Best Practices Guide for Water Laboratories

SOPAC Technical Report 405

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World Health Organization

nzaid New Zealand's International Aid & Development Agency

SOPAC

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PREFACE

This Manual is written as part of the New Zealand's International Aid and Development Agency (NZAID) funded Regional Water Quality Monitoring Capacity Building Programme (2006-2008) coordinated by the Pacific Islands Applied Geoscience Commission (SOPAC), the World Health Organization (WHO) and the Institute of Applied Sciences (IAS) in response to the Pacific Regional Plan on Sustainable Water Management.

The Manual is intended to be a resource guide for small Pacific Island water laboratories on best practices that can be employed in water laboratories.

The Manual is comprised of two parts:

(1) Part I - outlines the 10 key features that an ideal water monitoring laboratory is recommended to have in order to successfully conduct its role.

(2) Part II - outlines other aspects that are to be addressed in supporting the development of a water monitoring laboratory

The following manuals and technical documents were used to compile this Manual:

(i) SPACNET Generic Quality Assurance Manual, 13/03/2003
(ii) IAS Quality Manual 2007
(iii) IAS Standard Operating Procedures 2007
(iv) NZS/ISO:IEC 17025:2005 General requirements for testing and calibration laboratories
(v) IANZ Technical Guide – Balances
(vi) IANZ Technical Guide – Thermometers
(vii) General requirements for Standford University Laboratories, Environmental Health and Safety, October 2003

INTRODUCTION

The principal function of a water analytical laboratory is to produce high quality data that are accurate, reliable and adequate for the intended purpose. This can only be achieved if the key features of an ideal water Laboratory such as Proper Laboratory Space, Calibration of Equipment, Reagent Supply and others as those outlined in Part I of the Manual are present. The funding of the Laboratory as addressed in Part I is also important in ensuring the implementation of these key features.

The Part II of the Manual addresses other aspects of the Laboratory operations that could be implemented to ensure that its functions are managed in a thorough, safe and cost effective manner such as defining Staff Responsibilities, Control of Nonconforming Work, Safety in the Laboratory and Internal Audits. Part II of the Manual also contains certain important technical procedures such as Sampling, Handling and Storage of Samples and Analytical Standards Preparation.

PART

I

Chapter 1 PROPER LABORATORY SPACE/DESIGN

1.0 PROPER LABORATORY SPACE

An ideal Water Laboratory will separate the chemical and physical analysis work from microbiological analysis. Ideally, the Microbiological analyses are to be conducted in a separate room from physical and chemical analysis work. This minimizes the contamination of samples for Microbiological analysis and also ensures that chemicals and media are segregated and better organized.

2.0 PROPER LABORATORY DESIGN AND LAYOUT

2.1 Building Design Issues

Due to the handling and storage of hazardous materials which carry a higher risk of exposure and injury, laboratory and non-laboratory activities (e.g. laboratory staff meetings and meeting with customers) are to be segregated as a greater degree of safety results when non laboratory work and interaction is conducted in a space separated from the laboratory.

2.2 Windows

If the laboratory has windows that open, they must be fitted with insect screens. Insects, particularly flies, are known to be a potential carrier of disease. To keep insects out of the laboratory, the doors must be closed while an experiment is in progress, and windows shall be screened if they are capable of being opened.

2.3 Sinks

Each laboratory room must contain a sink for handwashing and glassware washing.
Exposure to hazardous materials and/or pathogenic organisms can occur by hand-to- mouth transmission. It is extremely important that hands are washed prior to leaving the laboratory. For this very reason, the sink should be located close to the egress.

2.4 Chemical/Waste Storage

Chemical storage shelves are not to be placed above laboratory sinks. Incompatible chemicals or materials which in combination with other substances may cause a fire, explosion, or may liberate a flammable or poisonous gas, must be stored separately. It is recommended that solvent storage not be located under the laboratory fume hood, as this is a location where fires are most likely to occur in laboratories.

2.5 Furniture Design and Location/Exit Paths

It is important that all work surfaces (e.g., bench tops and counters) are impervious to the chemicals used. For example, many microbiological manipulations involve concurrent use of chemical solvents such as formaldehyde, phenol, and ethanol as well as corrosives. The laboratory bench must be resistant to the chemical actions of these substances and disinfectants.

2.6 Aisle/Workstations and Exits Clearance

Clear aisles and exits are necessary to facilitate departure in the event of an emergency. In practice, laboratory aisles must be designed wider than 24" (0.6 m) so that even with the presence of laboratory stools and other miscellaneous items, a clearance of 24" (0.6 m) is always maintained.

Laboratory benches must not impede emergency access to an exit. This is also applicable to placement of other furniture and appliances such as chairs, stools, refrigerators, etc.

The space between adjacent workstations and laboratory benches should be 5 ft (1.5 m) or greater to provide ease of access.

Spaces between benches, cabinets, and equipment must be accessible for cleaning and to allow for servicing of equipment.

2.7 Laboratory Doors

Where possible, all Laboratory doors should swing out. Doors which swing in the direction of egress will facilitate occupant departures from laboratories during emergencies.

2.8 Laboratory Desks

Laboratory desks should be located near exit ways and in the path of fresh make up air. This will ensure that in the event of an emergency, employees working in "clean" areas (i.e., their desk) do not have to pass through more hazardous areas to exit the laboratory.

2.9 Illumination

Laboratory areas should have adequate natural or artificial illumination to ensure sufficient visibility for operational safety.

2.10 Cleanability

The laboratory should be designed so that it can be easily cleaned. Bench tops must be a seamless one-piece design to prevent contamination. Wooden and wood finish walls or floors are not appropriate because they can absorb hazardous and/or potentially infectious material, particularly liquids, making decontamination virtually impossible.

2.11 Electrical

The laboratory should be fitted with an adequate number of electrical outlets which can accommodate electrical current requirements.

The laboratory may have several pieces of equipment, which require large amounts of electrical current. Such items include freezers, bio-safety cabinets, centrifuges, and incubators whose electrical demand if overloaded onto a minimum number of electrical outlets can lead to potential power failure.

Circuit breakers should be located outside the laboratory. In the event of an emergency, the laboratory may be unsafe to enter. Hence, the circuit breakers for key electrical appliances should be located outside the laboratory.

2.12 Plumbing

Auxiliary valves for gas and vacuum lines should be located outside the laboratory.

In the event of an emergency, the laboratory may be unsafe to enter. Hence, the valves for gas and vacuum lines should be located outside the laboratory.

2.13 ACCESS CONTROL

Access to the laboratory is to be restricted to essential personnel only. Visitors must be warned of any hazards relating to work in progress, and should be accompanied at all times. Any conditions of entry e.g. (proper shoes, safety glasses) should be posted on doors and enforced. Casual visits to the laboratory area by other staff are to be discouraged.

The laboratory is to be kept locked outside normal working hours
All visitors must be signed in at Reception and issued with a Visitor pass tag, and a laboratory staff member must take the responsibility for their presence. This includes any external service personnel.

2.14 TEMPERATURE & VENTILATION

The environment in the working areas of the laboratory is controlled by normal methods of ventilation and air conditioning. Temperature and humidity are measured using battery operated data loggers and an assigned technician will be responsible for recording the readout.

Adequate control of humidity, dust and temperature is important to:

(i) protect samples which are stored in this room;

(ii) have a clean work environment, without major fluctuations in these conditions, during analyses of extracts;

(iii) staff comfort;

(iv) instrumental performance;

(v) have a safe working environment.

3.0 REFERENCES

3.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). *Standard Methods for the Examination of Water and Wastewater*, 21st Edition. American Public Health Association (APHA), Washington, D.C; 9020B.2 Facilities

3.2 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

3.2.1 SOP No. MM 300 Environmental Monitoring

3.2.2 Quality Manual, QMS 8.0 Environment

Chapter 2 QUALITY ASSURANCE and QUALITY CONTROL

1.0 IMPORTANCE OF QUALITY ASSURANCE IN THE LABORATORY

Data obtained from laboratory analyses are often used for making decisions. These decisions could be:

- of considerable economic significance
- used for improving the environment and community health and safety
- related to regulatory and legal matters

Quality assurance (QA) includes all the activities undertaken by a laboratory to ensure that reliable and accurate testing or measurement will be undertaken at all times. These activities (will be covered in Part II of this Manual) include Document Control, Laboratory Internal Audits, Management Review, Sampling, Handling and Storage of Samples, Control of Non-conforming Work, Complaints and Corrective Action Procedures, Technical and Quality Records.

2.0 IMPORTANCE OF QUALITY CONTROL IN THE LABORATORY

Quality Control (QC) on the other hand includes those activities that are undertaken to confirm that test and measurements results are accurate and reliable. These activities include, but are not limited to participation in proficiency tests and other inter-laboratory comparisons, regular use of certified standard reference materials, secondary or sub-reference materials, in-house reference standards, testing or measurement of multiple samples (duplicates or replicates).

QC procedures are critical to maintain and improve the accuracy, precision and reliability of the data produced in any laboratory analysis. These should be implemented in each laboratory to ensure that appropriate sampling and analytical procedures are followed, laboratory and field equipment are regularly checked and calibrated, and staff are adequately trained and supervised. QC checks are what a Laboratory does to ensure that its QA program is working.

3.0 QUALITY CONTROL CHECKS IN WATER - PHYSICAL AND CHEMICAL ANALYSES

3.1 INITIAL METHOD VALIDATION

The two most important criteria for method acceptability are accuracy and precision.
Before being used in the analysis of a test material all methods must first undergo in-house validation which will involve first establishing the accuracy followed by establishing the precision of the method.

3.1.1 **Accuracy:** Accuracy is getting the right result.
A Standard Reference Material (SRM) is to be analysed at least 2 times using the method to be validated; this can be conducted independently of each other. The values obtained are then compared with the predetermined value of the SRM. Determined values should fall within the value's uncertainty.

Note 1: If SRMs are not available, the following can also be used:

- Use of proficiency program (refer to 5.0) samples to benchmark the laboratory results against those of other laboratories

- Checking the same sample at another laboratory expert in the method, or,

- Use of high quality analytical reagents.

3.1.2 **Precision:** Precision is getting the same result in repeat analyses.
The precision of a method is determined through replicate analysis of an appropriate material (usually the In-house Reference) in a number of separate runs. This is to be conducted at least 10 times. The mean and the standard deviation of the data collated are then determined (refer to 3.6). The statistics obtained can be used to produce quality control charts (refer to 3.7).

3.2 ON-GOING VALIDATION

On-going validation of test-methods is necessary to check that the method continues to work properly. The checks will take the form of some or all of the Quality Control procedures discussed in 3.3 – 3.8.

3.3 LABORATORY BLANKS

3.3.1 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 towards error in the measurement.

- Include two reagent blanks with every batch of samples.

- Analyse the blanks immediately after calibration.

- Record the blank readings in Worksheets as these data are useful in tracking reagent quality and analyst technique.

3.3.2 A sample blank consists of the sample without the reagents added, this is especially necessary for colorimetric work.

3.4 DUPLICATE DETERMINATIONS

This is the inclusion of duplicate determinations on the same test material in an analytical batch. For results to be acceptable, the % difference between duplicates is as outlined in the Quality Control Section of each SOP (refer to Chapter 7, Appendix I).

To calculate the % difference between duplicates:

$$\%\ Difference = \frac{(D_2 - D_1)}{(D_2 + D_1)/2} \text{ x } 100$$

Where D_1 = Duplicate 1
D_2 = Duplicate 2

3.5 IN-HOUSE REFERENCE

This is a relatively large amount of the matrix containing the analyte at an appropriate level. It is homogenized and analysed several times to check homogeneity and determine a "target mean". It is then kept under conditions where the analyte and the matrix are stable and is used for checking the self-consistency of the data obtained from a large number of similar analyses conducted over a period of time. It is used to test for the precision of a method.

3.5.1 **Preparation of In-house Reference:** The in-house reference can be prepared using the following:

- Sealed bottled water belonging to the same batch is obtained and the contents emptied into a clean 20 L container and mixed thoroughly to obtain a homogeneous mixture. Fifteen water samples can then be taken from this container and tested for analytes such as Electrical Conductivity, Total Dissolved Solids, Sodium, Potassium, Magnesium, Calcium and so on. The values obtained can then be collated, and the mean and standard deviation are determined using the formula in 3.6. The remaining water can then be filled into the original bottles for use as In House Reference.

Example: Potassium results obtained from analysis of 15 samples of water taken from the homogenous mixture of bottled water.

Results No.	Potassium values
1.0	7.82
2.0	7.84
3.0	7.82
4.0	7.83
5.0	7.86
6.0	7.84
7.0	7.82
8.0	7.86
9.0	7.86
10.0	7.87
11.0	7.85
12.0	7.86
13.0	7.87
14.0	7.83
15.0	7.84
Mean	**7.84**
Std Dev	**0.02**
2 x Std Dev	**0.04**

The In-house Reference results can also be the basis of a control chart (refer to 3.7).

3.6 QC CALCULATIONS

The following is a compilation of equations frequently used in QC calculations.

3.6.1 For the calculation of the mean:

Mean (x) = $\Sigma x/n$

Where: Σx = Sum of data
n = total number of data

3.6.2 For the calculation of the standard deviation:

SD (σ) =

$$\sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

Where: x_i = data obtained
x = mean of the set of data
n = number of data.

3.7 QUALITY CONTROL CHARTS

Quality Control Charts are useful for monitoring the performance of an analytical method, reagents and standard quality, analysts' techniques and the status of the in-house reference being used.

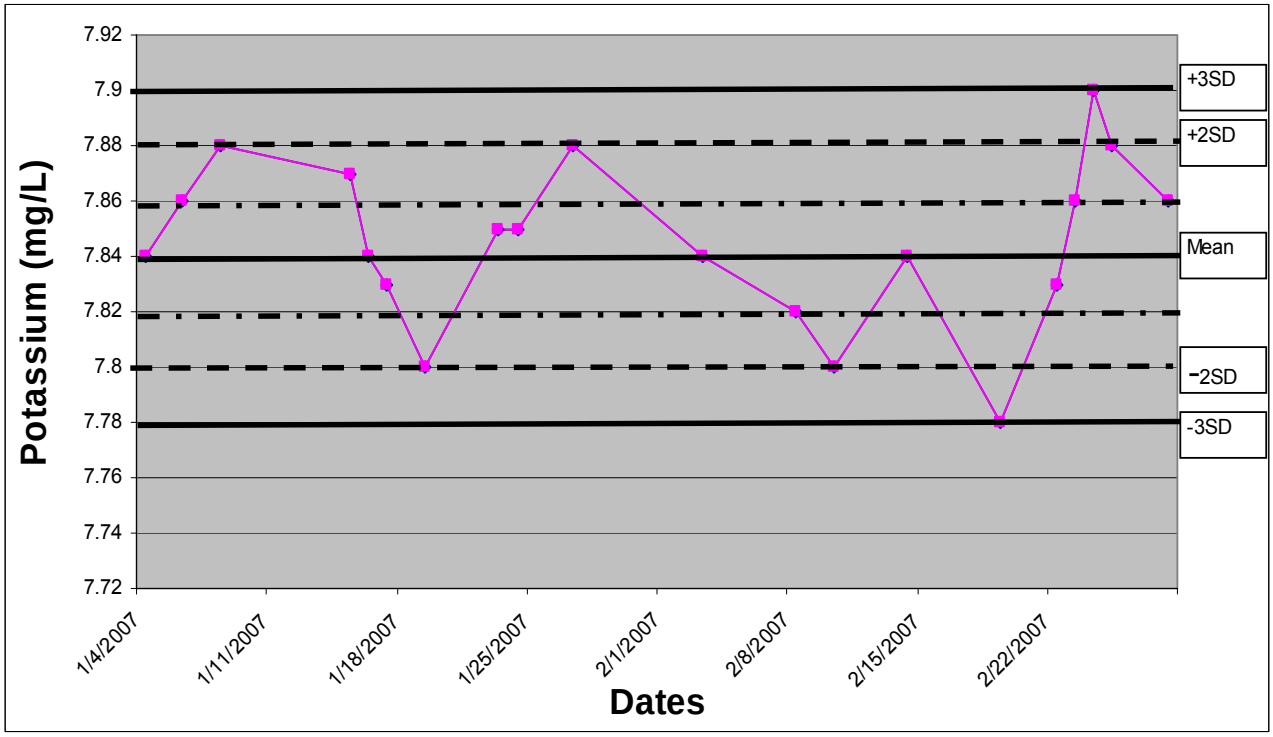
3.7.1 The results of each analysis of the in-house standard are plotted on the x-axis versus the accepted mean and Standard deviation (SD) on the y-axis. The 2 x SD value is taken as the Warning Limit and the 3 X SD value is taken as the Action Limit.

Using the results of the Example in 3.5 for Potassium analysis in Water, the Quality Control Chart would be as follows for the data obtained from the In-House Reference from 04 January to 28 February:

Table 1: Quality Control Data – Potassium in Water

Date	Potassium (mg/L)
4-Jan	7.84
6-Jan	7.86
8-Jan	7.88
15-Jan	7.87
16-Jan	7.84
17-Jan	7.83
19-Jan	7.8
23-Jan	7.85
24-Jan	7.85
27-Jan	7.88
3-Feb	7.84
8-Feb	7.82
10-Feb	7.80
14-Feb	7.84
19-Feb	7.78
22-Feb	7.83
23-Feb	7.86
24-Feb	7.9
25-Feb	7.88
28-Feb	7.86

Potassium in Water Quality Control Chart



3.7.2 Table 2 outlines the conditions on the control chart that indicate that the method is probably no longer "in control" and actions to check the accuracy and precision should be taken.

Table 2. Some indications and possible causes of a method going out of control:

OBSERVATIONS	POSSIBLE CAUSE
1 or more point outside 3xSD	Analyst performance Poor technique Deviation from procedure
2 consecutive points outside 2xSD	As above
4 consecutive points outside 1x SD	As above
7 consecutive points all outside 2xSD	Incorrect preparation of standards Incorrect preparation of reagents Analyst error, contamination
7 or more consecutive points outside 2xSD all increasing	Deterioration of standard Deterioration of reagents
7 or more consecutive points outside 2xSD all decreasing	Solvent evaporation from standard, Deterioration of reagents

3.8 CALIBRATION

Regular calibration and checking of instruments against Reference standards is essential. For example, at the start of conductivity or pH analysis the meter should be checked against a known standard and re-calibrated if necessary. Refer to Chapter 3 for more information on Calibration of Instruments.

3.9 EXAMPLE OF THE USE OF QUALITY CONTROL IN WATER TESTING - pH ANALYSIS IN WATER.

3.9.1 Calibrate the pH meter prior to use for analysis with the Buffers References: pH 7.00 ± 0.02, 4.00 ± 0.02 and check the calibration of the pH meter with Buffer Reference 9.22 ± 0.02.

3.9.2 Analyse samples in duplicate.

3.9.3 Duplicate determinations should agree within 4% of their average.

3.10 DOCUMENTATION OF QUALITY CONTROL DATA

3.10.1 Quality Control data is to be recorded on the appropriate Worksheet. Refer to Appendix I to Chapter 2, Example I for an example of how the QC data for pH analysis in Water is recorded on the pH in Water Worksheet.

4.0 QUALITY ASSURANCE AND QUALITY CONTROL CHECKS IN WATER - MICROBIOLOGICAL ANALYSES (Ref: Section 9020B APHA, 21st ed)

A quality assurance program for microbiological analyses must emphasize the control of laboratory operations and analytical procedures because the tests measure the living organisms that continually change in response to their environment. Analytical standards, QC charts and spiked samples are not applicable for Microbiological analysis. For this reason, a QC program in Microbiological analyses applies practices necessary to minimize systematic and random errors resulting from:

(i) Staff (refer to Chapter 6)

(ii) Facilities (refer to Chapter 1)

(iii) Equipment (refer to Chapters 3 & 4)

(iv) Reagents supplies (refer to Chapter 5)

(v) Sampling (refer to Part II of this Manual)

(vi) Standard Operating Procedures (refer to Chapter 7)

(vii) Data handling and reporting (refer to Chapter 9 and Part II of this Manual)

4.1 Analytical Quality Control Procedures

4.1.1 **New methods –** Conduct parallel test with the standard procedure and a new method to determine applicability and comparability. The new method can also be subjected to a Proficiency program sample check (refer to 5.0)

4.1.2 **Comparison of plate counts –** For routine analyses, repeat counts on one or more positive samples at least monthly and compare the counts with those of other analysts testing the same samples. Replicate counts for the sample analyst should agree within 5% and between analysts should agree within 10%. Refer to 3.6 for the calculation of duplicates.

4.1.3 **Duplicate analyses –** Perform duplicate analysis on 10% of samples and on at least one sample per test run. A test run is defined as an uninterrupted series of analyses. If the laboratory conducts less than 10 tests/week, make duplicate analyses on at least one sample each week.

4.1.4 **Sterility checks**

4.1.4.1 **Procedural Blank:** For membrane filtration tests, check sterility of media, membrane filters, buffered dilution and rinse water, pipettes, flasks and petri dishes, and equipment as a minimum at the end of each series of samples using sterile reagent water as the sample. For multi-tube and presence-absence procedures, check sterility of media, dilution water and glassware. If contaminated, check for the source.

4.1.4.2 **Media Blank:** To test sterility of media, incubate a representative portion of each batch of media prepared at the appropriate temperature for 24 to 48 h and check for growth.

4.1.4.3 **Field Blank:** A field blank is prepared to test the sterility of the sampling containers, the aseptic techniques of the person conducting the sampling and that the samples are not contaminated during transportation of sample to the Laboratory. A field blank is sterile water transferred into a sterile container at the sampling site which is analysed along with the samples. Its temperature is also taken upon receipt at the Laboratory to check whether the samples are maintained as specified during transportation to the Laboratory.

4.1.4.4 **Positive and Negative Control Cultures:** For each lot of medium, check analytical procedures by testing with a known positive and negative control cultures. Refer to Table 3 for examples of Control Cultures. Prepare successive dilutions of the relevant positive reference organism until a dilution with a number of organisms (20 – 50 colonies) is obtained. For example, for the validation of a batch of Lauryl Sulphate Tryptose broth, positive reference culture used would be *E. coli*.

Table 3 Examples of Control Cultures

	Control Cultures	
Group	**Positive**	**Negative**
Total Coliforms	*Escherichia coli*	*Enterococcus faecalis*
	Enterobacter aerogenes	*Pseudomonas sp*
Faecal coliforms	*E. coli*	*E. aerogenes*
		Streptococcus faecalis
Escherichia coli	*E. coli*	*E. aerogenes*
Enterococci	*S. faecalis*	*S. mitis/salivarius*

4.2 EXAMPLE OF THE USE OF QUALITY CONTROL IN WATER MICROBIOLOGICAL TESTING – TOTAL COLIFORMS ANALYSIS IN WATER (MEMBRANE FILTRATION).

4.2.1 A field blank (sterile water transferred into a sterile container at the sampling site) and a procedural blank (prepared at the laboratory for detecting any contamination of reagents or equipment used) are analysed along with the samples.

4.2.2 The temperature of the field blank is measured using a thermometer as soon as the samples reach the laboratory to check that the samples are being maintained at the specified temperature. This temperature is then recorded in the relevant Worksheets.

4.2.3 A procedural blank (prepared at the laboratory for detecting any contamination of reagents or equipment used) is analysed along with the samples (mentioned in 4.2.1- repetition).

4.2.4 Test each medium lot for satisfactory performance by conducting the following steps:

4.2.4.1 Make dilutions (1×10^{-5} dilution) of the Positive Control – Reference Culture *E.coli* and filter appropriate volumes to give 20 to 60 colonies per filter.

4.2.4.2 Before use, test each batch of laboratory prepared media with positive and negative controls. Also do the same for pre-prepared media.

4.2.5 Check for coliform contamination at the beginning and end of each filtration series by filtering 20 – 30 mL of dilution or rinse water through filter.

4.2.6 If controls indicate contamination, reject all data from affected samples and request for resampling.

4.2.7 The table below outlines the morphological characteristics of the Reference Control Cultures used on the media of this procedure:

Morphological Characteristics of the Control Organisms

	E. coli	*E. faecalis*
1. m-ENDO Agar (blank: pink)	The colonies are round, shiny, circular and are maroon in color with or without a green metallic sheen.	No growth.
2. m-ENDO Broth MF (blank: pink)	Smooth, shiny, round colonies that are pink in color with or without a green metallic sheen.	No growth.
3. LST Broth (blank: yellow)	Broth is turbid and the gas produced is collected in the Durham tube.	Broth is turbid but NO production of gas.

4.3 DOCUMENTATION OF QUALITY CONTROL DATA

4.3.1 Quality Control data are to be recorded on the appropriate Worksheet. Refer to Appendix I to Chapter 2, Example II for an example of how the QC data for Total Coliform analysis in Water (Membrane Filtration) is recorded on the TC in Water Worksheet.

5.0 PROFICIENCY PROGRAM

Participation in proficiency programs 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 standards.

The laboratory may participate in the following external quality assurance programmes outlined in Table 5a or other similar programs.

Table 5 a: Proficiency Testing Programs in Australia and New Zealand

PROGRAM	SCHEDULE	CONTACT DETAILS
Wide range of chemical & microbiological tests for food including: Dairy products Meat/food Water Pathogens	 Monthly Monthly 6/year 6/year	Richard Leong//Ms Sharon Taylor AgriQuality Ltd PO Box 10222, Te Rapa Hamilton NEW ZEALAND Ph: 647 849 9990 Fax: 647 849 4215 E-mail: TaylorSh@agriquality.co.nz or admin@proficiency.co.nz
Water (natural, synthetic and effluent)	5/year 4/year 4/year	Mr D Tebbutt International Accreditation New Zealand Level One, 630 Great South Road Greenlane, Auckland 1005 New Zealand Ph: 649 525 6655 Fax: 649 525 2266

PROGRAM	SCHEDULE	CONTACT DETAILS
Potable waters, effluent, saline waters	quarterly	Joanne Bedford Proficiency Services Ltd 11/5 Pukete Road, Hamilton Ph: 64 7 850 4483 Fax: 64 7 850 4487 Email: manager@proficiencynz.co.nz
Water - wide range of tests	2/year	Mr W J Emmett Australian Water Quality Centre Private Mail Bag 3, Salisbury SA 5108 Australia Ph: 618 8259 0319 Fax: 618 8259 0228
Low level nutrients in freshwater and seawater	yearly	Mr D Wruck Queensland Health Scientific Services 39 Kessels Road, Coopers Plains, QLD 4108 Australia Ph: 617 3274 9062 Fax: 617 3274 9119
Soft drinks - preservatives, artificial sweeteners, colours, caffeine Water - anions, cations pH	2/year 2/year 1/year	Mr D Siyali Quality Assurance, Division of Analytical Laboratories, Institute of Clinical Pathology and Medical Research PO Box 162 Lidcombe NSW 2141, Australia Ph: 612 9646 0222 Fax: 612 9646 0333

6.0 REFERENCES:

6.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). *Standard Methods for the Examination of Water and Wastewater*, 21st Edition. American Public Health Association (APHA), Washington, D.C;Method 1020B Quality Control, 9020 Quality Assurance.

6.2 Thompson, M, Ellison R.L.S, Wood R "Harmonized Guidelines for Single Laboratory Validation of Methods of Analysis" Pure and Applied Chemistry, Volume 74, No. 5, International Union of Pure and Applied Chemistry 2002.

6.3 Institute of Applied Sciences Analytical Laboratory Standard Operating Procedures Nos:

6.3.1 AS 8 Validation of Test Methods

6.3.2 AS 15 Determining of Methods Detection Limits

6.3.3 MM 310 Microbiological Media, Reagents, Purchasing, Preparation, Storage, Validation

Appendix I to Chapter 2

Example of Worksheet for pH in Water

pH Worksheet

Report No. 245

Site/Customer Name: ABC

Date Received: 12/02/07

Date Sampled: 12/02/07

Date Analysed: 12/02/07

Date Reported: 13/02/07

Lab Id.No(s): 07/2809 - 2812

Number Of Samples: 4

Analysts: ASK

QA / QC

Reference Material	**Reference Value**	**Value Obtained**	**Comments**
Buffer Reference 1	**9.22 + 0.02**	**9.21**	**ok**
Buffer Reference 2	**7.00+ 0.02**	**7.01**	**ok**
Buffer Reference 3	**4.00 + 0.02**	**4.01**	**ok**

			pH Reading			
Lab Id No.	Sample Description	Sample Id	Reading 1	Reading 2	Reading 3	Average pH Reading
07/2809	Tap Water	Workshop	6.49	6.50	6.51	6.5
07/2810	Tap Water	Kitchen	6.19	6.20	6.21	6.2
07/2811	Pool Water	Swimming Pool	7.24	7.25	7.26	7.3
07/2812	Water	Dam	6.45	6.46	6.47	6.5

Analyst:: _____ Checked By: _____ Authorized By _____

Date : _____ Date: _____ Date: _____

Appendix II to Chapter 2

Example of Worksheet for Total Coliforms (TC) in Water (Membrane Filtration)

RESULT #: 150

Site/Customer Name: AX	Date/Time Received : 02/05/07, 10.30 am	Date/Time Analyzed : 02/05/07, 10.45 am
Analysis Required: TC	Sample type: Bottled Water	Total # of samples: 2

FIELD BLANK (ºC):

Sample ID and Description		WC 2344 Bottled Water	WC 2345 Bottled Water	Field Blank		Procedural Blank	+ve control *E.coli -5 dilution*	-ve control *E. faecalis*
Lab # 2007/		1087	1088	----		----	----	----------
TC Dilutions: (step1)	1 mL	142	140	----		**Clear**	**75**	**No growth**
	10 mL	TNTC	TNTC	----				
	100 mL	TNTC	TNTC	0				
TC/100 mL		1.4 x 10^4	1.4 x 10^4			----	----	----------
√ or 10% of count		14	14			----	----	----------
# +ve in LST Broth	24h check (step 2a)	0/14 +ve	0/14 +ve			Clear	+ve gas	No gas
	48h check (step 2b)	1/14 +ve	1/14 +ve			Clear	+ve gas	No gas
# +ve in BGB Broth	24h check (step 3a)	----	----					
	48h check (step 3b)	----	----					
Verified TC/100 mL		1.0 x 10^3	1.0 x 10^3			----	----	----------

Step 1 by: AD	Date/Time in: 02/05/07, 11.30 am		Results:	AN	Date/Time out: 03/05/07, 1.15 pm
Is the TC Count higher than the FC Count, for ALL samples? If no, then inform the KTP immediately.				Sample(s):	
Step 2a by: AN	Date/Time in: 03/05/07, 1.30 pm	Results (24 h): AN	Date/Time out: 04/05/07, 11.45 am	Results (48 h): AD	Date/Time out: 05/05/07, 12.20 pm
Step 3a by: ---	Date/Time in: ----		Results: ----		Date/Time out: ----
Step 3b by: ----	Date/Time in: ----		Results: ----		Date/Time out: ----
Checked by: GL			KTP: AP		
Date: 05/05/07			Date: 05/05/07		

Comments:_____

Chapter 3 CALIBRATED INSTRUMENTS

All instruments of testing are to be calibrated before being put into service, and are to be further calibrated in accordance with the following programme and duly recorded.

The Laboratory Manager is to be responsible for the monitoring and implementing of the Equipment Calibration Programme.

Note:
* Items that may be calibrated by staff of the Laboratory, if the staff are competent to perform such calibrations.

Type of Equipment	**Maximum Period Between Successive Calibrations**	**Procedures**
Automatic Burettes, Dispensers and Pipettors	Initial and three months	Accuracy of, and repeatability at volumes in use
Balances	Initial calibration and 3 yearly recalibrations Accompanied by: (a) Each weighing (b) One Month (c) Six months	By an accredited calibration laboratory or; *Calibration using traceable certified masses. Refer to IANZ Technical Guide 2. Staff members performing calibrations need to be formally trained. Annual servicing is recommended. Zero check. One point check using a known mass close to balance capacity. Repeatability checks at the upper and lower ends of the scale. The standard deviation of the results can be compared against the results recorded on the last external certificate.
Biological Safety Cabinets	One year	By an accredited laboratory. Documented procedures need to be in place for on-going monitoring.
Conductivity Meter	Each use Note: If a temperature compensation probe is used, it must be calibrated. See thermometers.	Checked using appropriate standards in each of the scale ranges of the meter in use.

Type of Equipment	Maximum Period Between Successive Calibrations	Procedures
Filters (membrane)	Each manufacturer's batch	Manufacturer's certification of conformance to USEPA standards; and/or verification checks as per APHA "Standard Methods for the Examination of Water and Wastewater, 21st edition"
Masses (Stainless steel, or nickel-chrome alloys)	Initial calibration Three years (first recalibration) Five years (successive recalibrations)	By an accredited calibration laboratory By an accredited calibration laboratory By an accredited calibration laboratory
pH meter	Daily or before use Note: If a temperature compensation probe is used, it must be calibrated. See thermometers.	Calibrate using at least two appropriate standard buffers. Buffers need to be stored in appropriate containers and marked with an expiry date.
Refrigerators	Daily	Monitor the temperature and record.
Sterilisers Autoclaves	Initial and following repair or maintenance Each use	*Check heating profiles of typical loads with respect to chamber temperatures to determine lag times using appropriately calibrated equipment following a fully documented procedure. Annual servicing of steam sterilizers is strongly recommended. Check the time and temperature of the cycle. Discard loads should be autoclaved for at least 30 minutes at 121°C.
Thermocouples • Reference • Working	Three years or 100 hours use (whichever is sooner) Six months	By an accredited laboratory. Single point within the working range against a reference thermometer or thermocouple.

Type of Equipment	Maximum Period Between Successive Calibrations	Procedures
Thermometers (Liquid in glass) • Reference	Five years (complete)	By an accredited calibration laboratory, followed by an ice point check on receipt.
	Six months	Ice point
• Working	Initial	Check against reference thermometer/thermocouple across working range or at points of use.
Thermostatically Controlled Equipment (Incubators, water baths, ovens)	Daily	Monitor the temperature and record.
	Two years	Temperature variation within the working space by an accredited calibration laboratory or; *Using appropriately calibrated equipment following a fully documented procedure
Timers (Stopwatches) • Mechanical	Three months	Comparison against radio time “pips”
• Electronic	One year	Comparison against radio time “pips”
Volumetric glassware (flasks, pipettes, burettes)	Initial only	Using distilled water at critical graduations.
Turbidity meters	Initial and when required	Using supplied standards
TDS meters	Initial and when required	Using supplied standards
HACH DR series colorimeters	Initial check and following repair or maintenance	Using supplied standards

The methods for the calibration of some of the above-mentioned equipment are attached as Appendices to Chapter 3:

- Appendix I to Chapter 3 Operation and Calibration of pH meter
- Appendix II to Chapter 3 Operation and Calibration of Electrical Conductivity Meter
- Appendix III to Chapter 3 Operation of Turbidity Meter
- Appendix IV to Chapter 3 Balances Six-monthly repeatability
- Appendix V to Chapter 3 Operation and Calibration of Automatic Pipettes & Microbiological Media Dispensers

6.1 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 and copies of the calibration reports furnished by them will also be kept on file in the Laboratory Manager's office. External calibration records are to contain the following information:

(i) Report number.

(ii) Identification number of the calibration standard to which the report pertains.

(iii) Environmental conditions under which the calibration was performed.

(iv) Estimated uncertainty of the calibration value.

(v) Deviations or corrections.

Records of these subcontracted laboratories are to be maintained by the Laboratory (refer to Part II of this Manual).

6.2 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

6.2.1 CALIBRATION RECORDS

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

For each meter or instrument a Calibration Record Form must record the following information:

(i) Laboratory Equipment Number

(ii) Description.

(iii) Manufacturer's name.

(iv) Equipment serial number.

(v) Model number.

(vi) Location or section where used.

(vii) Calibration frequency.

(viii) Date calibration is performed.

(ix) Name of person performing the calibration.

(x) Results of calibration.

6.2.2 Example of Calibration Record

An example of a Calibration Record for the Electrical Conductivity meter would be as follows:

Instrument Calibration Record

EC Meter No.: 01 **EC Meter Model: Orion 325**

Location: Microbiology Laboratory **Serial Number: 326589**

Date	Standard used for calibration	Measured value(s)	Pass/Fail	Recalibrated instrument (Y/N)	Analyst
14/05/07	84 ± 2.0 µS/cm	83 µS/cm	Pass	N	AK
15/05/07	84 ± 2.0 µS/cm	87 µS/cm	Fail	Y	AK

Note:
Instrument should be recalibrated if measured value for a particular standard deviates from the standard check acceptable value.

7.0 REFERENCES:

7.1 International Accreditation New Zealand, Specific Criteria for Chemical Testing, 2005

7.2 Institute of Applied Sciences Analytical Laboratory Standard Operating Procedures Nos:

7.2.1 IO 649(R1) Operation and Calibration of pH Meter

7.2.2 O 629 Operation and Calibration of Electrical Conductivity Meter

7.2.3 IO 609 Operation and Calibration of Turbidity Meter

Appendix I to Chapter 3

SOP 1 OPERATION AND CALIBRATION OF HANNA pH METER, MODEL pH211.

1.0 SCOPE

This method describes the operational procedure for the Hanna pH meter, Model pH211. It also describes the pH meter's calibration procedure that is to be conducted prior to using the meter for analysis as well as the temperature probe calibration that will be conducted by the Senior Technician on a six-monthly basis.

2.0 APPLICATION

2.1 This method is suitable for an analyst who has been instructed and understands the basic principles involved in using and calibrating a pH meter and who has read the Hanna pH meter, pH211 Operation Manual.

2.2 It is also to be used by the Senior Technician for calibrating the temperature probe on a six-monthly basis.

3.0 PRINCIPLE

3.1 The basic principle of electromagnetic pH measurement is the determination of the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. The instrument is calibrated using two buffers and its performance is checked using a third buffer.

4.0 APPARATUS

4.1 The Hanna pH 211 is a microprocessor-based bench meter for pH and temperature measurement. The pH measurements are compensated for temperature effect automatically with the HI 7669/2W temperature probe. The meter has a large Liquid Crystal Display (LCD) which shows the pH and temperature simultaneously together with graphic symbols.

5.0 PROCEDURE

5.1 Instrument Setup

5.1.1 Power Connection: Plug the 12V DC adapter into the power supply socket.

5.1.2 Electrode and Probe Connections: For pH electrodes (with internal reference), connect the electrodes BNC to the socket on the back of the meter. For temperature measurements and automatic temperature compensation connect the temperature probe to the appropriate socket.

Note: To prevent damage to the electrode, remove the pH electrode from the solution before turning off the meter. If the meter is OFF, detach the electrode

5.1.3 The instrument is to be calibrated:

(i) Whenever the pH electrode is replaced.

(ii) At least once a week.

(iii) After testing aggressive chemicals.

(iv) After pressing RESET.

(v) If higher accuracy is required.

5.2 Preparation of Buffers

5.2.1 Prepare fresh buffers on a two monthly basis. Before changing buffers, perform old versus new buffers checks, and record results on the pH meter Calibration Log Book. Also record the batch numbers of newly prepared buffers.

Notes:

(i) *Prepared buffers maybe used past the two months expiration date provided there is data to show they are still within the acceptable pH range.

(ii) pH Buffers are to be kept free of contamination by keeping its bottles tightly sealed when not in use.

(iii) Use volumetric glassware to prepare solutions accurately.

* Th*is is a modification of the reference method that recommends that prepared buffers have a shelf life of one month.*

5.3 Calibration Procedure

Manual calibration with two buffers is described below. The performance of the pH meter will be checked with the third buffer.

5.3.1 Use pH 7.00 as the first buffer. If you are measuring in the acid range, use pH 4.01 as second buffer, if you are measuring in the alkaline range, use pH 9.2 as second buffer.

5.3.2 Rinse both the pH and temperature electrode with distilled water, blot dry using tissue papers. Immerse both the probes approximately 4 cm into the buffer 7.00 solution and stir gently. The temperature probe should be close to the pH electrode.

5.3.3 Press CAL. The “CAL” and BUF indicators will be displayed on the secondary LCD (smaller numbers displayed on the right of the LCD).

5.3.4 The “NOT READY” indication will blink on LCD until the reading has stabilised.

5.3.5 When the reading is stable, “READY” and “CFM” will blink. Press CFM to confirm the calibration. Record the displayed pH reading on the

primary LCD (bigger size numbers on LCD) in the pH meter Calibration Log Book.

5.3.6 If the reading is close to the selected buffer, the meter stores the reading. The calibrated value is then displayed on the primary LCD and the secondary LCD will display the second expected buffer value.

5.3.6.1 If the value measured by the meter is not close to the selected buffer, "WRONG BUF" and "WRONG I" will blink alternately. In this case check if the correct buffer has been used or clean the electrode (see 5.6.3). Change the buffer if necessary.

5.3.7 After the first calibration is confirmed, rinse electrodes with distilled water, blot dry using tissue papers. Immerse the pH electrode and temperature probe approximately 4 cm in the second buffer solution and stir gently. The temperature probe should be close to the pH electrode.

5.3.8 If necessary press "Δ°C" or "∇°C" to select a different buffer value.

Note:
The meter will automatically skip the buffer used for the first point. It will skip 7.01 if 4.01 or 9.01 was used as the second buffer.

5.3.9 The "NOT READY" indication will blink on LCD until the reading has stabilised.

5.3.10 When the reading is stable, "READY" and "CFM" will blink. Press CFM to confirm the calibration. Record the reading on the primary LCD in the pH meter Calibration Log Book.

5.3.11 If the reading is close to the selected buffer, the meter stores the reading and returns to normal operational mode.

5.3.11.1 If the value measured by the meter is not close to the selected buffer, "WRONG BUF" and "WRONG I" will blink alternately. In this case check if the correct buffer has been used, or regenerate the electrode by following the cleaning procedure below (see 5.6.3). Change the buffer if necessary.

5.3.12 Press RANGE to display the temperature reading on the LCD during calibration. Record the temperature reading on the pH Meter Calibration Log Book.

5.3.13 Remove electrode from second buffer, rinse thoroughly with distilled water and dry electrode with tissue papers. Immerse in a third buffer (after rinsing with third buffer - either 9.22 or 4.00; and measure pH value. The reading should be within 0.1 unit for the pH of the third buffer. Record the pH and reading on the primary LCD in the pH meter Calibration Log Book.

5.3.14 If meter response shows a difference greater than 0.1 unit from the expected value, electrode may need to be cleaned (refer to 5.6.3).

5.4 pH Measurement Procedure

5.4.1 Once pH meter is calibrated, remove electrode from buffer, rinse with distilled water and rinse with sample solution to be measured. Submerge the tip (4 cm) of the electrode and temperature probe into sample to be tested.

Note:

The pH reading is affected by the temperature. In order for the meter to measure the pH accurately, temperature must be taken into consideration. To use the Automatic Temperature Compensation feature, connect and submerge the HI 7669/2W temperature probe into the sample as close to the electrode as possible and wait for a couple of minutes.

5.4.2 Establish equilibrium between electrode and sample by stirring the sample gently to ensure homogeneity. Allow time for the electrode to stabilise.

5.4.3 pH is displayed on the primary display and temperature on the secondary one. Record both the pH and temperature readings on Worksheets/Note Books.

5.4.4 If measurements are taken successively in different samples, it is recommended to rinse the electrode thoroughly with distilled water and then with some of the next sample to condition the electrode before immersing it in the sample.

5.4.5 Record two more readings of the sample by repeating steps 5.4.2 and 5.4.3. Report results to one decimal place.

5.5 Calibration of Temperature Probe

The temperature probe will be checked against the externally calibrated Reference Thermometer on a six-monthly basis by the Senior Technician. The Reference Thermometer (with a stainless steel probe) has a resolution of 0.1°C.

5.5.1 Prepare a beaker containing ice and water and another one containing hot water (at a temperature around 50°C). Place insulation material around the beakers to minimise temperature changes.

5.5.2 Immerse the temperature probe in the vessel with the ice and water as near to the Reference Thermometer probe as possible. Allow a couple of minutes for the probe to stabilise.

5.5.3 Record the readings of both the Reference Thermometer and the pH Meter Temperature Probe in the pH Meter Calibration Log Book.

5.5.4 Calculate the temperature difference (∆Temperature):

$$\Delta\text{Temperature } (^\circ\text{C}) = \text{pH T} - \text{Ref T}$$

where Ref T = Reference Thermometer reading (°C)
pH T = pH Meter Temperature Probe reading (°C)

The calibration passes if the ΔTemperature is less than ±1°C. If calibration fails, repeat calibration, if it fails twice, inform the Laboratory Manager.

5.6 Care of Electrode

Proper electrode storage maximises the electrode performance and extends the electrode life.

5.6.1 Electrode Storage: To ensure a quick response and free-flowing liquid junction, the sensing element and reference junction must not be allowed to dry out. The electrode must be stored by soaking in pH Electrode Storage Solution. As an alternative, use potassium chloride (KCl) solution (which is prepared by adding 1 gram of KCl to 200 mL of pH 7 buffer (non-coloured). If KCl solution is not available, appropriate substitutes in order of preference are pH 4 buffer or tap water. **Never store the electrodes in distilled water.**

5.6.2 Electrode Maintenance: The Senior Technician is to inspect electrode for scratches, cracks, salt build-up, or membrane/junction deposits on a monthly basis. Rinse any salt build-up with distilled water. Record details of monthly inspection on the pH meter Calibration Log Book.

5.6.3 Electrode Cleaning Procedure: The solution used to clean a pH electrode depends on the possible contaminants:

5.6.3.1 For general cleaning, soak in electrode cleaning solution for 15 minutes and rinse four times with distilled water. Alternatively, soak the electrode in 0.1 M HCl or 0.1 M HNO_3 for 30 minutes.

5.6.3.2 For removal of protein deposits, soak the electrode in 0.1 M tetra sodium EDTA for 15 minutes.

5.6.3.3 For oil and grease removal rinse with mild detergent or 10% methanol solution.

5.6.4 After any of the cleaning procedures, thoroughly rinse the electrode with distilled water, drain & refill the reference chamber. Soak the electrode in storage solution for at least 1 hour.

6.0 REFERENCES

6.1 Hanna Instruments, pH 211 Microprocessor-based Bench pH/mV/°C Meters Manual

Appendix II to Chapter 3

SOP 2 OPERATION AND CALIBRATION OF ELECTRICAL CONDUCTIVITY METER.

1.0 SCOPE

1.1 This Standard Operating Procedure (SOP) describes the operational and calibration procedure for the EC 215 Bench Conductivity Meter.

2.0 APPLICATION

2.1 This SOP is suitable for a technician and other users who have been instructed and understand the basic principles involved in using the EC 215 Conductivity Meter and who have read the EC 215 Conductivity Meter Operation Manual.

2.2 This SOP must be followed when performing routine analysis in conjunction with SOP No. WP 202.

2.3 This SOP must be followed by the Senior Technician when performing six-monthly calibrations of the EC 215 Conductivity Meter.

3.0 PRINCIPLE

3.1 The measurement of electrical conductivity (EC) in water results from ions in solution from dissolved salts. Measurement of conductivity gives an estimate of the concentration of these dissolved salts.

3.2 Conductivity of an aqueous solution is the measure of its ability to carry an electric current by means of ionic motion. This ability depends on the concentration, mobility and valence ions present in solution and on the temperature of measurement.

4.0 APPARATUS

4.1 EC 215 Conductivity Meter

4.2 Conductivity Probes – 4 –ring probe which has built-in temperature sensor that automatically compensates for temperature changes in the liquid tested.

5.0 PROCEDURE

5.1 **Power Connection**

5.1.1 Plug the 12VDC adaptor into the power supply socket.

Note: Make sure the main line is protected by a fuse.

5.1.2 **Probe Connection**

5.1.3 Connect the conductivity probe to the socket provided.

Note: The instrument has to be calibrated before taking conductivity measurements.

6.0 CALIBRATION PROCEDURE

6.1 **Selection of conductivity standard solutions -** The conductivity standard solutions to be used will depend on the conductivity units and the conductivity measurement ranges selected:

6.1.1 When measuring in the mS ranges, use standard solution 12.88 mS at 25ºC or 80 mS at 25ºC.

6.1.2 When measuring in the µS range:

6.1.2.1 Use conductivity standard solution 1413 µS at 25ºC when calibrating in the range of 0 to 1999 µS.

6.1.2.2 Use conductivity solution 84 µS at 25ºC when calibrating in the 0 to 199 µS range.

6.2 Rinse the probe thoroughly in distilled water. This is to minimize contamination of the calibration solution and secure higher accuracy. Where possible use plastic beakers to minimize any EMC interferences. Pour a small quantity of the conductivity standard solution (refer to 6.1) into a plastic beaker.

6.3 Immerse the probe in the solution submerging the holes of the sleeve (0.5cm below) water level.

6.4 Tap the probe lightly on the bottom of the beaker to remove any air bubbles trapped inside the sleeve.

6.5 Adjust the **"TEMPERATURE COEFFICIENT"** knob to 2%/ ^{0}C.

6.6 Select the appropriate range (refer to 6.1)

"199.9 µS"	for 84 µS
"1999 µS"	for 1413 µs
"19.99 mS"	for 12.88 mS
"199.9 mS"	for 80 mS

Note: If the display shows "1", there is an over-range condition. Select the next higher range.

6.7 Allow a few minutes for the reading to stabilize and adjust the **"CALIBRATION"** knob to read on the Liquid Crystal Display (LCD), the value of the buffer solution at 25^{0}C (77^{0}F), e.g.12.88 mS/cm. Record the reading on the EC Meter Calibration Logbook.

6.8 All subsequent measurements will be referenced to 25^{0}C (77^{0}F).

Note: To reference the measurements to 20^{0}C (68^{0}F), adjust the **"CALIBRATION"** knob to read on the (LCD), the value of the buffer solution at 20^{0}C (68^{0}F), e.g. 11.67 mS/cm.

7.0 CONDUCTIVITY MEASUREMENTS

7.1 Switch the instrument on by pressing **"ON/OFF"** key.

7.2 Rinse the probe with distilled water and also rinse the probe with the sample. Pour the sample into a clean beaker. Tap the probe lightly on the bottom of the beaker to remove any air bubbles trapped inside the sleeve.

7.3 Adjust the **"TEMPERATURE COEFFICIENT"** knob to the temperature coefficient value of the sample.

7.4 Select the appropriate conductivity range.

Note: If the display shows "1", there is an over-range condition.
Select the next higher range.

7.5 Allow a few minutes for the reading to stabilize. The LCD will display the temperature compensated conductivity reading. Record the EC reading.

7.6 Rinse the probe with distilled/deionised water after every series of measurements.

8.0 PROBE MAINTENANCE

8.1 The Senior Technician will on a six-monthly basis clean the probe thoroughly with a non abrasive detergent. This is to be recorded on the EC Meter Logbook.

9.0 IN BUILT TEMPERATURE SENSOR (Refer to 4.2)

9.1 Calibration of In-Built Temperature Sensor (Within the Conductivity Meter Probe)

The Built-In Sensor will be checked against the externally calibrated Reference Thermometer on a six-monthly basis by the Senior Technician. The Reference Thermometer (with a stainless steel probe) has a resolution of 0.1°C.

9.9.1 Prepare a beaker containing ice and water and another one containing hot water (at a temperature around 50°C). Place insulation material around the beakers to minimise temperature changes.

9.9.2 Immerse the conductivity meter probe in the vessel with the ice and water as near to the Reference Thermometer probe as possible. Allow a couple of minutes for the probe to stabilise.

9.9.3 Record the readings of both the Reference Thermometer and the EC Meter Built-In Temperature Sensor in the EC Meter Calibration Log Book.

9.9.4 Calculate the temperature difference (∆Temperature):

$$\Delta\text{Temperature } (^{o}C) = \text{EC T} - \text{Ref T}$$

where Ref T = Reference Thermometer reading (ºC)
EC T = EC Meter Built-In Temperature Sensor reading (ºC)

The calibration passes if the ∆Temperature is less than ± 1ºC. If calibration fails, repeat calibration, should it fail twice, inform the Laboratory Manager.

Appendix III to Chapter 3

SOP 3 OPERATION AND CALIBRATION OF TURBIDITY METER

1.0 REFERENCE:

1.1 Lovibond Turbidity Meter Manual.

2.0 SCOPE

This Standard Operating Procedure (SOP) describes the operation of the Lovibond Turbidity meter. This instrument is suitable for use by a technician who has been advised of its operation and who has thoroughly read and understands the operation manual for the Turbidity meter.

3.0 PRINCIPAL

This Standard Operating Procedure is based on the comparison of the intensity of light scattered by a standard reference suspension under the same condition. The higher the intensity of scattered light, the higher the turbidity.

Dirty glassware, the presence of air bubbles and the effects of vibrations that disturb the surface visibility of the sample will give false results.

4.0 APPARATUS

4.1 Turbidity meter consisting of a light source for illuminating the sample. The scatter of the incident infrared light is measured in standardized manner at an angle of 90º.

4.2 Vials and Caps

5.0 CALIBRATION - For supplied standards with defined values.

5.1 Calibration Mode – For supplied standards with defined values.

5.2 Mode – press mode key and keep depressed.

5.3 Switch unit on using ON/OFF Key. Release MODE key after approx. 1 second.

5.4 CAL : Press mode to change the measuring range
CAL E1 → CAL E2 → CAL E3 → CAL E4 → … (scroll)

5.5 Position vial (with required standard – see supplied turbidity standards) with alignment of □ and □ marks. Close the sample chamber using the sample chamber cover.

5.6 Press the ZERO/TEST key.
Range the measuring range symbol flashes for approx. 9 second.
The display shows : : - confirmation of calibration adjustment.

5.7 Switch the unit off using ON/OFF key. The new correction factor is stored.

5.8 Repeat 5.4 – 5.6 for the remaining standards.

6.0 CALIBRATION - For Interim Values (Prepared Standard)

6.1 Press Mode and Zero/Test keys and keep both depressed.

6.2 Switch unit on using ON/OFF key, release MODE and ZERO/TEST keys after approx. 1 second.

6.3 CAL – Press MODE key to change the measuring range
E1 →E2 → E3 → E4 (Scroll)

6.4 Rinse and fill a clean and dust-free vial with the standard up to the mark (pour the standard) along the inner wall of the vial to avoid air bubbles, see Note 4. Screw the cap on and align the □ mark on the instrument.

6.5 Close the sample chamber using the sample chamber cover.

6.6 Press the ZERO/TEST key. The method symbol flashes for approx. 9 seconds. The result is shown in the display alternating with CAL.

6.7 If the result corresponds with the value of the calibration standard used (within the tolerance quoted), exit by pressing the ON/OFF key. Otherwise pressing the MODE key once increases the display unit by 1 digit. Pressing ZERO/TEST key once decreases the displayed value by 1 digit.

6.8 Press the relevant key until the displayed value equals the value of the calibration standard

6.9 By pressing ON/OFF key, the new correction factor is calculated and stored. The unit then switches off itself

6.10 Repeat 5.1 – 5.10 for the rest of the standards

6.11 **USER MESSAGES**

E10 – calibration factor out of range

E71 – E1 User calibration incorrect/erase

E73 – E2 User calibration incorrect/erase

E75 – E3 User calibration incorrect/erase

E77 – E4 User calibration incorrect/erase

CAL - After calibration on the interim values there will appear `CAL' when new calibrating as an indication to he former used calibration mode.

7.0 OPERATION

7.1 Switch the unit on using the ON/OFF switch. Display shows E1.

7.2 Select measuring range using the MODE key
E1 → E2 → E3 → E4 → E (Scroll)

Measuring range E1 0 - 2 NTU
E2 2 - 20 NTU
E3 20 – 200 NTU
E4 200 – 2000 NTU

7.3 Rinse and fill a clean and dust free vial with the water sample up to the mark (pour the sample along the inner wall of the vial to avoid bubbles, see Note 4). Screw the cap on and align the □-mark on the vial with the □-mark on the instrument. Close the sample chamber using the sample chamber cover.

7.4 Press the ZERO/TEST key. The measurement symbol flashes for approx. 9 seconds. The display shows the result in formazin FNU turbidity units.

Note : If the ambient temperature of the last calibration does not deviate by more than ± 3ºC from the current temp. the measurements are accepted by the unit. If the temperature changes are greater than this, the unit must be re-calibrated.

7.5 If recalibration is necessary, the display shows the following:
Performance of analysis with reduced accuracy

7.6 Press the ZERO/TEST key again.

7.7 Re-calibrate instrument, see 6.0.

8.0 ERROR MESSAGES

EO1 – Light absorption to great reason e.g. dirty optics

+Err – Measuring range exceeded [E24: Hardware limited]

-Err – Result below the lowest limit of the measuring range

oBAT – Replace 9V battery immediately, no further analysis possible

IMPORTANT NOTES

i) Vials must be clean and dry (free of dust). Clean the inside and the outside of the vial using a clean fuzz-free cloth. Finger prints or droplets of water as well as scratches on the sides of the vial can result in errors.

ii) Correctly position vials in the sample chamber.

iii) Conduct tests using closed vials. Always use black vial caps. Completely cover the sample chamber with it's cover.

iv) Always keep sample chamber closed.

v) Bubbles on the inside of the vial may also lead to errors.

vi) Avoid spillage of water in the sample chamber. Water leakage into the housing of the turbidity meter can result in damages to the electronic components and also causes corrosion.

vii) Contamination of the optical components such as light source and photo sensor in the sample chamber can result in errors. Check the condition of the optics at

regular intervals; use a moist cloth and cotton buds for cleaning the optics. Re-calibrate the unit each time it is cleaned.

viii) Large temperature differentials between the turbidity meter, the sample and the operating environment can lead to incorrect measurement due to factors such as the formation of condensate in the area of the lens or on the vial.

ix) Vials and caps should be cleaned thoroughly after each analysis to prevent errors being carried over. Even minor residues can cause errors in the test results.

x) Avoid errors caused by stray-light, do not use the instrument in bright sunlight.

Appendix IV to Chapter 3

SOP 4 BALANCE CHECK & CALIBRATION

1.0 SCOPE

1.1 This Standard Operating Procedure (SOP) describes the six-monthly repeatability checks of all the laboratory balances.

2.0 INTRODUCTION

2.1 Balances in use in the analytical and microbiology laboratories vary in capacity, sensitivity and reproducibility. Routine calibration checks are carried out monthly and repeatability checks are carried out six-monthly on each balance by the Senior Technician.

2.2 The repeatability check is conducted to determine a balance's ability to provide closely similar results for repeated weighing of the same measurand performed under the same conditions of measurement. These conditions are know as repeatability conditions and include:

2.2.1 The same measurement procedure

2.2.2 The same observer

2.2.3 The same balance, used under the same conditions

2.2.4 The same location

2.2.5 Repetition over a short period of time

2.3 Repeatability check will be conducted with two masses i.e. a mass resembling half of the balance's weighing capacity, and a mass resembling the maximum weighing capacity of the balance. For example a balance with a weighing capacity of 0.0 – 200.0 g will be checked at 100 g and at 200 g.

2.4 The error distribution on repeated loading of the same test specimen corresponds to a normal distribution (random error). The measure of the repeatability is thus the standard deviation of the individual measurements.

3.0 PROCEDURE

3.1 The Senior Technician will obtain an empty "Six-monthly Repeatability Check Sheet" (10.0) and the Standard Masses required for the calibration check (refer to "Six-monthly repeatability Standard Masses" (8.0). The Standard Masses are kept in the balance room of the Analytical Laboratory.)

NOTE: Always handle the Standard Masses with a soft tissue or anti-static glove to prevent residual build up of grime. Carefully wipe each Standard Mass with "Snowtex" or "Kleenex" tissue before and after each weighing.

3.2 Clean the weighing surface of the balance with methanol and ensure the immediate area surrounding the balance is clear of dust and other material.

3.3 Check the balance level by checking the "eye" of the balance. If necessary level the balance using adjustment wheels.

4.0 To perform the Repeatability Check, the Senior Technician will:

4.1 Make sure the weighing pan is empty.

4.2 Zero the balance and adjust the scale with the "CAL-MODE'' feature

4.3 Place the first appropriate check weight (as listed on Appendix III) onto the balance and record its weight on the repeatability test worksheet (Appendix II).

4.4 Remove the weight from the balance.

4.5 Without rezeroing the balance, replace the checkweight onto the balance and record its weight again.

4.6 Repeat procedure 4.3 – 4.5 until ten readings are obtained.

4.7 Place the second appropriate check weight (as listed on Appendix III) onto the balance and record its weight on the repeatability test worksheet (Appendix II). Proceed with steps 4.4 – 4.6.

4.8 Input the data to the spreadsheet 6monthlyrepeatability.xls (available in the Chem Lab Computer, My Desktop/My Documents/QC&QA/Calibrations/Balances) for calculation of means and standard deviations for the repeatability test on each of the balances.

5.0 To Pass or Fail a Balance Calibration Check, the Senior Technician will:

5.1 Record a "Pass" in the Pass/Fail column on the "Six-Monthly Balance Repeatability Check Sheet" if the standard deviation is greater than 1.8 times the Reference standard deviation (the acceptable limits) otherwise record "Fail".

Note:
The Reference standard deviation is the standard deviation of the repeatability values obtained at the latest external calibration that will be conducted by a technician from an accredited laboratory or equivalent.

5.2 If calibration passes, attach a completed "Calibration Record" sticker (9.0 (a)) to the balance.

5.3 Inform the Laboratory Manager of any Fail result immediately and attach a "Not in Calibration" sticker (9.0(b)) to the balance.

6.0 To complete the calibration, the Senior Technician will:

6.1 Have Calibration Check Sheets checked and approved by the Laboratory Manager.

6.2 Keep all "Six-Monthly Balance Repeatability Check Sheet"" in the Balance Calibration Folder, which is kept in the Laboratory Manager's office.

7.0 Where a balance calibration does not comply, the Manager Analytical Services will arrange for an accredited laboratory or equivalent to service the balance if necessary.

8.0 List of IAS balances and required masses:

QA Balance No	Model	Serial No.	Weighing Range	Calibrating Mass 1 (g)	Calibrating Mass 2 (g)	Location
7	Shimadzu AEX 200G	D419902028	0.0 – 200.0 g	100	200	Chem Lab
8	Shimadzu AEX 200G	D419902030	0.0 – 200.0 g	100	200	Chem Lab
11	Sartorius BL 3100	12808561	0.0 – 3100.0 g	2000	3000	Chem Lab
4	Sartorius CP 622	15204999	0.00 – 650 g	200	500	Micro Lab

9.0 CALIBRATION RECORD STICKERS

(a)

BALANCE REPEATABILTY CHECK RECORD
QA/QC 01
Balance #
Calibration Performed: / /
Next Calibration Due: / /
Done By:
PASS □ FAIL □

(b)

BALANCE REPEATABILTY CHECK RECORD
QA/QC 01
Balance #
Calibration Performed: / /
Next Calibration Due: / /
Done By:
NOT IN CALIBRATION

9.0 SIX-MONTHLY REPEATABILITY CHECK RECORD

BALANCE TYPE: _____ **QA BALANCE No.:** _____

WEIGHING RANGE: _____ ±_____ g **SERIAL No.:** _____

LOCATION: _____

Calibration Date: _____

Calibrating Weight 1:		Calibrating Weight 2:		
No. of weighing	**Display Weight**	**No. of weighing**	**Display Weight**	**Recalibration Due:**
1)		**1)**		
2)		**2)**		
3)		**3)**		**Calibrated By:**
4)		**4)**		
5)		**5)**		
6)		**6)**		**Checked By:**
7)		**7)**		
8)		**8)**		
9)		**9)**		
10)		**10)**		
(Obtain the mean and standard deviation of data from the spreadsheet….				
Mean		**Mean**		
Std dev		**Std dev**		
Ref Std dev		**Ref Std dev**		
Ref Std dev x 1.8		**Ref Std dev x 1.8**		
PASS/FAIL		**PASS/FAIL**		

TO PASS CALIBRATION, THE STANDARD DEVIATION MUST BE LESS THAN 1.8 TIMES THE REFERENCE STANDARD DEVIATION (FROM LATEST EXTERNAL CALIBRATION CERTIFICATE). If balance fails the calibration check, inform the Laboratory Manager of the results immediately.

Appendix IV to Chapter 3

SOP 5 OPERATION AND CALIBRATIN OF AUTOMATIC PIPETTES AND DISPENSERS

2.0 SCOPE

2.1 This Standard Operating Procedure (SOP) describes the operation and calibration of automatic pipettes and microbiological media dispensers.

2.2 To ensure that the accuracy of the automatic pipettes is maintained, the Senior Technician conducts a calibration check on each pipette on a 3-monthly basis.

2.0 INTRODUCTION

2.1 Pipettes and dispensers are designed for the transfer of known volumes of liquid from one container to another.

3.0 INTERFERENCES

3.1 Air drafts and temperature fluctuations should be eliminated prior to pipette and dispensers calibration.

4.0 APPARATUS AND MATERIALS

4.1 Automatic pipette and plastic tips.

4.2 Analytical balance, capable of weighing to within 0.0001 g.

4.3 Thermometer (0-50°C), calibrated.

4.4 Two glass beakers.

4.5 Dispensers

4.6 1 L Duram Bottle

5.0 REAGENTS

5.1 Deionised water without air bubbles and at ambient temperature.

6.0 GENERAL PIPETTE OPERATION PROCEDURE

6.1 Place the CORRECT plastic pipette TIP firmly on the tip of the automatic pipette. The yellow tips are used on pipettes that deliver less than 100 μL of solution, the blue tips for 100-1000 μL of solution, and the large tips for 1000-5000 μL of solution.

6.2 PUSH the operating button on the top of the pipette down to the FIRST STOP ONLY.

6.3 IMMERSE the PIPETTE TIP vertically in the liquid to a depth of about 1cm.

6.4 SLOWLY RELEASE the operating button and wait for a few seconds.

6.5 SLOWLY PULL the tip out of the liquid, wiping it by touching it to the side of the vessel as you do so.

6.6 PLACE THE TIP against the side of the vessel you are pipetting into and DISPENSE the liquid by PUSHING the operating button SLOWLY down to the FIRST STOP and then FURTHER to the SECOND STOP to empty the tip completely.

6.7 STILL HOLDING THE OPERATING BUTTON DOWN, remove the pipette from the vessel.

6.8 RELEASE the operating button and PRESS the EJECTOR button on the side of the pipette to remove the tip if necessary.

6.9 Make sure that droplets do not stick to the outside of the tip. This is especially important at the low end of the 10-100 μL pipettes and can be avoided by pipetting rapidly and using a clean dry pipette tip.

7.0 CALIBRATION PROCEDURE FOR AUTOMATIC PIPETTES

7.1 Calibration is carried out at a room temperature of about 22 +/- 3°C, constant to +/- 0.5 °C.

7.2 Set the DESIRED PIPETTE VOLUME by rotating the dial on the pipette. Place the correct plastic tip on the pipette.

7.3 Fill a beaker with deionised water and pipette and dispense 3 volumes of water to RINSE THE PIPETTE TIP.

7.4 TARE a separate beaker on the analytical balance.

7.5 PIPETTE the desired volume into the tarred beaker on the balance. Document the weight on the calibration record (see Appendix II).

7.6 REPEAT PROCEDURES ***7.4*** and ***7.5*** ten times for the volume you are calibrating on.

7.7 CALCULATE THE VOLUME delivered in the calibration record form using the known density of water at the temperature you are measuring at (see table below and use the nearest temperature). Alternatively, this can be calculated using the spreadsheet automatic pipettes.xls available in the Chemistry Lab Computer at (My Desktop/My Documents/QC&QA/Calibrations/Automatic Pipettes). This can then be printed out and attached to the Calibration Record on Appendix II.

Temperature (°C)	Density of Water (g/cm^3)	Temperature (°C)	Density of Water (g/cm^3)
20	0.9982	26	0.99681
21	0.9980	27	0.99654
22	0.9977	28	0.99626
23	0.9975	29	0.99597
24	0.9972	30	0.99564
25	0.9970		

8.0 TO PASS/FAIL AN AUTOMATIC PIPETTE CALIBRATION

The Senior Technician will:

8.1 Record a "PASS" in the Pass/Fail Column on the Calibration Record if the accuracy is between 99 – 101%, and the delivery variability is less than 3 %. Attach a completed "Calibration Record" on the pipette **(13.0 (a)).**

8.2 Record a "FAIL" if it does not meet these criteria and retest the automatic pipette. If the retest value still does not meet the criteria, then, refer to the maintenance section of this SOP (9.0) and retest the pipette. If it still does not meet the criteria, then attach a "Not in Calibration" sticker **(13.0(b))** and inform the Laboratory Manager.

9.0 To complete the calibration, the Senior Technician will:

9.1 Have the calibration records checked and approved by the Laboratory Manager.

10.0 MAINTENANCE

10.1 Refer to the manual for the pipette you are using for assembly and maintenance procedures. If you are pipetting corrosive liquids (e.g. acids) frequently, disassemble the pipette, rinse all parts with distilled water and dry before reassembly. If your pipetted volume is wrong the mechanical parts within the pipette may need greasing or some of the seals replaced. If this still does not fix the problem the pipette may need to be re-adjusted, inform the Laboratory Manager (Refer to 8.2).

11.0 CALIBRATION PROCEUDRE FOR MICROBIOLOGICAL MEDIA DISPENSERS

11.1 Fill up a 1 L Duran bottle with distilled water.

11.2 Mount the dispenser on the bottle and adjust the dispenser volume to 9.0 mL

11.3 Dispense 3 volumes of water into a beaker to rinse the dispenser tip.

11.4 Tare a beaker on the analytical balance.

11.5 Dipense the desired volume into the beaker and weigh. Record the weight on the calibration record (**12.0**).

11.6 Repeat procedure **11.5** ten times.

11.7 Repeat procedures **11.2 – 11.6** at a dispenser volume of 10.0 mL and record weights on a separate Calibration record (Appendix II).

11.8 CALCULATE THE VOLUME delivered on the calibration record using the known density of water at the temperature you are measuring at (refer to table on 7.7and use the nearest temperature). Alternatively, this can be calculated using the spreadsheet automatic pipettes.xls available in the Chemistry Lab

11.9 Follow procedure **8.0 – 10.0** to complete the calibration procedure for dispensers.

12.0 AUTOMATIC PIPETTE/DISPENSERS CALIBRATION RECORD

Pipette Serial #: _____

Date Calibrated: _____ **Balance QA No.:**_____

Water Temp (^oC): _____ **Water Density (g/cm^3):** _____

	Calibration Volume ……..cm^3	**Calibrated By:**
Reading #	**Weight (g)**	
1		
2		
3		
4		**Checked By:**
5		
6		
7		
8		
9		
10		**Next Calibration due:**
Average (g)		
Standard Dev. (g)		
Accuracy %		
PASS/FAIL		
Reproducibility %		
PASS/FAIL		

$$\text{Accuracy } (\%) = \frac{\text{weight} \quad \text{x density of water } (g/cm^3)}{\text{calibration volume}(cm^3)} \text{ x } 100$$

$$\text{Delivery variability } (\%) = \frac{\text{standard deviation (g)}}{\text{weight (g)}} \text{ x } 100$$

Note: Automatic Pipettes/Dispensers will be calibrated on a 3-monthly basis.

13.0 CALIBRATION RECORD STICKERS

(a)

CALIBRATION RECORD
QA/QC 01
Autopipette Serial #:
Calibration Performed: / /
Next Calibration Due: / /
Done By:
PASS ☐ FAIL ☐

(b)

CALIBRATION RECORD
QA/QC 01
Autopipette Serial #:
Calibration Performed: / /
Next Calibration Due: / /
Done By:
NOT IN CALIBRATION

Chapter 4 BASIC EQUIPMENT/INSTRUMENTS

In order for a water laboratory to be operational, basic equipment are to be obtained for the analysis of water samples.

1.0 BASIC EQUIPMENT FOR WATER PHYSICAL AND CHEMICAL ANALYSIS

1.1 An water laboratory is recommended to have the following equipment for basic Physical and Chemical Analysis:

(i) Distillation/Deionization Water Apparatus
(ii) pH Meter
(iii) Electrical Conductivity Meter
(iv) Residual Chlorine Meter
(v) TDS meter
(vi) Turbidity meter
(vii) Analytical Balance
(viii) Volumetric Glassware – pipettes
(ix) Glassware – Beakers, Measuring Cylinders, Glass stirrers
(x) Sampling bottles – plastic and glass
(xi) Dissolved Oxygen Meter
(xii) Fume Hood
(xiii) Thermometers
(xiv) Basic colorimeter for some chemical tests

1.2 When the water laboratory is fully functional and ready for expansion, the following equipment may be added to the above list:

(i) Atomic Absorption Spectrophotometer
(ii) UV/Vis Spectrophotometer
(iii) Oven
(iv) Digestion Block
(v) Muffle Furnace
(vi) Temperature/Humidity Meters
(vii) Reference Thermometers and Standard Masses
(viii) Volumetric Glassware – volumetric flasks, burettes

2.0 BASIC EQUIPMENT FOR WATER MICROBIOLOGICAL ANALYSIS

2.1 A water laboratory is recommended to have the following equipment for basic Microbiological Analysis:

(i) Distillation/Deionization Water Apparatus
(ii) Hot air oven (for sterilization)
(iii) Membrane Filtration Units
(iv) Vacuum pump
(v) Petri dishes (can get either pre-prepared and pre-sterilized ones nowadays)
(vi) Incubators
(vii) Automatic Pipettors and Dispensers
(viii) MacCartney Bottles (for Multi-tube analysis, biochemical testing and media preparation)

(ix) Filter Funnels
(x) Glassware – 50 mL and 100 mL beakers, 1 L Conical Flask (with a Sidearm) for Membrane Filtration, 1 L Measuring Cylinders
(xi) Sampling Bottles – 250 mL , 500 mL , 1 L and 2L
(xii) Refrigerator
(xiii) Eskis or chill bins

2.2 When the water laboratory is fully functional and ready for expansion, the following equipment may be added to the list in 2.1:

(i) Autoclave
(ii) Biological Safety Cabinets
(iii) Bunsen burner and gas supply

3.0 INVENTORY

The laboratory is to maintain an inventory of major equipment in the form of an electronic database. Each piece of equipment is to be identified with a unique number of the form XYZ Laboratory No. 01, 02, 03 and so forth.

Details such as description, supplier, contact details for servicing, purchase date, serial number, location, consumables required etc; are to be recorded.

4.0 COMMISSIONING OF NEW EQUIPMENT

Before beginning routine use, new equipment is to be fully evaluated by the Laboratory Manager 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
- Manufactures claims on performance.

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

5.0 LOGS & STANDARD OPERATING PROCEDURES (SOPs)

All instrumental equipment operated in the laboratory comes supplied with operating instructions. These manuals are primary source of information about the operation, calibration and maintenance of the equipment. Original copies of all instructions are to be stored in files in the Laboratory Manager's office. However, because supplied manuals are usually too detailed and complicated for everyday use, abbreviated operating instructions are to be written up as SOPs (refer to Chapter 7 and Appendices to Chapter 3) and a laminated copy of the SOP is to placed next to the equipment.

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

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

6.0 AUTHORISATION

Once they have been fully trained, all laboratory staff are authorized to operate equipment.

7.0 CALIBRATION

For calibration of equipment/instruments, refer to Chapter 3.

8.0 MAINTENANCE

The Laboratory Manager is responsible for the proper maintenance of all laboratory equipment. Proper maintenance of laboratory equipment is a key ingredient to its performance and reliability. 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 to be prepared. 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 the Laboratory Manager.

Items that fail maintenance checks are to be tagged with a sticker and taken out of use until repaired.

9.0 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.

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 authorized service agents themselves.

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

10.0 TRANSPORT AND STORAGE

Major equipment items, such as instruments, are not to be moved. If relocation is necessary, then the manufactures 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 should be carried around. If any other balance has to 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 carried out of use for a significant length of time it should be prepared for storage according to the manufactures instructions. Before use gain, the proper flushing and/or conditioning steps need to be carried out before recalibration.

11.0 REFERENCES

11.1 International Accreditation New Zealand, Specific Criteria for Chemical Testing, 2005

11.2 Institute of Applied Sciences, Standard Operating Procedures, QM 7.0 Equipment

Chapter 5 REAGENT AND MICROBIOLOGICAL MEDIA SUPPLY

1.0 REAGENTS AND MEDIA

Correct reagents and media are essential in achieving accurate results. Reagents refers to all chemicals including analytical standards (refer to Part II of this Manual) and media refers to either dehydrated microbiological or pre-prepared media. The Laboratory is to obtain reagents and media that have the formulation specified by the analysis procedures. Chemicals of Analytical Reagent (AR) grade should be obtained for preparing analytical standards (refer to Part II of this Manual).

It is also important that an adequate supply of reagents and media are available in the Laboratory at all times. The use of expired reagents and media would compromise the integrity of the analytical data.

2.0 INVENTORY OF REAGENTS AND MEDIA

It is important to have an inventory of all the reagents and media present in the laboratory. This inventory should be carefully drawn. It should examine the monitoring programme for the water lab and decide the amount of reagent and media required for analysis between supply orders. For example, if the laboratory does 10 Total Coliforms analysis per month then it should have around 20 media broth, plates and so on present in the laboratory which are not expired. By referring to the inventory on how much reagent/media is present in the lab (and expiry dates), the laboratory would ensure that there is always a constant supply of reagents/media present. It is advisable to have spare or extra reagents/media present in the laboratory for unforeseen monitoring needs that may arise.

3.0 SUPPLIERS OF REAGENTS AND MEDIA

The Laboratory may purchase its Reagents and Media from the following suppliers;

No	Supplier	Items Supplied
1	Biolab Ltd Private Bag 102 922 North Shore Mail Centre Auckland New Zealand Ph: +64 9 980 6700 Fax: +64 980 6788	Chemicals, Equipment, Microbiological Media and Reagents
2	Livingstone International 106 – 116 Epsom Road Roseberry NSW 2018 Australia Ph: +61 2 8344 7333 Fax: +61 2 9313 6444	Chemicals, Equipment, Microbiological Media and Reagents
3	Banksia Scientific Ltd P O Box 529 Bulimba Queensland 4171 Australia Ph: +61 7 3902 3000 Fax: +61 7 3217 9869	Equipment and Chemicals

No	Supplier	Items Supplied
4	Sigma Chemicals Pty Ltd 228 Balcatta Road Balcatta Perth Western Australia Ph: +61 8 9345 2233 Fax: +61 8 9345 4012	Chemicals

4.0 RECEIPT OF REAGENTS AND MEDIA

The following steps are applicable for both chemicals and media.

4.1 It is important for the laboratory staff member receiving the chemicals to be aware of the proper handling, storage, and disposal of the chemical. This information can be obtained from the Material Safety Data Sheet(MSDS) which is usually sent with the chemicals. All chemicals MSDS are also to be stored in a common place in the Laboratory where they can be easily accessed.

4.2 Retrieve packing slips (normally on top of carton) and check for any visual damage to cartons or packing materials. If there is any visual damage record this on packing slip.

4.3 Remove packing material carefully from the inside of the carton and place all chemicals on a bench. Check each chemical for quantity ordered and supplied. Record any discrepancies on the packing slip which will denote the quantity ordered and supplied.

4.4 Make a copy of the packing slip and forward this slip to the Accounts Clerk for further checking against the original order.

4.5 Stick a "Chemical Stores" label (see below) on each bottle of chemical and fill in the necessary details, i.e., fill in the date when the chemical was received. Refer to 6.0 for the policy on the “Expiry Date”. Date taken into stores should be filled in once the chemicals are placed in the Stores.

Laboratory Stores Label
CHEMICAL/MEDIA

Date In Stores:

Date Out Stores :

Date Opened :

Expiry Date :

4.6 Record the chemicals received in the Chemicals or Media Register.

5.0 STORAGE OF REAGENTS AND MEDIA

5.1 REAGENTS STORAGE

5.1.1 Chemicals should be stored in a manner that minimizes exposure to hazardous substances, chemical spills, the possibility of fire or explosions, and reactivity hazards. The storage of chemicals will depend on its nature, whether it is radioactive, flammable, corrosive or combustible. Always refer to its package instructions or its MSDS on its storage guidelines.

5.1.2 Chemicals in the Laboratory or Stores are to be stored in appropriate cabinets and shelves according to the following guidelines:

5.1.2.1 Chemicals should be stored in an uncluttered manner on shelves with solid back and side frames that are firmly secured.

5.1.2.2 Avoid storing chemicals in passageways, under tables, on bench tops, in hoods or stored as to block emergency equipment or exit areas.

5.1.2.3 Volatile toxic substances and odoriferous chemicals should be stored in a ventilated cabinet.

5.2 MEDIA STORAGE

5.2.1 Dehydrated media is to be stored in a cool, dark, minimal humidity environment e.g. not near autoclaves. The higher the environment moisture content, the greater the possibility of degradation of the various constituents of the medium. If stored under desirable conditions, most dehydrated media will remain in good condition for several years. However, a few products, which contain ingredients of high sensitivity, are less stable especially if the moisture is allowed to rise.

5.2.2 Do not open Media containers for prolonged periods. Carefully close the media containers lid/closure when closing containers to maximize shelf-life. Do not use dehydrated media that are caked, cracked or have shown colour change.

5.2.3 Store supplements and additives appropriately e.g. under refrigeration where this is required.

5.2.4 Store light sensitive chemicals in the dark or wrap in aluminium foil.

6.0 EXPIRY DATES OF CHEMICALS AND MEDIA

6.1 The Expiry Date for dry, inorganic chemicals and aqueous acids, organic solvents or aqueous solutions will not be noted on the label. The quality of these chemicals will be monitored through the use of Quality Control checks such as in-house references, Standard Reference Materials and Proficiency Program samples. If these values are not satisfactory, then the chemicals used are to be validated. If unsatisfactory values are still obtained after two attempts, and other possible reasons for the unsatisfactory results are eliminated, then the chemical used is to be discarded.

6.2 Dehydrated media are not to be used beyond their labeled expiry dates.

7.0 REFERENCE

7.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

7.1.1 SOP No. SSS 17(R1) Solutions, Reagents, Standards Preparation, Validation and Documentation

7.1.2 SOP No. SSS 18(R1) Chemical Procurement, Distribution and Storage

7.1.3 SOP No. AS 4 Suppliers Performance Appraisal

7.1.4 MM 310(R1) Media, Reagents Purchasing, Preparation and Validation

Chapter 6 STAFF KNOWLEDGE and TRAINING

1.0 STAFF KNOWLEDGE

1.1 **For water physical and chemical analysis,** staff members are to possess the following knowledge and skills:

1.1.1 preparation of standards and reagents

1.1.2 interpretation of analysis data

1.1.3 proper sample collection and preservation

1.1.4 operation and calibration of instruments

1.1.5 routine analytical testing

1.1.6 use of quality controls during analysis.

1.2 **For water microbiological analysis,** staff members are to possess the knowledge and skills outlined in 1.1 in addition to the following:

1.2.1 sterilization techniques

1.2.2 media and glassware preparation

1.2.3 counting of Colonies

It is therefore important that all laboratory staff member are trained to acquire these knowledge and skills upon recruitment in a Laboratory.

2.0 NEW LABORATORY STAFF MEMBERS TRAINING

New staff members are to undergo a training schedule that includes the following:

(i) A briefing from the Laboratory Manager on the functions of the laboratory, its place in the overall organization, the services it provides, the laboratory's structure staffing arrangements.

(ii) A tour of the laboratory and introduction of new staff.

(iii) An opportunity to study the laboratory manuals and other laboratory documentation.

(iv) Instructions on the duties required, including a copy of the relevant job description.

(v) Basic safety instructions and a description of safety policy and equipment available.

(vi) The Safety Manual must be read and signed off.

(vii) 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 is to be provided as required throughout the training period.

3.0 ONGOING TRAINING

A development/training programme is to be developed 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. The Laboratory Manager/Human Resources section is to maintain records of prior educational and professional qualifications and hold records of subsequent training in the Staff Training File. These records include:

(i) Educational and Qualifications, other experience and starting date

(ii) Training courses attended, including certificates awarded

(iii) Proficiency in laboratory operating procedures.

Each year annual staff appraisals are to 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 (refer to 4.0) will be reviewed at this time and new training need 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 members are to be encouraged to develop specialized skills, for instance in flow injection analysis, laboratory management, Word and Excel Spreadsheet use and processing, by attending external courses and seminars where appropriate, and by contact with staff in similar organizations.

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

4.0 STAFF TRAINING RECORDS

All staff members are to have a training record such as that outlined in Appendix I to Chapter 6, where levels of competency can be assigned by the Laboratory Manager upon assessment of the Laboratory staff members. The competency level may be categorized as follows:

4.1 Level 1 – Staff member has been trained in the analytical procedure

4.2 Level 2 – Staff member has conducted the analysis under supervision

4.3 Level 3 - Staff member can conduct analysis unsupervised and has achieved satisfactory results from Proficiency Programs (refer to Chapter 2, section 5.0)

5.0 REFERENCES

5.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures:

5.1.1 QM 6.0 Staff

5.1.2 AS 54(R4) Laboratory Staff Training

Appendix I to Chapter 6

Laboratory Staff Member Training Record

Name : _____

Position: _____

Date Commenced: _____

Supervisor: _____

Title of Standard Operating Procedure	SOP Numbers	Level 1	Level 2	Level 3
pH	WP 01			
Total Suspended Matter in Water	WP 02			
Determination of Total Dissolved Solids in Water at 103 - 105°C.	WP 03			
Total Coliform in Water (Membrane Filtration)	MM 345			

Chapter 7 ANALYSIS STANDARD OPERATING PROCEDURES (SOPs)

1.0 POLICY

A Water Laboratory is to document any tests conducted in the Laboratory in the form of an Analysis Standard Operating Procedure (SOP) and to ensure that staff members are trained in the use of the SOP (refer to Chapter 6).

The SOPs are also to be available for use to all staff members and are to maintained under a Document Control System (refer to Part II of this Manual, Section 1)

2.0 ANALYSIS SOPs FORMAT

All analysis SOPs should be written according to the following format and should include the following sections:

- Identification (title and method number)
- Reference Method - the source of the method
- Scope
- Definitions
- Principle
- Reagents/Media
- Apparatus
- Quality Control
- The uncertainty and or detection limit
- Safety Notes - Any safety precautions to be taken
- Sampling/Sample Preparation - Handling, transporting, storing and preparation of test items
- Environmental conditions required
- Preparation of reagents
- Preparation of standards
- Procedure - a step-by-step description of the procedure
- Calculation - full description of calculations
- Interpretation of Results
- Acceptance Criteria - criteria required for release of results
- Recording of Results
- Reporting of Results
- Issue To – Laboratory Sections to which copies of the SOP is issued
- Appendix I – Reason for Revision, contains any revision changes to the SOP

3.0 SOURCES OF ANALYTICAL SOPS

It is preferable that methods have been published either in international, regional or national standards, or by reputable organisations, or 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 the customer agreement must be obtained and must include specifications of customer requirements.

Commercial test kits (kits) will require further validation (refer to Chapter 2) if the Laboratory is unable to source the validation data from manufacturers with a recognised quality assurance system; reputable validation based on collaborative testing e.g. AOAC Official Methods or independently reviewed methods e.g. AOAC Performance Tested Methods.

4.0 ANALYTICAL METHOD DEVELOPMENT & VALIDATION

All new analytical procedures are to be validated before being used in the Laboratory. For the initial validation of the procedure, which involves the determination of the accuracy and precision of the method, refer to Chapter 2, section 3.1). The accuracy and precision data are to be filed in a Method Validation File and kept in the Laboratory.

5.0 STAFF TRAINING IN ANALYTICAL SOPs

Staff members are to be trained in how to perform analytical methods and such training is to be recorded in their individual training record (refer to Chapter 6)

6.0 EXAMPLES OF SOPS

The following examples of SOPs are outlined in Appendix I:

6.1 SOP No. XX01 Determination of pH in Water

6.2 SOP No. XX02 Determination of Orthphosphate in Water (Molybdenum Blue Method)

6.3 SOP No. XX03 Total Coliforms in Water (Membrane Filtration)

7.0 REFERENCE

7.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). *Standard Methods for the Examination of Water and Wastewater*, 21st Edition. American Public Health Association (APHA), Washington, D.C;Method 1020B Quality Control, 9020 Quality Assurance.

7.2 Institute of Applied Sciences Analytical Laboratory Standard Operating Procedures Nos:

7.2.1 WP 201(R2) Determination of pH in Water

7.2.2 WC 250(R2) Determination of Orthophosphate in Water

7.2.3 MM 282(R3) Total Coliform (Filtration Method)

Appendix I to Chapter 7

SOP No. XX 01 DETERMINATION OF pH IN WATER

1.0 REFERENCE METHOD

1.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). Standard Methods for the Examination of Water and Wastewater, 21st dition. American Public Health Association (APHA), Washington, D.C;Method 4500-H+ B.

2.0 PRINCIPLES

2.1 The basic principle of electromagnetic pH measurement is the determination of the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. The instrument is calibrated using two buffers and its performance is checked using a third buffer.

Samples must be dilute aqueous simple solutions (<0.2M). Determination of pH cannot be made accurately in non-aqueous media, suspensions, colloids, or high-ionic-strength solutions.

3.0 INTERFERENCES

3.1 The sensitivity can be reduced by the presence of oil in the samples. Measurement errors in oil-containing waters may be prevented by washing the electrode before each measurement is taken, as in 3.2.

3.2 First rinse the electrode with soap or detergent, then rinse with water. After this, rinse the electrode with methanol (10%), followed by deionised water, which in turn is followed by dilute HCl rinse (0.1N) for approximately 10 seconds, and finally with more deionised water.

3.3 The sensitivity of the electrode may also be affected if the pH measured is either very low or very high. Measurement errors can be prevented by washing the electrode as mentioned above (3.2).

3.4 Sodium ion is the principal interference of the pH electrode, causing increasing error at high pH (pH>10) and at high temperature. Because the pH membrane is composed of low sodium error glass, error due to sodium is negligible when measuring at pH values less than 12.

3.5 Good care of the electrode is of paramount importance: see IAS SOP PMET 8.00. The electrode should be stored in electrode Storage solution or alternatively in pH buffer 7.0. Never store electrode in deionised or distilled water.

4.0 SAMPLE COLLECTION/PRESERVATION

4.1 Teflon (TFE) bottles are the best containers for collecting water samples but in the absence of TFE, polyethylene bottles with polyethylene caps can be used.

4.2 All containers need to be rinsed with concentrated HCl or soaked for 24 hours in 10% HCl bath. To prepare 10% HCl bath, use 1:9 ratio of concentrated HCl to deionised water. Upon removal, rinse thoroughly at least 5 times with deionised water.

4.3 pH readings can be taken on site but if samples are being collected, rinse the container at least twice with sample before filling to the brim.

4.4 Do not filter or acidify samples for pH measurements.

4.5 Samples have to be analysed on the same day of collection and immediately after receipt.

5.0 PRECISION/BIAS AND DETECTION LIMIT

5.1 By careful use of a pH meter with a good electrode, a precision of ± 0.02 pH unit and an accuracy of ± 0.05 pH units can be achieved. Detection limit is not applicable in this case.

6.0 QUALITY CONTROL

6.1 Calibrate the pH meter prior to use for analysis with the Buffers References: pH 7.00 ± 0.02, 4.00 ± 0.02 and check the calibration of the pH meter with Buffer Reference 9.22 ± 0.02.

6.2 Analyse samples in duplicate.

6.3 Duplicate determinations should agree within 4% of their Analyse samples in duplicate.

7.0 APPARATUS

7.1 pH Meter:

7.2 Beakers:

Preferably use polyethylene or Teflon (TFE) beakers.

7.3 Stirrer:

Use either a magnetic, TFE- coated stirring bar or a mechanical stirrer with inert plastic- coated impeller.

8.0 REAGENTS

All reagents should be kept in polyethylene, polypropylene, polycarbonate, or polystyrene containers. Only analytical grade (AR grade) reagents are to be used, unless otherwise stated.

8.1 pH Buffers:

pH buffers may be prepared using the following methods:

- **Method 1: Use of Commercial Tablets**

BDH Laboratory Supplies commercial tablets are available in the laboratory, and these may be used to prepare buffer solutions. In general, the instructions (for this particular brand of tablets) are described as follows:

8.1.1 pH 4.00 ± 0.02 Buffer:

Dissolve one tablet in a small quantity of deionised water in a 50 mL beaker. Once dissolved, transfer the solution quantitatively into a 100 mL volumetric flask and make up to the mark using deionised water. Thus, a solution of pH 4.00 is produced at 20°C. This solution has a shelf life of 1 month.

8.1.2 pH 7.00 ± 0.02 Buffer:

Dissolve one tablet in a small quantity of deionised water in a 50 mL beaker. Once dissolved, transfer the solution quantitatively into a 100 mL volumetric flask and make up to the mark using deionised water. Thus, a solution of pH 7.00 is produced at 20°C. This solution has a shelf life of 1 month.

8.1.3 pH 9.22 ± 0.02 Buffer:

Dissolve one tablet in a small quantity of deionised water in a 50 mL beaker. Once dissolved, transfer the solution quantitatively into a 100 mL volumetric flask and make up to the mark using deionised water. Thus, a solution of pH 9.22 is produced at 20°C. This solution has a shelf life of 1 month.

NOTE: The instructions for solution preparation may vary, therefore, always check the bottle labels for instructions and expiry dates of the tablets.

- **Method 2: Alternative to Commercial Tablets**

8.1.4 Commercially prepared buffer solutions (of ~4.00, ~7.00, and ~9.00 pH) can be used.

9.0 PROCEDURE

Follow the IAS Standard Operating Procedure for the pH Meter (SOP No. IO 650).

9.1 Instrument Calibration:

9.1.1 Before use, remove the glass electrode from the storage solution, rinse with deionised water, and blot dry with soft tissue.

9.1.2 Calibrate the pH meter with the pH 7 buffer using the standard operation procedure.

9.1.3 Make preliminary reading of sample.

9.1.4 If pH is < 7, set slope using pH 4 and pH 7 buffers. If pH > 7, set slope with pH 7 and pH 9.22 buffers (Refer to Operational SOP for pH meter, Appendix I to Chapter 3).

9.2 Sample Analysis:

9.2.1 Remove electrode from buffer, rinse with deionised water and rinse with sample solution to be measured, blot dry, and place in test solution/sample.

9.2.2 Establish equilibrium between electrodes and sample by stirring the sample to insure homogeneity; stir gently using a stirrer to minimise CO_2 entrapment. Press **measure**.

9.2.3 Record pH reading when **READY** sign appears. Record two more readings of the same sample by repeating step 9.2.2.

10.0 CALCULATION

10.1 Since the pH meter gives direct pH readings, pH calculation is not necessary. Report pH as the mean of the three readings with an accuracy of 0.05 pH units for values between 2.00 and 12.00. Values below 2.00 and above 12.00 should be reported with an accuracy of 0.1 pH unit.

11.0 RECORDING OF RESULTS

All analysis data are to be recorded on the pH in Water Worksheet (Refer to Chapter 2, Appendix I.

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Appendix II to Chapter 7

SOP No. XX02 DETERMINATION OF ORTHOPHOSPHATE IN WATER (Molybdenum Blue Method)

1.0 REFERENCE METHOD

1.1 Norsk Institutt for Vannforskning. Minirintest 8306; Ortofosfat, total fosfor, nitrat, ammonium og totalnitrogen. 0-8101402, Oslo, **Danish Standard 291**.

2.0 PRINCIPLES

2.1 In an acidified (0.2 mol/L sulphuric acid) solution, orthophosphate forms antimony-12-molybedophosphoric acid with antimony (III) and molybdenum. This complex is then reduced with ascorbic acid to a heteroploycomplex (molybdenum blue complex). The complex's absorbance at 880 nm is proportional to the orthophosphate content.

3.0 INTERFERENCES

3.1 Silicate forms a blue complex with molybdate. Under usual circumstances, colour will develop so slowly that concentrations lower than 5 mg/L Si will not interfere. Sulfide, nitrite, and arsenate interfere when their contents are higher than 2 mg/L sulfide, 1 mg/L nitrite and 2 μg/L arsenate respectively. Treatment of samples containing these substances is described in the Danish Standard 292, section 5.5.

4.0 SAMPLE COLLECTION/PRESERVATION

4.1 Teflon (TFE) bottles are the best containers for collecting water samples. Polyethylene bottles with caps can also be used.

4.2 All containers need to be rinsed with concentrated HCl or soaked for 24 hours in 10% HCl bath.

4.3 If the sample has to be filtered this has to be done as soon as possible, and preferably on the sampling site. The sample must be filtered before preservation.

4.3.1 For filtration use G/FC or 0.45μm millipore filters. Rinse the filtration setup with deionised water. Place G/FC filter with tweezers and filter the sample. While filtering, discard the first 20 mL of the filtrate.

4.4 Add, as soon as possible, 1 mL sulphuric acid (4 mol/L) for each 100 mL of sample. Store the preserved samples in a refrigerator.

5.0 QUALITY CONTROL

5.1 Analyse samples in duplicate.

6.2 Duplicate determinations should agree within 4% of their average.

6.3 Analyse IAS In-house Standard Reference Water # 006. Enter value in Phosphate Quality Control File which contains the latest mean and standard deviation of the $P\text{-}PO_4$ values. The value should fall within 2 standard deviation (2SD) of the mean.

6.0 APPARATUS

6.1 Spectrophotometer:

Shimadzu UV-Vis Spectrophotometer, UV 2501 PC or Perkin Elmer UV/VIS spectrophotometer (Lambda 3B) for use at 880 nm, providing a light path of 100 mm.

6.2 Vacuum Pump and Filtration Unit:

This includes G/FC paper and Millipore filter papers.

6.3 Acid Washed Glassware:

Glassware should not be used for any other analysis. Clean all glassware with hot hydrochloric acid or soak it in 10% hydrochloric acid for 24 hours and rinse thoroughly. Glassware used for developing color must be rinsed before analysis with 4 M sodium hydroxide to remove molybdenum blue that can be adsorbed on the walls. Rinse thoroughly 5 times with water again.

7.0 REAGENTS

All reagents should be kept in polyethylene, polypropylene, polycarbonate, or polystyrene containers. Only analytical grade (AR grade) reagents are to be used, unless otherwise stated.

7.1 Sulphuric Acid (4 mol/L):

Cautiously add, while stirring, 110 mL concentrated sulphuric acid, H_2SO_4, to approximately 350 mL water. Let the solution reach room temperature and make up to 500 mL.

7.2 Sulphuric Acid (0.04 mol/L):

Dilute 10 mL 4 mol/L sulphuric acid to 1 L.

7.3 Ascorbic Acid Solution (5%):

Dissolve 5 g L-ascorbic acid, $C_6H_8O_6$, in 100 mL water. Store this solution in a brown bottle in the refrigerator. This solution is stable for several weeks as long as it is colourless.

7.4 Reagent Solution:

The reagent solution is made up of the following solutions:

Sulphuric Acid: Add 120 mL concentrated sulphuric acid, H_2SO_4, to 170 mL of water in a 600 mL beaker cautiously and while stirring. Let the solution reach room temperature.

Molybdate Solution: Dissolve 13 g ammonium molybdate, $(NH_4)_6MoO_{24}.4H_2O$, in 100 mL water.

Antimony Solution: Dissolve 0.35 g potassium antimony tartate, $K(SbO)C_4H_4O_6$, in 100 mL of water.

Reagent Solution: Add to the sulphuric acid while stirring, first the molybdate solution and then the antimony solution. Mix thoroughly and pour the solution in a 500 mL volumetric flask and make up to 500 mL. Store the reagent solution in a dark glass bottle made of resistant glass. The solution is stable for at least 2 months.

7.5 Stock Phosphate Solution (50 mg/L P):

Dissolve 0.2197 g of oven dried (1 hour at 105^{o}C) potassium dihydrogen orthophosphate, KH_2PO_4, in water in a 1 L volumetric flask, add 10 mL sulphuric acid (4 mol/L) and make up to 1 L.

7.6 Standard Phosphate Solution (1 mg/L):

Dilute 10 mL of the stock phosphate solution to 500 mL in a volumetric flask. This solution must be made on the same day it is to be used.

7.7 Hydrochloric Acid (≈ 2 mol/L):

Add 160 mL of concentrated hydrochloric acid, HCl, to approximately 500 mL of water and make up to 1 L. Used for cleaning.

7.8 Sodium Hydroxide (≈ 4 mol/L):

Dissolve 160 g sodium hydroxide, NaOH, in 800 mL of water while stirring and cooling in ice bath; make up to 1 L. Used for cleaning.

8.0 <u>PROCEDURE</u>

8.1 Calibration:

Pipette 5, 15, 25, 30 and 40 mL of the 1 mg/L P standard phosphate solution into five different 250 mL volumetric flasks and add 2.5 mL sulphuric acid (4 mol/L) to each volumetric flask and mix well. Make up to 250 mL. The solutions contain 20, 60, 100, 120 and 160 μg/L P. Pipette 25 mL of the acidified standard solutions to 150 mL Erlenmeyer flasks. Use 25.0 mL of sulphuric acid (0.04 mol/ L) as reagent blank.

8.2 Colour Development:

Add to each flask (standard or sample), first 1.0 mL of the ascorbic acid solution, mix and wait for 30 seconds then add 1.0 mL of the reagent solution and mix again.

8.3 Measuring:

Between 10 and 30 minutes after the last reagent was added, measure the five calibration solutions absorbance at 880 nm in a 100 mm cell by following Standard Operating Procedure for Perkin Elmer Lambda 3B UV/VIS Spectrophotometer.

8.4 Analysis:

Pipette 25 mL of the fresh or preserved sample to an Erlenmeyer flask. Include two sample blanks in addition to standard blank (25 mL of 0.04 mol/L). Develop colour as described above and measure the blank and samples absorbances at 880 nm in a 100 mm cell. Be aware of any dilution.

9.0 CALCULATION

9.1 PECSS software program reports sample values as μg/L $P\text{-}PO_4$ and can be read off directly. Before reporting subtract the sample blank (average of 2 blank values) from each sample value, that is,

μg/L $P\text{-}PO_4$ = $P\text{-}PO_4$ (from PO_4 column) - Blank

Note: If sample has been diluted, DF should be entered as the FACTOR into PECSS program.

9.2 Calculation can also be carried out by reading off μg/L $P\text{-}PO_4$ from calibration curve and using the following equation:

μg/L $P\text{-}PO_4$ = Graph Reading x DF

where DF is the dilution factor. If values are to be reported as PO_4, then the value obtained from the formula in 10.2 above should be multiplied by 3.06.

9.3 Report results as mg/L $P\text{-}PO_4$. For concentrations up to 0.5 mg/L, round off the figure to 0.01 mg/L and for concentrations between 0.5 and 1.0 mg/L, round off the figure to 0.02 mg/L. Up to 10 mg/L, the value should be rounded off to 0.1 mg/L. Values above 10 mg/L round off to whole numbers. Values below 0.018 mg/L should be reported as <0.018 mg/L $P\text{-}PO_4$.

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Appendix III to Chapter 7

SOP No. XX03 Microbiological Analysis of Water: Total Coliform Count – Membrane Filtration Method

1.0 REFERENCE METHOD

1.1 Main Reference:

1.1.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(1998). *Standard Methods for the Examination of Water and Wastewater*, 20th Edition. American Public Health Association (APHA), Washington, D.C;Method 9222 B Standard Total Coliform Membrane Filter Procedure.

1.2 Supplementary Reference(s):

1.2.1 Queensland Health Scientific Services, (May 1996), SHW2 Water Microbiology – Coliforms – Membrane Filtration Method.

2.0 SCOPE

1.1 This method is designed for the detection and enumeration of coliform bacteria in water by means of membrane filtration.

Note:
Membrane filtration is suitable for enumerating microorganism when the turbidity of the water is low.

3.0 DEFINITIONS

3.1 Coliforms: Gram negative, non-sporing, oxidase negative rod-shaped bacteria, belonging to the family Enterobacteriaceae, capable of aerobic and facultatively anaerobic growth, and able to produce acid from lactose within 48 hours at 37°C (i.e. possess β-galactosidase). This definition is not taxonomic, but rather defines in a practical working sense a group of bacteria with public health significance in relation to the microbiological quality of water.

4.0 PRINCIPLE

4.1 Samples of water are filtered through a 0.45 micron filter and placed on coliform selective agar plates. Alternatively, the filter may be placed on a coliform liquid medium pipetted on treated absorbent pad. Colonies demonstrating the appropriate morphology are counted and the results are reported.

5.0 EQUIPMENT/REAGENTS/MEDIA/CONTROL CULTURES

5.1 Bunsen burner

5.2 Colony counter (Quebec type)

5.3 Filtration manifold

5.4 Forceps

5.5 Incubator adjusted to 35 ± 0.5°C

5.6 m-Endo Agar LES

5.7 m-Endo Broth MF

5.8 Lauryl Sulphate Broth (LST)

5.9 HCl (1M)

5.10 NaOH (1N)

5.11 Screw cap jars

5.12 Sterile 0.45 micron grid filters specific for water testing

5.13 Sterile 0.85% saline

5.14 Sterile water

5.15 Sterile 60 x 15 mm petri dishes

5.16 Sterile 9 mL 0.1% peptone water

5.17 Sterile 0.1 mL, 1.0 mL and 5.0 mL and 10.0 mL pipette tips

5.18 Sterile 47 mm absorbent filer pads

5.19 Vacuum source with trap

5.20 Waterbath adjusted to 46 $\pm$ 2.0°C

5.21 Control Cultures:

5.21.1 Positive Control: *Escherichia coli* NZRM 2577. This microorganism produces typical coliform colonies and gives reactions of a typical coliform when used with the media and tests of this method (see sections 13.3, 13.6.4.1 and 14.4.2).

5.21.2 Negative Control: *Enterococcus faecalis* NZRM 1106. This microorganism does not grown on m-Endo media (see section 13.3).

6.0 QUALITY CONTROL

6.1 A field blank (prepared by autoclaving a 250 mL bottle of distilled water) is kept under similar conditions as the samples to be analysed during transportation to the laboratory. The temperature of the field blank is measured using a thermometer as soon as the samples reach the laboratory to check that the samples are being maintained at the specified temperature. This temperature is then recorded in the relevant Worksheets. The field blank is also to be analysed along with the samples.

6.2 A procedural blank (prepared at the laboratory for detecting any contamination of reagents or equipment used) is analysed along with the samples.

6.3 For coliform verification (Confimatory Tests), see 13.6.

6.4 Test each medium lot against previously acceptable lot for satisfactory performance by:

6.4.1 Make dilutions (1 x 10^{-5} dilution) of the Positive Control – *E.coli* and filter appropriate volumes to give 20 to 60 colonies per filter.

6.4.2 Before use, test each batch of laboratory prepared m-Endo media with positive and negative controls (Refer to MM 310). Check for coliform contamination at the beginning and end of each filtration series by filtering 20 – 30 mL of dilution or rinse water through filter.

6.4.3 If controls indicate contamination, reject all data from affected samples and request for resampling.

6.5 Refer to SOP No. MM 297 for the preparation and maintenance of Reference Control Cultures.

6.5.1 The table below outlines the morphological characteristics of the Reference Control Cultures used on the media of this procedure:

Table 1 Morphological Characteristics of the Control Organisms

	E. coli	*E. faecalis*
4. mENDO Agar (blank: pink)	The colonies are round, shiny, circular and are maroon in color with or without a green metallic sheen.	No growth.
5. mENDO Broth MF (blank: pink)	Smooth, shiny, round colonies that are pink in color with or without a green metallic sheen.	No growth.
6. LST Broth (blank: yellow)	Broth is turbid and the gas produced is collected in the Durham tube.	Broth is turbid but NO production of gas.

6.6 For the Acceptance Criteria of the analyses results, see section 15.0 of this SOP.

7.0 SAFETY NOTES

7.1 Take care when alcohol flaming forceps.

7.2 Check that gas taps have been closed and hot plates switched off, both at the appliance and the power point, when no longer required.

8.0 SAMPLE PRESERVATION AND STORAGE

8.1 General:

8.1.1 Start Microbiological analysis of water samples as soon as possible after collection to avoid unpredictable changes in the microbial population.

8.2 Drinking water for compliance purposes:

8.2.1 Preferably, hold samples at <10°C during transit to the laboratory.

8.2.2 Analyse samples on day of receipt whenever possible and refrigerate overnight if arrival is too late.

8.2.3 Do not exceed 30 h holding time from collection to analysis for coliform bacteria.

8.3 Non-potable water for compliance purposes:

8.3.1 Hold source water, stream pollution, recreational water and waste water below 10°C during a maximum transport time of six h.

8.3.2 Refrigerate these samples upon receipt in the Laboratory.

8.4 Other water types for non-compliance purposes:

8.4.1 Hold samples at <10°C during transit and until time of analysis.

8.4.2 Do not exceed 24 h holding time.

8.5 Exceptions to the above guidelines:

8.5.1 Section 8.3.1 allows a maximum transport time of 6 h. However, water samples from certain clients in the Western Division (such as Ba and Tavua), at times take a maximum transport time of 6 – 8 h. However, historical data of analysis of field blanks transported at <10°C for up to 8 h have showed no growth.

9.0 SAMPLE PREPARATION

9.1 Mix the sample by repeatedly inverting or shaking at least 10 times. Ensure there is sufficient headspace to allow for effective mixing – a small amount of sample may need to be discarded to allow this. Prepare dilutions if required. Ensure that the dilutions prepared in 0.1% peptone are thoroughly mixed by inverting or shaking at least 25 times or by using the vortex.

9.2 Selection of sample size for filtration:

9.2.1 Size of sample depends on the expected bacterial density. An ideal sample volume will yield 20 – 80 coliform colonies and not more than 200 colonies of all types on a membrane filter surface.

9.2.2 For drinking water analysis, sample size is limited only by the degree of turbidity or by the non-coliform growth on the medium.

9.2.3 The table below outlines the required sample volumes for filtration.

Water source/Sample type	Sample Volume (X) to be Filtered mL							
	100	50	10	1	0.1	0.01	0.001	0.0001
Drinking water	X							
Swimming pools	X							
Wells, springs	X	X	X					
Lakes, reservoirs	X	X	X					
Water supply intake			X	X	X			
Bathing beaches			X	X	X			

Water source/Sample type	Sample Volume (X) to be Filtered mL							
	100	50	10	1	0.1	0.01	0.001	0.0001
River water				X	X	X	X	
Chlorinated sewage				X	X	X		
Raw sewage					X	X	X	X

9.2.4 Analyse drinking water by filtering 100 to 1000 mL or by filtering replicate smaller sample volumes such as 50 ml duplicates or four replicates of 25 mL portions.

9.2.5 Analyse other waters by filtering three different volumes, 1mL, 10 mL, 100 mL (diluted or undiluted) depending on the expected bacterial density.

9.2.6 When less than 10 mL of sample (diluted or undiluted) is to be filtered, add approximately 10 mL sterile water to the funnel before filtering or pipet the sample volume into a sterile dilution bottle, then filter the entire dilution. This increase in water volume aids in uniform dispension of the bacterial suspension over the entire effective filtering space.

Note:
Water, once in the Laboratory, must be tested within 1 h of receipt or within 4 h if refrigerated (at 4°C).

10.0 MEDIA PREPARATION

Safety Note: Use caution when heating m-Endo broth or agar as it may boil quicker than expected.

10.1 Prepare m-Endo Agar LES according to manufacturer's directions (as outlined on bottle label). Do not autoclave. Hold in waterbath. Dispense 4 mL into petri dishes. Use immediately, otherwise refrigerate for no more than two weeks. Discard any unused m-Endo Agar plates after two weeks of refrigeration.

10.2 Prepare m-Endo Broth MF according to manufacturer's directions and dispense in 2.0 mL quantities on sterile 47 mm petri dishes with absorbent pads. Cool to room temperature. Use immediately or store refrigerated for no more than 96 h. Discard any unused m-Endo Broth MF after 96 h of refrigeration.

10.3 Prepare 0.85% saline solution using distilled water and adjust pH to 6 – 7 using 1N NaOH, or 1M HCI. Dispense sufficient quantities in screw top jars, sterilize and cool to room temperature before use. This can be stored for 1 month at room temperature after which it is to be discarded.

11.0 FILTRATION PROCEDURE

Note:
The filter funnel is rinsed with sterile water and 0.85% saline before filtering a new sample from the same sampling site. For analysis of samples from different sites, a different sterile filter funnel is used for each site.

11.1 Use sterile filtration unit at the beginning of each filtration series as a minimum precaution to avoid accidental contamination.

11.2 Using sterile forceps, place a sterile membrane filter (grid side-up) over porous plate of receptacle.

11.3 Carefully place matched funnel unit over receptacle and lock it in place.

11.4 For the procedural blank, before beginning sample analyses, the filter unit is washed with a 100 mL of sterile water, followed by 20 to 30 mL of sterile 0.85% saline. The filter is placed on an appropriate media and incubated under the same condition as the sample.

11.5 Aseptically add sample and filter under partial vacuum. With filter still in place, rinse the interior surface of the funnel by filtering three 20 to 30 mL portions of sterile 0.85% of saline solution.

Note:
Rinsing between samples prevent carry over contamination.

11.6 After the final rinse and filtration, slowly turn off the vacuum source, unlock and remove funnel. Immediately, aseptically remove membrane filter with sterilised forceps and transfer to the selected medium in the following manner.

11.6.1 If m-Endo Agar LES is used:

11.6.1.1 Gently roll the filter, grid side up, onto the surface of a m-Endo Agar LES plate. Be sure to avoid trapping air bubbles.

11.6.1.2 Cover the plate, invert and incubate for 22 to 24 h at 35 ± 0.5°C.

11.6.2 If m-Endo Broth MF is used:

11.6.2.1 Place an absorbent filter pad into a petri dish and pipette 1.8 – 2.0 mL of m-Endo Broth MF onto the pad.

11.6.2.2 Carefully remove excess medium by decanting the dish.

11.6.2.3 Gently roll the filter, grid side up onto the saturated absorbent filter pad. Be sure to avoid trapping bubbles.

11.6.2.4 Cover the petri dish, invert and incubate for 22 to 24 h at 35 ± 0.5°C.

Note:
The petri dish lids must fit tightly to prevent moisture loss. If lids fit loosely, tape lid closed to seal. .

11.7 After filtration of a series of 10 samples, filter 100 mL sterile water using the procedure in 11.1. This is to check for possible cross-contamination or contaminated rinse water.

11.8 Incubate the rinse water control membrane culture under the same conditions as the sample.

11.9 Preparation of Reference Culture:

11.9.1 Pick a single isolated colony of a T2 culture of *E.coli*, with a sterile loop and inoculate into 9 mL of 0.1% peptone. This is regarded as the stock solution of *E.coli.*

11.9.2 Using 9 mL 0.1% peptone, make a 1 x 10^{-5} dilution of the stock culture.

11.9.3 Filter 1 mL of this dilution as outlined in the procedure, sections 11.1 to 11.6.

11.9.4 The count should be between 20 -60 colonies/dilution. This range has been determined in-house (Ref: Quantitative Recovery of Bacterial Cultures – Membrane Filtration Method, Appendix XII(c), SOP NO. MM 310).

11.9.5 For the negative control culture, *E.faecalis*, streak a loopful of T1 onto a plate of mENDO Agar and incubate with the other plates as in section 11.6.1.2 above.

12.0 RECORDING OF RESULTS

12.1 All results data and observations are to be recorded on the Worksheet (refer to Chapter 2, Appendix II for the Worksheet for Total Coliform (TC) in Water (Membrane Filtration Method).

13.0 COUNTING OF COLONIES

13.1 As colonies are liable to change colour on cooling and standing, count colonies within a few minutes of removal from incubator.

13.2 Use the colony counter to count colonies on membrane filters.

13.3 The typical coliform colony has a pink to dark-red colour with a metallic surface sheen. Count both typical and atypical coliform colonies. Atypical coliform colonies can be dark, red, mucoid or nucleated without sheen. Generally pink, blue, white, or colourless colonies lacking sheen are considered non coliforms.

13.4 Report results as the number of coliform species/100 mL.

13.5 If no colonies meet the criteria, report as <1 coliform species/100 mL.

13.6 Quality Control (Confirmatory Tests) – Verification of colonies

13.6.1 Verify all typical and atypical colony types. If the total coliform count is <100, then verify √ of the total coliform count. If the total count is >100, then 10% of the total coliform count needs to be verified. In a similar manner atypical colonies of different morphological types can be verified.

13.6.2 For drinking water, **verify all suspect colonies** from a given membrane filter culture.

13.6.3 For waters **other than** drinking water, at a minimum, verify representative sheen colonies and representative atypical colonies of different morphological types from a positive water sample **on a monthly basis**.

13.6.4 Verification tests are as follows:

13.6.4.1 Lactose fermentation – Transfer growth from each colony with a sterile inoculating loop. Inoculate each colony, calculated in 13.6.1 above, into separate tubes of LST broth. Incubate at 35 ± 0.5°C for 48 h. Gas formed in LST within 48 h confirms the colony as a coliform.

14.0 CALCULATION OF COLIFORM DENSITY

14.1 Compute the count, using membrane filter with 20 – 80 coliform colonies and not more than 200 colonies of all types per membrane, by the following equation:

$$\text{Total Coliforms/100 mL} = \frac{\text{Coliform colonies counted} \times 100}{\text{ml sample filtered}}$$

If no coliform colonies are observed, report the coliform colonies counted as "<1 coliform/100 mL".

14.2 For verified coliform counts, adjust the initial count based upon the positive verification percentage and report as the **verified coliform count/100 mL.**

$$\text{\% Verified coliforms} = \frac{\text{number of verified colonies} \times 100}{\text{Total number of coliform colonies subjected to verification}}$$

If confluent growth occurs, covering either the entire filtration area of the membrane, report results as "confluent growth with/without coliforms".

14.3 If the total number of bacterial colonies, coliforms plus noncoliforms, exceeds 200 per membrane, or if the colonies are not distinct enough for accurate counting, report results as "Too Numerous to Count" (TNTC).

14.4 Water of drinking water quality:

With water of good quality, the occurrence of coliforms genera will be minimal. Therefore, count all coliform colonies (disregarding the lower limit of 20 cited above) and use the formula given above to obtain coliform density.

14.4.1 For drinking water, the presence of coliforms in such cultures showing no sheen, has to be confirmed by transferring the representative number into sterile tubes of LST broth. For TNTC, brush the entire filter surface with a sterile loop, applicator stick, or cotton swab and inoculate this.

14.4.2 If gas is produced from the LST broth tube within 48 h at 35 ± 0.5°C, coliforms are present.

14.4.3 Report TNTC with at least one detectable coliform colony (which is verified) as a total coliform positive sample.

14.4.4 Report TNTC without detectable coliforms as invalid. For invalid samples, request a new sample from the same location within 24 h and select more appropriate volumes to be filtered per membrane, observing the requirement that the standard drinking water portion is 100 mL. Thus to reduce interference from overcrowding, instead of filtering 100 mL per membrane, filter 50 mL portions through two separate membranes, 25 mL portions through each of four membranes, etc. Total the coliform counts on all membranes and report as number per 100 mL.

14.5 Water of other than drinking water quality:

As with potable water samples, if no filter has a coliform count falling in the ideal range, total the coliform counts on all filters and report as number per 100 mL.

Examples are given below:

14.5.1 If duplicate 50 mL portions were examined and the two membranes had 5 and 3 coliform counts, respectively report the count as 8 coliform colonies per 100 mL.

$$\frac{[(5+3)\,x\,100]}{(50+50)} = 8\ \text{coliforms}/100\ \text{mL}$$

14.5.2 Similarly, if 50, 25, and 10 mL portions were examined and the counts were 15, 6, and <1 coliform, respectively, report the count as 25/100 mL.

$$\frac{[(15+6+0)\,x\,100]}{(50+25+10)} = 25\ \text{coliforms}/100\ \text{mL}$$

14.5.3 On the other hand, if 10, 1.0, and 0.1 mL portions were examined with counts of 40, 9 and <1 coliform colonies, respectively, select the 10 mL portion only for calculating the coliform density because this filter had a coliform count falling in the ideal range. The result is 400/100 mL.

$$\frac{(40\,x\,100)}{10} = 400\ \text{colonies}/100\ \text{mL}$$

14.5.4 Report TNTC as described in 14.4 above. Request a new sample and select more appropriate volumes (or dilutions) for filtration or utilise the MPN method.

15.0 ACCEPTANCE CRITERIA

15.1 The QC of the media must be acceptable as described in SOP No. MM 310.

15.2 Each time this test is performed, Daily Media Control for this agar is performed as described in this procedure using *E.coli* NZRM 2577 and *E.faecalis* NZRM 1106. These microorganisms should be prepared according to SOP No. MM

297 and should perform to acceptable levels as described in SOP No. MM 310.

15.3 Test results are accepted if any of the following do not apply:

15.3.1 The results of a quality assurance procedure invalidate the test results. For example, sterility blanks indicate that contamination of membrane filters may have occurred, or daily media control indicates an unacceptable level of performance for the batch of media used.

15.3.2 An accident or error has occurred during the test procedure that in the opinion of the scientist invalidates the test results.

15.4 Whenever any of the above occurs, the report shall state “No results due to laboratory error” or “No results due to laboratory accident”, whichever is appropriate.

16.0 ISSUE TO

Master Copy File
Laboratory Bench Copy

Chapter 8 SAMPLE TRACEABILTY (LOG IN)

1.0 SAMPLE LOG IN (REGISTRATION)

All samples to be analyzed in the lab are registered in the Samples Register Logbook in the following recommended manner:

- Sampling Site Identity or Client Name

- Laboratory Number – a number unique to the sample that is assigned by the Laboratory. This can be in the following format: YY/### (where YY = Year and ### = sequential numbers starting from 1 for the first sample of the year. This number is written in waterproof pen. For example, 07/005 is the fifth sample registered in 2007.

- Date Samples Received

- Date Sampled (for a client this will only be entered if the information is made available to the Laboratory)

- Sample Type

- Client/Sampling Site ID (this is the sample identification by the client)
- Date Due (this is the date the results are due for reporting)

- Analysis Required

- Notes (for any notes on sample & packaging condition)

- Analyst - who the work is assigned to

2.0 SAMPLES LABELING

All samples bottled are to be labeled if the staff will be conducing the water sampling. Refer to Part II of this Manual, Section 2 on how the bottles are to be labeled.

If clients submit samples to the Laboratory, then the samples are to be labeled with a Samples Label after registration. The following information is to be noted on the label:

- Laboratory Number

- Date Sample Received

- Analysis Required

- Initials of Staff Member who is registering the sample.

Laboratory Sample Label Lab No: 2007/ *Date Recd: _____ Due Date: _____* ***Analysis Reqd :***

An exception to this is if the samples are already labeled with a Sampling Label (refer to Part II of this Manual, Section 6) where the Lab ID no is to be noted on the label.

3.0 SAMPLE TRACEABILITY

The sample details such as the Sampling Point ID/client, Laboratory Number, Date Received and Date Sampled are to be recorded on the Worksheets (refer to Chapter 2, Appendices I & 2. These details are also to be recorded on Test Reports (refer to Part II of this Manual, Section 8).

4.0 REFERENCE:

4.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures, SOP No. SSS 13 Analysis Requests, Sample Registration and Login Environmental Monitoring

Chapter 9 DATA MANAGEMENT

1.0 DATA MANAGEMENT

Data Management includes the documentation and storage of laboratory data to ensure that there is a traceability of samples data from when the samples are registered (refer to Chapter 8), analyse (refer to 2.0) and reported (refer to Part II of this Manual, Section 8).

Laboratory Data also includes Equipment Calibration Data (refer to Chapter 3), Quality Control Charts (refer to Chapter 2), and Quality Records (refer to Part II of this manual, Section 9.0)

2.0 WORKSHEETS

Observations, data and calculations are to be recorded on Worksheets 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. Using white-out correction fluid is forbidden in the Laboratory.

- 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 Laboratory.

Refer to Chapter 2, Appendices I and II for examples of Worksheets.

3.0 CHECKING

It is recommended that all data entered into Worksheets are to be checked by another technician and also the Laboratory Manager before final approval is given for release of results. The compilation of Test Reports is conducted after the checking of data (refer to Part II of this Manual, Section 11.0).

4.0 ACCESS

All Laboratory Data are to be filed appropriately in labeled Files and are to be easily accessible to all laboratory staff members.

5.0 LONG-TERM STORAGE

The period of storage of data will be at the discretion of the water Laboratory. At the end of each financial year, all Laboratory Data are to be transferred from their filing cabinet into a file box clearly labeled with the contents and the year covered.

Any electronic data back-up are to be stored in the fireproof safe.

6.0 REFERENCE

6.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

6.1.1 Quality Manual, QMS 4.0 Management of the Quality System

6.1.2 Quality Manual, QMS 12.0 Test Records

Chapter 10 FUNDING

1.0 FUNDS AVAILABILITY

Funds are to be made available to the Laboratory for successful implementation of the necessary measures outlined in Chapters 1 to 9. The achievement of accurate, precise and reliable analytical results that may have public health implications is only possible if these measures are implemented.

2.0 COMMITMENT OF GOVERNMENT MINISTRIES

The Government and the Ministry under which the Laboratory operates are to ensure that a high priority is placed on the allocation of funds for the Laboratory. Water is a basic necessity and it needs to be ensured that the resources are available to the Laboratory for its proper monitoring.

3.0 PROPER UTILISATION OF FUNDS

The Laboratories are to ensure that funds are properly utilized for the purchase of Reagents, Equipment, Reference Materials and other items outlined in Chapters 1 to 9. This will ensure that these are available to staff members for the proper monitoring and analysis of water.

PART

II

Section 1 SAFETY IN THE LABORATORY

1.0 Responsibility for Safety

The organization, Laboratory Manager and all the Laboratory staff members are responsible for the achievement of a safe and healthful workplace. All laboratory staff members must make every effort to protect themselves and their fellow workers by adhering to the Safety Rules and Practices that is developed and documented specifically for their Laboratory.

2.0 Chemicals and Reagents Handling

2.1 DO NOT use any chemicals or raw materials without being aware of the hazards involved (available on the Material Safety Data Sheet; MSDS).

3.0 Personal Protective Equipment

3.1 Personnel apparel – Wear laboratory coats, safety glasses and shoes at all times in the Laboratory. **DO NOT** wear sandals or perforated shoes. Any visitors to the Laboratories are also to wear these protective clothing.

4.0 General Safety Rules

4.1 Accidents and spills:

4.1.1 **Eye contact –** Promptly flush eyes with water for a prolonged period (minimum of 15 min) and seek immediate medical attention.

4.1.2 **Ingestion –** Encourage victim to drink large amounts of water.

4.1.3 **Skin contact –** Promptly flush affected area with water for approximately 15 min and remove any contaminated clothing. If symptoms persist after washing, seek medical attention.

4.1.4 **Clean-up –** Promptly clean up spills, using appropriate protective apparel and equipment and proper disposal procedures.

4.1.5 **Working alone –** Avoid working alone in a building; do not work alone in a laboratory if the procedures to be conducted are hazardous.

5.0 Work Practices/Rules

5.1 Work habits – Develop and encourage safe habits, avoid unnecessary exposure to chemicals and avoid working alone whenever possible.

5.2 Exhaust ventilation – DO NOT smell or taste chemicals. Vent any apparatus that may discharge toxic chemicals (vacuum pumps, distillation columns, etc) into local exhaust devices.

5.3 Eating/Smoking, and related activities – DO NOT eat, drink, smoke, chew gum, or apply cosmetics in areas where laboratory chemicals are present. Always wash hands before conducting any of these activities.

5.4 Food storage – DO NOT store, handle, or consume food or beverages in storage areas, refrigerators, or glassware and utensils that are also used for laboratory analysis.

5.5 Equipment and Glassware – Handle and store laboratory glassware with care to avoid damage. **DO NOT** use damaged glassware. Use equipment for its designed purpose only.

5.6 Mouth suction – DO NOT use mouth suction for pipetting or starting a siphon.

5.7 Personal housekeeping – Keep work area clean an uncluttered, with chemicals and equipment properly labeled and stored. Clean up work areas on completion of an operation at the end of the each day.

6.0 REFERENCE:

6.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). *Standard Methods for the Examination of Water and Wastewater*, 21st Edition. American Public Health Association (APHA), Washington, D.C; 1090B. Safe Laboratory Practices

Section 2 SAMPLING, HANDLING AND STORAGE OF SAMPLES

1.0 IMPORTANCE OF PROPER SAMPLING

Sampling is a vital part of monitoring the quality of water. A major source of error in the whole process of obtaining water quality information often occurs during sampling. Poor management decisions based upon incorrect data may result if sampling is performed in a careless and thoughtless manner.

Water sampling should therefore be conducted in such a way that:

- the analytical results represent the actual sample composition
- that the sample is protected from contamination or alteration

2.0 SAFETY CONSIDERATIONS

2.1 Always wear protective clothing, safety shoes, gloves and safety glasses when collecting water samples.

3.0 SAMPLING FOR WATER PHYSICAL AND CHEMICAL ANALYSES

3.1 **Sampling Materials**

3.1.1 Teflon (TFE) bottles - Teflon (TFE) bottles are the best containers for collecting water samples but in the absence of TFE polyethylene bottles with polyethylene caps can be used.

3.1.1.1 All sampling bottles are to soaked in 10% HCl bath. To prepare 10% bath, use 1:9 ratio of concentrated HCl to deionsed or distilled water. Upon removal rinse thoroughly at least 5 times with deionised or distilled water.

3.1.1.2 Distilled Water

3.1.1.3 Esky with ice/ice blocks

3.1.1.4 Sample Labels (refer to 7.0)

3.1.2 **Conducting Sampling**

3.1.2.1 When sampling rinse container at least twice with the sample before filling to the brim. **Note:** An exception to this is when the sample bottles contain a preservative agent as required by the test method SOP.

3.1.2.2 Label sample bottles as outlined in 7.0

3.1.3 **Transportation Condition of Samples**

3.1.3.1 Samples are to be kept cool in an esky during transportation to the Laboratory.

3.1.4 **Samples Receipt at the Laboratory**

3.1.4.1 Refer to Part I of Manual, Chapter 8 for the registration of samples in the Laboratory.

3.1.4.2 Samples are to be analysed immediately upon receipt.

4.0 SAMPLING MATERIALS AND TECHNIQUES FOR WATER MICROBIOLOGICAL ANALYSES

4.1 General considerations

4.1.1 Water sample must be representative of the water under evaluation and must be protected against extraneous contamination.

4.1.2 Samples are to be maintained at chill temperature (<4°C) during transportation from the sampling site to the Laboratory.

4.1.3 Protect sampling instrument from exposure and contamination before and during use. When opening the sterile sampling container, open it sufficiently to admit the sample, then close and seal it immediately.

4.1.4 Do not touch the inside of the sterile container lid.

4.1.5 Do not allow open lid to become contaminated.

4.2 Sampling Equipment and Reagents

4.2.1 **Sample Bottles**

Sterilized Bottles are to be used for the collection of water samples. Bottles for sampling chlorinated water must contain sterile sodium thiosulphate to nullify the disinfectant effect of the chlorine.

4.2.2 **Field Blanks:**

This is used to measure the temperature of samples received at the Laboratory and the sterility of the sampling containers.
A bottle of sterile water is to be transferred to an empty sterile container at the sampling site. The temperature of this water will be measured and at the Laboratory during the receipt of samples to gauge the temperature of the samples upon receipt. The Field Blank will also be analysed along with the samples.

4.2.3 **Thermometers**

Thermometers that measure -20 to 100°C with graduation intervals not exceeding 1°C.

4.2.4 **Surface Disinfection**

4.2.4.1 Ethanol (70%) is used for sterilizing hands and the mouth of water taps (refer to 5.0) if sampling from water taps.

4.2.4.2 An alternative to 70% ethanol is Sodium Hypochlorite solution (100 mg/L).

4.2.5 **Labeling supplies**

Marking pens and labels (refer to 6.0)

4.2.6 Esky containing ice or freezer blocks

For the transportation of samples from the sampling site to the Laboratory.

5.0 SAMPLING TAP WATER MICROBIOLOGICAL ANALYSIS and pH and Chlorine

The sampling of tap water is to be conducted as follows:

5.1 Open tap **fully** and let water run to waste for 2 or 3 minutes, or sufficient time to permit clearing the service line. Close tap.

5.2 Rinse the sampling tap with 70% ethanol or sodium hyphochlorite. Be sure to get some inside the opening. A portable plastic hand sprayer filled with the disinfectant should be used to ensure it getting inside the tap.

5.3 Flush water through the tap at full force. Use the bucket, if necessary, to prevent water from getting on the floor. The quantity will depend on the size of the port. A quantity of 15 to 20 L is usually sufficient to remove the residual disinfectant.

5.4 Reduce the flow of the water to one stream taking care not to close off the tap completely.

5.5 Using aseptic technique collect at least 250 mL of water. Collect samples by carefully removing cap of a sterile sample bottle and holding bottle near the base. Fill slowly and ensure no water is splashing out of the bottle, that is, water from tap MUST be running out in one clear stream. Replace cap securely. Do not rinse bottle (i.e., retain sodium thiosulfate). Make sure there is some air (about 2 – 3 cm) left in the container to improve mixing which needs to be done prior to analysis.

5.6 Close the water port.

5.7 If sampling water from mixing tap (taps with HOT and COLD water) remove tap attachments such as screen or splash guard, run hot water for 2 minutes, then cold water for 2 minutes, disinfect as in 5.2 and collect sample.

6.0 SAMPLING RECREATIONAL WATERS FOR MICROBIOLOGICAL ANALYSIS

The sampling of recreational water is to be conducted as follows:

6.1 Collect samples during periods of maximum bather load. Information on number of bathers may be helpful in subsequent interpretation of laboratory results.

6.2 Collect samples by carefully removing cap of a sterile sample bottle, plunge below the surface of the water (to avoid the surface scum layer) and holding bottle near the base at an angle of 45 degrees. Fill in one slow sweep through the water, with the mouth of the bottle always ahead of the hand. Immediately pour out a little water to give a 2 – 3 cm head space (to aid in mixing the sample in the laboratory). Replace cap securely. Do not rinse bottle.

7.0 SAMPLES LABELING

All water sampling bottles are to be labeled with a Sample Label sticker as shown below:

Description/ Site: ----------

Location: ----------

Date: ----------

Analysis Type: ----------

Time: ----------

Sampler: ----------

8.0 SAMPLES RECEIPT IN THE LABORATORY

8.1 For the receipt of samples and registration in the Laboratory, refer to Part I of this Manual, Chapter 8.

9.0 STORAGE OF WATER SAMPLES FOR PHYSICAL AND CHEMICAL ANALYSIS

9.1 For the storage of water samples for physical and chemical analysis, refer to 3.0.

10.0 STORAGE OF WATER SAMPLES FOR MICROBIOLOGICAL ANALYSIS

For Water samples for Microbiological analyses the following steps are to taken:

10.1 General

10.1.1 Start Microbiological analysis of water samples as soon as possible after collection to avoid unpredictable changes in the microbial population.

10.2 Drinking water for compliance purposes:

10.2.1 Preferably, hold samples at <10°C during transit to the laboratory.

10.2.2 Analyse samples on day of receipt whenever possible and refrigerate overnight if arrival is too late.

10.2.3 Do not exceed 30 h holding time from collection to analysis for coliform bacteria.

10.3 Non-potable water for compliance purposes:

10.3.1 Hold source water, stream pollution, recreational water and waste water below 10°C during a maximum transport time of six h.

10.3.2 Refrigerate these samples upon receipt in the Laboratory.

10.4 Other water types for non-compliance purposes:

10.4.1 Hold samples at <10°C during transit and until time of analysis.

10.4.2 Do not exceed 24 h holding time.

11.0 REFERENCES

11.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). *Standard Methods for the Examination of Water and Wastewater*, 21st Edition. American Public Health Association (APHA), Washington, D.C; 1060B Collection of Samples, 9020B.6 Sampling, 9222 B Standard Total Coliform Membrane Filter Procedure.

11.2 Mosley L, Singh S & Aalbersberg B, SOPAC Technical Report 381

11.3 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

11.3.1 SSS 13(R2) Sample Receipt & Login

11.3.2 SSS 15 (R1) Water Sampling Methods

11.3.3 Water Physical, Chemical and Microbiological SOPs

Section 3 REAGENTS AND ANALYTICAL STANDARDS PREPARATION

1.0 REAGENTS PREPARATION & LABELING

1.1 Prepare reagents as outlined in the relevant test methods Analytical Standard Operating Procedure. The reagent details are also to be documented in the Worksheets as follows:

1.1.1 Name of Solution being prepared

1.1.2 Mass of Reagent

1.1.3 Final Volume of Reagent

2.0 PREPARATION OF ANALYTICAL STANDARDS AND OTHER REAGENTS

2.1 Chemicals of Analytical Reagent (AR) grade should always be used while preparing analytical standards.

2.2 The preparation of stock solutions, working standards, and calibration standards must be documented in the relevant Worksheet. Preparation details are to include the following:

2.2.1 **For Standard Preparation by Standardisation:**

2.2.1.1 Name of Solution being Standardised

2.2.1.2 Primary Standard

2.2.1.3 Number of Trials (titrations)

2.2.1.4 Mass of Primary Standard for each Trials

2.2.1.5 Volume(s) of Titrant (mL)

2.2.1.6 Molarity obtained from each Trial

2.2.1.7 Calculation details – Average molarity, Standard deviation, 2 x Standard deviation (2SD)

2.2.2 **<u>For Working Stock and Working Standard Solution</u>**

<u>Definitions:</u>

Stock Solution – the solution from which the Working Stock Solution is prepared. This is prepared from dissolving a dry chemical or purchased from suppliers e.g. National Institute of Standards and Technology (NIST) (refer to Part II of Manual, Section 4).

Working Stock Solution – can be kept up to 3 months, this might vary between solutions.

Working Standards – these are prepared from the Working Stock solution and are used for calibrating the instruments.

2.2.2.1 Working Stock Solution

2.2.2.1.1 Name of Working Stock Solution

2.2.2.1.2 Compound/Stock – Name and concentration of Stock Solution

2.2.2.1.3 Expiration Date of Stock Solution

2.2.2.1.4 Date Prepared

2.2.2.1.5 Volume (cm^3) or Amount (g) of Stock Solution used

2.2.2.1.6 Final Volume of Working Stock Solution

2.2.2.1.7 Dilution Factor

2.2.2.1.8 Final Concentration

2.2.2.1.9 Balance/Pipette number

2.2.2.1.10 Expiration Date of Working Stock Solution

2.2.2.2 Working Standards

2.2.2.2.1 Name of Working Standard

2.2.2.2.2 Date Prepared

2.2.2.2.3 Compound/Working Stock – name and concentration of Working Stock Solution from which the Standard is prepared.

2.2.2.2.4 Expiration Date of Working Stock Solution

2.2.2.2.5 Volume (cm^3) or Amount (g) of Stock Solution used

2.2.2.2.6 Final Volume of Working Standard Solution

2.2.2.2.7 Final Concentration of Working Standard Solution

2.2.2.2.8 Expiration Date of Working Standard

2.2.2.2.9 Balance/Pipette number

2.2.3 **<u>For Other Reagents (e.g. 0.1% Lanthanum Chloride, indicators</u>**)

2.2.3.1 Name of Reagent

2.2.3.2 Volume (mL) or Amount (g) of Chemical

2.2.3.3 Volume Prepared

2.2.3.4 Date Prepared

3.0 LABELING OF PREPARED REAGENT, STANDARD, STOCK AND REAGENT SOLUTIONS

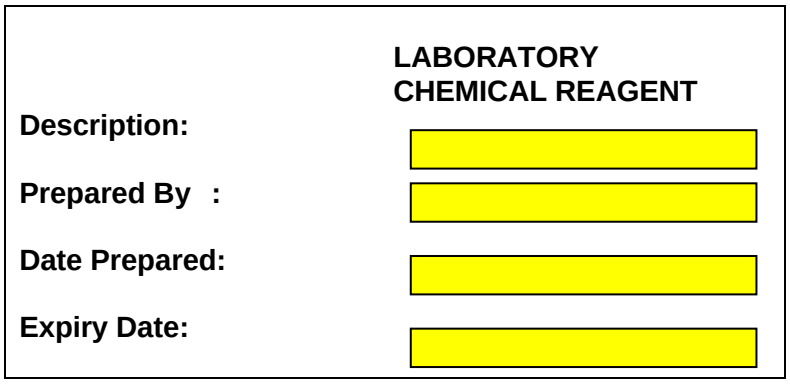
3.1 All prepared Standard, Stock and Reagents solutions are to be labeled using the Chemical Reagent label and are to have the following information:

3.1.1 Description – name of solution and its concentration e.g. 0.1 M HCl

3.1.2 Prepared By – name of analyst who prepared the solution

3.1.3 Date Prepared – the date of preparation

3.1.4 Expiry Date – expiration date of solution which will vary according to the nature of the solution (refer to 4.0).



3.1.5 If the vial is too small for all information to be included on the label, label the vial with only the serial number. Place the vial into a larger container, and label the container with all the required information.

4.0 EXPIRATION DATES OF REAGENTS AND ANALYTICAL STANDARDS

4.1 For in-house prepared reagents and solutions, the expiration dates will vary as follows:

4.1.1 Chemicals that do not have a direct effect on the accuracy of the results will use expiration dates as stated on its original package. These maybe used beyond their due dates without any validation.

4.1.2 For prepared Standards, Working Stock Solutions and Reagents that have a direct effect on the accuracy of the results, these will be given an expiration date according to the historical performance of the solution. After the expiration date, the solution or reagent may be re-assayed according to the test method or any other appropriate procedures. If the solution is deemed acceptable for continuing use, then the expiration date maybe extended.

4.1.2.1 Standards that are Standardized by Titration will be labeled with the words “Standardise before use” in the Expiration Date section on the Chemical Reagent Label.

4.1.2.2 For Working Standards that are used for calibrating instruments, newly prepared working standards will be compared against the old working standards, the results of which will be recorded on the Worksheet.

5.0 REFERENCE

5.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures, SOP No. SSS 17(R1) Solutions, Reagents, Standards Preparation, Validation and Documentation

Section 4 REFERENCE MATERIALS SUPPLY

1.0 REFERENCE MATERIALS

Reference Materials refers to any item used for checking the accuracy of the test method (refer to Part I of this Manual, Chapter 2) and equipment (refer to Part I of this Manual, Chapter 3).

The laboratory is to ensure that it obtains the correct reference material and also maintain an inventory similar to that done for reagents (refer to Part I of this Manual, Chapter 5.)

1.1 Reference Testing Materials

This includes Standard Reference Materials, pH buffer solutions, Electrical conductivity meter standards and Reference Cultures used for Microbiological analysis.

1.2 Reference Calibration Materials

This includes equipment with a high accuracy that have been calibrated by an internationally accredited Laboratory such as Digital Thermometers, Standard Weights.

2.0 SUPPLIERS OF STANDARD REFERENCE MATERIALS

The Laboratory may purchase its Standard Reference Materials:

No	Supplier	Items Supplied
1	National Institute of Standards and Technology 100 Bureau Drive STOP 2322 Gaithersburg MD 20899 – 2322 USA Ph: (301) 975 6776 Fax; (301) 948 3730	Standard Reference Materials for Water Testing
2	Biolab Ltd Private Bag 102 922 North Shore Mail Centre Auckland New Zealand Ph: +64 9 980 6700 Fax: +64 980 6788	pH Buffers, Conductivity Meter Standard Solutions Digital Thermometers, Standard Masses
3	Banksia Scientific Ltd P O Box 529 Bulimba Queensland 4171 Australia Ph: +61 7 3902 3000 Fax: +61 7 3217 9869	pH Buffers, Conductivity Meter Standard Solutions

No	Supplier	Items Supplied
4	New Zealand Reference Culture Institute of Environmental Science & Research Limited Kenepuru Science Centre P O Box 50-348 Porirua, New Zealand Ph: +64 4 914 0700	Microbiological Reference Cultures
5	Calibration Services 32 Shelter Drive, Greenhithe Auckland New Zealand Phone: +64 9 413 9335	Calibration of Digital Thermometers and Standard Masses

3.0 STORAGE OF REFERENCE MATERIALS

Standard Reference Materials are to be stored as outlined in the accompanying Data Sheets.

Reference Calibration Materials are to be stored in a cool environment.

4.0 REFERENCE

4.1 Institute of Applied Sciences, Standard Operating Procedure, QM 7.0 Equipment

Section 5 LABORATORY HOUSEKEEPING AND ENVIRONMENTAL MONITORING

1.0 HOUSEKEEPING

Housekeeping must be of a high standard at all times, for reasons of safety as well as for quality of results. The Laboratory is to be cleaned daily and the cleaning can be recorded in a Housekeeping Logbook.

For the Microbiology Laboratory the following practices are essential:

(i) workbenches are to be swabbed with 70% alcohol twice daily i.e. before and after work commences

(ii) Laboratory rooms, shelves, floor and windows are to be regularly cleaned

(iii) Floors are to be wet-mopped and treated with a disinfectant solution such as Savlon or Virkon.

2.0 LABORATORY ENVIRONMENTAL MONITORING

2.1 The temperature and humidity of the Laboratory Rooms are to be taken daily and recorded in a Logbook.

2.2 The Microbiology Laboratory room air is to be monitored monthly for Total Aerobic Count and Yeast and Mould Count using the following procedures:

2.2.1 Prepare Plate Count Agar (PCA) and Malt Extract Agar (MEA) agar plates according to instructions on the media containers. Plate Count Agar (PCA) will be used for enumerating Total Aerobic and Malt Extract Agar (MEA) will be used for enumerating Yeasts and Mould Counts.

2.2.2 Expose a PCA plate and a MEA plate for 15 minutes at any selected sites in the Laboratory e.g. at the membrane filtration area, near the incubators, near the autoclave and near the fridge.

2.2.3 Close the lids after 15 minutes exposure and incubate the PCA plates at 35°C for 48 h, the MEA plates at 25°C for 5 days.

2.2.3.1 Count the number of colonies on each plate. Results are deemed satisfactory if the total number of colonies per plate is less than 15 for Total Aerobic Count and less than 10 for Yeast and Mould Count (Ref: APHA (20 Ed) 9020B). If the total number of colonies is more than the acceptable level, conduct the following:

2.2.3.1.1 Suspend all laboratory work, disinfect all laboratory surfaces with 70% ethanol and/or Savlon solution.

2.2.3.1.2 Repeat plates exposure procedure until acceptable results are obtained

2.2.3.2 If unsatisfactory results persist, inform the Air-conditioning technicians to check that the Air-conditioning has had an annual filter replacement.

Refer to Appendix I to Section 5 for an example of a Microbiology Environment Monitoring Worksheet.

3.0 WASTE DISPOSAL

The Laboratory is to show its commitment to the protection and enhancement of the environment by the following actions:

- Hazardous substances are to be handled in a manner that minimizes the possibility of adverse environmental effects for e.g. all Microbiological wastes are to be sterilized before disposal.

- Discharge to the sewer is to comply with the conditions of any trade waste permit or regulatory bylaws for the site.

- Wastes are to be disposed of in a manner that does not adversely impact on the environment.

- Reduce energy costs and the impact of the energy use on the environment through energy conservation, efficient energy use and fuel substitution.

- Minimise water wastage.

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

4.0 REFERENCES:

4.1 Clesceri, L.S., Eaton, A.D., and Greenberg, A.E. (Ed).(2005). *Standard Methods for the Examination of Water and Wastewater*, 21st Edition. American Public Health Association (APHA), Washington, D.C; 9020B.2 Facilities

4.2 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

4.2.1 SOP No. MM 300 Environmental Monitoring

4.2.2 Quality Manual, QMS 8.0 Environment

Appendix I to Section 5

Example of a WORKSHEET FOR MONITORING OF AIR IN LAB ENVIRONMENT (Expose Plates)

Frequency of Monitoring: **MONTHLY** Date of Test:

Site #	Total Aerobic Count @ 35 °C/ 24 hours	**Pass/ Fail**	Yeast & Mold Count @ 25 °C/ 5 days	**Pass/ Fail**
1.Near the balance	7	P	4	P
2.Near the 2nd MF set-up	5	P	2	P
3.Near original MF set-up	1	P	4	P
4.RHS of laminar flow	5	P	2	P
5.Near the stomacher	10	P	6	P
6.Inside media cabinet	5	P	7	P
Blank	0	P	0	P
Acceptable Levels	**<15 colonies/ plate/ 15 minutes**		**<10 colonies/ plate/ 15 minutes**	

Media Used	**Date Prepared**	**Date Prepared (if re-tested)**
PCA	02/01/07	
MEA	02/01/07	

Analyst: RS	Results (TAC): RS	Results (Y/M Count): RS	Checked by: AP
Date: 03/01/07	Date: 04/01/07	Date: 08/01/07	Date: 10/01/07

Action Taken (if site has failed):

Site #:	Test that failed: TAC	Y/M

Follow – up Procedure:

Area cleaned:	By:

Area re-tested: Date	Analyst:

Results:

Analyst:	Read by:	Date:	**Pass:**	**Fail:**
Checked by:	Date:			

Section 6 STAFF RESPONSIBILITIES

1.0 STAFF RESPONSIBILITIES

It is important that a Laboratory clearly defines its staff member's responsibilities so that everyone is aware of their roles in ensuring that the Laboratory operations are conducted successfully. It needs to be clearly defined as to who is responsible for obtaining reagents and media, calibrating the instruments, conducting the analysis, checking data, approving the release of results, general housekeeping and other aspects described in Part I and II of this manual. The staff reporting line also needs to be clearly identified. The staff responsibilities could be outlined in the form of Job Descriptions as in 2.0.

2.0 STAFF JOB DESCRIPTIONS

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.

2.2 DIRECTOR

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

The key function of the Director in the **Quality System** of the XYZ Laboratory is to keep an overall check on the performance of the system. The other key functions are:

(i) To chair the annual review of the Management System, review both internal and external audits, client complaints and non-conformances and intervene in the case of inadequate corrective action.

(ii) To sit on the Appointments Committee for new employees' recruitment and comments on the annual laboratory staff performance review that is conducted by the Laboratory Manager.

Absence: In the case of planned leave (annual leave, conferences, and duty travels), the Director recommends to the Permanent Secretary a staff member to deputize for the period of absence.

2.3 LABORATORY MANAGER

The Laboratory manager is a principal technician who reports to Director. 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 Laboratory. This involves the supervision and safety of staff, the safe operation and maintenance of laboratory instrumentation, and the supply of consumables.

The Laboratory Manager is also responsible for the conduct of the Laboratory's **Quality Management System** by taking or recommending measures to ensure the fulfillment of the quality objectives of management. This includes the carrying out of Quality Policies in the most effective and economical manner to ensure continuing accuracy and precision of data produced.

The detailed job descriptions of the Laboratory Manager are:

(i) To conduct internal training for laboratory staff and ensure that they are competent in their designated analysis or other related activities. To ensure that staff training records are updated as assessed.

(ii) To participate in the Committee for new employees' recruitment and to conduct annual laboratory staff performance review.

(iii) To liaise with the Laboratory customers for clarification of analysis requests and results.

(iv) To assess and review non-conforming tests and complaints and decide on the appropriate corrective actions. The Laboratory Manager has the authority to halt suspend tests, withhold test reports and authorise the resumption of halted tests.

(v) To monitor and keep a record of all **quality system** activities (including the external auditing) of the laboratory and to make appropriate recommendations for correction and improvement as may be necessary.

(vi) To develop and carry out the designated quality control programme including coordinating the internal auditing of the Laboratory.

(vii) To organize Management Reviews of the Quality System.

(viii) To ensure that all equipment used in the Laboratory is functional and calibrated.

(ix) To ensure spare parts are kept in stock to ensure all laboratory equipment is operational with a minimum of downtime in cases of failure.

(x) To coordinate the inter-laboratory proficiency testing programme.

(xi) To check Analysts Worksheets.

(xii) To advise on the suitability of reagents, chemicals, reference materials and other consumables to be purchased and on the functionality of the same when received and when in stock.

(xiii) To monitor the use of all laboratory consumables and initiate timely ordering of supplies, and ensure the timely order of all consumable materials used by the laboratory.

(xiv) To keep a record of all incoming equipment and consumables and prepare the receiving report as outlined in Part I of this Manual, Chapter 5.

(xv) To perform other related duties, commensurate with qualifications and training, as assigned by the Director.

At all times, for safety and accountability reasons, there must be no ambiguity as to who is in control of the Laboratory.

Absence: In the event of the absence of the Laboratory Manager the most senior and experienced staff remaining must be formally authorised to take control of the Laboratory.

2.4 ANALYSTS

Analysts have a responsibility to:

(i) Follow appropriate and analytical methods and standard procedures

(ii) Ensure all appropriate QC activities are performed as described by the SOP

(iii) Ensure the Laboratory Manager is notified when QC indicators do not meet the required criteria

(iv) Ensure all analytical and QC activities are properly documented, and documentation is placed appropriately

(v) Show a commitment to compliance with the Quality Policies and procedures of the Laboratory.

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

3.0 REFERENCE:

3.1 SPACNET Generic Quality Assurance Manual, 13/03/2003

Section 7 QUALITY SYSTEM DOCUMENTATION

1.0 QUALITY AND TECHNICAL RECORDS SYSTEM

The Laboratory could create a hierarchy of manuals and procedures that document the policies of each aspect of the Laboratory's operations including test procedures.

1.1 Quality Manual

The Quality Manual describes the Laboratory system for accomplishing the Quality assurance activities such as Internal Audits, Management Review, Control of Nonconforming work, and Data Management.

1.2 Standard Operating Procedures (SOP) Manual

The SOPs manual describes the laboratory test procedures written up as Standard Operating Procedures and may comprises sections for:

(i) Administration,
(ii) Samples/Sampling/Storage
(iii) Instrument Operation
(iv) Instrument Calibration Methods
(v) Water Methods – Physical and Chemical
(vi) Microbiology & Miscellaneous

1.3 Standards and Reference Documents

The laboratory could obtain current version of the following standards and reference documents:

(i) APHA (American Public Health Association) Standard Methods.) This could be obtained from:
Standard Methods Customer Service:
Ph: 1-703-684-2469 (outside U.S./Canada)
email: support@standardmethods.org

1.4 Technical Records

Test records are accumulations of data and information that result from carrying out tests and calibrations. They may include forms, contracts, Worksheets, notebooks, control charts, test reports (refer to Section 11), calibration certificates and customer notes.

1.5 Quality Records

Quality records include reports from internal audits and management reviews as well as records of corrective and preventative actions.

2.0 DOCUMENT CONTROL

The Quality Manual and SOPs are to be treated as controlled documents so as to allow the use of only an existing version of the SOP to be used at all times in the Laboratory.

2.1 Master Copy

A Master Copy of the Quality Manual and SOPs is to be created and identified with a Master Copy stamp. The Master Copy is to be kept in the Laboratory Manager's office and is not to all documents.

2.2 Laboratory Benchcopy

Copies of the Master Copy could be made and laminated for use on the Laboratory bench by Analysts. These could be stamped as "Laboratory Benchcopy".

2.3 Documentation Indexing and Format

2.3.1 Indexing

The Quality Manual and SOPs are to be assigned a unique number and properly indexed, for example, the Water Microbiology SOPs could be numbered as WM 01, WM 02 and so forth. An updated index is to be placed in the Manuals (refer to 2.1 and 2.2).

2.3.2 Format

The Quality Manual and SOPs are to have the following standardized format. This can be inserted in the Headers of each QM or SOP section:

XYZ LABORATORY
QUALITY MANUAL

Title of Section: **QUALITY POLICY AND QUALITY SYSTEM DOCUMENTATION**	**Section:**	**QMS 2.0**
Date Issued: **10 March 2007**	**Page**	**104 of 3**

The fields in the control section are completed as follows:

(i) **Document Title:** e.g.XYZ Laboratory Quality Manual

(ii) **Title of Section:** e.g. Quality Policy and Quality System Documentation

(iii) **Document code: Section #** e.g. QMS 2.0

(iv) **The page number of the section:** e.g. 1 of 3

(v) **Issue Date:** in the format **DD MM YY** e.g. 15 April 2007

(vi) **Authorised by:** The signature of the Director

2.4 Changes to Documents

Any changes to the Quality Manual or SOPs are to be authorized by the Laboratory Manager. Any staff member can request for changes which are to be recorded by the Laboratory Manager on a SOP Revision Logbook. The summary of changes is also to be documented in the "Revision Summary" section of each SOP.

3.0 STORAGE OF TECHNICAL AND QUALITY RECORDS

The proper storage of technical and quality records is important for traceability and retrievability of data. The minimum length of storage of the records will be at the discretion of the Laboratory Manager as this is subject to available storage space in the laboratory.
Refer to Appendix I to Section 7 for an example of the storage, location and retention period of Quality Documents & Technical Records.

4.0 REFERENCE:

4.1 International Accreditation New Zealand, 2005, General requirements for the competence of testing and calibration laboratories.

4.2 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

4.2.1 SOP No. AS 10(R1) Writing and Issuing SOP

4.2.2 Quality Manual, QMS 4.0 Management of the Quality System

Appendix I to Section 7

STORAGE, LOCATION AND RETENTION PERIOD OF QUALITY DOCUMENTS & TECHNICAL RECORDS.

	CODE	Document Title	Storage area	Length of Storage	Length of storage in Archive
1)	QM	Quality Manual & Standard Operating Procedures – Master Copy	Laboratory Manager's Office	Subject to review of procedures	Obsolete copies are stored for 5 years.
3)		Laboratory Notebooks & Worksheeets	Laboratory	3 years	5 years
4)		Test Reports	Laboratory Manager's Office	3 years	5 years
5)		Audit Reports – external Audit Reports – internal	Laboratory Manager's Office	3 years 3 years	5 years 5 years
6)		Sample Register	Laboratory	3 years	5 years
		Change Control (Documents) Hand Amendments	Laboratory Manager's Office	3 years	5 years
		Non-conformances/Customer Gifts Non-conformances for Proficency Program results	Laboratory Manager's Office	4 years 4 years	6years 6 years
		Proficiency Tests Results:	Laboratory	5 years	10 years
		SRM's Certificates	Laboratory	5 years	10 years
		Calibration Records (internal): ▪ Thermometers ▪ Incubators/Waterbaths ▪ Automatic Pipettes/Dispensers ▪ Ovens ▪ Balances ▪ Glassware	Laboratory	3 years	5 years
		Calibration Records (external): ▪ Reference Thermometers ▪ Standard Masses	Laboratory Manager's Office	5 years	10 years

Section 8 NON-CONFORMING WORK

Quality control is primarily aimed at the prevention of errors. However, despite all efforts it remains inevitable that errors are made; therefore the quality 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 non-conforming 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 customer, 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 analysis purpose. There may be potential safety problem in the laboratory, performance in a proficiency 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.

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.

1.0 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 Non-conformance Report (refer to Appendix I) 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.

2.0 CORRECTIVE ACTION

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

2.1 The Laboratory Manager will discuss the problem with the analyst and may include arranging for another sample to be analysed.

2.2 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 customers to be contacted/reports recalled.

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

2.4 Corrective actions identified in the Non conformance Report shall be implemented immediately. These may include:

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

2.5 The Laboratory Manager is responsible for authorizing 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.

2.6 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.

2.7 Affected areas should be fully audited (refer to Section 9) 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.

3.0 RECORDS

Document the investigation fully, including discussions with staff. Records of the complaint or sub-standard work and the corrective action taken will be kept in the Nonconforming Work folder in the Laboratory Manager's office. This will record all details, including dates, client/sampling point, sample identification and laboratory numbers, discussions with the staff 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 non conformance report file will be presented as a component of the Management Review process (refer to Section 10); and the cause of any non-compliance will be established and remedied.

4.0 PREVENTATIVE ACTION

In the evaluation of nonconforming work, and in the Non conformance procedure, consideration should be given to what might be done to prevent a recurrence of the problem.

The non conformance report contains 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.

5.0 REFERENCES

5.1 Non conformance Report

5.2 SOP No. AD 11 Non conformances and Corrective Action

Appendix I to Section 8

Non Conforming Work Report

Report No.	**Date:**	**Initiated By:**
1 Type of problem □ Client enquiry (report or result) □ Failed Standard Reference Material Check □ Internal Audit non compliance □ Unsatisfactory Performance in Proficiency Program □ Other		
2 Client/Sampling Point		
3 Description		
4 Investigation		
5 Outcome		
6 Corrective Action		
7 Preventative Action/Opportunity for improvement		
8 Follow-up		
9 Conclusion		
Signed: _____ Date: _____		

Section 9 INTERNAL AUDITS

1.0 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 the test methods
- Internal safety audits
- 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.

2.0 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 organizing 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 Non conformances and dealt with through the Non conformance investigation system (refer to Section 8) When the audit findings cast doubt on the effectiveness of the operations or on the 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 annual Management Review will monitor (record and verify) the implementation and effectiveness of correct 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 Appendix I 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 non conformance for action. The results of the implementation of corrective actions will be reviewed at the next Management Review meeting.

3.0 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 summarizing the Laboratory Control Samples 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.

4.0 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 non conformance for action (refer to section 8). The results of the implementation of corrective actions will be reviewed at the next Management Review meeting.

5.0 REFERENCE

5.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

5.1.1 AS 14 Internal Audits

Appendix I to Section 9

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 amendments up to date?		
Do the manuals contain the current versions of documents?		
Are manuals & SOPs (except Laboratory Manager's) free of hand-written amendments?		
Has the Quality Manual review been completed to date?		
Has the Standard Operating Procedures review been completed to date?		

Comments:

	Yes	No
Are staff resource levels adequate?		
Is there an internal training program to maintain job competency?		
Are staff training records up to date?		
Are the educational and training backgrounds of all analysts sufficient to perform testing on submissions?		
Are staff training needs being identified and addressed?		

Comments:

2. ENVIRONMENT

	Yes	No
Is adequate space available for the type of testing performed?		
Is the housekeeping schedule being adhered to?		
Are temperature and humidity records up to date?		
Microbiology Laboratory: ▪ is a monthly environmental reading being conducted?		

Comments:

3. EQUIPMENT

	Yes	No
Do all equipment items have an inventory number?		
Are records of new instrument commissioning in the XYZ Laboratory Equipment File?		
Does every instrument have a operation/service/maintenance log?		
Is instrument servicing being recorded in logs?		
Is the calibration reminder wall planner in place in the Laboratory?		
Is the calibration schedule being adhered to:		
▪ Thermometers		
▪ Incubators		
▪ Ovens		
▪ Automatic pipettes		
▪ New volumetric glassware		
▪ Balances		
▪ Muffle furnace		
▪ Digestion Block		
Are calibration records up to date?		
Reference Equipment Standards: Are the following Reference Equipment Standards (Standard masses & reference thermometers) stored appropriately? Were they calibrated by an accredited laboratory (ISO/IEC 17025) and is the calibration certificate available?		

Comments:

4. 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:

5. SAMPLE HANDLING

	Yes	No
Are samples stored in the appropriate locations?		
If refrigeration is required, is the temperature monitored?		
Are samples identified with unique lab numbers?		
Can the job the samples belong to be identified?		
Is sample disposal up to date?		
Can a set of samples currently under analysis be located?		

Comments:

6. TEST METHODS

	Yes	No
Are laboratory copies of Standard Operating Procedures(SOPs) Manuals in good condition?		
Do laboratory copies of SOPs Manuals contain hand-written amendments?		
Do any new methods have validation data in the Method Development File?		
Are method uncertainties documented?		

Comments:

7. TEST RECORDS

Select 3 Analysis Request Forms and check the Worksheets of required analysis noted in these forms.

	Yes	No
Are sample receipt details present?		
Are client discussion records present?		
Are instrument printouts present?		
Are the Worksheets dated and initialed by the Analyst?		
Are the Worksheets free form the use of “White Out” or “Twink”?		
Can the Test Report be located for these Analysis Request Forms?		

Comments:

8. ANALYTICAL QUALITY CONTROL PROGRAMME

	Yes	No
Are Laboratory Control Samples (LCS) results being entered in the appropriate spreadsheet?		
Are appropriate actions being taken & recorded du to LCS results?		
Are control charts functioning and up to date?		
Are Proficiency program results available?		
Is action taken as a result of proficiency program results?		

Comments:

9. NONCONFORMING WORK

Locate the Client Complaints File in the Laboratory Manager’s Office.

	Yes	No
Have any incidents/complaints been recorded in the last year?		
Have all action items been carried out?		

Comments:

10. SAFETY

	Yes	No
Are monthly audits being conducted?		
Are non-compliance items being actioned?		
Are laboratory accidents recoded in the Site Accident Register?		

Comments:

11. INTERNAL AUDIT

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

Comments:

12. MANAGEMENT REVIEW

	Yes	No
Has a review been carried out since the last aduit?		
Have all items raised been actioned?		

Comments:

Section 10 MANAGEMENT REVIEW

1.0 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, Non conformance file and Customer Feedback 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.

2.0 RESPONSIBILITY

The Laboratory Manager is responsible for coordinating the Management Review. . The review team will compromise the Director, Laboratory Manager, Laboratory Manager and the Senior Technical Officers.

3.0 CONTENT

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

4.0 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 Appendix I. 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.

5.0 REFERENCE

5.1 SPACNET Generic Quality Assurance Manual, 13/03/2003

Appendix II to QMS 16.0

MANAGEMENT REVIEW AGENDA

XYZ LABORATORY

Management Review Agenda

Date: _____

Time: _____

Place: _____

Agenda Items

1. Review of objectives of 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

Section 11 TEST REPORTS

1.0 REPORT CHECKING

Once an analysis request has been completed, the Laboratory Manager will conduct the following checks before signing the Worksheets to ensure that:

- Data is entered correctly (refer to Part I of this Manual, Chapter 9)
- Quality Control Checks are within acceptable ranges (refer to Part I of this Manual, Chapter 2).

2.0 REPORT FORMAT

A report will be generated for the analysis and the Laboratory Manager will sign all reports.

In general, each report shall contain the following:

- On the cover page:
    - A title e.g. Water Analysis Report
    - The name and address of the laboratory
    - Unique identification of the test report, such as the RPT YY/### where RPT = report; YY = year in which the report was issued, #### = sequential numbering of the issuing of report, for example, RPT 2007/0003 will be the third test report issued in 2007.
    - Pagination, in the form of “page x of y” on each page
    - Name and address of the customer
    - Name, Signature and Position of Certifying Officer (usually the Laboratory Manager)
- On the Data Page
    - Dates of receipt of samples
    - A note stating the “Results apply to samples as received”
    - Identification of the reference methods used. This may be in the form of a separate sheet referred to in, and accompanying, the report.
    - Client/Sample Identification
    - Laboratory Sample ID Number (refer to Part I of this Manual, Chapter 8)
    - Test results with appropriate units

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

- Sampling details, including date, sample identification, location of sampling, sampling procedures, relevant environmental conditions

- The sample condition as recorded on receipt should be noted.

- Any deviations from the test methods

- A statement of compliance/non-compliance with specifications

- Opinions and interpretations as needed by customers - 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 bear the same Test Report number but with a clear “Amended Report” title.

3.0 REPORTS STORAGE

A hard copy of each Test Report is to be stored in the Test Reports file and will be stored at the Laboratory Manager’s office as outlined in Appendix I to Section 7.

An electronic copy of the report can be stored in a disk and maintained in a fire-proof cabinet for 5 years.

4.0 REFERENCE

4.1 Institute of Applied Sciences, Analytical Laboratory Standard Operating Procedures

4.1.1 Quality Manual, QM 14.0 Test Reports