Contents

**PART I QUALITY MANUAL**

**PART II STANDARD OPERATING PROCEDURES**

# PREFACE

The first version of this manual was produced as part of the SPACNET (South Pacific Agricultural Chemistry laboratory Network) programme to improve quality assurance in member laboratories. This updated version was compiled to bring the manual in line with the new requirements of the international standard ISO/IEC 17025:1999 "General requirements for the competence of testing and calibration laboratories."

It is intended to be a resource for small Pacific Island laboratories that do not have the time or resources to write their own manual from the beginning. It is expected that considerable modification and additions will be made to this template manual to make it specific for each laboratory, for example with the equipment operating procedures. The manual is comprised of a Quality Manual and a section of Standard Operating Procedures, which complement existing SPACNET Methods Manuals and Safety Manual.

In-house manuals from Landcare Research New Zealand (B K Daly and L F Hill), Fiji Agricultural Chemistry Laboratory (B K Daly & P T Hine), and PNG's National Agriculture Research Institute - National Agricultural Chemistry Laboratory (F Grieshaber & P S Corbett) were used to compile this template manual. Also used was the comprehensive work compiled by L P van Reeuwijk, 'Guidelines for quality management in soil and plant laboratories', FAO Soils Bulletin 74, 1998: 144p. The version of ISO/IEC 17025:1999 used was from the booklet 'General criteria for accreditation' International Accreditation New Zealand, 2000: 26p

# INTRODUCTION

The principal function of an analytical laboratory is to produce high quality analytical data that are accurate, reliable and adequate for the intended purpose. The aim of a quality assurance programme is to ensure the achievement of this objective in a thorough and cost effective manner. This manual is intended to detail the mechanisms and tools by which the laboratory's quality assurance systems operate in relation to the:

- Organisation, laboratory and facilities
- Documentation system
- Staff
- Health and safety system
- Job and sample control system
- Analytical quality control programme
- Equipment operating procedures.

The quality assurance manual describes a major part of the laboratory operating system, but is not the entire documentation system. Method manuals, instrument-operating manuals, standard operating procedures, staff training records, correspondence with clients, analytical worksheets and reports are also part of the system.

Like all the other laboratory documentation the QA manual is a living document and must be continually subject to review and updating. This is done in a formal way as part of the annual internal audit and management review procedures.

# PART I

# QUALITY MANUAL

# 1 QUALITY POLICY

## 1.1 QUALITY POLICY STATEMENT

The XYZ laboratory operates a quality assurance system that undertakes to comply with the criteria set out in ISO/IEC 17025:1999 General requirements for the competence of testing and calibration laboratories. The laboratory management is committed to compliance with this standard, to good professional practice and to maintaining the quality of its service to meet the needs of clients. This Quality Manual sets out the policies and procedures to meet these ends.

The laboratory defines its standard of service by aiming to:

- Complete 95% of all work within the time schedule agreed upon with the client
- Keep level of complaints to below 2% of report numbers.

In addition, the laboratory is committed to achieving a high standard of analytical work. This commitment is attained by:

- An active internal analytical quality control programme that covers all the testing carried out in the laboratory
- Participation in external sample exchanges and proficiency programmes
- Development of staff, aimed at improving their technical abilities and professional knowledge
- Regular review and improvement of the overall quality assurance system.

Laboratory management and personnel are free from any undue internal and external commercial, financial or other pressures and influences that may adversely affect the quality of their work. The laboratory actively avoids involvement in any activities that would diminish confidence in its competence, impartiality, judgement or operational integrity. The avoidance of conflicts of interest is taken seriously.

It is the responsibility of all laboratory staff to familiarise themselves with the content of the Quality Manual and to comply with its policies and procedures at all times.

This Quality Policy Statement is made on the authority of

Name
*Permanent Secretary or equivalent position*
Ministry/Company etc.

# 2 QUALITY SYSTEM

## 2.1 QUALITY SYSTEM

The Quality Policy Statement drives the quality system for the XYZ Laboratory. All laboratory functions are brought together in this Quality Manual, which complies with ISO/IEC 17025: 1999.

This manual covers the activities of the XYZ Laboratory as defined by the site plan in Figure 1 section 3 of this manual, and at associated field/temporary/mobile facilities.

## 2.2 QUALITY MANAGEMENT

The Laboratory Manager is the Authorised Representative for the XYZ Laboratory. The Deputy Laboratory Manager deputises for the Laboratory Manager on all matters, including safety and quality.

The Laboratory Manager acts as the Quality Manager and is responsible for ensuring that the day-to-day operation of the Laboratory complies with ISO/IEC 17025. He will take the following actions:

- Initiate action to prevent the occurrence of quality problems in testing work
- Identify and record any quality problems in testing work
- Initiate, recommend or provide solutions for quality problems in testing work
- Verify the implementation of solutions
- Control further testing work until deficiencies have been rectified
- Maintain a bring-up system to monitor periodic quality requirements.

Departure from documented policies is permitted only at the discretion of the Laboratory Manager, and must be clearly documented and endorsed by him. Where documented policies and procedures have not been followed, the variation from specified procedures and policies will be documented and brought to the attention of the Laboratory Manager, who will decide the course of action to be taken.

## 2.3 DOCUMENT CONTROL

The following procedures serve to ensure all documentation in the Quality System is maintained:
The Laboratory Manager, in the role of Quality Manager, must approve documents that comprise the Quality System before issue. The document control folder in the Laboratory Manager's office contains a master list of documents with distribution and revision status. All Quality System documents must carry a unique identification in the form of standard headers and footers on every page. The header shall include the laboratory name, the manual in which the document belongs, and the document name. The footer shall contain the document version number, issue date and pagination in the form "page … of…"

- The Laboratory Manager must approve the decision that a controlled document is to become obsolete. This is recorded in the document control folder, together with the date and reason for obsolescence. The master list is then used to ensure the obsolete document is removed from all hard copy sources. A hard copy set of obsolete methods will be retained for knowledge preservation purposes, and will be marked "Historical." The electronic copy will be transferred to the appropriate Historical subdirectory.
- All documents in the Quality System shall be reviewed annually as part of the management review, and changes made where required. Changes may be made at any other time where necessary.
- All changes must be justified to and approved by the Laboratory Manager, who will then ensure the affected documents are changed. The document control master list is used to ensure all copies are updated.
- The Manual Amendment sheet in the front of each Manual will record the date of change, the unique identifier of the changed document, the new version number, and the reason for the change. A verbal explanation will also be given at the next laboratory users group meeting.
- Handwritten amendments to Quality System documentation may not be made.
- All changes must be justified to and approved by the Laboratory Manager. This control will ensure that amendments are only made once the proposed changes have been fully validated or justified.
- If a change is considered necessary, then the case must be presented to the Laboratory Manager. Write the proposed changes on a photocopy of the section in question and submit it to the Laboratory Manager. If the change is found to be justified then the Laboratory Manager will ensure that formal re-issue and notification will be done as soon as practical.
- Each copy of a manual will be updated once the Laboratory Manager has approved the amendment. The author of the amendment will be responsible for ensuring that all copies of the manual are updated, and that this process is recorded in the document control folder.
- Electronic versions of Quality System documents are Read-only. The Laboratory Manager and Deputy are the only personnel authorised to change this status.
- All documents and records that are part of the Quality System will be kept for a minimum of 7 years. This includes records of staff training, equipment calibration, management review, internal audits, equipment printouts, and test reports, etc.

## 2.4 QUALITY MANUAL

The Quality Manual identifies the general organisation, staff, responsibilities, range of facilities and equipment of the XYZ Laboratory. It defines all the procedures set up to ensure the quality policy objectives are met. The electronic version of the document is stored in Quality Assurance \ Manuals \ Quality.

The responsibility for the Quality Manual rests with the Laboratory Manager, and compilation and amendments to this manual will be authorised by him. The author of any amendment will be responsible for ensuring that all copies of the manual are updated, and that this process is recorded in the document control folder.

The Quality Manual will be reviewed annually as part of the Management Review (see section 16). Invalid and obsolete documents/sections will be promptly removed from the Manual, while others may be amended to reflect current practice.

## 2.5 METHOD MANUALS

Three methods manuals exist – the Soils Method Manual, the Plant Method Manual and Waters Method Manual. They consist of a binder containing individual methods stored in plastic envelopes. This provides protection when being used at the bench and also allows easy updating where necessary.

Notice of amendments to methods, and the reasons for them, is discussed at the weekly laboratory users meeting.

Each Method Manual will be reviewed annually as part of the Internal Audit (see section 15). This will evaluate whether the laboratory is following the method as defined. Corrective action will be taken on any deviations – this may take the form of staff training or amendment of the method.

## 2.6 EQUIPMENT & OPERATING PROCEDURES MANUAL

The equipment section of the Equipment and Laboratory Operating Procedures Manual contains the operating instructions and calibration/verification procedures for all laboratory instruments and equipment. The operating procedures section provides instruction on tasks ranging from changing gas cylinders to preparation of the Laboratory Control sample.

## 2.7 LOCATION OF DOCUMENTS IN THE QUALITY SYSTEM

The Document Control folder in the Laboratory Manager's office contains a master list of the Quality System documents with distribution, revision status and approval signatures. The following outlines manuals and files in the Quality System and their location:
Laboratory Manager's office:

- Approved Suppliers Register
- Client Feedback file
- Employment Contracts
- Equipment Calibration file
- Instruments file
- Interlaboratory Proficiency file
- Laboratory Audit file
- Laboratory Safety Audit file
- Laboratory Suppliers file
- Management Review file
- Method Validation file
- Plant Methods Manual
- Quality Manual
- Quality Problem file
- Soil Methods Manual
- Staff Training file.
- Water Methods Manual

Main Laboratory:

- Analytical standards file
- Chemical MSDS sheets
- Daily Temperature records
- Equipment and Laboratory Operations Manual
- Plant Methods Manual
- Quality Manual
- Safety Manual
- Individual procedures from the Equipment and Apparatus Operating Manual, usually beside the piece of equipment they relate to
- Soil Methods Manual.
- Water Methods Manual

# 3 ORGANISATION AND MANAGEMENT

## 3.1 ORGANISATION

The Environmental Chemistry Laboratory is part of the XXX department of YYY. Its focus is on ZZZ. There are approximately # staff working directly in the laboratory.

The laboratory offers a range of services to both ministry and external clients, including:

- Chemical analyses of soils and plants for extension officers and farmers for soil fertility and plant nutrition purposes.
- Chemical analyses of soils and plants for researchers for agronomic and other purposes.
- Chemical analyses of soils for soil classification / soil survey purposes.
- Chemical analysis of water for decisions on suitability for irrigation.
- Limited interpretation service for advice on the need for fertilisers *etc.*

The physical location is:

The mail and other contacts for the laboratory are:

Phone:
Fax:
Email:

## 3.2 STAFF

A staff organisation chart is given below in Figure. 1.

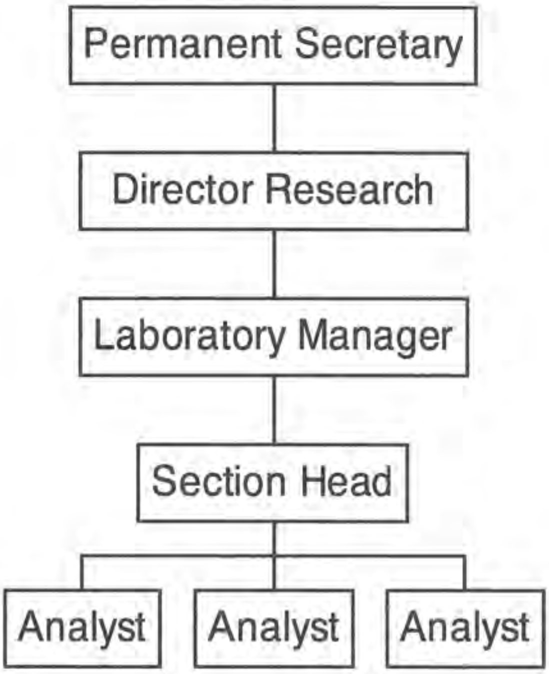

**Figure 1. XYZ laboratory staff organisation chart**

### 3.2.1 PERMANENT SECRETARY

The Permanent Secretary has overall responsibility for management and financial decisions covering the direction, safety and working environment of the Ministry. Decisions involving the hiring of staff and the purchase of equipment for the laboratory are made by or through the Permanent Secretary. The Permanent Secretary reports to the Minister.

### 3.2.2 DIRECTOR, RESEARCH

The Director, Research has overall responsibility for the safety and work of the XYZ Laboratory. The XYZ Laboratory Manager and staff report to the Director. The Director reports to the Permanent Secretary.

### 3.2.3 LABORATORY MANAGER

The Laboratory Manager is a principal technician who reports to the Director, Research. In his absence, the Deputy Laboratory Manager will make decisions. The Laboratory Manager has the overall authority for technical operations, and has the resources needed to ensure the smooth running of the laboratory.

The Laboratory Manager is responsible for the management and day-to-day running of the XYZ Laboratory. This involves the supervision and safety of staff, the safe operation and maintenance of laboratory instrumentation, and the supply of consumables. In the role of Quality Manager, he is responsible for ensuring compliance with ISO/IEC 17025, and implementing Good Laboratory Practice (GLP). This involves fulfilling of quality assurance requirements and GLP in line with current requirements for laboratory accreditation, maintaining a clean and tidy laboratory, conducting analysis to the highest scientific standards, and ensuring the integrity of data.

The Laboratory Manager:

- Provides leadership promoting a work culture that stresses the importance of safety, integrity, data quality and customer service
- Has limited financial responsibility for the purchasing of laboratory supplies and equipment (Refer to section 7 Purchasing Services & Supplies)
- Accepts work for the laboratory, issues jobs to individual analysts, and monitors overall workload
- Has the authority and resources needed to identify and initiate actions to prevent or minimise departures from the Quality System and testing procedures
- Ensures appropriate actions are taken as a result of quality control indicators
- Is responsible for all data produced by the laboratory
- Is responsible for performing a final overview of each job before it is reported
- Serves as the primary contact on analytical issues.

The Laboratory Manager is A.B. CDEFG. The deputy Laboratory Manager, who assumes the above responsibilities in the absence of the Laboratory Manager, is H.I. JKLMNOP.

### 3.2.4 ANALYSTS

Up to # analysts work in the XYZ Laboratory, involved in carrying out analyses to GLP standards and in recording and storing results and raw data. Analysts have a responsibility to:
- Follow appropriate analytical methods and standard procedures
- Ensure all appropriate QC activities are performed as described by the method
- Ensure the Laboratory Manager is notified when QC indicators do not meet the required criteria
- Ensure all analytical and QC activities are properly documented, and documentation is placed in the job file
- Show a commitment to compliance with ISO/IEC 17025.

Job descriptions for laboratory staff are kept in the Laboratory Manager's office.

## 3.3 APPROVED SIGNATORIES

Test reports will be signed by an approved signatory who will take responsibility for the content of that report. Approval is held for the following signatories and classes of test:

## 3.4 AUTHORISATION FOR STAFF

The work required of the laboratory is spread over a wide range of sample types and analyses. To maintain flexibility of staff placements, job descriptions define staff responsibilities so that specialisation is avoided. No specific personnel are required to be authorised to perform particular activities.

## 3.5 STAFF TRAINING

### 3.5.1 INITIAL TRAINING

New staff undergo a training schedule that includes the following:
- A briefing from the Laboratory Manager on the functions of the laboratory, its place in the overall organisation, the services it provides, the laboratory's structure staffing arrangements
- A tour of the laboratory and introduction to staff
- An opportunity to study the laboratory manuals and other laboratory documentation
- Instructions on the duties required, including a copy of the relevant job description
- Basic safety instructions and a description of safety policy and equipment available. The Safety Manual must be read and signed off
- Graduated training over a period of months in relevant analytical methods, completion of time sheets and worksheets, use of calculation spreadsheets, filing of results, etc. Appropriate supervision will be provided as required throughout the training period.

### 3.5.2 ONGOING TRAINING

A development/training programme is set up in consultation with each member of staff associated with the laboratory. This programme will include development courses, training in standard operating procedures for sampling and testing, and methods for equipment calibration. Appropriate members of the staff will give these. The Human Resources section maintains records of prior educational and professional qualifications. The Laboratory Manager holds records of subsequent training in the Staff Training File. These records include:

- Education and qualifications, other experience and starting date
- Training courses attended, including certificates awarded
- Proficiency in laboratory operating procedures.

Each year annual staff appraisals will be held. This is an opportunity to check progress on targets and to discuss and document the tasks and training to be set for the coming year. Training records will be reviewed at this time and new training needs identified. It should be a joint procedure involving both the Laboratory Manager and staff member to discuss and set training for the ensuing year.

Staff will be encouraged to develop specialized skills, for instance in flow injection analysis, laboratory management and GLP, word processing and spreadsheet use, by attending external courses and seminars where appropriate, and by contact with staff in similar organisations.

As new administrative or analytical procedures are introduced, the laboratory staff are instructed either individually or by groups in meetings, demonstrations or seminars held within the laboratory.

# 4 ENVIRONMENT

## 4.1 LAYOUT

The XYZ Laboratory is located in the Abc Building in the town of Qprst. A floor plan of the laboratory area is shown in Figure 2 below.

**Figure 2. Floor plan of the XYZ Laboratory**

The purpose of each of the rooms in is as follows:

e.g.

| Room | Activity / Equipment |
|---|---|
| Plant grinding | Plant drying oven, plant grinder |
| Soil grinding | Soil drying cabinets, soil grinders |
| Extractions | Centrifuge, shakers |
| Workshop | Tools |
| Digestion | Fume cupboard, furnace, digestion block, acid storage |
| Balance | Sample weighing, storage of "current" samples |
| Main lab | Analytical work area |
| Chemistry store | Chemical storage |
| Instruments | Laboratory instruments |
| Field gear store | Field gear storage – boots, augers, bags etc |
| Preparation room | Sample receipt and unpacking. |
| Store | "Completed' samples, laboratory archived records |
| Cold store | Walk-in cold store for sample storage |
| Laboratory manager's office | Laboratory manager's office |

## 4.2 ACCESS CONTROL

The building reception staff control access to the XYZ Laboratory. All visitors must be signed in at Reception and issued with a Visitor pass tag, and a laboratory staff member must take responsibility for their presence. This includes any external service personnel.

Access to the laboratory is restricted to essential personnel only. Visitors must be warned of any hazards relating to work in progress, and should be accompanied at all times. Casual visits to the laboratory area by other staff are discouraged.

The laboratory is kept locked outside normal working hours.

## 4.3 TEMPERATURE

Temperature is important in methods involving extractions, so room temperature should be checked both at the start and finish of an extraction. If this is outside of the range specified in the method the Laboratory Manager should be notified immediately and all extractions stopped until the problem has been rectified. Daily temperature record graphs are maintained to track fluctuations; these are stored in a file in the Main Laboratory. The Extraction Room and Instrument Room are air-conditioned.

## 4.4 VENTILATION

The plant and soil grinding rooms are equipped with ventilation systems that operate both to protect the health of personnel working in the rooms and to minimise the spread of dust to other work areas.

## 4.5 CROSS-CONTAMINATION PREVENTION

Grinding of soil and plant samples is restricted to their designated rooms. To prevent the spread of dust, the extraction systems must be used at all times when grinding samples. At the end of use, grinding rooms and equipment should be cleaned (vacuumed and wet-wiped) as indicated in the grinding procedures.

Working with soil or plant samples is restricted to the preparation rooms, the soil and plant grinding rooms and the balance room. Samples are never to be worked with in the Main Laboratory.

Chemicals are to be kept in the appropriate storage locations unless currently in use.

## 4.6 HOUSEKEEPING

Housekeeping must be of a high standard at all times, for reasons of safety as well as for quality of results. The Laboratory Safety Manual and procedures in the Equipment and Laboratory Operations Manual detail the housekeeping practices expected.

## 4.7 WASTE DISPOSAL

The XYZ Laboratory is committed to the protection and enhancement of the environment by the following actions:

- Hazardous substances shall be handled in a manner that minimises the possibility of adverse environmental effects.
- Discharges to the sewer shall comply with the conditions of any trade waste permit or regulatory bylaws for the site.
- Wastes will be minimised through reuse, recycling or reducing the quantity of waste.
- Wastes will be disposed of in a manner that does not adversely impact on the environment.

- Minimise air emissions wherever possible and obtain consents as required.
- Reduce energy costs and the impact of energy use on the environment through energy conservation, efficient energy use and fuel substitution.
- Minimise water wastage.

At all times however, conditions must ensure the quality of work is not compromised in any way.

# 5 EQUIPMENT

## 5.1 INVENTORY

The laboratory maintains an inventory of major equipment in the form of an electronic database. Each piece of equipment is identified with a unique number of the form XYZ###. Details such as description, supplier, contact details for servicing, purchase date, serial number, location, consumables required etc., are recorded.

## 5.2 COMMISSIONING OF NEW EQUIPMENT

Before beginning routine use, new equipment is fully evaluated by the laboratory staff in collaboration with the equipment supplier and/or instruction manual to ensure compliance with appropriate performance standards. Evaluation is based on:

- Past results and experience
- Desired future performance
- Manufacturers claims on performance.

Results are stored in the Instruments file in the Laboratory Manager's office.

## 5.3 LOGS

All instrumental equipment operated in the laboratory comes supplied with operating instructions. These manuals are the primary source of information about the operation, calibration and maintenance of equipment. Original copies of all instructions are stored in box files in the instrument room. However, because supplied manuals are usually too detailed and complicated for everyday use, abbreviated operating instructions have been written up as procedures in the Equipment & Laboratory Operations Manual. The manual is stored in the main laboratory.

In addition, beside each instrument or piece of equipment a folder is kept that contains:

- A copy of the individual operating procedure
- Maintenance schedule and instructions
- Operating and maintenance logs.

## 5.4 AUTHORISATION

Once they have been fully trained, all laboratory staff are authorised to operate equipment. Some aspects of use, such as access to calibration or maintenance areas, are restricted by password protection.

## 5.5 CALIBRATION

Most instruments are calibrated at the beginning of an analytical run following instruction given either in the method manual or in the relevant equipment-operating procedure. Procedures for use of other apparatus or items requiring calibration or verification, such as ovens and glassware, are contained in the Equipment & Laboratory Operations Manual. Some of the analytical instruments have built-in calibrations that are drift-corrected before/after/during each use by the operator. These calibrations are password protected so they cannot be accessed or altered other than by authorised staff.

Wherever practical, all equipment requiring calibration is labelled with a small sticker indicating the date when recalibration is due. This includes pipettors and dispensers.

Ongoing checks on the operational efficiency of equipment are obtained through testing of Laboratory Check Samples (LCS) as described in section 12.4. Unscheduled checks are conducted if the performance of the instrument is suspect.

## 5.6 MAINTENANCE

Proper maintenance of laboratory equipment is a key ingredient to longetivity, reliability and performance of this equipment. All equipment users are required to conduct a pre-use check of equipment as described in the relevant operating manuals.

A wall-planner showing the due dates for maintenance and calibration checks is located on the wall of the Instrument Room. Maintenance procedures for major equipment items are described in the equipment operating procedures. Any maintenance carried out is recorded in the log section of the individual operating manual for that piece of equipment.

A conservative inventory of critical spare parts is maintained for high-use instrumentation. Other parts are ordered as and when required. Scheduled maintenance and trouble-shooting of equipment is carried out by laboratory staff who have had specialised training in that area either by the equipment service agents or by other trained laboratory staff.

Workshop staff carry out a bi-annual electrical safety check of all electrical equipment. Items that fail this check are tagged with a sticker and taken out of use until repaired.

## 5.7 SERVICE AND REPAIR

Equipment that has been subject to overloading or mishandling, gives suspect results or has been shown to be defective or outside specified limits, will be taken out of service. If possible, it will be isolated, clearly tagged as being out of service, and an explanation for this will be provided.

After repair, the equipment will be tested and recalibrated before the tag is removed and the equipment placed back in service. The effect of the defect or departure from specified limits on previous tests using that equipment will/should be examined, and the "Control of nonconforming work " procedure (see section 14) instituted.

Similarly, if an equipment item is lent outside the laboratory it must be tagged and not returned to service until it has been tested and recalibrated if necessary.

Laboratory staff who have had suitable training may carry out minor service jobs. This is normally done under instruction from the service agents. More complex jobs are done by the equipment's authorised service agents themselves.

Any repairs involving mains or high voltage components are to be performed only by personnel holding current electrical registration.

## 5.8 TRANSPORT AND STORAGE

Major equipment items, such as instruments, are not to be moved. If relocation is necessary, then the manufacturers instructions should be consulted to secure the instrument so that no damage is incurred. After a move, the equipment shall be tested to check performance, and recalibrated if necessary.

The portable balance should be used where a balance must be carried around. If any other balance has to be moved, the analyst responsible for balances should be consulted to ensure calibration checks are conducted before use both in the new location and on return.

If any equipment item is to be out of use for a significant length of time it should be prepared for storage according to the manufacturers instructions. Before use again, the proper flushing and/or conditioning steps need to be carried out before recalibration.

# 6 TRACEABILITY AND CALIBRATION

## 6.1 EQUIPMENT CALIBRATION PROGRAMME

The Laboratory Manager is responsible for the monitoring and implementation of the Equipment Calibration Programme. All items of testing equipment will be calibrated before being put into service, and are further calibrated in accordance with the following programme:

| Item | Interval |
|---|---|
| Volumetric glassware | Before initial use |
| Automatic pipettors | 3-monthly |
| Dispensers | 3-monthly |
| Timers | 1-yearly |
| Working thermometers | 6-monthly |
| Reference thermometer - ice point<br>- full | 6-monthly<br>5-yearly |
| Balances - comparison<br>- repeatability<br>- full calibration | 1-monthly<br>6-monthly<br>3-yearly |
| Ovens - operating temp<br>- spatial variability | Before each new temperature<br>2-yearly |
| Muffle furnace – single position<br>- spatial variability | 6-monthly<br>2-yearly |

The methods for the testing of these items are contained in the Equipment & Laboratory Operations Manual. Results are stored in the Equipment Calibration Data folder in the Laboratory Manager's office.
Equipment Maintenance and Calibration File.

## 6.2 EXTERNAL CALIBRATIONS

Certain calibrations, namely the 5-yearly calibration of the reference thermometer and the 3-yearly full calibration of balances, are performed externally. These are carried out by laboratories accredited for these specific calibrations. Records of these subcontracted laboratories, including details of their accreditation status and competency, will be held as part of the Approved Suppliers Register.

## 6.3 TRACEABILITY OF CALIBRATIONS

The following national standards of measurement will be used for equipment calibration:

| | |
|---|---|
| Time | Standard radio time signals |
| Temperature | Certified thermometer |
| Mass | Certified masses |

Records for all equipment calibrations, including external and internal certificates, will be kept in the Equipment Calibration Data folder in the Laboratory Manager's office.

## 6.4 ANALYTICAL STANDARDS

In the Equipment & Laboratory Operations Manual, Procedure - Preparation of Standards describes the procedure for preparation, checking and storage of standards. A copy of this procedure is kept in the Analytical Standards file in the Main Lab, along with the log for recording standard preparation details and results from the checking process.

### 6.4.1 PRIMARY STANDARDS

Chemicals used for preparations of standards are of the highest grade available, such as Aristar, AnalaR or UniVar. Commercially prepared SpectrosoL standards will be used for metals analysis.

### 6.4.2 WORKING STANDARDS

Intermediate and working standards are prepared by dilution from primary standards in the appropriate matrix.

## 6.5 REFERENCE MATERIALS

The use of reference materials is described in section 12. The laboratory uses secondary reference materials that are in-house samples. These have been calibrated against certified reference materials where possible and have also been analysed by other laboratories as part of the sample exchange round robin.

# 7 PURCHASING SERVICES AND SUPPLIES

## 7.1 PURCHASING POLICY

The aim is to secure goods and services at the most economically favourable terms, while ensuring health and safety and environmental and quality requirements are being met by:

- Actively seeking to purchase goods and services at the best value for money
- Maintaining a good working relationship with suppliers, continually improve knowledge of products, and keep up with the market
- Providing full and fair opportunity for local suppliers, without compromising sound commercial practices
- Ensuring no adverse health or safety effects would arise from the use of the product or service.
- Assessing the environmental effects that could arise from the use of the product or service. Where an alternative with the potential for lower environmental impact is available, this is to be preferred
- Ensuring approvals for purchasing comply with financial delegations and purchases are made in the interest of the company and not the individual
- Having at least two independent quotes wherever possible
- Regularly reviewing sources of supply.

## 7.2 APPROVED SUPPLIERS

A register of approved suppliers of critical consumables, supplies and services that affect the quality of the laboratory's testing work is maintained in the form of an electronic database. The ongoing performance of suppliers is monitored as part of the Internal Audit process. The Approved Suppliers Register is kept in the Laboratory Manager's office.

## 7.3 PURCHASING DOCUMENTS

The Internal Requisition for Supplies/Services form is filled in, giving the quantity and full description of supply or service, and the required supplier. The description should include type, class, grade, specification, quality required or quality system standard.

## 7.4 PURCHASING

The completed Internal Requisition for Supplies/Services is given to the Laboratory Manager for initialling to indicate technical approval. It must then be passed to the Research Director for financial approval. Once approved, the Accounts section use the requisition to draw up an official company Purchase Order and submit this Order to the requested supplier. A copy of the Purchase Order is sent to the requisitioning officer, who should check it immediately for any errors.

## 7.5 RECEPTION

All inwards goods are delivered to Reception, who then advises either the laboratory staff member named on the packing documents or the Laboratory Purchasing Officer. If the goods are described as Dangerous then the laboratory staff must endeavour to collect them as soon as possible.

## 7.6 VERIFICATION

All purchased supplies that affect quality are segregated and not used until verified as complying with defined specifications. Records are kept of the actions taken to demonstrate this compliance.

The goods shall be checked as follows:
- Examine packaging for any signs of damage or leakage
- Open packaging
- Check contents for signs of damage
- Check off contents against packing slip
- Check off contents against original Purchase Order
- Initial and date beside goods received on invoice. Invoice to go to Accounts department
- For most consumables, write the date received on the container or packaging
- Conduct any activities necessary for verification of compliance with specifications.

## 7.7 STORAGE

When verification of compliance has been completed, the goods can then be stored in their appropriate location in the laboratory:
- Chemicals must be stored in the chemical pantry according to their hazard class
- Flammable liquids are stored in the flammables cabinet
- Alkalis are stored
- Acids are stored
- Consumables such as sample containers should be kept in their transport boxes to keep them free of dust
- Instrument spares and consumables should be stored in the cupboard unit near the instrument.

# 8 SERVICE TO CLIENTS

## 8.1 REVIEW OF REQUESTS AND CONTRACTS

Before undertaking any sample analysis, it is critical to understand the exact requirements of the client for analyses, timing, and cost. These requirements must be matched with the capabilities and limitations of the laboratory, and a clear agreement must be arranged between the client and laboratory. Unnecessary or overly expensive testing should not be undertaken, as it serves neither the interest of the client nor the laboratory.

The following should be discussed:

- Test methods to be used. Does the client understand exactly how each test is performed?
- Other work necessary as part of the test. All soils must be prepared before testing – dried, ground and moisture factor must be determined. This adds to the cost and the time taken.
- Will these tests meet the client's requirements? For example, are the detection limits low enough?
- Sample requirements. The sampling process should be discussed to ensure the sample is collected, preserved, stored and transported properly. Special requirements, such as quarantine permits for overseas samples.
- How much the requested work is likely to cost.
- How long the requested work will take to carry out.

All discussion points should be documented and filed in the job envelope. When samples are delivered without written instructions, a telephone call or email discussion should be conducted with the client. The responsibility for this negotiation process lies with the Laboratory Manager.

Before accepting new work, it is the responsibility of the Laboratory Manager to ensure the laboratory has the capability and resources (both equipment and personnel) to carry out the work requested and within the agreed timeframe.

## 8.2 CHANGES TO CONTRACTS

The client must be informed (and records made of this notification) if there are any changes made to what was originally agreed upon for the job. This includes any changes to tests used, or delays in carrying out the work. If a contract has to be amended after work has begun, the same review process set out above in 8.1 must be carried out.

## 8.3 SUBCONTRACTING

If a request is made for tests the laboratory does not perform, the client will be referred to other laboratories known to have this capability. In some circumstances, the laboratory may subcontract certain tests within a job to another laboratory. Similarly, if an unusually long delay occurs e.g. due to equipment breakdown, the laboratory will look at subcontracting affected tests. In all cases, the client will first be consulted for agreement about use of a subcontractor. Preference will be given to using an accredited laboratory wherever possible.

Tests performed by subcontractors will be clearly identified on the test report. The report will contain the endorsement "Tests marked "**" have been performed by a subcontracted laboratory which is/is not accredited", and the relevant tests will be marked accordingly in the body of the report.

The laboratory will maintain records, including details of the subcontractors' accreditation status and/or competency, of all subcontractors it uses for testing work. These records will be held in the Approved Suppliers Register.

## 8.4 CLIENT LIAISON

Clients value the maintenance of good communication, advice and guidance in technical matters, and opinions and interpretations based on results. Communication with the client, especially in large assignments, should be maintained throughout the work. The laboratory should inform the client of any delays or major deviations in the performance of tests.

At all times the client must be given due respect, which means that unnecessary delays or sloppiness in sample analysis or report preparation cannot be excused. Wherever possible, the laboratory will endeavour to allow clients to monitor the laboratory's performance. This may be in the form of visits to the lab for witnessing of tests. To maintain confidentiality for other clients, visiting clients shall be accompanied at all times by the Laboratory Manager.

Before the Management Review process, an annual survey of both internal and external clients will be carried out to ensure we are performing satisfactorily and to identify what could be done to improve both our service to our clients and our quality system.

## 8.5 POSITIVE FEEDBACK

This is usually in the form of cards or emails from satisfied clients, and is a sign that the Quality System is performing properly. Such material should be filed in the Client Feedback folder in the Laboratory Manager's office, as it is important for gauging the laboratory's performance in the Management Review.

## 8.6 CONFIDENTIALITY AND OWNERSHIP OF RESULTS

Staff must observe the confidentiality of information acquired by them during their work with the laboratory. They must not discuss such information with other persons or firms nor make use of it for their own benefit or the benefit of others during or after their employment with the laboratory.

## 8.7 TRANSMISSION OF RESULTS

All reports are sent out as hard copy. If requested, they may also be sent by fax or email. To maintain client confidentiality with such electronic transmission, the statements below are added.

The fax template header contains the following statement:

> *This information is **CONFIDENTIAL** and may **be LEGALLY PRIVELEGED**. Any unauthorised use of this document is strictly prohibited. If you are not the addressee please telephone us immediately (collect) and destroy this document.*

All emails contain the following footer:

> *WARNING: This email and any attachments may be confidential and/or privileged. They are intended for the addressee only and are not to be read, used, copied or disseminated by anyone receiving them in error. If you are not the intended recipient, please notify the sender by return email and delete this message and any attachments.*

# 9 HANDLING OF TEST ITEMS

## 9.1 SAMPLING

The laboratory does not carry out sampling for clients. However, information sheets on appropriate sampling methods may be given to any clients who are interested or request them.

**Or**

Sampling may be carried out for clients by laboratory staff. The procedure is set out in Procedure ### Sampling, in the Equipment & Laboratory Operations Manual.

## 9.2 SAMPLE RECEIPT

Reception will notify the Laboratory Manager of the arrival of samples. The Laboratory Manager will assign an analyst to collect the sample and transfer them to the Preparation room, where they are unpacked and laid out in order on the bench.

The following information will be recorded:
Date and time of sample receipt

- Analyst's initials.
- Carrier and consignment note number if known
- Sender's details from packaging if not included on documents
- Any discrepancies between sample identification on accompanying documents and on actual samples
- Condition of samples.

Normally all samples received are analysed regardless of their condition, and a footer on the report states, "Results apply to samples as received." If, however, any unusual features were noted on the documentation when the samples were received, the client should be contacted and advised prior to commencing work on those samples. The results may not be meaningful because of deterioration or contamination occurring between sampling and receipt at the laboratory. The client shall be given the option of whether or not the affected samples should be analysed. The client's decision should be recorded and filed in the job envelope. If a client does wish to go ahead with the analysis of such a sample, then the sample condition as recorded on receipt should be noted on the final report.

The documentation is handed to Laboratory Manager, who carries out the job registration procedure.

## 9.3 REGISTRATION

Each separate batch of samples (i.e. job) coming into the laboratory is given a unique job number, and each sample within a job is given a unique sample number. The recording of these numbers is done electronically in a relational database, with job number as the relational field. The advantages of a database system are speed of entry and the convenience of being able to search and sort data to rapidly produce information for annual reports and other uses. As the database is on the company computer network, a full tape back-up service is provided. A hardcopy printout of the job and sample registers is also maintained.

## 9.4 STORAGE AND PROTECTION

Storage requirements depend on the nature of the sample and tests to be performed, with details specified in the Method Manuals. Generally the following applies:

Field-moist soils and fresh plant material for chemical analysis:
- Transfer to soil air-drying cabinets or plant drying oven within 2 days.

Field-moist soils for biochemical analysis:
- Refrigerated at 4°C.
- Analyse as soon as possible.

Air-dry soils and dry plant material.
- Transfer to appropriate, labelled sample containers and store ready for analysis

Preserved water samples:
- Storage refrigerated at 4°C.

Unpreserved water samples:
- Analyse as soon as possible.
- If not possible, storage frozen at -18°C.

## 9.5 ANALYSIS

The Laboratory Manager assigns the entire job or certain analyses within the job to individual analysts. Samples are analysed in accordance with the client's request using the methods specified on the job worksheet.

## 9.6 DATA INPUT

Results are entered directly into the appropriate job spreadsheet. This is either by manual entry e.g., for pH results, or by importing the instrument result file e.g. for flow injection work. Any worksheets e.g., pH results, or instrument printouts are filed in the job envelope.

The results for the Laboratory Control Sample (LCS) are entered into the LCS spreadsheet, and compared with the statistics for each analyte. If any are out of range, the Laboratory Manager is advised and will make a decision on further action as described in Section 12 Analytical QC Programme.

## 9.7 SAMPLE DISPOSAL

Soil and plant samples remain in the store for a month after the job has been reported, in case any queries result in a need for re-analysis. After this period they are transferred to the long-term storage area. After 2 years samples will be disposed of. Samples that are under Quarantine restrictions are to be stored on the Quarantined shelf in the store. After the 2-year storage period they are disposed of by either by handing to the local MAF inspector, or by autoclaving.

Water samples are kept refrigerated for 3 months after the job has been reported, then disposed of. If the samples are in XYZ Laboratory sample bottles then the bottles washed up for re-use.

## 9.8 HANDLING OF QUARANTINE SAMPLES

Samples from outside the country are subject to quarantine requirements under the Biosecurity Act. The XYZ Laboratory holds a permit allowing the importation of samples for analysis. When arranging a job involving overseas samples, a copy of the import permit is sent to the client. This must accompany the samples during transportation to the laboratory. The samples will be inspected by MAF upon entry to the country and a Biosecurity Authority/Clearance Certificate issued.

Upon arrival at the laboratory, the certificate details are entered in the Quarantine Register. As part of registration a note is made on the Job Worksheet to advise the analyst that the samples have quarantine status. Quarantine samples are processed in the same manner as other samples except that all residues are retained and autoclaved before disposal.

## 9.9 HANDLING OF EFFLUENT-TREATED SAMPLES

Soils, particularly those treated with effluent, can contain potentially hazardous organisms. Again, as part of registration a note is made on the Job Worksheet to advise the analyst that the samples have a hazardous status. Gloves should be worn when handling the samples, and a dust mask as well when working with the dry material.

# 10 TEST METHODS

## 10.1 POLICY

Tests carried out in this laboratory will be validated test methods and procedures set out in the Method Manuals and will be the most up-to-date methods. Where appropriate, an estimation of the measurement uncertainty will be made. All analytical methods used in the laboratory are drawn from reliable published sources, and any modifications have undergone extensive validation processes.

The methods selected and used will meet the client's needs, and will be appropriate for the test. In particular, the relevance to the client's needs of the range and accuracy of the values (e.g., uncertainty of results, detection limit, selectivity, matrix interferences) obtained will be considered. The client will be informed of the method chosen if it is not the method specified, and the laboratory will also inform the client when the method proposed by the client is considered to be inappropriate or out of date.

Test Method Manuals will be reviewed annually to ensure that what is described is actually being done.

## 10.2 AVAILABILITY

Test Method Manuals are held in the Laboratory Manager's office, and a working copy is kept in the Main Laboratory. While full copies of methods will not be available to the client, a method outline is included with each test report and a full method summary, including references, is given on request.

## 10.3 METHOD DEVIATIONS

Modification of existing methods, e.g., by necessity based on lack of appropriate equipment (short-term changes only), can only be approved according to the following guidelines:

- Technical justification
- The carrying out of a documented investigation
- Authorisation by the Laboratory Manager
- Acceptance by the client.

## 10.4 METHOD DEVELOPMENT

At times it may be necessary to introduce new test methods, e.g., as a result of client demand for such a test. The introduction of such methods shall be a planned activity carried out by qualified personnel with adequate resources. Plans will be updated as development proceeds, and communicated to laboratory staff at the regular laboratory meetings. Staff will receive training in the how to perform the new methods, and such training will be recorded in their individual training records.

It is preferable that methods have been published either in international, regional or national standards, or by reputable organisations, or in relevant scientific texts or journals, or as specified by equipment manufacturers. Laboratory-developed or adapted methods shall be appropriately validated before use.

When the methods used are not covered by standard methods then the purpose of the test must be identified, the method validated before use, and client agreement must be obtained and must include specifications of client requirements.

## 10.5 INITIAL DEMONSTRATION OF PERFORMANCE (VALIDATION)

Validation is the confirmation by examination, and provision of objective evidence, that the particular requirements for a specific intended use are met. The full validation procedure is described in Procedure – New Method Validation, in the Equipment & Laboratory Operations Manual. The following parameters (based on USEPA recommendations) are determined:

- Linear calibration range (LCR)
- Laboratory control sample (LCS) or standard reference material (SRM) values
- The laboratory-fortified matrix (LFM or spike) is a sample aliquot to which a known quantity of analyte is added. Its purpose is to ascertain whether the sample matrix contributes bias to the results.
- The instrument detection limit (IDL) for an analyte is established using a sample extract with known low levels of the analyte in question, preferably at a concentration of two to three times the estimated detection limit. 20 replicate samples are taken through the instrumental part of the method and the IDL calculated statistically using student t-values.
- The method detection limit (MDL) differs from the IDL in that samples themselves are processed through the complete analytical method i.e. extraction/digestion as well as instrumental determination. It is more practical than the IDL as it includes the results from different analysts over different days.
- The initial recovery is determined by taking 20 replicate samples of a mid-range standard through the entire method.
- The initial precision is taken as the standard deviation of the 20 replicate aliquots of mid-range standard in the recovery determination above.

Other techniques used in validation include comparison of results achieved with other methods, and inter-laboratory comparisons. The validation procedure is fully documented and records include the procedure used, the results obtained, and a statement that the method is fit for its intended use.

## 10.6 TEST METHOD FORMAT

All analytical test methods should be written according to the format adopted in the Test Methods Manuals, and should include the following sections:

- Identification (title and method number)
- Introduction – explaining the relevance of the test, the scope, the type of material being tested and typical or comparative values
- Preparation of reagents
- Preparation of standards
- Apparatus – special equipment requirements, including technical performance requirements
- Procedure — a step-by-step description of the procedure, including
- Environmental conditions required
- Handling, transporting, storing and preparation of test items
- Checks to be made before work is started
- Checks that equipment is working properly, and calibration and adjustment of equipment before use
- The method of recording observations and results
- Any safety precautions.
- Calculations – full description of calculations and accuracy required for results
- The uncertainty and or detection limit.
- References – the source of the method or other information.

## 10.7 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

An assessment of the uncertainty of results for each test method is made using a combination of an understanding of the theoretical principles and the results from repeated analysis of the Laboratory Control Sample (where available.)

For trace analyses, the limits of detection are estimated by statistical means using the method detection limit (MDL) approach.

Uncertainties and detection limits are detailed in under each method in the Method Manuals.

## 10.8 CALCULATIONS AND DATA TRANSFERS

Spreadsheets are used to help interpret and calculate results from raw data. A separate spreadsheet page is used for each test, with the final results copied to the report on the front-page. Each test is identified by the tab at the bottom of the page and by a heading that gives the method name and method number (as in the Method Manual). There are also spaces to record the date and analyst's initials. Because the actions of spreadsheets and the formulae they use are mostly hidden, and are not immune to accidental corruption, a system is required to ensure that spreadsheets give the required and correct results in the first place, and continue to do so in the longer term.

Template spreadsheets are checked annually or if a problem is suspected. Procedure - Template Spreadsheet Validation, in the Equipment & Laboratory Operations Manual, describes how this is done using a standard set of manually calculated data.

The only cells in a spreadsheet where an analyst is required to input data are coloured. In addition, when a job spreadsheet is set up from the templates the formula cells will be protected to prevent inadvertent changes.

To avoid errors occurring during data collection and transfer, most laboratory equipment is connected to the computer network so data may be downloaded directly from the instrument to the appropriate spreadsheet. This avoids transcription errors where a value may be read incorrectly from an instrument, written down in a notebook or worksheet incorrectly or typed incorrectly into a spreadsheet.

Weights captured directly from the balances are transferred directly into the appropriate spreadsheet cells, while other instrumental results are pasted into columns to the right of the main spreadsheet. The relevant data is then copied and pasted directly into the appropriate spreadsheet cells

Results from instruments not networked are written onto printed worksheets at the time of measurement then manually typed into the spreadsheet.

# 11 TEST RECORDS

Technical records are accumulations of data and information that result from carrying out tests and calibrations. They may include forms, contracts, worksheets, notebooks, control graphs, test reports, calibration certificates and client notes. Quality records include reports from internal audits and management reviews as well as records of corrective and preventative actions.

## 11.1 INDEXING

Job numbers are of the form LJ######, where LJ stands for Lab Job, the first two digits refer to the current financial year and the remaining digits increment sequentially for each new job. For example, LJ01025 is the twenty-fifth lab job registered in the 2000/2001 financial year.

## 11.2 JOB FILE – HARDCOPY

The hardcopy job file is the primary job management and documentation tool. It consists of a foolscap envelope that contains all information relating to every individual job, such as:

- The job quotation
- Notes of phone calls discussing the job
- Any correspondence (including hard copies of e-mails) from or to the client
- Documentation accompanying the samples
- All primary records from analyses including worksheets and hard copy printouts from instruments
- Any observations
- Any derived data
- The analysis report
- A copy of the invoice request form.

The documentation should contain sufficient information such that together with the electronic file an audit trail may be established, that the test could be repeated using original conditions and that any factors affecting the uncertainty can be identified.

The hardcopy job file is stored in the filing cabinet in the Laboratory Manager's office.

## 11.3 JOB FILE – ELECTRONIC

The electronic component of each job is a directory containing the spreadsheet and result files from on-line instruments. The spreadsheet has the form of a pre-formatted report on the front page where the final calculated results are copied from worksheets on other pages.

**Or**

The electronic component of each job is maintained as part of the Laboratory Information Management System (LIMS)

Electronic files or the LIMS data are backed up nightly, weekly and monthly.

## 11.4 RECORDING

Observations, data and calculations are to be recorded at the time they are made, in a legible manner. They must be identifiable to the specific task. Any changes made to the original records (including electronic records) must be such that:

- The original record is not obscured. For hand written entries this means crossing out with a single line rather than erasing or using white-out correction fluid. For electronic entries, the original entry should be left where it was, and the correct value entered or pasted to a cell in a column further to the right, together with the formulae
- The alterations (both hand-written and electronic) must be initialed and dated by the person making the correction.

Personal workbooks are not used in the XYZ Laboratory. Where a test requires manual recording of data (pH and conductivity for example) then the appropriate worksheet can be printed from the job spreadsheet. The completed worksheet is filed in the job file hardcopy envelope.

**Or**

Where personal workbooks are used then a photocopy/carbon copy of each page containing data relating to that job must be filed in the job file hardcopy envelope.

## 11.5 CHECKING

All data entered into spreadsheets (both hand-written and electronic) must be checked for transcription errors. The main points are that:

- Every hand-written entry is checked from hardcopy against the electronic entry
- A red electronic script signature of the person checking is placed beside the analyst's initials when such checking is complete
- For electronically captured data, at least two entries per page on screen are checked, comparing the data copied to the input column against the raw data file out to the right of the worksheet
- A red script electronic signature is placed in the same row as the checked cell, out to the right of the worksheet.

## 11.6 ACCESS

The location of the hard copy job files in the Laboratory Manager's office serves to control access to these records. Access to the computer network or LIMS is restricted to authorised laboratory staff by password protection.

## 11.7 LONG-TERM STORAGE

At the end of each financial year, the hard copy job files are transferred from their filing cabinet into a file box, labelled for contents, and the year covered. The boxes are kept in the storeroom. These will be kept for as long as storage space permits, after the minimum legal period of seven years.

At the same time, all the electronic job spreadsheets are moved to one directory and kept forever.

**Or**

The LIMS data is archived and kept forever.

The electronic back-ups are stored in the fireproof safe.

## 11.8 QUALITY RECORDS

The raw data relating to the Analytical Quality Control Programme is by necessity stored as part of each individual job file. The QC results are entered by the individual analyst into the appropriate spreadsheet or LIMS file, which is in turn maintained by the Laboratory Manager.

The following documents are maintained in the Laboratory Managers office:

- External and internal audit records and responses
- Management review summary and responses
- Summaries of the Analytical Quality Control Programme
- Records relating to control of nonconforming work and associated corrective and preventative actions
- Client feedback file.
- Certification of thermometers and weights
- Verification of glassware records
- Initial demonstrations of capability and method validation studies.

# 12 ANALYTICAL QUALITY CONTROL

## 12.1 POLICY

The XYZ Laboratory is committed to achieving a high standard of analytical work. A major part of meeting this commitment is by conducting an internal analytical quality control programme. This programme consists of an initial demonstration of capability followed by regular analysis of a range of samples or solutions as a continuing check on performance. Records that define the data quality are kept so that trends are detectable, and statistical reviews may be carried out.

## 12.2 VALIDATION

The initial performance demonstration, also known as method validation, is used to characterise instrument and laboratory performance before routine introduction of the method.

## 12.3 ONGOING PERFORMANCE CHECKS

### 12.3.1 CALIBRATION

12.3.1.1 Perform calibration as described in the instrument-operating manual.

12.3.1.2 Where linear regression is used to fit a calibration line, check that the correlation coefficient is at least 0.995 and repeat the calibration if it is not. The instrument log contains records of the $r^2$ values for each analysis that should be used as a guide as to the expected value.

12.3.1.3 Check the residuals for each standard for a further check as to how well the line fits the data. Higher %RSD's can be tolerated for lower standards. Again, the instrument log records the mid-standard residuals for each analysis and should be used as a guide.

12.3.1.4 Compare the raw data for the standards e.g. peak area, against values previously logged.

### 12.3.2 LABORATORY REAGENT BLANK (LRB)

A blank consists of all the reagents that are in contact with the sample during the analytical procedure. It is used to determine the contribution of the reagents and preparative steps to error in the measurement.

12.3.2.1 Include two reagent blanks with every batch of samples.

12.3.2.2 Analyse the blanks immediately after calibration.

12.3.2.3 Record the blank readings in the QC spreadsheet. This data is useful in tracking reagent quality and analyst technique.

### 12.3.3 LABORATORY CONTROL SAMPLE (LCS)

The frequency of LCS inclusion is usually specified in each method; however as a general rule include one LCS sample for every 40 samples. Appendix describes the preparation of soil and plant control samples.

12.3.3.1 Run the LCS sample after the blanks immediately following calibration. If the measurements exceed the upper or lower control limits, the analysis run must be terminated and the reason determined. Start off with an instrument recalibration, followed by the standard check, then the reagents check, etc.

12.3.3.2 For measurements such as pH and conductivity, one of the LCS samples should be analysed again at the end of the run to monitor any drift since the last calibration adjustments.

### 12.3.4 DRIFT STANDARD (DS)

The drift standard is a standard analysed periodically through the course of a run to demonstrate that the initial calibration remains valid. This varies with each instrument - a general description is given below with more details available in the individual operating procedures.

#### 12.3.4.1 FLOW INJECTION/CONTINUOUS FLOW ANALYSIS

Typically a drift standard should be scheduled to run every 15 samples and at the end of a run. Set up a DQM plan that tell the instrument which standard is to be used as the drift standard, where the standard is located and what the expected concentration is. The DQM plans should have three levels:

- If the drift standard is within 4 % of the initial calibration value the instrument continues the run
- If the drift standard is between 4 to 8% of the initial calibration value the instrument recalibrates then continues the run
- If the drift standard is beyond 8% of the initial calibration value the instrument recalibrates then re-analyses all samples back to the last acceptable drift standard.

#### 12.3.4.2 ATOMIC ABSORPTION SPECTROPHOTOMETRY

Atomic absorption spectrophotometer analyses utilise a reslope standard at programmed intervals, and use the value obtained to shift the initial calibration up or down accordingly. Most methods have a reslope scheduled every 10 samples and a recalibration every 60. Ensuring that the flame, burner head temperature and lamps have been allowed to stabilise for 10 minutes prior to starting the run can minimize drift.

#### 12.3.4.3 CN ANALYSER

A drift correction standard should be run together with an LCS sample every 25 samples, and again at the end of the run.

#### 12.3.4.4 UV-VIS SPECTROPHOTOMETER

Read a mid-range standard after every 20 samples and at the end of the run.

#### 12.3.4.5 pH METER

Check both the pH 4 and 7 buffers after every 40 samples and adjust the meter if necessary. Read an LCS sample again after the buffer check.

#### 12.3.4.6 CONDUCTIVITY METER

Check the conductivity standard after every 40 samples and adjust the meter if the reading has drifted by more than 1% . Read an LCS sample again after the standard check.

### 12.3.5 EXTERNAL QC PROGRAMMES

The internal Quality Control (QC) Programme described in sections 12.3.1 – 12.3.4 involves the systematic day-to-day checking of the produced data to ensure whether it is reliable enough to be released. This is however only a relative check, and may lead to serious bias of results as a result of systematic errors. An external QC programme is a means of avoiding or discovering such errors.

Participation in interlaboratory exchange programmes allows an evaluation of the analytical performance of a laboratory by comparison with the results of other laboratories. In addition, the schemes are a useful source of reference samples e.g. they can be used as an internal laboratory control sample (LCS).

The laboratory participates in the following external quality assurance programmes:

- A round robin programme involving laboratories analysing two samples on a monthly basis.
- Australasian Soil and Plant Analysis Council (ASPAC) soil and plant proficiency programmes that involve 40+ laboratories in Australasia and issue proficiency certificates for laboratories that achieve certain standards.
- The Wageningen soil and plant proficiency programmes that involve laboratories internationally.

### 12.3.5.1 PERFORMANCE ASSESSMENT

The Laboratory Manager is responsible for examining the laboratory's performance. Care must be taken when comparing results, taking into account different methods used.
For the less formal schemes where limited numbers of participants make statistical analysis impossible, summarise each result by comparison to the others ( a subjective decision made on the basis of experience) using highlighter pen:

| Colour | Interpretation |
|---|---|
| Green | Satisfactory |
| Yellow | Questionable |
| Red | Unsatisfactory |

Where a statistical analysis is available the z-scores (based on the bias relative to the mean of all laboratories) should be used as follows:

| z-score | Interpretation |
|---|---|
| <1 | Excellent |
| Between 1 and 2 | Satisfactory |
| Between 2 and 3 | Questionable |
| >3 | Unsatisfactory |

If a result is flagged as questionable, that particular analysis will be closely observed during future runs. If two successive rounds give questionable results for an analyte, then a Quality Problem Report will be initiated.

If a result is flagged as unsatisfactory, then a Quality Problem Report will be initiated immediately.

Results are kept in the Proficiency Programmes file in the Laboratory Manager's office.

## 12.4 CONTROL CHARTS

Control charts are used to monitor the results for the Laboratory Control Sample (LCS).

Once the target value (mean) and upper and lower limits have been established, the values obtained for each run by every analyst are recorded directly into the appropriate QC spreadsheet. The result, date and the analyst's initials are entered on the page for that test. The latest result is automatically plotted on the control chart and must be inspected in light of the criteria in 12.4.2. The laboratory manager is notified immediately if any criteria fail, and may instigate a Quality Problem Report (see section 14 of this manual.)

The control chart shows any trend in results caused by biases not large enough to put the result out of range. Such biases are revealed by most or all the values lying above or below the mean, rather than being evenly distributed about the mean. The reasons for any bias are investigated, again by use of the Quality Problem Report system.

### 12.4.1 PREPARATION

To prepare the control chart, sufficient data (usually 10 – 20 results) on an analyte is accumulated and used to calculate a target mean and standard deviation. These are then used to draw five lines on the control chart – the mean, upper and lower warning levels (+/- 2 standard deviations) and the upper and lower control levels (+/- 3 standard deviations)

12.4.1.1 At the end of a run, transfer the data to the appropriate spreadsheet for calculation of results.

12.4.1.2 Copy the LCS result then paste (as values) into the appropriate page on the QC spreadsheet.

12.4.1.3 Fill in the analysis date and your initials.

12.4.1.4 Inspect the value, which is automatically plotted on the control chart, in light of the criteria in 12.5 below. The laboratory manager should be notified immediately if any criteria fail.

### 12.4.2 CRITERIA FOR REJECTING DATA

- LCS result outside upper or lower warning limit: Data require careful inspection but may be acceptable. Discuss with the Laboratory Manager.
- LCS result outside upper or lower control limit: Data are rejected and analyses are repeated.
- Two successive results beyond the same warning limit: Data are rejected and reason for bias is investigated.
- 10 successive LCS results on the same side of the mean: Data are rejected and reason for bias is investigated.

### 12.4.3 CORRECTIVE ACTION

*Data outside the acceptance limits or exhibiting a trend are evidence of unacceptable error in the analytical process. Corrective action needs to be taken promptly to determine and eliminate the source of error. Data is not to be reported until the cause of the problem is identified and either corrected or qualified. Procedure 756 Quality Problem Reports, in the Administration Manual, gives further details on how such problems should be investigated and rectified.*

### 12.4.4 DRIFT

Something less conspicuous and therefore perhaps more dangerous than incidental errors is gradual upward or downward trends, where biases may not large enough to put the result out of range. Such biases are revealed by most or all the values lying above or below the mean, rather than being evenly distributed about the mean. The reasons for any bias should be investigated. The main cause of drift is often LCS instability – pH and phosphate retention have been observed to change over time with soils. Other causes may include new stock standard solutions that are a small percentage in error, or a balance that has developed a small error.

### 12.4.5 IMPROVEMENTS IN PRECISION

Another important function of the control chart is an assessment of improvements in precision. If measurements rarely exceed the warning limits then recalculate the limits using the 10 – 20 most recent data. Trends in precision can be detected if running averages are kept.

# 13 TEST REPORTS

## 13.1 REPORT CHECKING

Once a job has been completed, a signatory, normally the Laboratory Manager, will make an internal verification check ensuring that items such as:

- Manually recorded data, such as pH measurements, have been entered correctly.
- Electronically captured data has been transferred into the correct position.
- Results for the in-house laboratory control samples are within acceptable ranges.
- An assessment of "correctness" in terms of expected ranges and correlation with other properties.

### 13.1.1 DATA ENTERED CORRECTLY?

13.1.1.1 For data that has been recorded manually then transcribed into the spreadsheet, locate the data hardcopy in the documentation envelope. Check each entry against the hard copy, and place a red script electronic signature in the cell to the right of the analyst's initials.

13.1.1.2 For electronically captured data, check two entries per page on screen comparing the data copied to the input column against the raw data file out to the right of the worksheet. Place a red script electronic signature in the same row as the checked cell, out to the right of the worksheet.

### 13.1.2 LABORATORY CONTROL SAMPLE RESULTS IN RANGE?

13.1.2.1 Check the LCS result on each page against the accepted limits and place a red script electronic signature to the right of the row.

### 13.1.3 ASSESSING DATA "CORRECTNESS"

A number of ranges have been determined to indicate results that need to be repeated or examined carefully because, while they may be correct, are outside the usual range. There are also internal relationships that should be expected. These are outlined in Figure 13.1 below.

13.1.3.1 Examine the data as a whole in terms of the ranges and relationships in Figure 13.1 below, looking for "oddball" results that don't tie in. Anything noticed should be recorded on the Notes page at the end, and a Quality Problem Report form filled in. This initiates the procedure for dealing with non-conforming work. No further work is done on reporting the job until the Laboratory Manager has cleared the Quality Problem.

13.1.3.2 Once the data has been found to be sound, a red script initial should be placed in the cell below the final row on the report page to indicate the checking has been done.

## 13.2 REPORT FORMAT

A report will be generated for each job, and an authorised signatory will sign all reports. In general, each report shall contain the following:

- A title e.g. Soil Analysis Report
- The name and address of the laboratory, and the location where the tests were carried out if different from the laboratory address
- Unique identification of the test report, such as the job number.
- Pagination, in the form of "page x of y" on each page
- Name and address of the client
- Identification of the methods used. This may be in the form of a separate sheet referred to in, and accompanying, the report.
- A description and unambiguous identification of the samples tested.
- A statement that results only apply to samples as received.
- A statement specifying that the report may not be reproduced, except in full, without the written consent of the signatory.
- Date of receipt of samples
- Dates of performance of tests
- Test results, with appropriate units
- Name, position and signature of person authorising the report

In addition, where necessary the following shall also be included:

- Sampling details, including date, sample identification, location of sampling, sampling procedures, relevant environmental conditions
- If, as described in section 9.2 any unusual features were noted when the samples were received and the client agreed to the analysis proceeding, then the sample condition as recorded on receipt should be noted.
- Any deviations from the test methods
- A statement of compliance/non-compliance with specifications
- A statement on the estimated uncertainties in the measurement
- Opinions and interpretations as needed by clients. Where included, the laboratory will document the basis upon which these have been made. Opinions and interpretations will be clearly marked as such on the report
- Any tests carried out by a subcontractor must be clearly identified as such.

If a test report needs to be re-issued for any reason, the re-issued report will be bear the same job number but with a clear "Amended Report" title.

## 13.3 RETENTION OF TEST REPORTS

A hard copy of each report will be stored in the hardcopy job file envelope, and will be kept for as long as storage space permits, after the minimum legal period of seven years.

An electronic copy of the report will be archived and stored forever.

# 14 NONCONFORMING WORK

## 14.1 POLICY

Quality control is primarily aimed at the prevention of errors. However, despite all efforts it remains inevitable that errors are made; therefore the control system must have checks to detect them.

The occurrence of sub-standard testing and complaints is a sign of quality breakdown in the laboratory, and must be urgently addressed. The Laboratory Manager is responsible for non-conforming work from the laboratory, and has the authority to manage the resolution of nonconforming situations, and to carry out actions to:

- Halt work and withhold reports where necessary
- Evaluate the significance of the nonconforming work
- Take immediate corrective action and decide on the acceptability of the nonconforming work
- Notify the client, if necessary, to recall work
- Define the requirements for the resumption of work

Quality problems are not restricted to the correctness of analytical data – results may be late or not adequate for the client's purpose. There may be a potential safety problem in the laboratory, performance in a sample exchange programme may be poor, calibration activities may be overdue, etc.

Sub-standard work may be due to inaccuracies in calculations, incomplete checking, use of the wrong techniques, standards, components, or some form of equipment failure. These are most likely to be picked up by the report signatory, but could be missed and result in a complaint by the client.

Sub-standard work may become apparent in the form of internal or external complaints, and may not necessarily relate to the quality of testing, e.g., results may be delivered late, or may not be adequate for the customer's purpose. All complaints of sub-standard work will be investigated and resolved at the earliest convenience; relevant test reports/testing work will be reviewed and, if inaccurate, will be recalled.

## 14.2 PROCEDURE

When errors are suspected or discovered, the following questions must be asked:

- What error was made?
- Where was it made?
- When was it made?
- Who made it?
- Why was it made?

The Quality Problem Report is a system designed to answer these questions in order to take proper action to correct the error, and prevent the same error being made again.

14.2.1 As soon as a Quality Problem is found or suspected it is reported to the Laboratory Manager by filling in sections 1 – 3 of the Quality Problem Report form. A compressed version of the blank form is reproduced in Figure 3 below.

**Figure 3. The Quality Problem Report form (compressed).**

## Quality Problem Report

QPR No: ……………….. Date: ……………….. Initiator: ………………..

1. Type of problem:
☐ Client enquiry re report or result
☐ Internally detected quality problem
☐ Interlab comparison result
☐ Client complaint re service
☐ Internal audit non compliance
☐ Safety problem/hazard identification
☐ Opportunity for improvement
☐ Other……………………………………………………….

2. Client / Job Number:

3. Description:

4. Investigation:

5. Outcome:

6. Corrective action: ☐ Affected report amended and re-issued
☐ Explanation given to client

7. Preventative action / Opportunity for improvement:

8. Follow-up: ☐ Not required
☐ Required - action……………………………………………………
- completed by…………………………………………
- date...……………………………………………………

9. Conclusion: ☐ Problem resolved satisfactorily
☐ Other……………………………………………………………

Signed:………………………… Date:…………………………

14.2.2 The Laboratory Manager sets up an investigation based on the preliminary details reported. The order and nature of the investigation depends on the type of problem.

14.2.3 Open the electronic job file.

14.2.4 Locate the result in question.

14.2.5 Check the spreadsheet for calculation or data transcription errors.

14.2.6 If no simple answer is found, arrange for the sample to be re-analysed, and continue the investigation.

14.2.7 Check that the Laboratory Control Sample result for that batch was in range.

14.2.8 Check the hard copy data in the job envelope against the electronic data.

14.2.9 Check the instrument calibration obtained for the run:

- Look at the raw areas/signals/counts/absorbances and compare them with those obtained for the same standards in other runs
- Look at the curve type (first order, second order, point-to-point)
- Look at curve algorithm used
- Look at weighting methods used in curve preparation
- Look at peak recognition (brackish /direct)
- Look at r2 and residuals
- Look at baseline (excessive noise/spikes/drift/shift compared to previous runs)
- Look at peak shapes (jagged tops/multiple tops tailing/flattening out/air spikes)
- Look at peak separation
- Look at retention times/run times/analysis times
- Look at drift standard results

14.2.10 Check the standards used:

- Were they within expiry dates?
- Were they stored correctly? Properly capped? Refrigerated? In the dark?
- Were flasks of the correct volume used?
- Check the Analytical Standards file to see if the correct salts/ correct grade were used in their preparation.
- Were the salts dried before use if necessary?
- Was the appropriate matrix used?
- Are there records of comparisons against previous standard sets?

14.2.11 Check the reagents used:

- Were they within expiry dates?
- Were they stored correctly? Properly capped? Refrigerated? In the dark?
- Were refrigerated reagents allowed to come to room temperature before use?
- Were the correct chemicals used in their preparation?
- Any signs of deterioration or contamination e.g. fungal growth, precipitates, “floaties”
- Check pH of any buffer reagents.

- Check cadmium column efficiency.

14.2.12 Check reagent blank values and compare to previous runs.

14.2.13 Check the apparatus calibration file:
- Were the pipettes/dispensers used calibrated according to schedule?
- Do another calibration check to ensure the calibration is still valid.

14.2.14 Check which dispenser was used.
- Any possibility of contaminations from previous reagents used in it?
- Is it capable of dispensing the volume specified in the method?

14.2.15 Check the quality of the deionised water:
- Check daily conductivity records for high levels/ spikes/drift.
- Check when the cartridges were last replaced. Ammonia can be produced by the breakdown of the resins used.
- Check where deionised water was sourced – direct from the unit or from lab storage containers? Ammonia and carbon dioxide levels are lowest in freshly deionised water.

14.2.16 Check the instrument:
- Any comments in the operating log which merit further investigation?
- Has maintenance been carried out according to schedule?
- Check wear items such as pump tubes (Varian SIPS unit, Lachat FIA pump)
- Visually check catalyst tubes.
- Visually check drying tubes.

14.2.17 Check the sample itself:
- Was the correct sample used?
- Are sample labels present/legible?
- Are sample numbers properly assigned?
- Was the correct particle size aliquot used - < 2mm or < 0.25mm?
- Are there any signs of sample deterioration?
- Was the sample correctly ground?
- Check sample homogeneity.
- Check for presence of roots in soils, or soil material in plant samples.
- Was the sample properly air-dried? Dry again and check weight change.

14.2.18 Check the sample extracts/digests if they are still available:
- Unusual colour?
- Clarity?
- Precipitates?
- Particulate matter?

## 14.3 CORRECTIVE ACTION

When the investigation determines that a significant problem has occurred then corrective action must be taken immediately.

14.3.1 The Laboratory Manager will immediately contact the affected client and arrange for recall of the test report in question.

14.3.2 The Laboratory Manager will discuss the problem with the client and advise them of action being taken. This may include arranging for another sample to be analysed.

14.3.3 The Laboratory Manager will review all other results in the affected batch, and preceding batches, to see if other samples are similarly affected. This may in turn mean further clients to be contacted/reports recalled.

14.3.4 The Laboratory Manager will stop all analyses of the type in question until it can be determined that the analysis is producing valid results.

14.3.5 Corrective actions identified in the Quality Problem Report shall be implemented immediately. These may include:

- Instrument/equipment servicing
- Instrument recalibration
- Preparation/purchase of new standards
- Preparation of new reagents
- Replacement of faulty consumables
- Application of another analysis method
- Modification of the current analysis method
- Staff training

14.3.6 The Laboratory Manager is responsible for authorising the resumption of work once the corrective action has been taken and it has been established that the corrective action has been successful in eliminating the problem.

14.3.7 All further testing will be monitored closely by the Laboratory Manager to ensure that the corrective actions have been successful and there is no recurrence of the problem.

14.3.8 The Laboratory Manager will issue an amended report if appropriate.

14.3.9 Affected areas should be fully audited if the non-conformance indicates the laboratory is not complying with its own quality system.

The investigation may however determine that no significant problem has occurred.
It may be that there is no problem with the laboratory's work and the problem has occurred prior to the samples being received, e.g. the investigation may find that the results are correct and indicate that a topsoil and subsoil have had their labels transposed during sampling by the client. The client must still be contacted and fully informed of the QPR findings.

## 14.4 RECORDS

Document the investigation fully, including discussions with clients and staff. The report is not confined to the boxes and questions on the QPR form. Records of the complaint or sub-standard work and the corrective action taken will be kept in the Quality Problem folder in the Laboratory Manager's office. This will record all details, including dates, client, job and laboratory numbers, discussions with the client and staff, trace back of spreadsheets and QC results, courses of action and the clearance of the problem.

The quality of the testing and reporting process as set out in the Quality Policy statement is monitored by these entries. Analysis of the QPR file will be presented as a component of the Management Review process, and the cause of any non-compliance will be established and remedied

## 14.5 PREVENTATIVE ACTION

In the evaluation of nonconforming work, and in the Quality Problem procedure, consideration should be given to what might be done to prevent a recurrence of the problem. The QPR forms contain a section in which the preventative actions may be described.

Preventative action is a pro-active process to identify opportunities for improvement, rather than simply a reaction to the identification of problems or complaints. Staff are encouraged to keep an open mind about such opportunities in their work, and to contribute to discussion on such matters at the weekly laboratory team meeting. Preventative action could include:

- Further staff training
- Changes to instrument operating procedures.

# 15 INTERNAL AUDIT

## 15.1 PURPOSE

To verify the laboratory's operations continue to comply with the requirements of the quality system, as well as with the criteria set out in ISO/IEC 17025, annual audits of activities will be carried out. These audits include:

- Internal audit of the quality system
- Internal audit of test methods
- Internal safety audit
- External audit.

Internal audits are conducted by peers, and are intended to be fact finding not fault finding; i.e. all parties should view them constructively. The areas of activity audited, the audit findings and the corrective actions that arise from them are to be fully recorded.

## 15.2 INTERNAL QUALITY AUDIT

The timing of the internal quality audit will be around October each year, 6 months prior to the external audit. The Laboratory Manager is responsible for planning and organising the audit, which should be carried out by trained personnel who are, if possible, independent of the activity to be audited. The Laboratory Manager is also responsible for ensuring internal audits are carried out.

Any non-compliances will be registered as Quality Problem Reports and dealt with through the QPR system. When the audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory will urgently carry out corrective action, and will notify clients if investigations show results may have been affected.

The Management Review, conducted around January each year, will monitor (record and verify) the implementation and effectiveness of corrective actions taken. This allows a maximum timeframe of approximately 3 months for corrective actions to be implemented.

The audit will be carried out using the checklist in Figure 4 below and will include all aspects covered by the Quality Manual. It will also include a review of previous non-compliances.

The audit checklist will be used to compile an audit summary that will be submitted for comment to the Laboratory Manager and Research Director. Each non-compliance will be registered as a Quality Problem for action. The results of the implementation of corrective actions will be reviewed at the next Management Review meeting.

## Figure 4. INTERNAL QUALITY AUDIT CHECKLIST

### XYZ LABORATORY
### INTERNAL QUALITY AUDIT CHECKLIST

**Date:** ________________________________

**Auditor:** ________________________________

### 1. DOCUMENT CONTROL

Locate the Document Control file in the Laboratory Manager's office.

| | Yes | No |
|---|---|---|
| Are the manuals located where stated? | ☐ | ☐ |
| Are the manual amendment sheets up to date? | ☐ | ☐ |
| Do the manuals contain the current versions of documents? | ☐ | ☐ |
| Are manuals (except Lab Manager's) free of hand-written amendments? | ☐ | ☐ |
| Has the Quality Manual review been completed to date? | ☐ | ☐ |
| Has the Method Manual review been completed to date? | ☐ | ☐ |

Comments:

________________________________________________________________

### 2 STAFF/TRAINING

| | Yes | No |
|---|---|---|
| Are staff resource levels adequate? | ☐ | ☐ |
| Do training files show all current personnel? | ☐ | ☐ |
| Are non-current files removed to the historical section? | ☐ | ☐ |
| Are staff training records up to date? | ☐ | ☐ |
| Are staff training needs being identified and addressed? | ☐ | ☐ |

Comments:

________________________________________________________________

## 3 ENVIRONMENT

| | **Yes** | **No** |
|---|---|---|
| Are temperature records up to date? | ☐ | ☐ |
| Are temperatures in controlled rooms within limits? | ☐ | ☐ |
| If not, has action been taken? | ☐ | ☐ |
| Are RO & MilliQ water records up to date? | ☐ | ☐ |
| Are RO & MilliQ water conductivities within limits? | ☐ | ☐ |
| If not, has action been taken? | ☐ | ☐ |
| Are grinding room dust extraction systems functioning? | ☐ | ☐ |
| Are grinding rooms being used for correct samples? | ☐ | ☐ |
| Plant drying oven not being used for sample storage? | ☐ | ☐ |
| Grinding rooms and equipment being cleaned after use? | ☐ | ☐ |
| Grinding room logs being used? | ☐ | ☐ |
| Overall housekeeping standard high? | ☐ | ☐ |

Comments:

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## 4 EQUIPMENT

| | **Yes** | **No** |
|---|---|---|
| Do all equipment items have an inventory no? | ☐ | ☐ |
| Are records of new instrument commissioning in Equipment & Calibration file? | ☐ | ☐ |
| Does every instrument have a operation/service/maintenance log? | ☐ | ☐ |
| Are instrument logs being used to record use? | ☐ | ☐ |
| Is instrument servicing being recorded in logs? | ☐ | ☐ |
| Instrument maintenance being carried out & recorded in logs | ☐ | ☐ |
| Is calibration reminder wall planner in place in Instrument Room? | ☐ | ☐ |
| Is calibration schedule being adhered to: | | |
| • Glass pipettes? | ☐ | ☐ |
| • Pipettors ? | ☐ | ☐ |
| • Dispensers? | ☐ | ☐ |
| • Timers? | ☐ | ☐ |
| • Thermometers? | ☐ | ☐ |
| • Balances? | ☐ | ☐ |
| • Ovens? | ☐ | ☐ |
| • Muffle furnace? | ☐ | ☐ |
| • New volumetric glassware? | ☐ | ☐ |
| Are calibration records (in Equipment Maintenance & Calibration file) up to date? | ☐ | ☐ |
| Are stock standards stored appropriately? | ☐ | ☐ |
| Are there no analytical stock standards older than 6 years? | ☐ | ☐ |
| Are records in Analytical Standards file up to date? | ☐ | ☐ |

| | | |
|---|---|---|
| Are fresh analytical standards being compared against old? | ☐ | ☐ |
| Are records in Analytical Standards file up to date? | ☐ | ☐ |
| Are working standards >100 mg/L no older than 3 months? | ☐ | ☐ |
| Are working standards 100 - 10 mg/L no older than 1 month? | ☐ | ☐ |
| Are working standards <10 mg/L no older than 1 week | ☐ | ☐ |
| Are standards labelled with i.d. & prep date? | ☐ | ☐ |
| Are suitable reference materials available? | ☐ | ☐ |
| Are template spreadsheets validation records up to date? | ☐ | ☐ |

Comments:

## 5 SUPPLIERS

| | Yes | No |
|---|---|---|
| Are purchases made only from approved suppliers? | ☐ | ☐ |
| Has the review of performance of approved suppliers been made to date? | ☐ | ☐ |
| Are purchasing verification records up to date? | ☐ | ☐ |

Comments:

## 6 SAMPLE HANDLING

| | Yes | No |
|---|---|---|
| Are samples stored in the appropriate locations? | ☐ | ☐ |
| Are sample identified with unique lab numbers? | ☐ | ☐ |
| Can the job the samples belong to be identified? | ☐ | ☐ |
| Is sample disposal up to date? | ☐ | ☐ |
| Are sample disposal records up to date? | ☐ | ☐ |
| Are quarantine records up to date? | ☐ | ☐ |
| Can a set of samples currently under analysis be located? | ☐ | ☐ |
| Can a set of samples completed within the last 3 months be located? | ☐ | ☐ |
| Do records indicate where to find a set of samples analysed a year ago? | ☐ | ☐ |
| Is sample storage in good consecutive order? | ☐ | ☐ |

Comments:

## 7 TEST METHODS

| | Yes | No |
|---|---|---|
| Are lab copies of method manuals in good condition? | ☐ | ☐ |
| Do lab copies of method manuals contain hand-written amendments? | ☐ | ☐ |
| Do any new methods have validation data in the Method Development file? | ☐ | ☐ |
| Are method uncertainties documented? | ☐ | ☐ |

Comments:

## 8 TEST RECORDS (JOB ENVELOPE)

Take a selection of 4 job envelopes at random.

| | Yes | No |
|---|---|---|
| Are sample receipt details present? | ☐ | ☐ |
| Are client discussion records present? | ☐ | ☐ |
| Are instrument printouts present? | ☐ | ☐ |
| Do manual worksheets have the date and analysts initials on? | ☐ | ☐ |
| Are manual worksheets free from use of pencil or Twink? | ☐ | ☐ |
| Is the test report present? | ☐ | ☐ |

Comments:

## 9 TEST RECORDS (ELECTRONIC)

Take a selection of another 4 jobs at random and open their electronic records.

| | Yes | No |
|---|---|---|
| Has the date and analyst initials been filled out for each test? | ☐ | ☐ |
| Is the imported instrument data available to the right of the spreadsheet? | ☐ | ☐ |

Comments:

## 10 ANALYTICAL QUALITY CONTROL PROGRAMME

| | Yes | No |
|---|---|---|
| Are LCS results being entered in the appropriate spreadsheet? | ☐ | ☐ |
| Are appropriate actions being taken & recorded due to LCS results? | ☐ | ☐ |
| Are control charts functioning and up to date? | ☐ | ☐ |
| Are proficiency programme results available? | ☐ | ☐ |
| Are proficiency programme results summarised? | ☐ | ☐ |
| Is action taken as a result of proficiency programme results? | ☐ | ☐ |

Comments:

## 11 TEST REPORTS

Take a selection of 4 job envelopes at random and check the test reports.

| | Yes | No |
|---|---|---|
| Are client identification details present? | ☐ | ☐ |
| Is the LJ job identification number present? | ☐ | ☐ |
| Is the sample receipt date noted? | ☐ | ☐ |
| Are the reports laid out in an easy to read manner? | ☐ | ☐ |
| Are the laboratory contact details present? | ☐ | ☐ |
| If IANZ endorsed, does an approved signatory sign the report? | ☐ | ☐ |

Comment:

## 12 NONCONFORMING WORK

Locate the Client Response file in the Laboratory Manager's office.

| | Yes | No |
|---|---|---|
| Have any incidents been recorded in the last year? | ☐ | ☐ |
| Have all action items been carried out? | ☐ | ☐ |

Comments:

### 13 SAFETY

| | Yes | No |
|---|---|---|
| Are monthly audits being conducted? | ☐ | ☐ |
| Are non-compliance items being actioned? | ☐ | ☐ |
| Have there been no laboratory accidents recorded in the Site Accident Register? | ☐ | ☐ |

Comments:

### 14 INTERNAL AUDIT

| | Yes | No |
|---|---|---|
| Have all items raised from the last audit been actioned? | ☐ | ☐ |

Comments:

### 15 MANAGEMENT REVIEW

| | Yes | No |
|---|---|---|
| Has a review been carried out since the last audit? | ☐ | ☐ |
| Have all items raised been actioned? | ☐ | ☐ |

Comments:

## 15.3 INTERNAL METHOD AUDIT

Method audits will be carried out annually and will be arranged by the Laboratory Manager. These audits will be aimed at all test methods, and will be carried out by summarising the LCS data performance and trends. In addition, up to 5 methods will be chosen and the full analysis procedure audited to detect any deviation from the method as documented in the manual. Opportunities for improvements to methods will also be examined.

## 15.4 INTERNAL SAFETY AUDIT

The purpose of the audit is to identify any hazards or unsafe practices that may exist. A senior staff member, using an available checklist contained at the back of the Safety Manual, carries out the audit annually. The audit also includes a review of the previous non-compliances. The completed checklist will be used to compile an audit summary that will be submitted for comment to the Laboratory Manager. Each non-compliance will be registered as a Quality Problem for action. The results of the implementation of corrective actions will be reviewed at the next Management Review meeting

More concise safety checks are carried out on a monthly basis by a nominated analyst. The purpose of these checks is to identify any potential hazards requiring urgent attention, as well as ensure safety equipment such as eyewashes and showers are functional. A checklist is held in the Laboratory Safety Audit file in the Laboratory Manager's office. The completed checklist is handed to the Laboratory Manager who will ensure that minor issues will be dealt with immediately and signed off on the checklist. More serious non-compliances will be registered as Quality Problems and actioned through the QPR system.

## 15.5 EXTERNAL AUDIT

An external audit shall be carried out annually by ..............

# 16 MANAGEMENT REVIEW

## 16.1 PURPOSE

The quality process can be described as follows:

- Objectives are established (Quality Policy)
- A plan to achieve these objectives is formulated (Quality Procedures)
- Performance is regularly monitored (Internal audits)
- Action is taken if the procedures are not being met (Corrective actions)
- The procedures are adjusted, if necessary (Document Control)
- The final outcome is reviewed (Management Review meeting)

Management reviews are held annually to establish the objectives regarding aspects such as customer complaints, external and internal audit findings, sub-standard testing work, timeliness of calibrations, number of errors or re-issued reports, staff turnover, etc. These objectives are a summary of the laboratory's performance, and are made quantifiable in terms of time, cost, quality or quantity. The annual internal audit reports, QPR file and Client Response file therefore form essential elements to be considered in the management review process.

The management procedures will be reviewed to ensure their continuing suitability and effectiveness, and to introduce any necessary changes or improvements.

## 16.2 RESPONSIBILITY

The responsibility for carrying out the management review will lie with the Laboratory Manager and will be arranged by him. The review team will comprise the Research Director, Laboratory Manager and Laboratory representatives.

## 16.3 CONTENT

The management review will include discussion of trends/developments in laboratory activity, an examination of recent audits, external assessments, records of customer feedback, complaints and non-compliances, problems with methodology and equipment, results of interlab and proficiency testing, changes in technology, and staffing requirements.

## 16.4 PLANNING AND DOCUMENTATION

Planning for an annual management review will include nominating the review team, gathering information on performance, and setting out an agenda of items to be covered. A sample agenda for the meeting is given in Figure 5 below. The management review process will be recorded by minutes taken. Documentation and records from the Review are located in the Management Review File held by the Laboratory Manager. Amendment to the Quality Manual may follow a Management Review.

## Figure 5. MANAGEMENT REVIEW AGENDA

XYZ LABORATORY
Management Review Agenda

Date: ______________________________

Time: ______________________________

Place: ______________________________

Agenda Items:

1. Recap of objectives set at last meeting
2. Review of the quality indicators from the last 12 months, namely:
   - Customer feedback (number & nature of complaints, positive and negative.
   - Reports/requests/feedback from staff.
   - Number & nature of corrective action issues raised.
   - Number & nature of accidents and other safety issues.
   - Equipment issues e.g. unplanned down time.
   - External audit findings.
   - Internal audit findings.
   - Proficiency programme results.
   - Staff issues e.g. morale, turnover.
   - Other key performance indicators e.g. number of reports issued late,, amount of chargeable time recorded.
3. Trend analysis of these quality indicators.
4. Were the objectives met? If not, why?
5. What changes are anticipated in the next year?
   - Workload increase/decrease?
   - Technology – new/changed tests?
   - Staffing
   - Equipment
   - Environment/premises
   - Customer expectations
   - Third party requirements e.g. accreditation agencies
6. Establish objectives for next year

# PART II

# STANDARD OPERATING PROCEDURES

# SOP1 BALANCE CHECK & CALIBRATION

## 1 INTRODUCTION

The maintenance of accurately calibrated scientific balances is an essential requirement for quality assurance. Internally, a monthly reading check and a six monthly repeatability check of all balances is carried out to check and monitor balance performance. An accredited service company conducts an annual professional service on all balances.

## 2 EQUIPMENT

2.1 A set of calibrated weights (50g, 100g, 200g) certified by an accredited service company is kept in the Laboratory Manager's office. An accompanying certificate for these weights is kept with the Annual Service Report in the box-file in the Instrument Lab.

2.2 A set of check weights are held in the Balance Room (0.2g - 100g, 1000g, 2000g).

2.3 Bone tipped tweezers are kept for handling weights <100g. Soft cloths are kept for use with weights >100g. These handling tools are kept in the boxes that contain the respective check weights.

2.4 A soft brush and tissues for general cleaning of balances.

2.5 Monthly and six monthly balance check sheets and a balance calibration worksheet are kept in the Balance Check File in the Balance Room. The worksheets list balance details, their inventory number, their location, and maximum weight, number of decimal places that it records to and check weight/s used.

## 3 ROUTINE BALANCE MAINTENANCE

Balances are left switched on all the time, unless used very infrequently. This is because electronic balances have a warm-up period of about 30 minutes. When constant temperature is reached the balance has increased accuracy. No balance check must be carried out until a balance has been turned on for at least 30 minutes. **All users should carry out general maintenance procedures.**

### 3.1 GENERAL MAINTENANCE

3.1.1 Balances must be kept in a clean, dry, dust-free environment. They must be positioned in an area away from direct sunlight or excessive draughts or temperature fluctuations.

3.1.2 Balances must be sitting level on the bench. Most of the balances are equipped with a level control and levelling feet. The feet are adjusted until the air bubble is in the middle of the level control. A balance will normally require levelling every time that it is moved.

3.1.3 Balances should be zeroed before each weighing.

### 3.2 ADJUSTMENT USING CAL-MODE

3.2.1 Balances should be regularly adjusted using the CAL-MODE feature. This is a scale adjustment, required due to temperature, humidity and pressure fluctuations in the balance environment. This feature varies between balances, and some balances do not have the feature at all.

Table 1. Procedure to Adjust Scale:

| **BALANCE** | **TYPE OF CAL-MODE** | **PROCEDURE** |
|---|---|---|
| Sartorius AC121S | Internal calibration weight | Press F1. The display will indicate "CAL" and then do an automatic calibration using internal weights |
| Mettler AE160<br>Sartorius A200S | Internal calibration weight | Press the control bar, until the display indicates "CAL". The display will then flash "100". Push the internal calibration slide on the right hand side of the balance into position. The display will then flash " 0". Push the slide back to its original position. The balance will automatically perform its own calibration. Wait until the display returns to " 0.0000" before use. |
| Mettler PB5001<br>Mettler PB153<br>Mettler BB244<br>Mettler AJ150<br>Mettler PM2500<br>Mettler PM3000 | External calibration weight | Press the control bar until the display indicates A-CAL-0. The display will then flash a number, indicating the weight that it requires. Place the appropriate calibrated weight onto the pan. The display will then flash " 0 ". Remove the selected calibrated weight. The balance will automatically perform its own calibration. Wait until the display returns to "0.0000" before use. |
| Mettler PC440<br>Mettler PC2200<br>Mettler PC4400 | External calibration weight and calibration screw | Adjust weight by inserting a screwdriver into calibration screw at rear of balance (see balance manuals for further information). Adjust weight for zero and 100g (PC440) and zero and 1000g (PC2200 and PC4400). |

For further information refer to the Operator Manuals for each balance, located in a box file in the Instrument Lab.

## 4 MONTHLY BALANCE CHECK

4.1 Zero the balance and adjust the scale with the "CAL-MODE" feature.

4.2 Obtain a monthly balance checksheet from the Balance Check File in the Balance Room.

4.3 Place the appropriate check weight (as listed on the checksheet) onto the balance and record its weight on the checksheet.

4.4 Calculate the difference between this weight and the reference balance reading. Put this value into the difference column.

4.5 The difference should be less than the acceptance limits for the balance. Use the comments column to report any problems.

## 5 SIX MONTHLY REPEATABILITY CHECK

5.1 Zero the balance and adjust the scale with the "CAL-MODE" feature.

5.2 Obtain a set of repeatability test worksheets and a 6 monthly balance checksheet from the Balance Check File in the Balance Room.

5.3 Place the appropriate check weight (as listed on the checksheet) onto the balance and record its weight on the repeatability test worksheet.

5.4 Remove the weight from the balance.

5.5 Zero the balance

5.6 Replace the check weight onto the balance and record its weight again.

5.7 Repeat procedure 5.3 – 5.5 until you have ten readings.

5.8 Repeat this procedure for each of the balances listed on the check sheet.

5.9 Input the data to the spreadsheet 6monthrepeatability.xls (available in QualityAssurance\Calibration\Balances) for calculation of means and standard deviations for the repeatability test on each of the balances.

If the standard deviation is greater than 1.8 times the reference standard deviation (the acceptance limits) then the balance probably requires servicing and recalibration.

## 6 IN-HOUSE BALANCE CALIBRATION

The Measurements Standards Laboratory of New Zealand recommends that a balance be professionally recalibrated every 1 to 3 years. The recalibration may also be done internally as described below. A "best accuracy" is calculated for each balance using the spreadsheet balcalibration.xls, located in QualityAssurance\ Calibration\Balances.

6.1 PAN POSITION ERROR.

**Never use a weight greater than half range of the balance, as this could damage the balance.**

6.1.1 Zero the balance.

6.1.2 Adjust the scale with the "CAL-MODE" feature.

6.1.3 Obtain an electronic balance calibration worksheet from the Balance Check File in the Balance Room, and complete page 1 with details of the balance being checked.

6.2 HYSTERESIS.

6.2.1 Zero the balance.

6.2.2 Adjust the scale with the "CAL-MODE" feature.

6.1.3 Obtain an electronic balance calibration worksheet from the Balance Check File in the Balance Room, and complete page 1 with details of the balance being checked.

6.3 REPEATABILITY.

6.3.1 Zero the balance.

6.3.2 Adjust the scale with the "CAL-MODE" feature.

6.1.3 Obtain an electronic balance calibration worksheet from the Balance Check File in the Balance Room, and complete page 1 with details of the balance being checked.

6.4 DEPARTURE FROM NOMINAL VALUE (LINEARITY ERRORS)

6.4.1 Zero the balance.

6.4.2 Adjust the scale with the "CAL-MODE" feature.

6.1.3 Obtain an electronic balance calibration worksheet from the Balance Check File in the Balance Room, and complete page 1 with details of the balance being checked.

## 7 REFERENCE

Clarkson, M., and B. May. 2001. Balances and Weighing. Published by Measurement Standard Laboratory of New Zealand, Industrial Research Ltd. (www.irl.cri.nz)

# SOP2 MUFFLE FURNACE VERIFICATION

## 1 INTRODUCTION

The muffle furnace is used to determine loss on ignition by heating a sample to 550 °C. A six-monthly check should be made to ensure that this temperature is being reached. In addition, the spatial variation of temperature within the furnace should be checked every two years in order to ensure there are no "cool spots" where incomplete combustion might occur. Checks are done using Tempil pellets, which show signs of melting when the temperature rating of the pellet has been achieved.

## 2 PROCEDURE

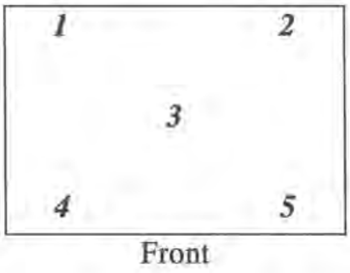
2.1 Obtain a set of Tempil pellets for 525 °C, 550 °C and 566 °C.

2.2 Line a set of 30 small porcelain crucibles with aluminium foil.

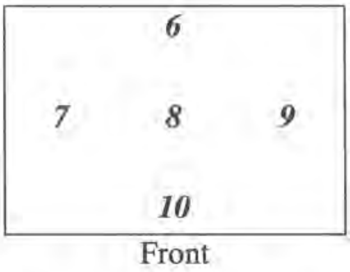
2.3 Place 10 pellets of each temperature rating into individual crucibles.

2.4 Place the crucibles in groups of three (one crucible of each temperature rating) into the different areas of the furnace as indicated on the diagrams below:

Top shelf



Bottom shelf



For the 6-monthly check, a single set of the 3 rated tempils at position 3 is sufficient.

2.5 Set the furnace to heat to 550 °C and maintain that temperature for 1 hour.

2.6 When the furnace has cooled, remove the shelves and examine the tempils for signs of melting.

2.7 Record the condition of each tempil at each position.

# 3 RESULTS

The 525°C tempils should melt completely, the 550 °C tempils should have begun to melt, and the 566 °C tempils should show no signs of melting.

If some of the 525°C tempils have not melted completely, or some of the 550 °C tempils have not begun to melt then the furnace is not getting hot enough and requires service.

If a large proportion of the 566 °C tempils have also melted then the furnace is getting too hot. While this is not a problem in the case of tests such as loss on ignition and ash determinations, a service check is in order.

Store the data records in the Ovens section of the Equipment Calibration file in the Laboratory Manager's office.

# SOP3 OVEN TEMPERATURE VERIFICATION

## 1 INTRODUCTION

The working temperature of the drying ovens and cabinets is monitored on a daily basis whenever the apparatus is in use. A numbered thermometer is kept with each oven/cabinet, either in a bung in the air vent opening on top or in a pottle of sand on the middle shelf inside. These thermometers are in turn recalibrated 6 monthly against the reference thermometer at the normal working temperature of the oven/cabinet.

It is the responsibility of the oven user to note the oven temperature daily and plot it on the daily temperature record charts. These are kept on a clipboard in the drying cabinet room. In addition, the spatial variation of temperature within the oven/cabinet must be checked every two years as described in the procedure below.

## 2 PROCEDURE

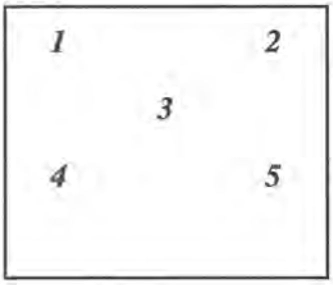
2.1 Set the oven to its normal working temperature.

2.2 Drill holes of sufficient diameter to take a thermometer in a numbered set of pottle lids.

2.3 Fill the pottles with dry quartz sand and screw on the lids.

2.4 Place the pottles into the positions within the oven where temperature is to be checked. The decision needs to be made for each oven/drying cabinet which and how many positions need to be checked, and this information is recorded on a standard worksheet.

e.g. for each shelf:



2.5 Leave for at least two hours for the sand temperature to stabilise (overnight for the soil drying cabinets.)

2.6 Place a thermometer through the hole in the pottle lid into the sand.

2.7 Shut the oven door and wait 2 minutes to allow the thermometer reading to stabilise.

2.8 Remove the pottle and thermometer and record the temperature.

2.9 Repeat 2.6 – 2.8 for all the pottles.

## 3 RESULTS

3.1 Check that the spread of temperatures is within the range specified as acceptable for that oven. Service may be necessary if the range is exceeded.

e.g.

| Oven Inventory No. | Oven Name | Normal temperature °C | Allowable Spread +/- °C |
|---|---|---|---|
| XYZ1 | Soil drying cabinet | 35 | 5.0 |
| XYZ2 | Incubator | 40 | 2.0 |
| XYZ3 | Plant drying oven | 80 | 5.0 |
| XYZ4 | Moisture factor oven | 105 | 2.0 |

3.2 Store the data records in the Ovens section of the Equipment Calibration Data folder in the Laboratory Manager's office.

# SOP4 CALIBRATION OF WORKING THERMOMETERS

## 1 INTRODUCTION

It is fundamental to good laboratory practice that the calibrations of both reference and working thermometers be traceable to national standards of temperature. The reference thermometer has a full calibration done by an approved calibration laboratory on a five-year cycle, with an ice-point check being performed internally every 6 months. The reference thermometer is then used to check the laboratory working thermometers. These are checked initially over the full temperature range, with subsequent 6-month checks of the ice point and one other temperature, usually the temperature at which the thermometer is most often used. The reference thermometer is kept in the Laboratory Manager's office, and is only to be used for calibration of other working thermometers.

## 2 APPARATUS

2.1 Water bath. Use a perspex plate cover, with five holes drilled to take the thermometers, and small o-rings to hold the thermometers at the correct height.

2.2 Oven.

2.3 Reference thermometer. Must be calibrated by an accredited laboratory.

2.4 Magnifying glass. To reduce parallax errors and improve readability.

2.5 Thermos flask.

## 3 PROCEDURE

Use the worksheet from QualityAssurance\Calibration\Thermometer.xls

3.1 VISUAL CHECK

3.1.1 Visually examine the thermometer for gross defects, such as non-uniform or missing graduations, missing numbers and improper etching.

3.1.2 Visually check the mercury column for defects such as gas inclusions and broken columns.

3.2 ICE POINT CHECK

When a thermometer is made the glass is in a highly stressed state. This stress slowly relaxes in time, causing the bulb to contract. This in turn pushes the mercury higher up the capillary, causing the ice point to rise. There is a consequent change to all other scale points. Such ageing slows with time, but an ice point check every 6 months is a good monitor of bulb contraction. In general, any rise detected can be subtracted linearly along the entire scale of the thermometer.

3.2.1 Prepare ice using deionised water in clean trays in a freezer.

3.2.2 Crush the ice by wrapping in a clean towel and using a hammer.

3.2.3 Place the ice into a thermos flask deep enough to immerse the thermometer to the correct level. The reference thermometer is a total immersion type, which means it needs to be immersed to the level of the reading. The working thermometers are partial immersion types that need to be immersed to 76 mm (specified on the stem).

3.2.4 Fill the thermos with deionised water until the whitish ice colour disappears and clear ice remains.

3.2.5 Drain off excess water, but not so much that the whitish colour returns.

3.2.6 Take the ice point reading by immersing the thermometer to the 0 °C mark (total immersion) or partial immersion mark as appropriate. Use a magnifying glass.

Any change in the ice point that has occurred between the current calibration and the initial calibration should be applied at all other calibrated points.

Example: The initial calibration report may be as follows:

| Calibration Point °C | Reading °C | Correction °C |
|---|---|---|
| 0.00 | 0.04 | -0.04 |
| 40.00 | 40.02 | -0.02 |
| 80.00 | 79.98 | +0.02 |
| 105.00 | 105.04 | -0.04 |

At the next ice point check, the new ice point values average 0.06 °C. The ice point has gone up by 0.02 °C, and the thermometer is therefore reading high by 0.06 °C. The corrections at the other temperatures must now be altered by -0.02 °C, thus:

| Calibration Point °C | Correction °C |
|---|---|
| 0.00 | -0.06 |
| 40.00 | -0.04 |
| 80.00 | 0.00 |
| 105.00 | -0.06 |

### 3.3 COMPARISON OF WORKING THERMOMETERS WITH REFERENCE THERMOMETERS

Up to four working thermometers can be checked at one time in the water bath, or singly in the oven. Check at several points along the scale during the initial calibration, typically 35 or 40 °C, 60 °C, 80 °C and 105 °C.

3.3.1 Set up the water bath

3.3.2 Insert the reference thermometer in the centre hole and up to four working thermometers in the surrounding holes. Slide them through o-rings to the depth appropriate for their type.

3.3.3 Allow the bath to stabilise at approximately the required temperature.

3.3.4 When the bath temperature is stable (or increasing slowly) read the thermometers.

Note: The 105 °C check has to be performed in an oven, with the thermometers side by side in a drilled bung in the air vent to hold them. Set the temperature using the digital controller.

For subsequent 6-month single temperature checks, use the temperature at which the particular thermometer is normally used.

## 4 CALCULATIONS

Use the spreadsheet in QualityAssurance\Calibration\Thermometer.xls

4.1 Correct the reference thermometer reading from its calibration certificate.

4.2 Subtract the corrected reference thermometer reading from the reading of the working thermometer to obtain the error in the working thermometer. The correction is found by changing the sign of the error.

Example for calibration at 40 °C:

| | Reference Thermometer | Working Thermometer |
|---|---|---|
| Reading (°C) | 39.8 | 39.3 |
| Correction to reference from calibration certificate - 0.04 °C | -0.04 | |
| Temperature in bath at calibration | 39.76 | |
| Subtract bath temperature from working thermometer reading to obtain error in working thermometer | | - 39.76<br>-0.46 |
| Correction to working thermometer | | +0.46 |
| Round to nearest 0.1 °C | | +0.5 |

4.3 Print off the summary page from the spreadsheet showing the correction to be applied to each thermometer at the temperatures checked, and place in the thermometer drawer in the instrument room.

## 5 APPLICATION OF CORRECTION FACTORS

Correction factors of less than 1°C do not need to be applied. Thermometers with correction factors greater than 1°C will be disposed of.

When a new thermometer is needed use the summary spreadsheet to select a thermometer with suitable accuracy for the temperature being measured.

## 6 REFERENCES

Working Thermometers Calibration Procedures. Telarc Technical Guide Number 3, August 1986.

# SOP5 CALIBRATION OF TIMERS

## 1 INTRODUCTION

Timers used in test measurements need to be checked against a standard time signal. The easiest method of doing this is to use the hourly time signals on the National Radio programme. Laboratory timers need to be accurate to within 2 seconds over a one-hour test period. Timers need to be tested upon purchase and then on an annual basis.

## 2 APPARATUS

2.1 Radio capable of receiving National Radio (1449MHz)

## 3 PROCEDURE

Use the worksheet from QualityAssurance\Calibration\Timers.xls to record measurements.

3.1 Listen for the commencement of the time pips on the hour on the National Radio programme.

3.2 Start the timer running in count-up mode on the sixth pip.

3.3 Stop the timer on the sixth time pip one hour later.

3.4 Carry out this check on one timer, and if it is within the 2 seconds limit then it can be used to test the other lab timers.

## 4 RESULTS

4.1 If any timer is outside the 2-second tolerance the batteries should be replaced and the test repeated. If the result is again outside the 2-second tolerance then the timer should be disposed of.

4.2 The raw data should be stored in the appropriate section of the Equipment Calibration folder in the Laboratory Manager's office.

# SOP6 VERIFICATION OF PIPETTORS AND DISPENSERS

## 1 INTRODUCTION

Measuring and transferring liquid volumes is one of the most frequently performed activities in a laboratory, and properly calibrated and performing volumetric measuring devices are essential to obtaining accurate analytical data. Verification is a comparison of the nominal volume against the volume actually contained or delivered. This is measured gravimetrically by weighing the quantity of deionised water contained or delivered, and measuring the temperature of the water at the same time to allow for a correction to be made for the density of the water.

Every pipettor or dispenser should be given a quick gravimetric test before use by pipetting the nominal volume four times. A full calibration check as described in this procedure should be carried out every three months.

## 2 APPARATUS

2.1 Balance

2.2 Thermometer.

2.3 Water. To avoid fluctuations in the temperature of the water used in the calibration process, fill a large container with sufficient deionised water for the task and leave to equilibrate overnight in the balance room.

## 3 PROCEDURE

### 3.1 PIPETTORS

3.1.1 Print off a worksheet from QualityAssurance\ Calibration\ Pipettor.xls

3.1.2 Inspect visually for any obvious physical defects such as scratches, damage to the nose cone or loose connections.

3.1.3 Perform a leak test by drawing in the maximum nominal volume of deionised water and holding steady for 20 seconds. There should be no visible drops at the tip.

3.1.4 Record the water temperature to the nearest 0.5 °C.

3.1.5 Place an appropriately sized weighing vessel such as a beaker on the balance.

3.1.6 Add sufficient deionised water to cover the bottom of the weighing vessel, and tare.

3.1.7 Pre-rinse the pipette tip and discard the rinse.

3.1.8 Draw up a set volume of deionised water from the bulk supply, deliver into the tared weighing vessel and record the value.

3.1.9 Repeat step 3.1.8 another 9 times at each volume to be checked.

### 3.2 DISPENSERS

3.2.1 Print off a worksheet from QualityAssurance\ Calibration\ Dispensers.xls.

3.2.2 Record the water temperature to the nearest 0.5 °C.

3.2.3 Place an appropriately sized weighing vessel such as a beaker on the balance and tare.

3.2.4 Ensure the dispenser is at least half full either with deionised water or the liquid normally dispensed.

3.2.5 Set to the check volume and dispense two aliquots to waste to ensure there are no air bubbles.

3.2.6 Dispense the set volume into the weighing vessel ten times, recording the weight and then re-taring each time.

## 4 CALCULATIONS

All data should put input into the spreadsheets QualityAssurance\ Calibration\ Pipettor or Dispenser.xls. Cells requiring data are coloured green.

### 4.1 PIPETTORS

The mean volume at the test temperature for pipettors is calculated in the spreadsheet from the mean weighing result by a factor incorporating density corrections. The mean volume is used to calculate the inaccuracy:

$$U\% = ((v_m - v_n) / v_n) \times 100$$

where:

$U$ = inaccuracy (mL)
$v_m$ = mean volume of liquid delivered (mL), from equation (1)
$v_n$ = nominal volume of liquid delivered (mL)

The individual weight readings are used to calculate the coefficient of variation, which is a measure of precision.

### 4.2 DISPENSERS

The individual weights dispensed are used to calculate the coefficient of variation, which is a measure of precision of the dispenser. Accuracy is not considered as part of this checking process as the volume is set each time by the user.

The raw data should be stored in the appropriate section of the Equipment Calibration folder in the Laboratory Manager's office.

## 5 TOLERANCES

All pipettors should have a precision within 2% and a bias (inaccuracy) within 2%. Any pipettor outside these limits should be serviced and retested. If it still fails then it must be disposed of. The accuracy of a dispenser is governed by the setting made by the user at the time of use; however the precision should be within 2%.

The spreadsheets have a built-in formula which checks the parameters measured against these tolerances, and outputs either "Pass" or "Service" if the tolerances are exceeded. Notify the Laboratory Manager if service is required on any piece of equipment tested.

## 6 REFERENCES

Australian Standard. Verification and use of volumetric apparatus. Part 2:Guide to the use of piston-operated volumetric apparatus. AS 2162.2 - 1998.

# SOP7 VERIFICATION OF VOLUMETRIC GLASSWARE

## 1 INTRODUCTION

Measuring and transferring liquid volumes is one of the most frequently performed activities in a laboratory, and properly calibrated and performing volumetric measuring devices are essential to obtaining accurate analytical data. Before a piece of volumetric glassware (flask or pipette) is put into service it must be fully verified following this procedure.

Verification is a comparison of the nominal volume against the volume actually contained or delivered. This is measured gravimetrically by weighing the quantity of deionised water contained or delivered, and measuring the temperature of the water at the same time to allow for a correction to be made for the density of the water.

Other factors affect the accuracy; however these are largely negligible compared to the water density correction and are mentioned here for completeness only. They are:
- Glassware temperature.
- Glass type.
- Cleanness of glass surface.
- Altitude.

## 2 APPARATUS

2.1 Balance.

2.2 Thermometer.

2.3 Water. To avoid fluctuations in the temperature of the water used in the calibration process, fill a large container with sufficient deionised water for the task and leave to equilibrate overnight in the balance room.

2.4 Indelible pen.

2.5 British Standard Schedule for Tables for use in the calibration of volumetric glassware. BS 1797:1987. Kept on the manuals shelf in the laboratory.

## 3 PROCEDURE

3.1 Print off the appropriate worksheet (QualityAssurance\ Calibration \VolFlask or VolPipette.xls)

3.2 Use the indelible pen to place a unique serial number on the item.

3.3 Record the serial number and capacity.

3.4 If necessary, clean with deionised water. Volumetric glassware must drain in such a manner that a smooth film of solution adheres to the inside, with no beading or droplet formation. If necessary, soak briefly in a warm detergent solution. Avoid soaking for too long, as a rough area may develop at the glass/air interface. Rinse with 3 or 4 portions of deionised water.

3.5 Dry by allowing to drain, or blowing gently with compressed air. **Volumetric glassware should never be oven dried as this may cause permanent changes in volume.**

3.6 To avoid errors due to absorption or loss of moisture from the glass surface, wipe the vessel with a clean tissue, and wear cotton gloves when handling.

3.7 VESSELS GRADUATED TO CONTAIN e.g. volumetric flasks

3.7.1 Place the empty vessel on the balance pan and tare.

3.7.2 Remove the vessel and fill to just below the graduation line.

3.7.3 Remove any trapped air bubbles by gentle tapping.

3.7.4 Bring the bottom of the meniscus to sit on the graduation line by adding water dropwise with a disposable dropper pipette.

3.7.5 Replace the filled vessel on the balance and record the weight.

3.7.6 Remove the vessel from the balance and record the water temperature.

3.8 VESSELS GRADUATED TO DELIVER e.g. pipettes

3.8.1 Place an appropriately sized weighing vessel such as a beaker on the balance.

3.8.2 Add sufficient deionised water to cover the bottom of the weighing vessel, and tare.

3.8.3 Fill the pipette to be tested to a few millimetres above the graduation mark using a suction device.

3.8.4 Wipe any adhering water off the outside of the pipette tip with a tissue.

3.8.5 Set the meniscus to the graduation line by allowing the water to run out.

3.8.6 Remove the drop of water adhering to the tip by bringing the tip into contact with an inclined glass surface such as the inside of a beaker (not the weighing vessel one!)

3.8.7 Allow the pipette to drain naturally into the weighing vessel, with the pipette tip being maintained in contact with the weighing vessel.

3.8.8 Remove the pipette after a waiting time of 3 seconds. The small quantity of water that remains in the tip must not be added to the delivered volume.

3.8.9 Record the weight of the weighing vessel with the delivered water.

3.8.10 Record the water temperature.

# 4 CALCULATIONS

Use the British Standard Schedule for Tables for Use in the Calibration of Volumetric Glassware. Two sets of tables are included; set A relates to soda glass vessels and set B to borosilicate glass vessels.

Table 1A (or B as appropriate) gives a mass in g to be added to the weighed contained or delivered mass to correct for the density of the water at the test temperature.

An example is given below for the test of a 100 mL borosilicate volumetric flask:

| | |
|---|---|
| Mass of water contained | 99.87 g |
| Water temperature | 21.5 °C |
| Correction from Table 1B/100 (page 67) | +0.315 g |
| Therefore capacity at 20 °C | 99.87 + 0.315 = 100.19 mL |

# 5 TOLERANCES

Tolerances used are based on those prescribed by the American National Bureau of Standards (Vogel) as follows:

| Flask Capacity mL | 10 | 25 | 50 | 100 | 200 | 250 | 500 | 1000 | 2000 |
|---|---|---|---|---|---|---|---|---|---|
| Tolerance +/- mL | 0.05 | 0.10 | 0.10 | 0.15 | 0.30 | 0.30 | 0.50 | 1.00 | 1.00 |
| Tolerance (% of capacity) | 0.5 | 0.4 | 0.2 | 0.2 | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 |

| Pipette Capacity mL | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|
| Tolerance +/- mL | 0.02 | 0.02 | 0.02 | 0.03 | 0.03 | 0.03 | 0.03 | 0.04 | 0.04 |
| Tolerance (% of capacity) | 2 | 1 | 0.7 | 0.8 | 0.6 | 0.5 | 0.4 | 0.5 | 0.4 |

| Pipette Capacity mL | 10 | 15 | 20 | 25 | 30 | 40 | 50 | 75 | 100 |
|---|---|---|---|---|---|---|---|---|---|
| Tolerance +/- mL | 0.04 | 0.05 | 0.05 | 0.06 | 0.07 | 0.07 | 0.08 | 0.10 | 0.12 |
| Tolerance (% of capacity) | 0.4 | 0.3 | 0.3 | 0.2 | 0.2 | 0.2 | 0.2 | 0.1 | 0.1 |

If a flask or pipette does not meet the tolerance criteria then it shall be retested. If the retest value is also outside the tolerance limits then the item shall be removed from the laboratory.

Enter the results in the spreadsheet QualityAssurance/Calibration/Volflask.xls or /VolPipette.xls as appropriate. The raw data should be stored in the appropriate section of the Equipment Calibration file in the Laboratory Manager's office.

## 6 REFERENCES

Australian Standard. Verification and use of volumetric apparatus. Part 1: General - Volumetric Glassware. AS 2162.1 - 1996.

British Standard Schedule for Tables for use in the calibration of volumetric glassware. BS1797:1987.

Vogel, A.I. 1961. A textbook of quantitative inorganic analysis. 3rd Edition. 199 – 200. Longmans, London.

# SOP8 USE OF PIPETTORS, DISPENSERS AND PIPETTES

## 1 INTRODUCTION

While glass pipettes remain the most accurate means of delivering precise volumes of liquid, they are rapidly being replaced by piston-operated volume apparatus - pipettors and dispensers. These have a degree of accuracy and precision approaching that of glass pipettes; however this is very dependent on correct operating technique and regular maintenance.

## 2 PIPETTORS

A button pushes a piston into a cylinder, displacing the air from the internal cylinder through a disposable tip. In the case of pipettes with a variable volume setting, the stroke of the piston is adjustable. When the tip is then immersed in a liquid, this is drawn into the tip when the spring-loaded piston returns to its original position. Depressing the button again discharges the liquid.

Pipettors should be given a quick gravimetric check before each use, and in addition are checked on a 3-monthly basis for accuracy and precision. A coloured sticker attached to the barrel indicates when the next calibration is due.

### 2.1 NORMAL (FORWARD) MODE

2.1.1 Fit the tip.

2.1.2 Set the volume.

2.1.3 Hold the pipette in a vertical position.

2.1.4 Immerse the tip about 2 to 5 mm in the sample.

2.1.5 Depress the button with the thumb smoothly to the first stop.

2.1.6 Allow the button to retract smoothly and slowly to the rest position, observing the filling operation. There should be no turbulence developed in the tip.

2.1.7 After a pause of about one second, take the thumb off the button.

2.1.8 Slowly withdraw the tip straight up from the liquid.

2.1.8 If there are droplets adhering to the exterior of the tip, wipe the tip with a single downward stroke with a tissue, being careful not to allow the tissue to contact the tip orifice.

2.1.10 Place the tip against the wall of the receiving vessel at a 10 ° - 45 ° angle.

2.1.11 Place the thumb on the button and depress it smoothly to the first stop.

2.1.12 After a pause of one second, depress the button smoothly and swiftly to the second stop whilst sliding the tip up the wall and out of the vessel. Observe that the thread of liquid delivered is continuous.

2.1.13 Release the button and observe the tip for residual liquid.

### 2.2 REVERSE MODE

In reverse pipetting, the blowout stroke is used to draw in an additional volume of liquid. This technique gives better results on viscous or volatile liquids. It takes a good deal more skill to use this technique and there is a greater risk of getting liquid up inside the pipette itself.

2.2.1 Fit the tip.

2.2.2 Set the volume.

2.2.3 Hold the pipette in a vertical position.

2.2.4 Immerse the tip about 2 to 5 mm in the sample.

2.2.5 Depress the button with the thumb smoothly to the **second** stop.

2.2.6 Allow the button to retract smoothly and slowly to the rest position, observing the filling operation. This time a larger volume than the set value enters the tip.

2.2.7 After a pause of about one second, take the thumb off the button.

2.2.8 Slowly withdraw the tip straight up from the liquid.

2.2.9 If there are droplets adhering to the exterior of the tip, wipe the tip with a single downward stroke with a tissue, being careful not to allow the tissue to contact the tip orifice.

2.2.10 Place the tip against the wall of the receiving vessel at a 10 ° - 45 ° angle.

2.2.11 Place the thumb on the button and depress it smoothly to the **first stop only**.

2.2.12 Discard the liquid retained in tip by pressing the button to the second stop in a waste vessel.

## 2.3 TROUBLESHOOTING

| **Fault** | **Cause** | **Rectification** |
|---|---|---|
| Leaking and/or dripping | High vapour pressure liquid | Prewetting, reverse mode |
| | Tip not firmly on | Push tip firmly home |
| | Wrong tip | Use genuine tip |
| | Crack in tip | Replace tip |
| | Loose mechanical connection | Tighten securely |
| | Dirty nose cone | Clean |
| | Scratched nose cone | Replace lower part |
| | Dirty piston/cylinder | Clean and regrease |
| | Damaged piston seal | Replace seal and regrease |
| Residual liquid in tip | Pipetting too quickly | Pipette more slowly |
| | Sample with poor wetting properties | Reverse pipette |
| | Dirty tip | Use new tip |
| | Tip with poor wetting properties | Use genuine tip |
| Stiff pipetting button | Swollen seal | Ventilate seal |
| | Dirty piston/cylinder/seal | Clean and regrease |
| | Dried out grease | Clean and regrease |
| | Damaged piston/cylinder | Replace piston/cylinder |
| Volume dispensed inaccurate or not reproducible | Residual liquid in tip | See above |
| | Leaking pipette | See above |
| | Different conditions to when pipette was calibrated: | |
| | - Position of pipette | Hold vertically |
| | - Tip | Use genuine tip |
| | - Density of sample liquid | Re-calibrate |
| | - Temperature of sample | Allow sample to reach room temperature |
| | Calibration no longer correct e.g. after cleaning or replacing components | Re-calibrate |

# 3 DISPENSERS

To avoid cross-contamination, most dispensers and bottles are dedicated for use with a specific reagent. Dispensers are checked on a 3-monthly basis for precision. A coloured sticker indicates when the next calibration is due.

3.1 Empty the reservoir vessel and rinse with deionised water then a small amount of the liquid to be dispensed.

3.2 Fill the reservoir vessel with the liquid to be dispensed and screw the dispenser assembly in place on top.

3.3 Loosen the screw on the volume setting system and slide the indicator to the desired volume setting. Lock the screw again.

3.4 Prime with several slow up and down plunger strokes.

3.5 Smoothly lift the plunger until it meets the indicator stop.

3.6 Pause slightly to check that there is no air inside the tip then gently push the plunger down until it bottoms firmly. **With the yellow Optifix dispensers, allow the plunger to move down by its own weight without pushing**. Any droplets on the tip should be blotted off into the receiving vessel.

3.7 Dispense an aliquot into a measuring cylinder to check the volume dispensed and adjust if necessary.

3.8 When finished, empty the reservoir and rinse thoroughly with RO water.

3.9 Wash the dispenser assembly in RO water.

3.10 Fill the reservoir with RO water and screw the dispenser assembly back on.

3.11 Prime with several strokes to ensure the unit is left full of water.

# 4 PIPETTES

Every glass pipette is gravimetrically tested for accuracy and indelibly numbered before being put into use. Annual visual checks are made for chips or cracks which may affect the volume delivered.

Transfer pipettes have a single mark and deliver a constant volume. Graduated pipettes have a series of marks and are used to deliver predetermined variable amounts. All glass pipettes in this laboratory are "delivery" rather than "blowout" models i.e. do not blow out any liquid remaining in the tip.

**For safety reasons, all pipetting is to be done using a filling device, never by mouth suction.**

4.1 Rinse the pipette inside and out with a stream of deionised water from a wash bottle.

4.2 Dry the outside of the pipette with a clean paper towel.

4.3 Blot out the remaining liquid in the tip with a paper towel.

4.4 Compress the suction bellows of the Brand pipette filler.

4.5 Hold the pipette near its upper end and insert it into the adaptor until it fits tightly. Never use force; pipettes are thin and liable to break causing injury. Once secured, always hold the pipette in a vertical position with the tip down.

4.6 Immerse the pipette tip into the liquid.

4.7 Press the pipetting lever slowly upwards and draw up liquid to the base of the bulb section. The further up the lever is pressed the stronger the suction and the faster the liquid rises.

4.8 Remove the pipette from the adaptor and cover the top of the pipette with a finger.

4.9 Tip the pipette horizontally and rotate to rinse the inside with liquid, and then discard.

4.10 Repeat steps 4.4 to 4.7 to fill the pipette so that the level of liquid is slightly above the mark. Never allow the liquid to enter the filling device. If liquid does enter, change the filter.

4.11 Although lowering the liquid level to the mark can be achieved by pressing the filling lever downwards, better control is achieved by removing the pipette from the adaptor and covering the end with a finger tip.

4.12 Allow the liquid to run out by relaxing the finger pressure and gently rotating the pipette until the liquid meniscus is level with the mark. During this stage the pipette tip should be kept in contact with the container side.

4.13 Increase the finger pressure and shift the pipette over into the receiving vessel.

4.14 Lift the finger off fully to allow the liquid to run out into the receiving vessel, with the pipette tip touching the side of the receiving vessel all the time.

4.15 When the discharge stops, maintain contact with the side for a further 5 seconds to allow the pipette to drain fully. Do not blow out any drops remaining in the tip.

# 5 GOOD PRACTICE TIPS

## 5.1 GENERAL

5.1.1 To avoid cross contamination, transfer approximately the amount of liquid to be pipetted from its original container to a clean beaker or P35 vial to pipette from.

## 5.2 GLASS PIPETTES

5.2.1 Store pipettes on a stand to minimise the risk of tip breakage.

5.2.2 Always rinse a pipette with deionised water before use, and then wipe the exterior with a clean paper towel.

5.2.3 Rinse the pipette with a small quantity of liquid to be pipetted then discard.

5.2.4 To avoid sudden air intakes and liquid being sucked into the filling device, keep the tip of the pipette slightly angled but in contact with the bottom of the container.

5.2.5 A slightly moist finger tip gives more control when lowering the meniscus to the mark

## 5.3 PIPETTORS

5.3.1 Store pipettors in their boxes in a drawer.

5.3.2 Use only tips specified by the manufacturer. Poor quality tips may not fit properly, may not wet up evenly, or contain contaminants such as heavy metals.

5.3.3 Pipetting operations should be carried out at room temperature, with the sample liquid at room temperature.

5.3.4 The pipette should always be held vertically when drawing up sample otherwise the height of the liquid column will be lower than when the pipette was calibrated, causing too much liquid to be drawn in.

5.3.5 The immersion depth should never be more than 5 millimetres otherwise the pressure conditions in the tip are greater than when the pipette was calibrated, causing too much liquid to be drawn in. A low immersion depth also minimises carryover on the external surface of the tip.

5.3.6 When liquid is dispensed from a tip a thin film of liquid remains clinging to the wetted inner surface. Prewetting of the tip, especially for liquids other than water, is advisable to saturate the tip surfaces first.

5.3.7 If the same liquid type is pipetted at short intervals (less than 5 minutes) the tip does not need to be replaced as long as flow characteristics are problem-free i.e. no formation of droplets.

5.3.8 Liquids with a high vapour pressure, such as ethanol, give up some of their mass to the gaseous phase in the tip. This increases the gas pressure and liquid is forced out as drips. Prewetting the tip once is usually sufficient to saturate the vapour space. Reverse pipetting is better for these types of liquids.

5.3.9 Water is normally used for calibration. If a liquid of rather different density is to be pipetted the pipette must be calibrated specifically for this. For example with sulphuric acid the volume drawn in is too little. With dense liquids too it is advisable to fir the tip very firmly since there is greater risk of leakage.

5.3.10 Each pipettor should be given a quick gravimetric test before use by pipetting the nominal volume first. Remember, the weight will be a little less than the volume due to the temperature of the liquid. A full calibration check as described in Procedure 509 Verification of Pipettors and Dispensers should be carried out every three months.

### 5.4 DISPENSERS

5.4.1 Maximum reproducibility is achieved when a slow steady stroke is used. Excessive speed may cause the unit to pre-dispense or post-dispense a droplet.

5.4.2 Always lift or carry a dispenser using one hand beneath the bottle.

5.4.3 Regularly wipe the dispenser with a damp tissue to remove any salt deposits.

5.4.4 Never leave a dispenser empty without first cleaning thoroughly with water otherwise the valves may jam. If this does happen, soak in water to loosen.

## 6 REFERENCES

Australian Standard. Verification and use of volumetric apparatus. Part 2:Guide to the use of piston-operated volumetric apparatus. AS 2162.2 - 1998.

Operating Instructions. Gilson Pipetman P. Gilson Medical Electronics, France.

Good Pipetting Practice. Mettler Toledo GmbH, Switzerland.

Brand Macro Pipette Controller Operating Manual. Brand GmbH, Wertheim, Germany.

Optifix Bottle Top Dispenser Operating Manual. Poulten & Graf GmbH, D-97877 Wertheim, Germany.

# SOP9 PREPARATION OF STANDARDS & REAGENTS

## 1 INTRODUCTION

Correct standards and reagents are essential in achieving accurate results. This involves both the use of equipment calibrated or verified according to schedule, and good laboratory technique.

There are two types of standards – primary and working. Primary standards are normally a single element or compound at a specific high-level concentration such as 1000 mg/L, whereas working standards are at a lower level, covering the range of concentrations expected in the samples. Metal primary standards are usually purchased from a commercial supplier while non-metal primary standards are prepared in the laboratory from the purest grade of chemical available. Working standards are prepared by the analyst in the same matrix as the samples.

All chemicals, including standards, are dated upon receipt, and replacements are typically purchased before the last of the material is used up. To ensure traceability, a detailed log is kept recording the details of standard preparation such as the brand and grade of chemicals used, matrix, date and initials of the person preparing it.

## 2 PRIMARY STANDARDS

### 2.1 METALS

2.1.1 Use BDH Spectrosol commercial standards, 1000 mg/L.

2.1.2 Date upon receipt and store in the dark in a cupboard.

### 2.2 NON-METALS

2.2.1 Use the chemical compound specified in the method manual. Aristar, AnalaR or UniVar grades should be used if possible. Be very careful to check the formula on the container against the manual e.g. watch out for things like $Na_2HPO_4$ and $NaH_2PO_4$, di-sodium and tetra-sodium EDTA, and waters of hydration ($CuSO_4$ and $CuSO_4.5H2O$).

2.2.2 Follow the directions in the method manual as to pre-treatment - some chemicals need to be dried at 105 °C for 1 hour prior to weighing out. If this is the case, weigh out slightly more than the required weight into a clean glass weighing boat and dry, rather than drying the entire container.

2.2.3 Weigh the required amount of chemical into a disposable weighing boat on the 4-place balance.

2.2.4 Transfer the entire contents of the weighing boat via a funnel into the volumetric flask. Rinse both the weighing boat and funnel into the flask with the matrix solution.

2.2.5 Fill the flask approximately three-quarters full with matrix solution, stopper and mix by inverting until the solid has dissolved.

2.2.6 Make to the mark with matrix solution, using a disposable transfer pipette to add the final few drops accurately.

2.2.7 Label the flask with the name of the standard, its concentration in mg/L, the matrix, the date of preparation and your initials.

2.2.8 Write the expiry date (from Table 1 at the end of this procedure) on a yellow dot sticker and attach to the flask.

2.2.9 Store in the standards refrigerator unless specified otherwise in the method.

2.2.10 Record the preparation details in the Standards Log, kept on top of the fridge.

## 3 WORKING STANDARDS

Working standards are those used to prepare a calibration. Sometimes the step down in concentration from primary to working standard requires the use of an intermediate level standard.

3.1 Follow the instructions given in the method for preparing intermediate and working standards. Be sure to check the volumetric flask sizes carefully – some methods use one size for the full range whilst others may use a combination of flask sizes depending on the amounts used in calibration.

3.2 All transfers are to be made using pipettes or pipettors. Refer to SOP8 for the correct use of this equipment.

3.3 Use hand pipettors for volumes of 5-mL or less. Ensure that the pipettor calibration is current by referring to its sticker. Do a quick gravimetric check before use. Use a clean tip with each new standard pipetted.

3.4 Use a glass pipette for volumes above 5-mL. Check the pipette tip for signs of damage before use. Rinse with and discard a small amount of the liquid to be pipetted before filling to the mark.

3.5 Never pipette directly from volumetric flasks – transfer the approximate amount to a beaker or P35 vial.

3.6 Fill the flask approximately quarter-full with matrix solution first, then pipette the required amount of standard in.

3.7 Make to the mark with matrix solution, using a disposable transfer pipette to add the final few drops accurately.

3.8 Label the flask with the name of the standard, its concentration in mg/L and the matrix. Label the intermediate standard (if any) and the top standard in the working standard range with the date of preparation and your initials.

3.9 Write the expiry date (from Table 1 at the end of this procedure) on a yellow dot sticker and attach to the flask.

3.10 Store in the standards refrigerator unless specified otherwise in the method.

3.11 Record the preparation details in the Standards Log, kept on top of the fridge.

# 4 CHECKING NEW STANDARDS

Whenever a new set of standards is prepared, a comparison must be made between the old set and the new set. Some difference may be expected, especially if the old set are past their expiry date, due to factors such as concentration by evaporation, contamination (e.g. dissolution of atmospheric gases such as CO2 or ammonia) and biological activity (e.g. conversion of nitrite to nitrate.) These differences though should be small; what is being looked for is evidence of gross preparation errors such as use of the wrong volume pipette or flask.

4.1 Prepare a calibration using the new set of standards.

4.2 Run the old set of standards as samples.

4.3 Examine the calibration curve must be examined for linearity fit by checking the $r^2$ (should be better than 0.995) and the residuals.

4.4 Use the top standard from each set to calculate the difference between the old and new set:

$$\% \text{ difference} = \frac{\text{Conc (new)} - \text{Conc (old)}}{\text{Conc(new)}} * 100$$

If the difference is greater than 5% then the new standards must not be used until an investigation is carried out as to whether there has been an error in preparation or whether the old standards have deteriorated significantly. Record the investigation details as a quality problem report (see Section 14 – Non-conforming work in Part I, Quality Manual.)

4.5 File a copy of the curve and the results in the Standards Log, kept on top of the fridge.

# 5 REAGENT PREPARATION

In preparing reagents, accuracy is not as critical as for standards; however good laboratory practice and technique should still be used. Measuring cylinders, beakers, and calibrated lines on containers are adequate for volume measurements.

Small amounts of reagent remaining in a container should be discarded rather than "topping up" with fresh reagent.

When dissolving solid chemicals a magnetic stirrer should be used to ensure complete dissolution. Use about 10 % less than the final volume until the solid has dissolved, then make to the mark and stir briefly again. Remove the magnetic stirrer once preparation is complete, as the reagent may degrade it.

Similarly, when adjusting the pH of a reagent, use about 10 % less than the final volume till the correct pH is achieved then make to the mark and stir briefly again. Where reagents heat up during preparation, such as with some buffers, the reagent should be allowed to cool to room temperature before pH adjustment.

After preparation, write the expiry date (from the tables at the end of this procedure) on a yellow dot sticker and attach to the reagent container.

# 6 STORAGE TIMES

Storage times and conditions for standards, extractants, eluents and reagents vary depending on the chemical, the matrix, any preservatives used and the concentration. Critical storage times are normally specified in the individual methods, but Tables 1 - 3 below can be used as a general guide.

Some reagents require refrigeration while others do not. All standards, apart from commercial metal standards, should be stored in the refrigerator. Any refrigerated solutions should however be allowed to come to room temperature before use.

Table 1. Recommended maximum storage times for analytical standards.

| Concentration or type | Analyte | Maximum storage time |
|---|---|---|
| - | pH buffers | 1 month |
| 0.01 M KCl | KCl (conductivity ) | 1 month |
| 1000 mg/L | Metals – commercial stock | Expiry date on bottle |
| Intermediate | Metals | 3 months |
| Working | Metals | 1 week |
| 1000 mg/L | $NH_4$-N, $NO_3$-N, $PO_4$-P | 6 months |
| 500 mg/L | NO2-N | 1 month |
| Working | $NH_4$-N, $NO_3$-N, $PO_4$-P, NO2-N | 1 week |
| Low level | $NH_4$-N, $NO_3$-N, $PO_4$-P, NO2-N | 1 day |
| Primary | Kjeldahl N P K Ca Mg Al | 12 months |
| Working | Kjeldahl N P K Ca Mg Al | 6 months |
| 100 mg/L | Olsen P | 1 month |
| Working | Olsen P | 1 week |
| Working | 0.5M $H_2SO_4$ P | 6 months |
| Working | P retention | New per batch of P retention solution |
| 1000 mg/L | $SO_4$-S | 6 months |
| Working | $SO_4$-S | 1 week |
| Primary | Bases in ammonium acetate | 3 months |
| Working | Bases in ammonium acetate | 1 months |
| Primary | CEC | 6 months |
| Working | CEC | 3 months |
| Working | Extractable Fe, Al & Si | 6 months |
| Working | DTPA Fe, Mn, Cu & Zn | 1 week |
| Primary | Boron | 1 month |
| Working | Boron | 1 week |
| 1000 mg/L | Total carbon, inorganic carbon | 3 months |
| Working | Total carbon | 1 week |
| Working | Inorganic carbon | Fresh daily |
| 1000 mg/L | IC anions | 6 months |
| Intermediate | IC anions | 3 months |
| Working | IC anions | 1 week |

Table 2. Recommended maximum storage times for colorimetric reagents.

| **Method** | **Reagent** | **Maximum storage time** | **Storage** |
|---|---|---|---|
| Ammonia & CEC | Salicylate/citrate mixed reagent | 1 month | Room |
| | DCIC reagent | 2 weeks | Room |
| Bases | Strontium-cesium solution | 12 months | Room |
| Nitrate & nitrite | Ammonium chloride buffer | 3 months | Room |
| | Sulphanilamide | 1 month | Room |
| Phosphate | Ammonium molybdate solution | 3 months | Refrigerator |
| | Antimony potassium tartrate solution | 3 months | Refrigerator |
| | Molybdate colour reagent | 1 month | Refrigerator |
| | Ascorbic acid | 2 weeks | Refrigerator |
| Kjeldahl nitrogen | Buffer | 3 months | Room |
| | Salicylate/nitroprusside | 1 month | Room |
| | Hypochlorite solution | 2 weeks | Room |
| Boron | Buffer-masking agent | 1 month | Room |
| Boron | Azomethine-H | 2 weeks | Fridge |
| Boron | Mixed buffer/azomethine H reagent | Daily | Fridge |
| Chloride - plants | 0.02N mercuric nitrate | 6 months | Room |
| Chloride - plants | Diphenylcarbazone indicator | 1 month | Fridge |
| Chloride - water | Ferric ammonium sulphate solution | 6 months | Room |
| Chloride - water | Mercuric thiocyanate solution | 6 months | Room |

Table 3. Recommended maximum storage times for extractants, eluents & other reagents.

| **Method** | **Reagent** | **Maximum storage time** |
|---|---|---|
| pH | 1M KCl | 6 months |
| pH | 0.01M $CaCl_2$ | Daily |
| C & N extractions | 2M KCl | 6 months |
| Olsen P | 0.5M $NaHCO_3$, check pH before use | 1 month |
| Bray P | 0.3M $NH_4F$/0.1M HCl | 12 months |
| Acid sol & Ig P | 0.5M $H_2SO_4$ | 6 months |
| Extractable sulphate | 20 mM $KH_2PO_4$ | 1 month |
| IC anions | $NaHCO_3$ stock | 1 month |
| IC anions | $Na_2CO_3$ stock | 1 month |
| IC anions | Mixed $NaHCO_3$/$Na_2CO_3$ eluent | Daily |
| Exchangeable bases | 1M pH 7.0 ammonium acetate | 6 months |
| Exchangeable bases | Strontium-cesium solution | 12 months |
| CEC | 90% ethanol | 12 months |
| CEC | 1M NaCl | 6 months |
| Extractable Al | 1M KCl | 6 months |
| DTPA metals | 0.01M & 0.005M DTPA | 3 months |
| Oxalate metals | 0.2M ammonium oxalate pH 3.0 | 3 months |
| Pyrophosphate metals | 0.1M sodium pyrophosphate | 3 months |
| Dithionite-citrate metals | 22% sodium citrate | Daily |
| Biomass C, N & P | Washed chloroform | Daily |
| Biomass C & N | 0.5M $K_2SO_4$ | 12 months |
| Biomass N | 0.165M $K_2S_2O_8$ | Daily |
| Particle size | 0.11M sodium hexametaphosphate pH 8 | 6 months |
| Plant kjeldahl digest | Sulphuric/salicylic acid | 6 months |
| Lignin | Acid detergent | 12 months |
| Total N & P - water | 0.225M NaOH/0.15M $K_2S_2O_8$ | Daily |

# SOP10 SAFE USE OF COMPRESSED CYLINDER GASES

## 1 INTRODUCTION

Compressed gases can be toxic, flammable, oxidizing, corrosive, inert, or some combination of these hazards. In addition to the chemical hazards, the amount of energy resulting from the compression of the gas makes a compressed gas cylinder a potential rocket. Finally, cylinders are also very heavy. Appropriate care in the handling, storage and use of compressed gas cylinders is essential.

Know and understand the properties, uses, and safety precautions before using any gas or gas mixture. Consult Material Safety Data Sheets (MSDSs) for safety information on the gases that you will be using.

## 2 PRIMARY HAZARDS

The following is an overview of the primary hazards to be avoided when handling and storing compressed gases:

- **Asphyxiation:** Simple asphyxiation is the primary hazard associated with inert gases. Because inert gases are colourless and odourless, they can escape into the atmosphere undetected and quickly reduce the concentration of oxygen below the level necessary to support life.

- **Fire and Explosion:** Fire and explosion are the primary hazards associated with flammable gases, oxygen, and other oxidizing gases. Flammable gases can be ignited by static electricity or by a heat source, such as a flame or a hot object. Oxygen and other oxidizing gases do not burn, but will support combustion of flammable materials. Increasing the concentration of an oxidizer accelerates the rate of combustion. Materials that are non-flammable under normal conditions may burn in an oxygen-enriched atmosphere.

- **Chemical Burns:** Corrosive gases can chemically attack various materials, including fire-resistant clothing. Some gases are not corrosive in their pure form, but can become extremely destructive if a small amount of moisture is added. Corrosive gases can cause rapid destruction of skin tissue.

- **Chemical Poisoning:** Chemical poisoning is the primary hazard of toxic gases. Even in very small concentrations, brief exposure to these gases can result in serious poisoning injuries. Symptoms of exposure may be delayed.

- **High Pressure:** All compressed gases are potentially hazardous because of the high pressure stored inside the cylinder. A sudden release of pressure can cause injuries by propelling a cylinder or whipping a line.

- **Improper Handling of Cylinders:** Compressed gas cylinders are heavy and awkward to handle. Improper handling of cylinders could result in sprains, strains, falls, bruises, and broken bones. Other hazards such as fire, explosion, chemical burns, poisoning and cold burns could occur if gases accidentally escape from the cylinder due to mishandling.

# 3 REMOVING AN EMPTY GAS CYLINDER

3.1 Close the main cylinder valve.

3.2 Wind the regulator valve out to release the pressure. As there may still be pressurised gas in the line, vent any residual gas via the instrument if possible.

3.3 Use a cylinder spanner to undo the regulator where it screws into the main cylinder valve. Remember, all gases except acetylene are right hand thread i.e. turn in an anti-clockwise direction to loosen. If necessary, loosen with a gentle tap from a hammer on the spanner.

3.4 Place the regulator with its attached gas lines somewhere secure so that it will not fall and get damaged whilst the cylinders are being moved.

3.5 Undo the chain securing the cylinder to the bench.

3.6 With the cylinder trolley in front of you, tilt the cylinder back so that trolley can be slid underneath the base.

3.7 Lower the cylinder gently down onto the trolley and fasten the trolley chain.

3.8 Obtain the key to the cylinder cage from the Laboratory Manager's office.

3.9 Open the cage and ensure a clear path through to where the empty cylinder will be placed.

3.10 Wedge open the doors leading out to the cage.

3.11 Wheel the cylinder on the trolley out to the cage and up the ramp at a steady speed.

3.12 Position the cylinder against the wall and undo the trolley chain.

3.13 Tilt the cylinder back and free the trolley from beneath.

3.14 Manoeuvre the cylinder into position and secure the restraining chain.

3.15 Place an "Empty" tag inside the loop of restraining chain.

3.16 Notify the technician responsible for purchasing, preferably by email as well as verbally, that an empty cylinder has been placed in the cage. Make sure you specify what the gas was (cylinder size, gas, grade) and whether you wish a replacement to be ordered or just the empty to be taken away.

# 4 FITTING A NEW GAS CYLINDER

4.1 With the cylinder trolley in the cage in front of you, tilt the cylinder back so that trolley can be slid underneath the base.

4.2 Lower the cylinder gently down onto the trolley and fasten the trolley chain.

4.3 Wheel the trolley out of the cage at a steady speed.

4.4 Lock the cage, and return the key to the Laboratory Manager's office.

4.5 Wheel the trolley back to the lab, position the cylinder where it will be used and undo the trolley chain.

4.6 Tilt the cylinder back and free the trolley from beneath.

4.7 Manoeuvre the cylinder into position and secure the restraining chain.

4.8 Remove the plastic insert from the cylinder valve.

4.9 Wipe inside the valve with a clean tissue.

4.10 Connect the regulator head to the cylinder valve by closing the thread finger tight. The thread should move easily; if not it is either damaged or you are not holding it at the right angle. Remember, all gases except acetylene are right hand thread i.e. turn in a clockwise direction to tighten.

4.11 Use a cylinder spanner to tighten the connection, but don't use excessive force.

4.12 Open the cylinder valve and use a disposable pipette to drip detergent solution around the connection to check for leaks.

4.13 If there are no leaks, wind the regulator valve in to set the required delivery pressure.

4.14 Leak test all other connections on lines around the valve and regulator.

4.15 Note the cylinder change in the instrument log if appropriate, and record the initial cylinder pressure.

# 5 HANDLING, STORAGE, AND USE TIPS

- Only persons familiar with the hazards should handle compressed gas cylinders.
- Close the main valve and wind the regulator valve out when work is finished.
- Keep the number of gas cylinders in the laboratory to a minimum. Remove empty cylinders to the cage outside promptly.
- It is preferable to leave a slight positive pressure rather than empty a cylinder completely. Acetylene cylinders to the atomic absorption spectrophotometer must be changed when the cylinder pressure drops to 100 psi to prevent acetone from entering the gas line and damaging the instrument.
- All cylinder movement should be done using the cylinder trolley and using the chain to secure the cylinder.
- Place an "Empty" tag under the securing chain of an empty cylinder when placing in the cage for pick-up.
- Leave the cylinder cage locked at all times except when transferring cylinders. The key is kept in the Lab Manager's office.
- Wear appropriate work shoes when moving cylinders to avoid crush injuries.
- Store cylinders stored upright and secured at all time i.e. both when in use and when outside in the cylinder cage.
- Never use a cylinder without a regulator. The practice of "cracking" a cylinder to flush the valve is not acceptable.
- Wear safety glasses when removing and fitting regulators.
- Make sure the threads on both the regulator and cylinder valves are clean and in good condition.
- Don't use Teflon thread tape on connections. If a connection still leaks after cleaning the threads then service is needed.
- Leak test lines and regulators with a detergent solution after a cylinder change.
- Always open a cylinder only slightly to start gas flow, and then check for leaks before opening one full turn.
- Never open the main valve all the way. The valve may stick open and make it difficult to determine whether the valve is open or closed.
- Compressed gas cylinders should not be subjected to any mechanical shock that could cause damage to their valves or pressure relief devices. Cylinders should not be dropped, dragged, slid, or used as rollers for moving material or other equipment.
- Cylinder caps perform two functions. First, they protect the valve on the top of the cylinder from damage if it is knocked over. Second, if gas is accidentally released through the valve, the cap will vent the gas out of both sides, minimizing the likelihood that the cylinder will topple. If fitted, caps should not be removed until the cylinder is secured in place and ready for use.
- Cylinders should not be stored near radiators or other heat sources.
- Cylinders should not be exposed to sparks, flames, or high temperatures.
- Cylinders should not be places where they could come into contact with any electrical apparatus or circuits.
- Smoking and open flames is not permitted in or near the gas cage, nor any laboratory areas.
- Never permit oil, grease, or other combustible substances to come into contact with cylinders, valves, and systems.

- When returning an empty cylinder, close the valve before shipment.
- Replace the cylinder cap if originally shipped with the cylinder.
- If repair is needed on a cylinder or its valve, mark it and notify the technician responsible for gas purchasing.
- Repairs to regulators are only to be carried out by authorised BOC agents. Contact details are in the Approved Suppliers Register kept in the Laboratory Manager's office.

# 6 HANDLING OF LEAKING CYLINDERS

Most leaks occur at the valve in the top of the cylinder and may involve the valve threads, valve stem, valve outlet, or pressure relief devices. Do not attempt to repair leaking cylinders.

Where action can be taken without serious exposure, move the cylinder to an isolated, well-ventilated area (away from combustible materials if the cylinder contains a flammable or oxidizing gas) and contact the Fire Service.

If a large or uncontrollable leak occurs, evacuate the area immediately and contact the Fire Service.

# SOP11 PREPARATION OF IN-HOUSE CONTROL SAMPLES

## 1 TIMING

The following steps should be carried out at least six months before the previous Laboratory Control Sample (LCS) runs out, to allow time for the data to be accumulated and for the old and new LCSs to be run together for at least 10 times for each property measured. This gives confidence in the new values and ensures continuity of high quality results.

## 2 PROCEDURE

### 2.1 SOIL

The soil LC sample should be a soil that has mid-range values for most of the properties measured and is easily accessible. Collect a large sack full (approximately 50 kg).

- Mix the bulk sample in the sack or on a large bench top.
- Separate into a number of sample trays and dry at 40°C for 48 hours.
- Grind the sample to < 2 mm with a stainless steel roller mill.
- Mix well in a large container or on a bench top.
- Keep one bag in the sample weighing room and use to top up the LC sample jar before it gets empty.
- Place the remainder of the sample into a number of large plastic bags and store in a cool, dry place.

It is important to mix the sample in the bag before refilling the jar, and to mix the sample in the jar before weighing out aliquots for analysis because < 2 mm ground air-dry soil tends to separate according to particle size. This can lead to erroneous results if an atypical distribution of particle sizes is taken for an analysis.

### 2.2 PLANT

The plant LC sample should be a plant material that has mid-range values for most of the properties measured and be easily accessible. The sample should represent the same plant part, not a mixture of leaves and stems for example. A large quantity is needed so collect a number of large sacks full (approximately 20 kg).

- Dry the sample at 80°C for 16 hours.
- Grind in the Udy plant mill using the 1-mm screen.
- Mix the total sample well in a large container or on a bench top.
- Keep one bag in the sample weighing room and use to top up the LC sample jar before it gets empty.
- Place the remainder of the sample into a plastic bag in a large plastic bucket and store in the cold room.

It is important to mix the sample in the bag before refilling the jar, and to mix the sample in the jar before weighing out aliquots for analysis in case of any separation of the sample by particle size on standing.

## 3 SETTING TARGET VALUES

For each of the measured properties the new LC sample is analysed at least 20 times in different batches and by a number of different analysts. Where possible, include in a laboratory sample exchange round robin, or send to one or two other trusted laboratories which use similar methods.

Results are stored in the appropriate LCS spreadsheets, which are used to calculate mean and standard deviation for each parameter. Values that are considerably outside the range of the mean +/- 1 standard deviation are dropped from the data set.

The spreadsheets are used to calculate the upper and lower LC limits for each parameter. Typically, 2 standard deviations from the mean is used as a warning limit and 3 standard deviations from the mean as a control or reject limit. For some analyses these limits may be too wide and allow an unacceptable spread of results. Alternatively here, a lower limit is used for the warning limit, such as +/- 1.6 standard deviations from the mean. This represents the range that spans 90% of a normally distributed set of values and means that for one run in ten the LC sample will be outside the acceptable range even though the analyst has carried out the analysis correctly. If however the limits are relaxed to +/- 2 standard deviations, one in 20 runs will be outside the warning limits.

## 4 LCS MATERIALS CURRENTLY IN USE

- For most routine soil analyses, the standard check sample **X** is used. It is available in both < 2 mm and < 0.25 mm particle size, depending on how much sample is taken for analysis.
- For Olsen-soluble phosphorus, the standard check sample **Y** is used.
- For most routine plant analyses, the standard check sample **Z** is used.

# SOP13 CLEANING UP PROCEDURES

## 1 INTRODUCTION

The final step in all experimental work is cleaning up. It should be done promptly so that the equipment is available for the next user, and properly so that the quality of future results is not compromised. Good housekeeping also plays an important role in reducing the frequency of laboratory accidents.

## 2 DISPOSAL

- Used filter papers to go in a lined bin.
- Used needles must be placed in the Sharps container.
- Dry solids to go in a lined bin.
- Wet solids, such as soil/extractant mixtures to go into the labelled bucket (with a lid) and left to settle. Supernatant liquid is regularly decanted off down the sink with running water. Wet residue to be scraped out into a plastic bag and disposed of in a lined bin.
- Liquids to be poured down the sink with large volumes of running tap water.
- Water-immiscible liquids such as xylene should be placed in a shallow container, appropriately labelled as to the contents, and left to evaporate in the fume hood with the extraction fan running.
- Disposable plasticware, such as P35 vials and plastic test tubes, to be emptied in the sink with running water then to go in a lined bin.

## 3 CLEANING

3.1 Containers that have held a soil/extractant/digest mixture should be rinsed and then left to soak in a plastic basin containing detergent and hot water. (If the containers are dedicated for phosphate determination then Decon 90 should be used in place of detergent.) The containers must then be scrubbed with a bottlebrush to remove any adhering material.

3.2 Rinse all items three times with cold tap water and then twice with RO water. Alternatively, use the laboratory dishwasher.

3.3 Dry volumetric glassware by inverting on one of the drying racks beside the sink. Other containers and equipment can be placed in a basket and dried at 50 °C in the Equipment Oven beside the main door.

3.4 When dry, return the containers and equipment to the appropriate storage locations.

# EXAMPLE INSTRUMENT OPERATING PROCEDURE I

# SOP12 HANNA HI 8820 CONDUCTIVITY METER OPERATION

## 1 INTRODUCTION

The HI 8820 is a digital bench top conductivity meter for use in measuring the electrical conductivity in liquids. Four ranges of conductivity measurements are provided and the probe has a built-in temperature sensor to allow for automatic temperature compensation.

## 2 REAGENTS

2.1 STANDARD POTASSIUM CHLORIDE SOLUTION, 0.01 M KCl. Dissolve 0.7456 g of KCl, dried at 105 °C for 2 hours, in water in a 1 L volumetric flask and make to 1L. This is the standard reference solution, which at 25°C has conductivity of 1412 μS/cm.

## 3 CALIBRATION

3.1 Turn the meter on using the ON/Off key at the top left.

3.2 Pour about 75 mL of standard potassium chloride solution (reagent 2.1) into a 100 mL plastic tripour beaker.

3.3 Immerse the conductivity probe in the solution. The liquid level should be above the air escape hole.

3.4 Jiggle the probe up and down to force out any air trapped in the probe.

3.5 Select the conductivity range "1999 μS" by pushing the appropriate range key.

3.6 Check that the arrow on the temperature coefficient knob is pointing to 2.

3.7 Use a small screwdriver to adjust the trimmer until the display reads 1412 μS/cm. (The trimmer is the single slot screw at the top centre of the recessed panel on the rear of the meter.)

3.8 Remove the probe from the solution and rinse the electrode thoroughly both on the sleeve outside and via the air escape hole with deionised water from a squash bottle. For very high conductivity solutions, remove the outer sleeve cover and wash the sleeve and electrode separately.

# 4 SAMPLE MEASUREMENT

4.1 Immerse the probe in a suitable volume of the sample liquid; again making sure that the liquid level is above the air escape hole. This may mean transferring the sample from its extraction container into a narrower container such as a centrifuge tube.

4.2 Record the conductivity reading displayed, together with the units depending on the range selected (either μS/cm or mS/cm).

4.3 If the display shows only a "1" it means the sample is above the currently selected range; in which case the next range key should be selected.

4.4 Remove the probe from the solution and rinse the electrode thoroughly both on the outside and via the air escape hole with deionised water from a squash bottle.

4.5 Check the conductivity standard after every 40 samples and adjust the meter if the reading has drifted by more than 1% e.g. the 1412 μS/cm standard should be within 1398 – 1426 μS/cm. Read an LCS sample again after the standard check.

4.6 After all measurements have been made rinse the probe thoroughly and switch the meter off.

4.6 Fill in the log.

# EXAMPLE INSTRUMENT OPERATING PROCEDURE II

# SOP13 IEC MODEL K CENTRIFUGE OPERATION

## 1 INTRODUCTION

This procedure outlines how to use the IEC centrifuge with the 32-place swinging bucket rotor. It can be used at speeds up to 2,000 rpm with a tube volume of up to 50 mL.

## 2 PROCEDURE

2.1 Turn on power switch on at the wall.

2.2 Open the lid by depressing the brass knob on the top right of the lid and turning the handle clockwise to the 9 o=clock position.

2.3 Check rotor for cleanliness, signs of corrosion, or indication that its speed rating has been downgraded.

2.4 Place samples in tubes with lids in the buckets on the rotor, ensuring that opposing compartments are balanced to within 1g.

2.5 Ensure that the tachometer in the centre of the lid is in the raised position.

2.6 Close the lid and latch the cover by turning the handle anticlockwise to the 6 o=clock position.

2.7 Check that the Brake switch is set to **Off** on the front panel.

2.8 Set the timer to the desired run length.

2.9 Check that the switch is set to **On with timer** on the front panel.

2.10 Set the run length on the timer.

2.11 Set the speed control to 40%. The rotor will start spinning.

2.12 Slowly increase the speed control in about 5% increments, allowing the rotor noise to stabilise each time. Increasing too rapidly may cause the fuse to blow. At present 70% corresponds approximately to 2000 rpm.

2.13 When the rotor noise has stabilised, check the speed with the tachometer. Loosen the thumbscrew on either side and gently press the tachometer casing down until the needle gives a reading. Release the casing to raise the tachometer again and tighten the thumbscrews to secure it in the raised position.

2.14 Adjust the speed control if necessary and check the tachometer agin as above until the desired speed is achieved.

2.15 When the run time has elapsed the motor stops, and the rotor will coast to a halt. The lid will remain locked until the rotor stops spinning, indicated by a loud click. At this point, press the brass knob on the top right of the lid and turn the handle clockwise to the 9 o=clock position to open.

2.16 Remove samples from the buckets.

2.17 Return the speed control knob to zero.

2.18 Wipe out the chamber and lid with a damp cloth. If any liquid has spilled remove and clean the buckets also. Dry and replace in the correct position, as they have been carefully weight-matched.

# 3 TROUBLESHOOTING

3.1 THE CENTRIFUGE WILL NOT START:

3.1.1 The speed control must first be in the zero position. Return it to zero then try gain.

3.1.2 The timer is set to off. Set it to **On with timer**.

3.1.3 The speed sensor has been locked out. This may happen if the speed control has not been turned fast enough towards 40% to allow the rotor to start spinning within 10 seconds. Reset by pressing the timer switch to the **Off** position momentarily.

3.1.4 The fuse may have blown. Switch off the power. Inspect the fuse on the underside of the control box. If necessary, replace with a 5A 240V Slo-Blo, kept in the drawer to the left of the centrifuge.

3.2 THE COVER WILL NOT OPEN:

3.2.1 The cover handle must be turned within 5 seconds of the open knob being pressed. Try again.

3.2.2 The fuse has blown or the power failed during a run. Switch off the power. Remove the plug from the hole on the left side of the control box. Insert a screwdriver into the hole, depress the cover lock plunger and turn the cover handle.