

**specific criteria
for accreditation**

Chemical Testing

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AS LAB C 2

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1 Introduction

International Accreditation New Zealand's (IANZ) Specific Criteria are an elaboration of the General Criteria for Accreditation for specific fields of test and calibration, test technologies, products or materials. They address items that are essential or most important for the proper conduct of a test or calibration. Specific Criteria provide detail or add extra information to the generally stated requirements of the IANZ General Criteria for Accreditation, which remains the governing document. A list of all published Specific Criteria is available on www.ianz.govt.nz/publications or from IANZ on request.

This criteria document must be read in conjunction with current issues of NZS ISO/IEC 17025 and the IANZ publication *Procedures and Conditions of Accreditation*, the latter document describing the organisation and operation of the IANZ Laboratory Accreditation Programme.

NZS ISO/IEC 17025 is a general document designed to apply to all types of testing and calibration laboratories. This criteria document, on the other hand, provides information and interpretation on classes of test, staff, accommodation, equipment and other aspects of good laboratory management practice which are considered to be minimum standards for chemical testing laboratories being accredited against NZS ISO/IEC 17025.

2 Scope

This document sets out the specific requirements a chemical testing laboratory has to meet, in addition to the general requirements of NZS ISO/IEC 17025, if it is to be accredited by IANZ.

In addition to this document, there are Supplementary Criteria documents applicable to chemical testing laboratories working in specialised areas of testing which have their own set of unique criteria. At the time of publication the following additional criteria documents have been published:

AS LAB C9.0	<i>Specific Criteria for Accreditation – Dairy Testing</i>
AS LAB C10	<i>Specific Criteria for Accreditation – Laboratory Approval Scheme</i>
AS LAB C1.2/C2.2	<i>Supplementary Criteria for Accreditation – Ministry of Health Register of Water Testing Laboratories</i>

Please contact IANZ for more details.

3 Classes of Test

IANZ accreditation does not constitute a blanket approval of all a laboratory's activities. Therefore, a means of identifying those activities for which accreditation is granted, is necessary. The classes of test given in Appendix 1 provide the framework within which the scope of accreditation is expressed for chemical testing laboratories.

These classes are an arbitrary subdivision of the potential range of activities involved in chemical testing laboratories on the basis of the types of samples being tested, the scientific disciplines involved and the test methods employed. These classes and subclasses do not, however, constitute any restriction on the work a laboratory can perform but provide a convenient means of expressing an accredited laboratory's capabilities.

4 Laboratory Accommodation

Accommodation requirements for chemical testing vary widely depending on the nature of the testing involved. Some test measurements can be carried out in the field, while analyses for trace level concentrations require special precautions to prevent contamination of the sample during the analysis e.g. low level and high level areas.

Irrespective of where tests and measurements are performed, there must be adequate space and storage facilities for carrying out the tests, recording of test data, report preparation, etc.

Formal laboratory areas must have good lighting, adequate bench space, freedom from excessive dust and fumes, freedom from unwanted vibration and acoustic noise and, for some tests, control of temperature and humidity or light levels. The extent to which these environmental factors apply will vary according to the type and precision of the testing. Factors that may need to be considered include but are not necessarily restricted to:

- (a) Isolation from sources of stray electric and magnetic fields, mechanical vibration and shock likely to have a detrimental effect on sensitive instruments e.g. high accuracy balances
- (b) Adequate ventilation when fumes are created during the testing procedure
Note: Where perchloric acid digestions are conducted, water flushed fume hoods are expected
- (c) Suitable equipment and areas for the preparation of test samples
- (d) Any specified conditions in the referenced test methods such as air flow, light levels
- (e) Areas with subdued lighting may be needed for the performance of test procedures such as vitamin analysis, etc.

Calibrated and suitably maintained monitoring equipment and records are expected where conditions are specified by the test procedures.

It may be necessary in some cases to use clean-room facilities.

Field testing sites need to be selected so that the environment does not affect the testing results. Relevant monitoring records may be required to demonstrate this.

The suitability of the accommodation will be judged on whether it is likely to adversely affect the samples, equipment, staff performance or final test results.

References such as AS/NZ 2243 or an appropriate Code of Practice registered with the Department of Labour should be consulted when Laboratory Safety Procedures are being prepared and implemented.

5 Traceability of Measurement

Traceability of measurement in chemical and microbiological testing is the subject of much discussion and debate in the international testing community and readers are encouraged to familiarise themselves with current developments through sources such as those detailed in the References (7 & 8). The following discussion is provided to summarise the key issues associated with current approaches and provides laboratories with guidance on where to focus their efforts to improve the traceability of their measurements.

The *International Vocabulary for Metrology (VIM)* defines traceability as the:

“...property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.”

This definition is well understood and effectively applied in the traditional metrological areas where physical, electrical and other measurements can demonstrate traceability to “Système Internationale” (SI) units such as the kilogram (mass), the meter (length), the second (time), the amp (electric current) and the like. It also needs to be noted that traceability and uncertainty are closely aligned and neither have much meaning in the absence of the other.

5.1 Chemical Testing

Often in chemical testing, the parameter is an instrument response (peak height/area, absorbance, etc.) compared with a calibration from a “known” amount of substance – a reference standard, which may be a pure analytical standard or a matrix reference material. The mechanisms to ensure traceability of such reference material are, in most areas, not well developed. It is also recognised that availability of reference material complying with the generally accepted mechanisms to ensure traceability is limited.

Nevertheless, chemical testing laboratories are expected to source their reference materials (RMs) (which include analytical standards) from the following possible sources (generally in decreasing order of preference) where availability permits:

- (a) Certified Reference Materials (CRMs) from national measurement institutes which provide these e.g. National Measurement Institute (NMI) in Australia, NIST (USA), BCR (Europe)
- (b) CRMs from accredited (to ISO Guide 34) reference material producers
- (c) CRMs and RMs from well established and reputable reference material producers
- (d) Reputable chemical supply houses (particularly for pure analytical standards)
- (e) Customer supplied reference standards, preferably with certification
- (f) In-house produced reference standards.

For materials without formal evidence of traceability (and associated uncertainty information), it remains the laboratories' responsibility to demonstrate the materials are fit for their intended purpose.

CRMs are defined as a reference material that is characterised by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value(s) of the specified property(ies), its (their) uncertainty(ies) and a statement of metrological traceability.

A RM is a material sufficiently homogenous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.

In addition, for empirical methods, traceability relies on the laboratory complying in full with the method as published (as this defines the measurand). Other methods to determine this defined measurand are possible but must be validated or "calibrated" against the primary reference method defining the measurand e.g. measurands such as fat, protein or moisture in foods by near-infrared reflectance spectroscopy, need to have a clearly established relationship with results achieved by the reference method.

5.2 Traceability of Physical Measurements

Traceability requires that there is a chain of equipment whose calibrations to known levels of uncertainty are traceable from one item to the next and, eventually, to a national standard of measurement. The concept of traceability also includes the competence of all the people involved, the fitness of each measurement environment, the suitability of the methods used and all other aspects of the quality management systems involved at each step in the chain of measurements.

Traceability must be established for all critical* measurement and calibration equipment either:

- (a) Directly to the national standards laboratory (Industrial Research Limited - Measurement Standards Laboratory) or another such national body (e.g. National Physical Laboratory - UK, National Measurement - Australia, etc) acceptable to the Measurement Standards Laboratory, or
- (b) From a third party accredited calibration laboratory that is accredited by IANZ or an organisation with which IANZ has a mutual recognition arrangement.

The calibration certificates issued by accredited calibration laboratories must be endorsed in accordance with the requirements of the accreditation bodies concerned. This constitutes proof of traceability to national standards.

Endorsement to ISO 9000 standard is not considered acceptable.

**Critical measurements/calibrations are those that will significantly affect the accuracy or proper performance of tests.*

6 Equipment Management and Calibration

Laboratory equipment and its suitability ranks on a level equal to the competence of the staff using it. An accredited laboratory will be expected to possess and maintain, under a documented management system, all equipment necessary to carry out the tests requested for inclusion in the scope of accreditation.

Guidelines on calibration requirements and re-calibration intervals for specific items of equipment are detailed in Appendix 3. The guidelines set out maximum periods of use before equipment must be re-calibrated. These periods have been established by accepted industry practice and, in most instances, are the maximum permitted re-calibration intervals as laid down by international convention. Where a test method or operating environment requires a more stringent recalibration period than given here, the more frequent calibration will apply.

IANZ may accept reduced or extended calibration intervals based on factors such as history of stability, accuracy required and ability of staff to perform regular checks. It is the responsibility of the laboratory to provide clear evidence that its calibration and maintenance system will ensure that confidence in the equipment can be maintained.

Precision balances that are being used to their full readability (i.e. to the last place showing) will also require full re-calibration by an appropriate calibration authority (i.e. external calibration) if they are moved to a different location. Balances being used for less than their accuracy limit may be re-validated using appropriate QC methods i.e. single point and repeatability checks with standard check masses.

Records of calibrations carried out in-house must confirm traceability of measurement (see section 5.2 above). This is normally achieved by the record specifically identifying the reference item used, the date and the person performing the work using the documented procedure.

Calibration of an instrument as a whole rather than individual components of it is sometimes necessary, often as part of the test run. Chromatographic systems are examples.

6.1 Measurement Uncertainty in Calibration

Clause 5.4.6.1 of NZS ISO/IEC 17025:2005 requires testing laboratories which perform their own calibrations to have and apply a procedure to estimate the uncertainty of measurement in all calibrations. The full rigour of this requirement will be expected to be applied where the equipment item being calibrated has performance (accuracy and precision) requirements that are critical to the accuracy or proper performance of the test and which are approaching the performance specification of the equipment item. Examples would include the calibration of analytical balances, thermometers requiring a high level of (relative) accuracy and the like.

Chemical testing laboratories are recommended to have these items calibrated by an accredited external agency (see Section 5.2 above). If chemical testing laboratories wish to calibrate these items themselves, a full measurement uncertainty budget is expected to be estimated. This would normally be expected to be estimated in accordance with the *Guide to the Expression of Uncertainty of Measurement* (ISO,1995). The IANZ *Specific Criteria for Accreditation in Metrology and Calibration* (AS LAB C5) should be consulted for further information.

Uncertainty of measurement estimations for periodic checks conducted in-house on calibrated equipment (i.e. conducted between full calibrations) are not required.

7 Staff and Key Technical Personnel

An accredited laboratory must have at least one staff member who is competent in the testing being undertaken.

The qualification and appointment of Key Technical Personnel is an internal process in the laboratory under the responsibility of the laboratory management.

The expected roles and qualifications of a Key Technical Person are given in Appendix 2.

The following requirements in regard to Key Technical Personnel are reviewed as part of the assessment process:

- (a) Appointment of Key Technical Personnel will be the responsibility of a designated senior laboratory officer who is a member of the laboratory's senior management team. Laboratories are required to have a documented person/position specification for Key Technical Persons and a documented and formal process for their qualification and appointment
- (b) The laboratory will maintain a list of current Key Technical Personnel, including the technical scope of their areas of responsibility. This list may be included in the laboratory's quality manual or as a separate document but must be maintained up-to-date at all times. The technical scope for each individual will be described in a manner to suit the laboratory's circumstance and organisational structure but there must be at least one Key Technical Person appointed for each test or group of tests in the laboratory's scope of accreditation. The laboratory may choose to use the Classes of Test detailed in Appendix 1 with additional qualifiers as appropriate, but this is not mandatory
- (c) The list of Key Technical Personnel and their individual scope of responsibility must be notified to IANZ who will maintain this listing for each accreditation. IANZ will request this information in the Application for Accreditation or Reassessment documentation provided prior to the three yearly full reassessment. The list will also be reviewed with laboratories during their annual surveillance assessment
- (d) Changes to Key Technical Personnel listings (including individuals who have left the laboratory, new Key Technical Person appointments, or changes in the technical scope of responsibility) made between annual on-site assessments must also be notified to IANZ. This is the responsibility of the laboratory's Authorised Representative
- (e) In addition to the laboratory's usual training records, each Key Technical Person is required to have a brief CV-type summary of qualifications and experience. This CV information will be requested by IANZ for each appointed Key Technical Person in the Application for Accreditation/Reassessment documentation described above. This information is also expected to be provided to IANZ when new Key Technical Personnel are appointed and notified to IANZ outside of annual assessments
- (f) Where a laboratory loses the sole Key Technical Person for all or part of their scope of accreditation and no new appointment is made by the laboratory management, then the laboratory's accreditation (or part thereof) will be suspended until such time as a new appointment is notified to IANZ. Where new Key Technical Personnel appointments are made outside of routine reassessments, and particularly when a new appointment is the sole Key Technical Person for all or part of the accreditation, IANZ reserves the right to conduct an on-site assessment of the laboratory to be assured the laboratory's systems and integrity of the laboratory's tests results will continue to be maintained
- (g) All IANZ-endorsed test reports issued by an accredited laboratory must be signed or authorised by a Key Technical Person nominated by the laboratory. See Section 10.2.1.

The appointment of Key Technical Personnel effectively means the responsibility for qualification of key individuals within a laboratory lies with the laboratory management. IANZ Assessment Teams will no longer feel obliged to interview all appointed Key Technical Personnel. The Key Technical Personnel will still, generally, be expected to be the escorts for IANZ assessment teams during the course of an on-site assessment, with any of the appointed individuals being selected for the particular part of the scope of accreditation being assessed. The team may also choose to interview other levels of technical staff. In the case where a particular Key Technical Person is not able to demonstrate to the assessment team that the laboratory is continuing to maintain the requirements for accreditation, it is not the individual who is considered to have “passed” or “failed” but rather the laboratory as a whole on the grounds of inadequate, continuous technical supervision and it may be that the affected part of the scope of accreditation is suspended.

7.1 Generic Accreditations and Key Technical Personnel

The IANZ Chemical Testing Laboratory Accreditation Programme allows for laboratories with specialist expertise to gain more generalised scopes of accreditation under Classes of Test 2.70 – Instrumental Techniques and 2.71 – Non-Instrumental Techniques detailed in Appendix 1.

The ability of a laboratory to demonstrate the accreditation requirements for these Classes of Test are being met is dependent on the expertise of the individuals within the laboratory. Therefore, for accreditation in these two particular Classes of Test to be considered IANZ will continue to qualify nominated Key Technical Personnel by interview and assessment.

In essence, the laboratory management will nominate (rather than appoint) Key Technical Personnel for these Classes of Test. Each nominated individual will be assessed by an IANZ Assessment Team which will endorse (or otherwise) the named persons by the granting of accreditation with the Key Technical Person(s).

The requirements for Key Technical Personnel for these Classes of Test are included in Appendix 2.

For accreditation to be granted/continued in Classes of Test 2.70 and 2.71, the laboratory must have at least one Key Technical Person for each discipline whose nomination has been assessed and endorsed by IANZ. Should a sole Key Technical Person leave the laboratory or no longer fulfil the requirements, accreditation for these Classes of Test will be suspended. Laboratory management may nominate a replacement individual but accreditation will not be reinstated until such time as the nominated individual has been assessed by IANZ and the nomination endorsed.

IANZ-endorsed test reports issued under the umbrella of Classes of Test 2.70 and 2.71 must be signed by a Key Technical Person in the relevant discipline whose nomination has been endorsed by IANZ.

8 Test Methods

Accreditation is normally granted only for internationally or nationally accepted standard test procedures or non-standard procedures (in-house methods) that have been appropriately validated and which are performed regularly.

8.1 Standard Methods

Where standard methods are prescribed and followed, the laboratory is expected to maintain current versions of the standard methods (reference texts) and up-date laboratory bench methods in accordance with these.

Although full validation is not required, a laboratory must verify that it can properly operate the method and can demonstrate the specified limits of detection, selectivity, repeatability and reproducibility can be obtained.

8.2 Kits

Commercial test systems (kits) will require further validation 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 and/or associated JAOAC publications, or independently reviewed methods e.g. AOAC Performance Tested Methods.

8.3 In-house methods

In-house methods could include but not be restricted to:

- (a) Methods developed in the laboratory
- (b) Methods developed by a client
- (c) Methods developed for an industry group
- (d) Modified standard test methods
- (e) Methods from scientific publications but which have not been validated.

Validation procedures shall involve, as appropriate, the aspects referred to in Clause 5.4.5 of NZS ISO/IEC 17025:2005.

Appendix 4 provides a schematic guideline for method validation and a guide to the information that should be included in the validation report.

Standard test methods should be used whenever possible in order to ensure inter-laboratory reproducibility of test results. Laboratories are discouraged from seeking accreditation for test methods that depart from recognised published standards. If, however, approval of an in-house test method is required, the following information must be provided:

- (a) A copy of the fully documented test method
- (b) Details of the origin of the in-house test method
- (c) Details of the reason for its development and application
- (d) The results of comparative tests with standard methods (if possible) and other laboratories (if possible)
- (e) Full details of test method validation as described in Clause 5.4.5 of NZS ISO/IEC 17025:2005

Once a laboratory is accredited for a specific test method, the detailed procedures of that method must be adhered to at all times. Occasionally it may be necessary to deviate from the documented test method. Any departures must be reported with the test results and may invalidate accreditation status of that particular test.

Accreditation for opinions and interpretations in chemical testing is not offered under the IANZ Chemical Testing Laboratory Accreditation Programme.

9 Uncertainty of Measurement and Limits of Detection

9.1 Uncertainty of Measurement

It is important for testing laboratories to understand the concept of uncertainty of measurement. Laboratory management should be aware of the effect that their own uncertainty of measurement will have on test results produced in their laboratory.

Clause 5.4.6 of NZS ISO/IEC 17025:2005 requires testing laboratories to estimate their measurement uncertainty in the testing they conduct. While the concept and application of measurement uncertainty estimations have been well established in metrology and calibration laboratories, the same cannot be said for testing laboratories. The publication of NZS ISO/IEC 17025 has prompted rigorous discussion internationally on uncertainty of measurement in chemical testing and a consensus agreement on the definitive methodology to be used for estimating uncertainty is still to be finalised. Readers are encouraged to familiarise themselves with current developments through sources such as those detailed in the References 10, 11 & 17. It is noted that measurement uncertainty is closely related to the concept of traceability and, in metrological terms, it relates to the uncertainty inherent in the chain of comparisons from the laboratory's result to the SI or other defined reference.

The following details the current requirements for laboratories accredited under the IANZ Chemical Testing Laboratory Programme:

- (a) Laboratories need to make a formal estimate of measurement uncertainty for all tests in the scope of accreditation that provide numerical results. Where results of tests are not numerical or are not based on numerical data e.g. detected/not detected, pass/fail, positive/negative, or based on visual, tactile or other qualitative examinations, estimates of uncertainty are not required.
- (b) Where an estimate of measurement uncertainty is required, laboratories need to document their procedures and processes on how this is to be done.

There are various published approaches to the estimation of uncertainty in testing. NZS ISO/IEC 17025 does not specify any particular approach. All approaches which give a reasonable estimate and are considered valid within the chemical testing communities are equally acceptable and no one approach is favoured over others. For guidance, Appendix 5 sets out a possible approach which IANZ would recommend as being consistent with approaches internationally. This approach is not mandatory but alternative approaches would be expected to address the principles embodied within it. Laboratories are referred to the References 11, 13 & 17 for further information.

What is important is that laboratories document, with reference to published procedures, what their approach to estimating uncertainty in measurement will be. IANZ assessment teams will assess the suitability and rigour of these approaches during annual assessments.

- (c) Once a documented procedure is established, the laboratory needs to implement a programme for applying this procedure to all relevant tests within the scope of accreditation.

The procedures in (b) above may require a redesign of current quality control programmes and data may need to be collected over a reasonable length of time in order to make a sufficiently rigorous assessment of measurement uncertainty. Laboratories will need to maintain records of each test or type of tests to demonstrate full implementation of the procedure required by (b) above.

9.1.1 Reporting Measurement Uncertainty

Chemical testing laboratories are not required to report their estimated measurement uncertainty on test reports as a matter of routine.

However, Clause 5.10.3.1(c) of NZS ISO/IEC 17025:2005 requires reporting of measurement uncertainty when it is required for the correct application or interpretation of the test result. One such instance is where test results are used to determine if a sample conforms to a required numerical specification and where the specification limit falls within the limits of measurement uncertainty associated with the test result obtained.

9.1.2 Limits of Detection

For some chemical testing, method detection limits will need to be determined i.e. where laboratories are performing analyses at low analyte levels such as in residue testing and low level micronutrient testing.

As with measurement uncertainty, there are various published methodologies for the determination of method detection limits. In general, these different methodologies are associated with either:

- (a) The source of the published standard methods e.g. Codex, AOAC, etc., or
- (b) The specific industry sector the laboratory is operating within and in which the results are used.

Where the determination of method detection limits is applicable, laboratories will need to have a documented procedure on how they determine their detection limits. The procedure should be consistent with the source of methodologies normally used; or the conventions used within the industry sector.

In the absence of industry or test methodology conventions, there is an expectation that laboratories will determine method detection limits from a series of independent analyses of the analyte concerned in the matrix of interest at a level near the expected limit of detection. The method detection limit is calculated from the variation of results at this level and is not to be confused with a so-called instrument level of detection obtained from readings of a series of blanks. In some cases, the sensitivity of the instrument is such that the lowest calibration standard is more than two orders of magnitude below the regulatory limit. In this situation, laboratories may sometimes set the limit of detection at the level of the lowest calibration standard, but this approach does require that the laboratory consider the manner in which they report low positive results below the artificially high limit of detection to ensure that reports are not misleading to the users.

10 Test Records and Reports

10.1 Test Records

An adequate test records system in accordance with the various clauses of NZS ISO/IEC 17025 (e.g. 4.13, 5.4.7) is essential.

Most laboratories have developed forms (proforma sheets) for all of their routine testing. These are generally the preferred option as their use prompts the recording of all the required information, maintains consistency and increases recording efficiency.

Test records may also be contained in personal or test specific workbooks. Where such workbooks are free text (i.e. not bound proforma sheets), this type of records system is generally less efficient, and requires a greater level of management to ensure that records are not lost. For these reasons free text recording systems are now usually found only where a high level of non-routine testing is carried out, e.g. in research organisations.

10.2 Test Reports

Clauses 5.10.1, 2, 3, 6, 7, 8 and 9 of NZS ISO/IEC 17025:2005 sets out the requirements for test reports issued by testing laboratories.

Test reports must give the client all relevant information and every effort should be made to ensure that the test report is unambiguous. All information in a test report must be supported by the records pertaining to the test. All information required to be reported by the test specification must be included in the report.

It is important to note that in many instances the test standards, regulatory requirements and industry accepted practice will determine the report format and content.

Laboratories must retain an exact copy of all reports issued. These copies must be retained securely and be readily available for the time specified in the laboratory's documented policies.

10.2.1 IANZ-Endorsed Test Reports

Accredited laboratories are permitted to include reference to their accreditation in the test reports they issue. The general rules governing the use of IANZ endorsements are detailed in Appendix 1 of the IANZ publication *Procedures and Conditions of Accreditation* (AS 1).

For chemical testing laboratories, all test reports carrying the IANZ endorsement must be formally authorised by at least one of the laboratory's nominated Key Technical Personnel (see Section 7 and Appendix 2). This would normally be by a signature on the report itself (see also Section 10.2.2 below).

It is recognised that many of today's laboratories are multi-disciplinary in nature and, in some cases, very specialised within disciplines. Test reports pertaining to a particular sample or set of samples may include test results from several specialist areas and/or disciplines.

While the technical scope of nominated Key Technical Persons is expected to match their expertise in various specialist areas and/or disciplines, it is not practical to expect a number of Key Technical Personnel to sign a test report to cover each of the results that may be reported therein. In these instances it is acceptable that a multi-disciplinary test report is signed by only one of the laboratory's Key Technical Persons under the following conditions:

- (a) The individual authorising the test report is responsible for ensuring all results which are outside their technical scope as a Key Technical Person (and that are included in the test report) have been authorised or released internally by a Key Technical Person (or delegated staff – see Appendix 2, point (c) with these tests in their technical scope
- (b) There is a clear audit trail within the laboratory's system to demonstrate this.

10.2.1.1 Opinions and Interpretations

Clause 5.10.5 of NZS ISO/IEC 17025:2005 allows for test reports to include statements of opinion and interpretation related to the test results. In chemical testing, it is the policy of IANZ that accreditation is not granted to laboratories for providing statements of opinion and interpretation of test results.

Except where an interpretation is clearly factual e.g. a statement of compliance or otherwise with a specification, opinions and interpretations cannot be implied as being within the scope of the laboratory's accreditation on an IANZ-endorsed test report.

This does not preclude accredited laboratories from making such statements as an added value service to their clients. However, they should either be given in a (non-IANZ endorsed) separate document to the test report or, if included directly in IANZ-endorsed reports, a clear disclaimer made that the statements made are outside the laboratory's scope of accreditation.

10.2.2 Electronic Reporting

Traditionally, laboratories issued test reports in hard copy format with manuscript signatures (from IANZ Approved Signatories if the test report was IANZ-endorsed). With increased use of electronic media such as email and the internet and the use of electronic databases, laboratories are now being required to report electronically. Such practices challenge the generally accepted reporting criteria for accredited laboratories.

Clause 5.10.7 of NZS ISO/IEC 17025:2005 attempts in a very general way to specify the requirements for electronic reporting. While it is difficult to specify in detail a set of requirements to address every eventuality (as laboratories will tend to develop electronic reporting systems to suit their own circumstances and those of their clients), the following is intended to provide guidance on common issues of concern.

10.2.2.1 Transmission of Reports

It is the responsibility of the issuing laboratory to ensure that what is transmitted electronically is what is received by the client.

Email systems have proven to be robust in this regard, but laboratories need to consider whether clients will have the appropriate software and version to open attachments without corruption.

Laboratories should verify (at least initially and periodically thereafter is recommended) the integrity of the electronic link e.g. by asking the client to supply a copy of what was received and comparing it with what was transmitted. It is also important the laboratory and its client agree as to which part of the electronic transfer system they are responsible for and the laboratory must be able to demonstrate data integrity at the point the data comes under the control of the client.

10.2.2.2 Security

Laboratories should avoid sending test reports in an electronic format that can be readily amended by the recipient. Examples would be in word processing or spreadsheet software. Where possible, reports should be in a read only format e.g. pdf files.

Where this is not possible e.g. the client may wish to transfer the reported results file into a larger database, then laboratories are recommended to indicate these electronic reports have an interim status and are followed-up by a hard copy (or more secure) final report.

Laboratories must retain an exact copy of what was sent. This may be a hard copy (recommended) or an electronic copy. These copies must be retained securely and be readily available for the time specified in the laboratory's documented policies.

10.2.2.3 Electronic Signatures

All reports (whether hard copy or electronic) must not be released to the client until authorised by individuals with the authority to do so. For electronic reports there must be a clear audit trail with a positive authorisation record to demonstrate this is the case. Where this is managed through password access levels in the laboratory's electronic system, appropriate procedures should be in place to prevent abuse of password access.

The electronic report should show the identity of the individual releasing the report (a nominated Key Technical Person in the case of IANZ-endorsed reports). This may involve an electronic signature. The security of these signatures should be such as to prevent inadvertent use or abuse.

10.2.2.4 Electronic Report Formats

Clause 5.10.1 of NZS ISO/IEC 17025:2005 allows for simplified report formats for internal clients or in the case of written agreement from the client. This is often the case for electronic reports. While the laboratory may be accredited for the testing, it is usual such reports would not normally carry the formal IANZ-endorsement.

IANZ-endorsed reports, whether electronic or not, would normally be expected to comply with the requirements of Clause 5.10.2 and 5.10.3 (as appropriate) of NZS ISO/IEC 17025:2005.

11 Quality Control

It is essential that accredited chemical testing laboratories have developed, documented and implemented an appropriate quality control (QC) programme.

Clauses 5.9 and 5.4.7.1 of NZS ISO/IEC 17025:2005 suggest various quality control procedures that can be included in a laboratory's day-to-day activities and each laboratory is expected to implement the procedures most appropriate to their circumstances. Quality control data should be analysed and, where it is found to be outside pre-defined action criteria, the defined actions shall be taken to correct the apparent problem and to prevent incorrect results from being reported.

It is important for laboratories to understand where tests can go wrong so that steps can be taken to either eliminate the potential error point or put in an appropriate QC step for alerting the operator when the test has gone wrong. Quality control in some form is possible with any test being performed. A disciplined approach is required for the development of a suitable QC plan and this approach should be applied on a test-by-test basis.

The quality control programme should be designed in such a way as to demonstrate that the on-going control of both the accuracy and precision of each test is being maintained.

Where tests are performed infrequently, the laboratory should carry out regular performance checks to demonstrate its continuing competence to perform them or have in place a system for demonstrating proficiency prior to performing the test on a client sample.

12 Proficiency Testing

Proficiency testing is defined as the “determination of laboratory testing performance by means of inter-laboratory comparisons” (ISO Guide 43-1:1997) and is, thus, a very important tool in a laboratory’s quality control programme to demonstrate the validity and comparability of results.

In accordance with the policy of the Asia Pacific Laboratory Accreditation Co-operation (APLAC), to which IANZ is a full member of their Mutual Recognition Arrangement (MRA), (see Reference 13), it is IANZ policy that applicant/accredited chemical testing laboratories shall:

- (a) Demonstrate their technical competence by the satisfactory participation in proficiency testing activity where such activity is available, and that
- (b) The minimum amount of appropriate proficiency testing required per laboratory is one activity prior to gaining accreditation, followed by:
 - (i) Participation in as many inter-laboratory comparison programmes (where available) required to cover the scope of accreditation, and
 - (ii) For the programmes selected, to participate in all relevant rounds that are available. Where multiple programmes exist covering the same methodologies on similar sample types, participation in all rounds may be relaxed. This would need to be justified on a performance-based criteria and each case will be treated on its merits. The overall frequency must still be such as to demonstrate on-going proficiency.

Aside from the issues of coverage and frequency, laboratories are expected to select proficiency testing activities according to the following criteria (in a generally decreasing order of preference):

- (a) Mandated programmes. In some areas of chemical testing, participation in a particular programme is mandatory e.g. the National Asbestos Programme operated by NATA for laboratories conducting asbestos fibre counting
- (b) International inter-laboratory comparison programmes
- (c) National inter-laboratory comparison programmes
- (d) Proficiency testing programmes operated in accordance with ISO Guide 43 : Part 1
- (e) Formal inter-laboratory comparison programmes involving several independent laboratories
- (f) Less formal inter-laboratory comparison programmes between two or more laboratories.

The participation in a programme is of little value without the combined results being analysed to determine the nature of any discrepancies and the effect of this on any routine test results. Discrepancies may be in the order of expected uncertainty or they may indicate a serious shortcoming in a laboratory’s procedure. It is important for laboratories to have undertaken this analysis and to have adequately determined and implemented appropriate corrective action.

Records of the above analysis and any action taken of all proficiency testing results are required, including those for which no further action is considered appropriate i.e. satisfactory results.

The results from proficiency testing activities and their analysis will be viewed by IANZ at each assessment.

12.1 APLAC Proficiency Testing Programmes

From time to time, APLAC arranges for proficiency testing programmes to be run and expects accredited laboratories in all economies which are members of the MRA to participate.

On receipt of an invitation to participate, IANZ nominates (usually to a maximum of four) accredited laboratories to participate, provided the programme is relevant to their scope of accreditation. Nominated laboratories are expected to participate (usually no fee is charged) unless there are valid reasons for not doing so.

The results from these APLAC Proficiency Testing Programmes are required to be treated by IANZ in a formal manner. Both the participating laboratories and IANZ receive a copy of the report. Where a particular laboratory has outlier or non-conforming results, they will be required to submit to IANZ detail on the investigations conducted and any corrective action taken. (It should be noted that all

Chemical Testing

accredited laboratories in any inter-laboratory comparison programme are expected to do this but would not normally report it to IANZ. Such records would be reviewed at the next on-site visit).

IANZ staff will review the response and comment where appropriate. The records will also be reviewed at subsequent on-site assessments – particularly by a technical expert where appropriate.

It should be noted that APLAC Proficiency Testing Programmes are as much a measure of the IANZ performance in accrediting laboratories as they are a measure of the participating laboratories' performance. The co-operation of the nominated laboratories is appreciated by IANZ.

13 References

1. NZS ISO/IEC 17025:2005 – *General requirements for the competence of testing and calibration laboratories*
2. *Procedures and Conditions of Accreditation* (AS 1), IANZ
3. AS/NZS 2243 – *Safety in Laboratories*
4. *International Vocabulary of Basic and General Terms in Metrology (VIM)*, 3rd Ed.(2006), ISO/BIPM/IEC/IFCC/IUPAC/IUPAP/IOML
5. *Eurachem/CITAC Guide: Traceability in Chemical Measurement – A guide to achieving comparable results in chemical measurement*, 2003 (see www.eurachem.org).
6. King,B. *Meeting ISO/IEC 17025 Traceability Requirements: A new guide with worked examples*, CITAC News (February 2003), p.8
7. ISO Guide 34: 2000 – *General requirements for the competence of reference material producers*
8. *Guide to the Expression of Uncertainty in Measurement*, 1st Ed.,(1995), ISO/BIPM/IEC/IFCC/IUPAC/IUPAP/OIML
9. *Eurachem Guide: Quantifying Uncertainty in Analytical Measurement*, 2nd Ed.,(2000) (see www.eurachem.ul.pt)
10. ISO/IEC 5725:1994 – *Accuracy (trueness and precision) of Measurement Methods and Results*
11. APLAC TC005: *Interpretation and Guidance on the Estimation of Uncertainty of Measurement in Testing* (Issue No.1, March 2003), Asia Pacific Laboratory Accreditation Cooperation (APLAC), (see www.aplac.org)
12. ISO/IEC Guide 43-1: 1997 – *Proficiency Testing by Inter-laboratory Comparisons – Part 1: Development and operation of proficiency testing schemes*
13. APLAC MR-001: *Procedures for Establishing and Maintaining Mutual Recognition Arrangements amongst Accreditation Bodies* (Issue No.4, December 2002)
14. IANZ Technical Guide 2: *Laboratory Balances Calibration Requirements* (AS TG2)
15. IANZ Technical Guide 3: *Working Thermometers Calibration Procedures* (AS TG3)
16. IANZ Technical Guide 4: *UV/Vis Spectrophotometers Calibration Procedures* (AS TG4)
17. IANZ Technical Guide 5: *Uncertainty of Measurement, Precision and Limits of Detection in Chemical and Microbiological Testing Laboratories* (AS TG5)
18. American Society for Testing and Materials ASTM D3631: *Test Methods for Measuring Surface Atmospheric Pressure*
19. *Eurachem Guide: The Fitness for Purpose of Analytical Methods – A Laboratory Guide to Method Validation and Related Topics* (1998).

APPENDIX 1

CLASSES OF TEST

CHEMICAL TESTING

- | | | | |
|------|--|------|--|
| 2.01 | Metals and Alloys
(a) Ferrous materials
(b) Copper and copper alloys
(c) Aluminium and aluminium alloys
(d) Tin, lead and their alloys
(e) Magnesium and magnesium alloys
(f) Zinc and zinc alloys
(g) Precious metals
(h) Other metals and alloys | 2.13 | Lubricants
(a) Oils and greases
(b) Other lubricants |
| 2.02 | Metallic Coatings and Treatment Solutions
(a) Metallic and conversion coatings
(b) Plating solutions and metal finishing materials | 2.14 | Bituminous Materials
(a) Bitumens and asphalts
(b) Tars and tar products |
| 2.03 | Corrosion Tests | 2.15 | Petrochemicals and Related Products
(a) Waxes, petrolatums, white oils and similar products
(b) Antifreeze and de-icing fluids
(c) Hydraulic fluids
(d) Additives to fuels and lubricants
(e) Temporary corrosion preventives
(f) Electrical insulating oils and compounds
(g) Petrochemical feedstocks
(h) Trace contaminants |
| 2.04 | Ores and Minerals
(a) Ferrous ores
(b) Precious metal ores
(c) Radioactive ores
(d) Other non-ferrous ores
(e) Minerals
(f) Trace elements in geochemical prospecting samples | 2.16 | Solvents |
| 2.05 | Clays, Ceramics and Related Materials
(a) Clays, ceramics and refractories
(b) Lime and gypsum
(c) Sands
(d) Glasses | 2.17 | Paints and Surface Coatings
(a) Raw materials
(b) Formulated products and applied coatings |
| 2.06 | Cement and Concrete
(a) Portland cement
(b) Other cements
(c) Concrete and mortar
(d) Fly ash | 2.18 | Inks and Dyes
(a) Raw materials
(b) Formulated products |
| 2.10 | Oil Shale | 2.19 | Polishing Compounds |
| 2.11 | Crude Petroleum | 2.20 | Explosives |
| 2.12 | Fuels
(a) Gaseous fuels
(b) Liquid fuels
(c) Solid fuels | 2.21 | Rubber, Natural and Synthetic
(a) Raw materials
(b) Formulated products |
| | | 2.22 | Plastics
(a) Raw materials
(b) Formulated products |
| | | 2.23 | Leather
(a) Leather
(b) Pickled pelts
(c) Salt cured skins
(d) Wet blue leather
(e) Other related products |

- 2.24 Textiles and Textile Products
 (a) Identification of fibres
 (b) Quantitative analysis of mixtures and blends of fibres
 (c) Chemical tests
 (d) Colour fastness tests
 (e) Flammability
 (f) Other tests
- 2.25 Paper, Paperboard and Wood Pulp
 (a) Fibre composition
 (b) Chemical analyses
 (c) Other tests
- 2.26 Adhesives and Sealants
- 2.31 Foods
 (a) Cereals and cereal products
 (b) Edible oils, fats and their products
 (c) Nuts, fruits and vegetables and derived products
 (d) Sauces, herbs, spice and condiments
 (e) Sugars and sugar confectionery
 (f) Dairy products
 (g) Meat, poultry and derived products
 (h) Fish and fish products
 (i) Eggs and egg products
 (j) Alcoholic beverages
 (k) Non-alcoholic beverages
 (l) Food additives and supplements
 (m) Essential nutrients, including vitamins
 (n) Residues in foodstuffs
 (o) Other specified foods
- 2.32 Drugs and Pharmaceuticals
 (a) Uncompounded drugs
 (b) Other chemicals used in compounding pharmaceuticals
 (c) Antibiotics and their preparations
 (d) Enzymes and their preparations
 (e) Hormones and their preparations
 (f) Vaccines and sera
 (g) Vitamins and their preparations
 (h) Medicinal and veterinary preparations
 (i) Other products
- 2.33 Cosmetics, Perfumes and Essential Oils
 (a) Cosmetics
 (b) Perfumes
 (c) Essential oils
- 2.34 Fats, Oils and Waxes
- 2.35 Detergents
 (a) Soaps
 (b) Synthetic detergents, wetting and emulsifying agents
- 2.36 Agricultural Products and Agricultural Materials
 (a) Wheat and other cereal grains and by-products
 (b) Oil seeds and by-products
 (c) Stockfoods and licks
 (d) Essential nutrients in stock foods and licks including vitamins
 (e) Pesticides and herbicides
 (f) Fertilizers
 (g) Soils
 (h) Plants
 (i) Other agricultural products and related materials
 (j) Residues in agricultural products and related materials
- 2.37 Timber and Timber Treatment
 (a) Timber
 (b) Timber treatment solutions
 (c) Wood chips
- 2.38 Medical Devices
 (a) Contaminant residues
- 2.40 Weathering Tests
 (a) Outdoor tests
 (b) Accelerated tests
 (c) Other tests
- 2.41 Waters
 (a) Potable waters
 (b) Non-potable waters e.g. receiving waters, ground waters
 (c) Sewage
 (d) Effluents and trade wastes
 (e) Cooling tower and industrial waters
 (f) Swimming pools and spas
 (g) Marine waters
 (h) Boiler waters
- 2.50 Gases
 (a) Industrial gases
 (b) Gases for medical and life support use
 (c) Fumes and emissions
 (d) Atmospheric pollution
 (e) Other gases and gas mixtures
 (f) Instrument calibration

Chemical Testing

2.58	Environmental Monitoring (a) Waters (b) Air (c) Soils and sludges (d) Other materials	2.62	Forensic Biology (a) Physiological fluid identification (b) DNA typing
2.59	Motor Vehicles (a) Vehicle emissions (b) Fuel consumption tests	2.70	Instrumental Techniques (See Note 1) Such as atomic absorption/emission spectroscopy, colourimetry, high performance liquid chromatography, gas chromatography, mass spectrometry, etc.
2.60	New Zealand Shellfish Quality Assurance Programme (a) Shellfish toxin extraction (b) Shellfish toxin assay	2.71	Non-Instrumental Techniques (See Note 2)
2.61	Biological Specimens (a) Residues in specified human specimens (b) Residues in specified veterinary specimens (c) Other chemical tests	2.81	Other Specified Inorganic Materials
		2.82	Other Specified Organic Materials

Note 1 2.70 Instrumental Techniques

This class of test is not widely accredited in New Zealand laboratories. It was introduced to cover the situation of recognised experts working in a technique and able to apply it in an analytical development situation such as would arise in a large consulting, specialist or research laboratory. It is not applicable to a laboratory whose work is routine and on one instrument.

Accreditation is not granted as a stand alone Class of Test and will always be linked to other classes of test in the laboratory's scope of accreditation e.g. to 2.31 Foods or 2.41 Waters. It, therefore, allows the laboratory to claim accreditation for analytes which are not specifically detailed elsewhere in the scope of accreditation but which have been tested by the specified instrumental technique.

Accreditation is closely linked to the individual expertise within the particular laboratory. See Section 7 and Appendix 2.

For accreditation to be considered, laboratories are required to have a fully documented and operational protocol for the development and validation of new methods/analytes. Laboratories which provide IANZ-endorsed test results produced under the umbrella of their 2.70 accreditation i.e. for tests not specifically listed elsewhere in their scope of accreditation, must be able to demonstrate retrospectively that the method has been through the internal development and validation protocol prior to the results being issued.

Should the test become routine in the laboratory, it is expected it will be specifically included in the scope of accreditation during the course of a routine assessment.

Note 2 2.71 Non-Instrumental Techniques

This class of test is not widely accredited in New Zealand laboratories. It was introduced to cover the situation of recognised experts working in a particular field using a variety of non-instrumental classical analytical techniques and being able to apply them in an analytical development situation such as would arise in a large consulting, specialist or research laboratory. It is not applicable to a laboratory whose work is routine.

Accreditation is not granted as a stand alone Class of Test and will always be linked to other classes of test in the laboratory's scope of accreditation e.g. to 2.31 Foods. It will also indicate the common reference method sources from which the non-instrumental method is sourced e.g. AOAC

International, Codex, Pearsons, etc. It, therefore, allows the laboratory to claim accreditation for methods and analytes which are not specifically detailed elsewhere in their scope of accreditation but which have been tested by non-instrumental methods sourced from the references indicated.

Accreditation is closely linked to the individual expertise within a particular laboratory. See Section 7 and Appendix 2.

For accreditation to be considered, laboratories are required to have a fully documented and operational protocol for the development and validation of new methods or for the verification and commissioning of standard methods new to the laboratory. Laboratories which provide IANZ-endorsed test results produced under the umbrella of their 2.71 accreditation i.e. for tests not specifically listed elsewhere in their scope of accreditation, must be able to demonstrate retrospectively that the method has been through the internal development and validation/ verification and commissioning protocol prior to the tests result being issued.

Should the test become routine in the laboratory, it is expected it will be specifically included in the scope of accreditation during the course of a routine assessment.

APPENDIX 2

KEY TECHNICAL PERSONNEL

Supervisory staff in accredited laboratories must be competent and experienced in the technical areas covered by their accreditation. They must be able to oversee the operations and cope with any problems that might arise in their work or that of their colleague or subordinates. Such staff members, formally appointed by the senior management of the laboratory, are referred to as Key Technical Personnel.

The following sets out IANZ's expectations in relation to who the laboratory management should be appointing as Key Technical Persons.

- (a) Key Technical Persons would be expected to have:
 - (i) A tertiary qualification or equivalent professional recognition in the relevant discipline. Laboratories engaged in a restricted range of repetitive work may be able to appoint Key Technical Personnel with appropriate practical experience and specific training in that work but without formal qualifications
 - (ii) A position in the staff structure which provides for the authority to implement necessary changes in the laboratory operation to ensure the integrity of test results is maintained. The position in the staff structure should ensure the individual can maintain a working knowledge of the quality assurance and technical systems in operation in the laboratory on a day to day basis
 - (iii) A working knowledge of and commitment to the requirements for IANZ accreditation, including the quality and technical management principles embodied in NZS ISO/IEC 17025 and relevant Specific Criteria
 - (iv) The necessary scientific expertise and experience to be aware of and understand any limitations of the test procedures and to fully understand the scientific basis of the procedures
 - (v) Sufficient experience in the accredited laboratory to address all of the above points.
- (b) Key Technical Personnel are those individuals who are given both the responsibility and authority to:
 - (i) Develop and implement new operational procedures
 - (ii) Design quality control programmes, set action criteria and take corrective action when these criteria are exceeded
 - (iii) Identify and resolve problems
 - (iv) Take responsibility for the validity of the outputs.
- (c) Key Technical Personnel would normally be those individuals who authorise the release of all test results. However, in large laboratories such authorisations may be delegated to other supervisory staff on a day to day basis provided the delegations and the basis for them are clearly documented. Such delegation of authority does not absolve the Key Technical Person from taking full responsibility for the validity of the work. The authority to release results should not be confused with the authority to issue formal test reports. See Section 10.
- (d) Laboratory management may choose to appoint an individual engaged by the accredited laboratory as a consultant where their Key Technical Person responsibilities relate to work done within the scope of accreditation. There is an expectation that there would be a written agreement between the parties setting out the extent of the authority and responsibility of the consultant in relation to the services provided. The consultant's position in the laboratory organisation should be such that they can perform their role as a technical decision maker as effectively as if they were an employee.
- (e) Staff members of an accredited laboratory who are not engaged full-time could also be appointed as Key Technical Persons. However, the circumstances in which they are called

upon to exercise their Key Technical Person responsibilities and their access to and knowledge of the technical operations should be such that they are able to take full responsibility for the work they authorise or oversee.

Classes of Test 2.70 and 2.71

As detailed in Section 7, accreditation in Classes of Test 2.70 and 2.71 will only be granted provided nominated Key Technical Personnel for this work have been assessed by, and the nomination endorsed by IANZ.

Nominated persons will need to demonstrate to an IANZ Assessment Team that, in addition to the general expectations above, they:

- (a) Have significant experience (years) in the instrumental technique or in the non-instrumental classical analytical techniques (as appropriate) as they apply to a wide variety of matrices and components
- (b) Are totally familiar with all aspects of the instrumental or non-instrumental techniques (as appropriate) including various types of equipment, detectors, etc
- (c) Have access to current literature on and maintain an up-to-date knowledge of recent research developments in the techniques and how they are applied to relevant matrices
- (d) Could be considered by peer review to be one of the country's authorities in the instrumental techniques or general analytical methods (as appropriate)
- (e) Have a demonstrated history of method validation for the techniques involved, preferably in accordance with the laboratory's own procedures for this.

APPENDIX 3

RECOMMENDED CALIBRATION INTERVALS

The following table sets out the normal periods between successive calibrations for a number of reference standards and measuring instruments. It must be stressed that each period is generally considered to be the maximum appropriate in each case providing the other criteria as specified below are met:

- (a) The equipment is of good quality and of proven adequate stability, and
- (b) The laboratory has both the equipment capability and staff expertise to perform adequate internal checks, and
- (c) If any suspicion or indication of overloading or mishandling arises, the equipment is checked immediately and thereafter at frequent intervals until it can be shown that stability has not been impaired. Where the above criteria cannot be met, appropriately shorter intervals may be necessary.

IANZ is, however, prepared to consider submissions for extension of calibration intervals based on the factors outlined in Section 6.

Items marked (*) in the table are those which may be calibrated by staff of a laboratory if it is suitably equipped and the staff are competent to perform such recalibrations. Where the staff of a laboratory have performed calibrations, adequate records of these measurements must be maintained.

IANZ has produced a number of Technical Guides with further information on some calibration procedures e.g. balances, spectrophotometers, thermometers. Contact IANZ for further details. These can also be found at www.ianz.govt.nz/publications2/technical_guides.htm.

Type of equipment	Maximum period between successive calibrations	Procedures
Anaerobic Jars or Cabinets	*Each use	Check condition by suitable means such as an indicator, vacuum gauge, growth of known anaerobes, etc.
Automatic Burettes, Dispensers and Pipettors	*Initial and three months	Accuracy of and repeatability at volumes in use.
Balances	Initial calibration and three yearly recalibrations	By an accredited calibration laboratory or *Calibration using traceable certified masses. Refer CSIRO Division of Applied Physics paper <i>Calibration of Balances</i> and IANZ Technical Guide AS TG 2: <i>Laboratory Balances – Calibration Requirements</i> . Staff performing calibrations need to be formally trained. Annual servicing is recommended.

Type of equipment	Maximum period between successive calibrations	Procedures
Balances (continued)	Accompanied by (a) *Each weighing (b) *One Month (c) *Six months	Zero check. One point check using a known mass close to balance capacity. See CSIRO paper. Repeatability checks at the upper and lower ends of the scale. See CSIRO paper. The standard deviation of the results can be compared against the results recorded on the last external calibration certificate.
Barometer <ul style="list-style-type: none"> • Fortin • Aneroid 	*Initial then every five years *One year	ASTM D3631: side-by-side comparison at the nearest meteorology office. Telephone comparison with the nearest meteorology office.
Centrifuges	*One year (where the operating speed is specified)	Tachometer (mechanical stroboscope or light cell type).
Comparators and Comparison Charts	<i>Either</i> *Regular replacement <i>Or</i> *One year	From approved supplier (dependent on use). Comparison against a (protected) reference comparator or chart.
Computerised Systems	*Instruments with electronic readouts must be calibrated as a system, including the electronic readout. The period between calibrations will depend entirely upon the nature of the instrument and the use it is being put to. *Computer programmes used to manipulate data into test results must be validated against manually calculated data upon commissioning. The results of this validation must be retained on file in the same manner as a calibration record and may be used for on-going QC checks. The programmes need revalidation if the programme is reloaded, subjected to a voltage spike or if doubt of the integrity exists. In any event, it is recommended that they be revalidated periodically. It is insufficient for the laboratory to assume that proprietary programmes or programmes adopted from another accredited laboratory are inherently correct. The laboratory will need to run its own commissioning validations and subsequent QC checks.	

Type of equipment	Maximum period between successive calibrations	Procedures
Conductivity Meter	*Each use <i>Note: If a temperature compensation probe is used, it must be calibrated. See thermometers.</i>	Checked using appropriate standards in each of the scale ranges of the meter in use.
Digestion Blocks e.g. Kjeldahl	*Two years	Temperature variation check across working spaces using thermocouple or recovery check with a difficult to digest standard.
Dry Gas Meter	Two years	Check against an externally calibrated reference wet or dry gas meter.
Dumas instrumentation e.g. Leco (See Note 2)	*Each use	Calibration against a pure certified reference material e.g. EDTA, followed by regular with-in run monitoring of calibration stability.
Furnaces (for use at specified temperatures)	*On use *Six months *Two years	Monitor temperature with an appropriate sensor. Accuracy check of sensor using calibrated thermocouple or melting points of known materials. Temperature variation within working space (front to back) using reference standards e.g. calibrated thermocouple or melting points of known materials.
Hot Plates (for use at specified temperatures e.g. butter moisture)	*Six months	Temperature variation across the hot plate using calibrated thermometer(s).
Karl Fischer Titrators	*Each use	Known weighed amount of water.

Type of equipment	Maximum period between successive calibrations	Procedures
Manometer Reference (liquid) Working (liquid) Electronic	Every five years *Every three years Annual	By an accredited calibration agency. Check liquid cleanliness every five years. Check liquid cleanliness every three years. Checked against a reference manometer. Checked against an externally calibrated reference manometer.
Masses (integral, stainless steel or nickel-chrome alloys) <i>Note: For testing/calibration laboratories performing calibrations.</i>	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.
Mixers e.g. insolubility index and the like	*One year (where operating speed is specified)	Tachometer (technical stroboscope or light type cell).
Multi Gas Meter	Every two years *Six monthly *Each use	Full calibration, including maintenance. Five point (and zero) linearity check. Two point (and zero) for fixed or stationary instruments. Single point span check made on 75% to 90% full scale of range being used. <i>Note: For laboratories seeking accreditation for either US EPA 3A, 6C or 7E, the method specific calibrations need to be followed.</i>

Type of equipment	Maximum period between successive calibrations	Procedures
Near infra-red reflectance (NIR) instruments e.g. Infralysers (See Note 1 below)	*Initial *As required (seasonal recommended) *Following each calibration or recalibration *Routine	Stable and accurate calibration for each product/constituent combination against accredited reference method results. Recalibration using up-dated calibration set. Validation of calibration using independent set of reference method results. Monitoring of calibration stability against reference method results.
pH meter	*Daily or before use <i>Note: If a temperature compensation probe is used, it must be calibrated. See thermometers.</i>	Calibrate using at least two appropriate standard buffers. Buffers need to be stored in appropriate containers and marked with an expiry date.
Pitot Tubes	*Initial *Each use	Check dimensional compliance against BS 1042, section 2.1, Annex A or against a L type Pitot tube as per US EPA 2. Inspect tip for damage or blockage.
Refractometers	*Each use *Six months	Check against distilled water. Check against α -bromonaphthalene or other reference compound of known refractive index.
Refrigerators	*Daily	Monitor the temperature and record.
Rotameter <ul style="list-style-type: none"> • Reference • Working 	*Two years High flow i.e. >1 L/min Low flow i.e. <1 L/min *One year	By an accredited calibration laboratory or use ASTM D3195. Check with a Soap Bubble Flow meter. Check with a Soap Bubble Flow meter.

Type of equipment	Maximum period between successive calibrations	Procedures
Sieves	Endecott sieves (or other approved brands) supplied with certificates do not require checks. Working sieves can be compared against reference sieves using material typical of the samples normally subjected to sieve analysis in the laboratory.	
Spectrophotometers Calibration filters <ul style="list-style-type: none"> • Wavelength filters • Transmittance/Absorbance filters 	Six months (wavelength and absorbance accuracy) Five years Annually for two years, then two yearly once stability has been demonstrated	By an accredited calibration agency or; *Using traceable certified filters, or; *In accordance with IANZ Technical Guide AS TG 4 <i>UV/Vis Spectrophotometer Calibration Procedures.</i> By an accredited calibration laboratory By an accredited calibration laboratory
Sterilisers <ul style="list-style-type: none"> • Autoclaves • Hot Air Sterilising Ovens 	Initial and following repair or maintenance *Each use *Each use	Check heating profiles of typical loads with respect to chamber temperatures to determine lag times (see Appendix 7) by an accredited calibration laboratory or; *Using appropriately calibrated equipment following a fully documented procedure. Annual servicing of steam sterilisers is strongly recommended. Check the time and temperature of the cycle. Discard loads should be autoclaved for at least 30 minutes at 121°C. Check of time and temperature. At least 160°C for 2 hours.
Tachometers	Five years	By an accredited calibration laboratory.
Thermocouples <ul style="list-style-type: none"> • Reference 	Three years or 100 hours use (whichever is sooner)	By an accredited calibration laboratory.

Type of equipment	Maximum period between successive calibrations	Procedures
Thermocouples (continued) <ul style="list-style-type: none"> • Working 	*Six months	Single point within the working range against a reference thermometer or thermocouple.
Thermometers (Liquid in glass) <ul style="list-style-type: none"> • Reference • Working 	Five years (complete) *Six months *Initial *Six months	By an accredited calibration laboratory, followed by an ice point check on receipt. Ice point (See Technical Guide 3 AS TG3: <i>Working Thermometers Calibration Procedures</i>). Check against reference thermometer / thermocouple across working range or at points of use. (See Technical Guide 3 AS TG3: <i>Working Thermometers Calibration Procedures</i>). Check at ice point or at points of use.
Thermometers (Resistance) <ul style="list-style-type: none"> • Reference • Working 	Five years *Six months *Initial *Six months	By an accredited calibration laboratory, followed by an ice point check on receipt. Ice point. If outside five times the uncertainty of the calibration, complete recalibration is required. Check against reference thermometer / thermocouple across working range or at points of use. (See Technical Guide 3 AS TG3: <i>Working Thermometers Calibration Procedures</i>). Ice point. If outside five times the uncertainty of the calibration, complete recalibration is required.

Type of equipment	Maximum period between successive calibrations	Procedures
Thermometers (Handheld non-resistance electronic) <ul style="list-style-type: none"> • Working <p><i>Note: Handheld non-resistance working thermometers are generally considered of insufficient quality to be used as reference thermometers</i></p>	*One year	Check against reference thermometer / thermocouple across working range or at points of use (See Technical Guide 3 AS TG3: <i>Working Thermometers Calibration Procedures</i>).
Thermostatically Controlled Equipment (incubators, waterbaths, ovens)	*Daily *Two years	Monitor the temperature and record. Temperature variation within the working space by an accredited calibration laboratory; or *Using appropriately calibrated equipment following a fully documented procedure.
Timers (stopwatches, chart recorders) <ul style="list-style-type: none"> • Mechanical • Electronic 	*Three months *One year	Comparison against radio time “pips” or similar e.g. IRL Talking Clock (0900) 45678. Comparison against radio time “pips” or similar e.g. IRL Talking Clock (0900) 45678.
Viscometers <u>U Tube</u> <ul style="list-style-type: none"> • Reference • Working <u>Others</u> <ul style="list-style-type: none"> • Brookfield • Ferranti • Zahn 	Five years *Two years *Two years *Three months *One year	An accredited calibration laboratory, or; *Using standards oils. # Using quality oil against the reference tubes or using standard oils. Using standards oils. # Using standards oils. # Using standards oils. #

Type of equipment	Maximum period between successive calibrations	Procedures
Viscometers (continued) <ul style="list-style-type: none"> • Krebs Stormer • Flow Cup • Cone and Plate • Rotothinner 	*One year *One year maximum (depends on frequency of use) *Each use *Three months to one year (depending on frequency of use)	Using two standards oils. # Using three standards oils. # Using one standard oil (#) within 40-80% of working range, result within 5% of certified value. Using two standard oils. # <i># Standard viscosity oils must be stored in the dark in a closed clean container and may be used for two years from the date of opening.</i>
Volumetric glassware (flasks, pipettes, burettes, Dean and Stark traps, solubility tubes, butyrometers, organic syringes, etc.)	*Initial only	Using distilled water at critical graduations.

Notes

1. Near infrared reflectance (NIR) spectroscopy

(a) Initial calibration and recalibration

Stable and accurate calibration sets that cover each product/constituent combination sought for accreditation. The calibration must be against accredited reference methods of appropriate accuracy and precision and include data from throughout the expected range of product compositions. If the range in composition is small, such as would normally be the case for a component in a single product with a narrow range of acceptable compositions, then it would be expected that the calibration set would include at least 30 samples (preferably greater than 80) covering the range of results expected. Calibration sets covering products with a relatively wide range of compositions would be required to include a larger number of samples with these again covering the range in expected compositions. The records of this calibration set must be available along with the results of the appropriate data analysis such as regression analysis.

Documented procedures must contain the laboratory's policies and procedures detailing:

- (i) The minimum number of samples in a calibration set
- (ii) The required distribution of these samples across the calibration range
- (iii) Acceptance/rejection criteria on the regression statistics
- (iv) How often the calibration set is up-dated and regression statistics repeated, along with criteria for the acceptance or rejection of the new calibration. It is recommended calibration sets are reviewed annually.

(b) Validation of calibration sets

Each calibration or recalibration for (a) above needs validation against an independent set of samples. The validation set should be of at least 20 samples for a calibration set covering a narrow range in compositions and up to 50 or more for a calibration set covering a wider range of compositions and products. The validation set must cover the calibration and product range and have been analysed by the same accredited reference methods. Records of this validation set must be available for each calibration. The laboratory's documented policies and procedures need to detail this requirement along with acceptance/rejection criteria of the calibration set based on the results of the calibration set.

(c) Daily monitoring

An appropriate system for the daily monitoring (including comparisons against the reference methods and instrument parameters such as F-values) needs to be documented and implemented. These procedures are expected to detail:

- (i) Maximum allowable frequency of bias adjustments
- (ii) Maximum allowable magnitude of bias adjustments
- (iii) Required actions should these limits be exceeded
- (iv) Systems for on-going monitoring of bias adjustments and calibration stability
- (v) Authorities and responsibilities of each of the above.

In some situations daily monitoring may be provided by another group. In this case the laboratory staff needs access to records showing that the system of daily monitoring is effective.

2. Dumas Instruments

Calibration of these instruments requires the use of a suitably pure standard compound - usually EDTA. When setting-up the instrument, the following generic criteria are expected to be applied (in addition to the manufacturer's start up procedures):

- (i) The choice of individual determinations chosen for setting the blank must be from consecutive analyses (usually five) which agree within defined repeatability limits
- (ii) The choice of standard compound e.g. EDTA, determinations chosen for setting the calibration factor must be from consecutive analyses (usually five) which agree within defined repeatability limits
- (iii) Calibrations should be verified using appropriate matrix reference samples (where available)
- (iv) Standard compounds should be analysed at regular intervals throughout a run to monitor drift and always on start-up should the instrument have been sitting idle for some time
- (v) A record of all analyses (including blanks and standard compound determinations not chosen in the instrument calibration) must be retained.

APPENDIX 4

METHOD VALIDATION

Validation of chemical testing methods should only be carried out by laboratories with the appropriate knowledge, skills, experience and resources to do so in a competent and thorough manner. The requirements for method validation are detailed in Clause 5.4.5 of NZS ISO/IEC 17025:2005.

The following diagram provides for a very generalised approach to method validation that IANZ adopts when assessing the in-house validation of methods by individual laboratories and is considered to be consistent with the “fitness for purpose” principles embodied in Clause 5.4.5 of NZS ISO/IEC 17025:2005. It is not intended to be a comprehensive reference to validation requirements but rather a starting point to assist laboratories to ensure the key components are considered. In some instances, laboratories may need to do more to demonstrate full validation; in other instances, some of the elements may not need to be considered - depending on the purpose to which the method is to be applied.

Method Validation or Verification Reports

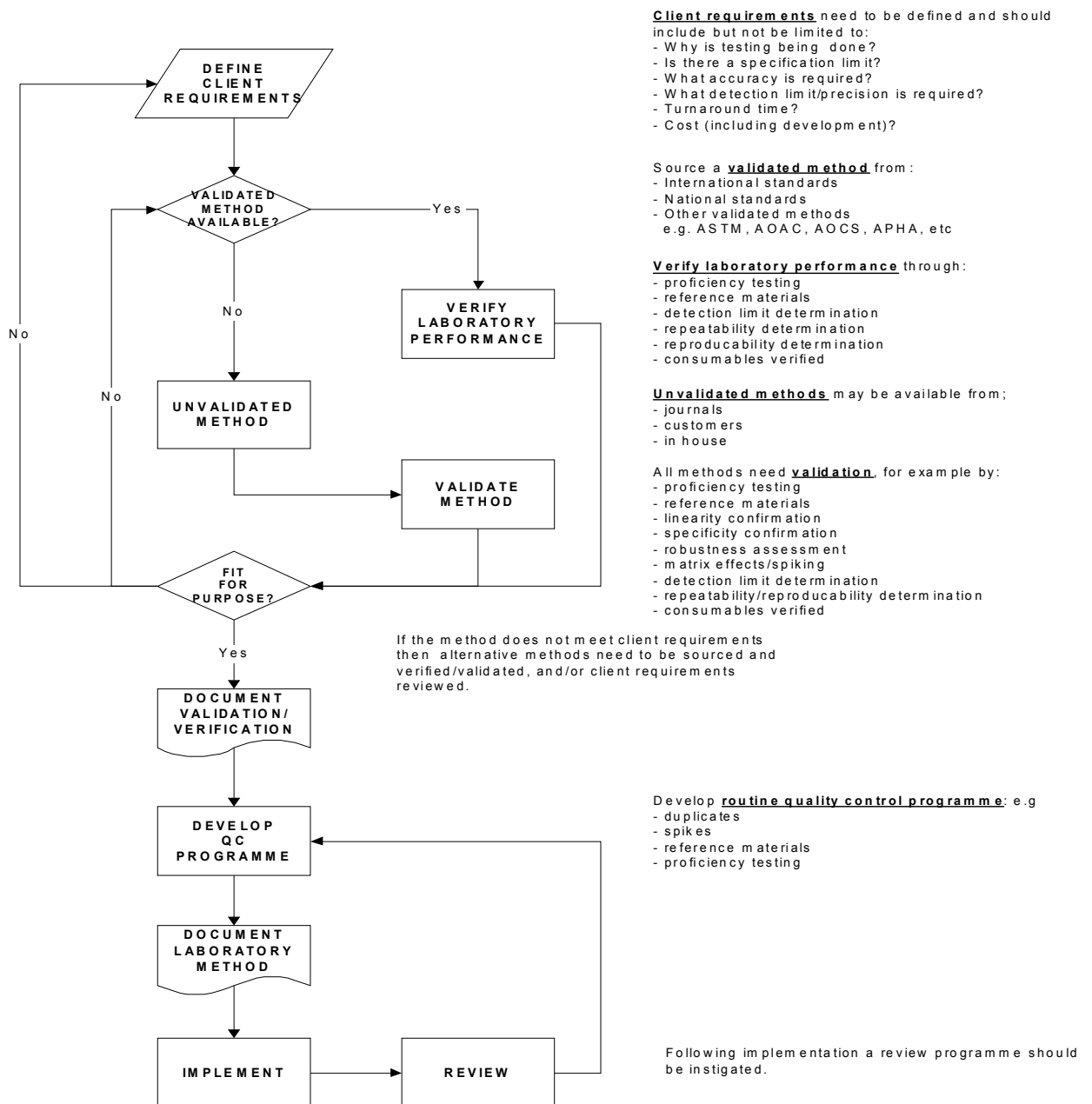
This provides some basic guidance for what should be included in a validation (verification) report for a new method for which accreditation is sought, (applicable to chemical testing).

- (a) Title e.g. study to determine method equivalence, validation of in-house method, etc
- (b) Author
- (c) Date
- (d) Summary
- (e) Objectives (analyte, scope, matrices etc)
- (f) Applicable reference methods (are these followed or used for guidance only to establish an in-house or modified method). Are methods internationally accepted, validated or manufacturers' instructions)
- (g) Method Performance:
 - (i) Precision, repeatability, intermediate reproducibility – with differences between the batches listed
 - (ii) Method accuracy from recoveries from spiked and/or real samples – with a description of how this was carried out
 - (iii) Matrix effects included for all matrices in the intended scope
 - (iv) Trueness of results by comparison with alternative methods, proficiency, certified reference materials
 - (v) Effect of method variation (robustness – acceptance criteria established for conditions found to be critical)
 - (vi) Effect of analyte levels (for unusually high or low levels – acceptable ranges should be determined)
 - (vii) Uncertainty of measurement, method limits of detection (where applicable), limits of quantitation (reporting)
 - (viii) Selectivity (are there interferences from other analytes)
 - (ix) Linearity (over the intended range).
- (h) Discussion (analysis of results)
- (i) Conclusion (was the objective achieved, include any limitations found to be necessary)
- (j) References
- (k) Appendices (raw data, methods, etc).

Method verification, that is determining that the laboratory can successfully perform in its own situation an analytical method previously validated and published by a recognised authority (AOAC, USEPA, etc), may not need to include all the above aspects if the laboratory is fully compliant with the reference method. To claim it follows a particular reference method does, however, imply that it can

match any method performance criteria given in the reference method and this needs to be demonstrated and included in the report.

Figure 1: General processes for method validation in chemical testing



APPENDIX 5

UNCERTAINTY OF MEASUREMENT

Section 9 sets out the IANZ policy for accredited chemical testing laboratories to make estimates of the uncertainty of measurements in their test results.

The following approach to estimating uncertainty of measurement is one that IANZ would suggest as being consistent with current published approaches in the international literature. It is not a mandatory specification and other approaches will be considered as equally valid provided they are sourced from published guidelines and meet the underlying principles of this process.

- (a) For each of the methods in the scope of accreditation providing numerical results, the laboratory should identify all components of the testing process which will contribute to the uncertainty in the final result. At this stage, it should not be necessary to quantify each component but rather just identify that it exists. Possible approaches to doing this exercise are:
- (i) By critically evaluating each step in the documented method to identify those actions/equipment etc. i.e. components that may affect the result
 - (ii) Using the method equation and critically evaluating each variable to identify the components that will affect its value.
- The use of fish-bone diagrams and the like may be a useful tool in this regard.
- (b) Identify and gather or collate all available data relating to the performance of the method. The sources of such data may be external to the laboratory or data generated internally i.e:
- (i) External data such as
 - Published validation data for the standard method (which may be published in the method itself or as a separate publication)
 - Results from formal proficiency testing or inter-laboratory comparison programmes e.g. reproducibility (R) figures
 - (ii) Internal data such as
 - In-house validation studies
 - Standard reference material results
 - In-house reference material results (generally giving rise to what is known as intermediate precision)
 - Precision or repeatability (r) data from duplicates
 - Uncertainty of measurement values from calibration certificates
 - Variability in spike recovery data.
- (c) Conduct a gap analysis to assess which of the components identified in (a) are incorporated in the data collected in (b). Care needs to be taken in this exercise. It is important to have a clear understanding of how the data collected in (b) are generated and what they mean. The following are a few examples which illustrate the type of issues that need to be considered:
- (i) Data from true duplicate sample testing will include components associated with taking the test portion from the submitted test sample (normally the taking of the test sample from the bulk is outside the control of the testing laboratory and, thus, the uncertainty component associated with sampling would not be considered) but will not normally include components of uncertainty associated with different equipment, different operators, different batches of media/reagents/standards, etc. The precision data from duplicates would in itself give an under-estimation of the overall uncertainty.

However, if as many of the testing variables (the components identified in (a) above) were to be varied in the analysis of each duplicate of each sample i.e. different analysts, different batches of diluents, media, etc; different pipettors, incubators, etc., then this data

(another form of intermediate precision) will provide a more realistic assessment of the measurement uncertainty.

Precision data from true duplicates gathered over a long period of time in which each of the components were varied may provide (following appropriate statistical analysis – details of which are outside the scope of this guidance) a possible estimate of uncertainty.

- (ii) In chemical testing, time issues also need to be considered – reference material data collected over weeks or months may be generated from only one batch of the analytical standard or only one batch of the stock solution. Thus, the uncertainty component associated with each of these i.e. purity of the analytical standard, mass and volume components associated with the stock solution, would not be included.
 - (iii) Reproducibility (R) data would generally give an over-estimation of an individual laboratory's uncertainty of measurement as it includes many different operators, types of equipment, batches of standards and often different methods, and some of these components are not relevant to a particular laboratory's circumstance. Estimates of reproducibility are dominated by the ability of the worst performing laboratories within the data set used to calculate the reproducibility and, therefore, make no allowance for the greater ability of the better performing laboratories. It may also not reflect the actual performance of the poorly performing laboratories and it needs to be noted that in most estimations any outliers results for poorly performing laboratories will have been removed from the data set. It should also be noted that reproducibility could also be an under-estimate as such data is normally generated from homogenous and stable samples, which may not reflect actual practices in working laboratories.
 - (iv) Spike recovery data needs to be carefully considered. The actual recovery itself is not a component of measurement uncertainty as it can be corrected for. However, variability in recoveries achieved is. Over time, this data will incorporate much of the measurement uncertainty components but in chemical testing may not include those associated with analytical standards (if only one batch is used – particularly for both the recovery experiment and calibration).
- (d) Where there are components identified in (a) which are not incorporated into the data collated in (b), these need to be independently estimated, their significance assessed and, where relevant, combined with the other uncertainty estimations.

Where these are significant, laboratories may need to review and redesign their quality control data collection programmes in order to incorporate as many of these additional components of uncertainty as possible.

Components of uncertainty which cannot be incorporated into the quality control data generated can be estimated by separate experiment, from published data, from calibration certificates, certificates of analysis or by professional judgement.

Statistical methods for the combination of components of uncertainty of measurement are outside the scope of this guidance document and readers should consult the referenced texts for further information.

The examples in (c) above suggest laboratories should be able to obtain data to sufficiently cover all significant identified components of uncertainty but these may come from different sources. It is important to ensure all major components of uncertainty are not double accounted.

Discussion

Chemical Testing

For the most part, professional judgement would suggest that the most significant contributions to measurement uncertainty in classical analytical chemistry would come from the extraction or separation of the analyte from the sample matrix. While other components should not be ignored, it is likely their contribution would be much less significant in comparison (but laboratories still need to demonstrate this). In addition, many chemical tests are empirical in nature (where the result is dependent on the method used) and, thus, if the method is followed, then method bias does not contribute to the measurement uncertainty.

The methodology suggested above for estimating measurement uncertainty will generally provide appropriate consideration of these issues and result in a reasonable estimate of the measurement uncertainty – provided the data are generated from samples of the same or similar matrix.

The model does have its limitations and, in particular, when the “extremes” of analytical chemistry are being used i.e. testing for purity of highly pure samples or testing for impurities or contaminants/residues at low levels. In these cases, components of uncertainty associated with effects, such as:

- (a) Purity of analytical standards
- (b) Balance performance when weighing small amounts of analytical/reference standards
- (c) Accuracy of volumetric glassware, etc.

(which would not normally be considered to have a significant effect) begin to have a significant contribution, which should not be ignored. Laboratories involved in such testing need to give extra consideration to the contribution of these effects.

In chemical testing, it is ideal if the uncertainty estimation is evaluated at selected levels across the range of application of the method. However, often a test is conducted to assess compliance with a particular specification, regulatory limit or the like. In these instances, laboratories should at least estimate an uncertainty value attributable to measurement results close to the specification limit i.e. to use the specification limit as the value at which the uncertainty is estimated.

The “number of significant figures” approach and that of Note 2 in Clause 5.4.6.2 of NZS ISO/IEC 17025:2005 should not be used as a substitute for evaluating measurement uncertainty in chemical testing.