



Ministry of Agriculture and Forestry
Te Manatu Ahuwhenua, Ngaherehere

DISCUSSION PAPER No. 37

MAF-approved Dairy Test Methods

Ministry of Agriculture and Forestry
New Zealand
September 2001
ISSN: 1174-961X
ISBN: 0-478-07645-2

Contents

1. Executive Summary	4
2. Purpose	5
3. Risk Assessment	5
4. Background	5
4.1 History	5
4.1.1 Before 1990	5
4.1.2 <i>Dairy Industry Regulations 1990</i>	5
4.1.3 Development of the MAF Standard D301	8
4.2 Current status.....	8
4.3 New Zealand’s legal requirements	9
4.4 International standards and guidelines,	9
4.4.1 Codex Alimentarius.....	9
4.4.2 ISO Standard 17025.....	10
4.5 Importing country requirements	10
4.5.1 European Union.....	10
4.5.2 USA	11
4.5.3 Other countries	11
5. Issues	11
5.1 Why approve test methods?.....	11
5.1.1 Role of test methods in product conformance decisions	11
5.1.2 What does test method approval provide?.....	11
5.2 Which test methods need to be approved?	12
5.3 Basis for test method approval	13
5.3.1 What is validation?	13
5.3.2 Is validation required?	13
5.3.3 Internationally/nationally recognised methods.....	13
5.3.4 Other methods.....	14
5.3.5 Method characteristics of interest.....	15
5.3.6 Fitness for purpose.....	16
5.4 Non-approved methods.....	17
5.4.1 Customer methods	17
5.4.2 Methods used for new products.....	17
5.4.3 Modified methods or those used outside of their scope	18
5.5 In-process testing.....	18
5.6 Process for test method approval.....	18
5.6.1 Overview of process	18
5.6.2 Data required for application.....	18
5.6.3 Assessment and recommendation by accreditation body	19
5.7 Status of currently used test methods	20
5.8 Verification of test methods	21

6. Implementation	21
6.1 Mechanism for implementing.....	21
6.2 Changes required for implementation	21
6.2.1 By MAF Dairy and Plant Products.....	21
6.2.2 By registered laboratories.....	21
6.2.3 By the accreditation body	21
6.3 Proposed implementation period.....	21
7. Proposal	22
8. Draft Standard	22
9. Consultation	23
10. References	23

1. Executive Summary

MAF provides a range of assurances, including official assurances, relating to the conformance of dairy produce and products to regulatory requirements. Along with laboratory capability and sampling, test methods contribute to the quality of the data and ultimately the conformance decision.

To enable MAF to have confidence, a mechanism is required to ensure that where assurances are required, dairy laboratories testing produce/product use only appropriate test methods. Appropriateness of a test method depends on the method's characteristics and the conformance limits and assurances.

This discussion paper considers the background, legislation, importing country requirements and issues relating to test methods used for verifying conformance of dairy produce and products to regulatory requirements and official assurances.

This paper proposes that:

- A MAF Standard relating to the approval of test methods (D301.1) be drafted and provided for industry consultation.
- For testing dairy produce or products' conformance with the *Dairy Industry Act 1952*, *Dairy Industry Regulations 1990*, MAF Standards, or official assurances provided by MAF, either in the form of memoranda of understanding/ agreements with other countries or export certificates this Standard requires that:
 - MAF only accept test results from tests conducted in MAF-registered laboratories;
 - MAF-approval is required for the test method used;
 - test methods from international standards, methods published in reputable international texts or national or regional standards, be approved by MAF provided they are used within their scope and are unmodified;
 - other methods be approved by MAF provided selected method characteristics are determined and the method is demonstrated to be fit for purpose;
 - where a test method is not approved, a MAF-approved method be run alongside the unapproved method.
- This Standard outlines the process for approval of test methods.
- A database of MAF-approved test methods be maintained and a list of approved methods be published on the MAF website.
- Assuming that the Standard is issued on or about 31 January 2002, it come into force 18 months later.

2. Purpose

This discussion document provides background information, discusses issues and makes recommendations relating to the requirements of test methods used by MAF-registered dairy laboratories for analysing testing dairy produce and dairy products.

3. Risk Assessment

A risk assessment is not required for this Standard.

4. Background

4.1 HISTORY

4.1.1 Before 1990

Under the *Dairy Industry Regulations 1977* MAF Dairy Division operated laboratories and also permitted the operation of agency certified laboratories in dairy companies. MAF Dairy Division published a series of Dairy Division Manuals (DDMs) containing the standard methods for sampling and chemical, physical microbiological and sensory analysis of dairy produce. These methods were mandatory for all Dairy Division and agency certified laboratories including process control laboratories. These manuals were later renamed the MQD manuals.

4.1.2 *Dairy Industry Regulations 1990*

In 1990 the *Dairy Industry Regulations 1990* were introduced (see below) and the DDM/MQD methods previously controlled by MAF were transferred to the industry and became the NZTM test methods (see below).

“Dairy Products Safe and True” Part A (section 2.6) required that:

“Laboratories that carry out testing for product safety or product certification purposes must be registered by MAF Reg.

Registered laboratories must carry out their testing properly and competently to ensure reliable test results.

Laboratories are registered for specified tests, test methods and products, and the name of the person who takes responsibility for the test results is listed.”

Further “Dairy Products Safe and True” Part A (section 6.6) listed the following approved methods of sampling and testing:

- “NZCP 13, “Development and Validation of Laboratory Methods Code of Practice”
- NZCP 14, “Salmonella and Listeria Testing Code of Practice”
- NZTM 2, “Microbiological Methods Manual”
- NZTM 3, “Chemical Methods Manual, Issue 2”
- NZTM 4, “Physical Methods Manual”
- NZTM 5, “Functional Methods Manual”
- NZTM 6, “Sensory Methods Manual”

Sampling and testing must be carried out according to these standards, as appropriate, or alternative standards must be offered for approval.”

4.1.2.1 NZCP13 Development and Validation of Laboratory Methods Code of Practice

Soon after the New Zealand Dairy Board became responsible for the NZTM methods, it recognised that method validation was critical. As a consequence, a code of practice NZCP13, “Development and Validation of Laboratory Methods Code of Practice” was developed and published in 1993. This was approved by MAF (Circular 13).

This manual provided an overview of the requirements of method validation. However it proved to be of limited use to dairy laboratory staff and other people involved with test method development and validation as it did not provide details of how methods were developed and/or validated.

Several attempts were made to expand NZCP13 to include this information. Recently the next version of NZCP13, including this “how to” information was finished. MAF’s approval for this code has not yet been applied for. This is possibly because the MAF Standard for method approval (D301) is still being developed and would when issued provide the outcomes against which NZCP13 would be assessed.

4.1.2.2 NZTM Test Method Manuals

In 1990 MAF provided the Dairy Division manuals, including the MQD/DDM Standard Methods Manuals to the New Zealand Dairy Board to manage on behalf of the New Zealand dairy industry. The MQD/DDM Standard Methods became the basis of the NZTM methods manuals.

In 1993, NZTM 2, “New Zealand Dairy Industry Microbiological Methods Manual” and NZTM 3, “New Zealand Dairy Industry Chemical Methods Manual” were published and approved by MAF (Circular 17) until 31 July 1994. The methods published were based on the MAF manuals, DDM 3, “Microbiology” and DDM 4, “Chemistry”, with improvements to bring the methodology up to date, to align where possible the methods with internationally-accepted ones, and to include available performance data.

In 1994, a dairy industry Working Group and consultants compiled NZTM 4, “New Zealand Dairy Industry Physical Methods Manual”, a manual of physical testing methods for milk and dairy products. The manual was published and approved by MAF until 31 May 1995. (Circular 19). The methods were based on the previously approved methods in DDM 4,

“Chemistry”, and included liquid milk methods previously held in MQD 10, “Standard Procedures for Milk Appraisal” and MQD 12, “Market Milk Code of Practice”. As with NZTM 2 and 3, improvements had been made to bring the methodology up to date, to align with internationally-accepted methods and to include available performance data.

Similarly, later that year, NZTM 5, “New Zealand Dairy Industry Functional Methods Manual”, and NZTM 6, “New Zealand Dairy Industry Sensory Methods Manual” were published and approved by MAF (Circular 20). Note that this approval related to truth of labelling and MAF export product certification and not to product safety.

When Issue 2 of NZTM 3, “New Zealand Dairy Industry Chemical Methods Manual”, was published it was approved by MAF without conditions, with a MAF approval statement appearing in the manual. This approval meant that separate approvals were not required for amendments to existing methods and new methods provided they were in accordance with the procedures laid down in the approved code NZCP 13, “Development and Validation of Laboratory Methods Code of Practice”. Once again, this approval was restricted to truth of labelling and MAF export product certification as well as product safety.

Similarly in July 1995, Issue 2 of NZTM 2, “New Zealand Dairy Industry Microbiological Methods Manual” was given the same unconditional approval as NZTM 3 (Circular 25).

From 1995 to 1998 the NZTM Working Groups continued to develop amendments to methods and add methods to the NZTM test methods manuals. MAF was represented on these Working Groups and the acceptance process required the MAF representative to accept the methods before they were issued. This became, in effect, the MAF-approval step. It is not clear whether the methods were validated in accordance with NZCP 13 as the unconditional approval had required.

In the 1996/97 season the New Zealand Dairy Board consolidated all its “customer methods” that had previously been uncontrolled and published these as restricted circulation methods in the relevant NZTM test method manual. Access to a restricted circulation method was by application. These methods were of variable quality and in some instances there was doubt about their fitness for purpose. Unfortunately because of the unconditional MAF approval of the NZTM manuals, some incorrectly perceived that these restricted circulation methods were approved by MAF.

In 1998 MAF requested that the New Zealand Dairy Board remove the generic MAF approval statements from the NZTM test methods manuals. This was done when the manuals were next issued. At a similar time, MAF’s involvement in the NZTM Working Groups ceased.

The NZTM test methods manuals are still maintained and published by the New Zealand Dairy Board on the Board’s intranet. Paper versions of the manuals are now obsolete. Access to the Board’s intranet and the NZTM methods is restricted. Laboratories outside the export aligned co-operative dairy industry can apply to the Dairy Board to obtain access to these methods.

4.1.2.3 Approval of other methods

It appears that MAF did not approve any test methods other than the NZTM methods in the period from 1990 to 1998. Only one test method has been approved by MAF since 1998.

4.1.3 Development of the MAF Standard D301

To have confidence in the data used and provided for the purposes of assurances given by MAF relating to dairy produce and products, MAF reconfirmed in 1999 that control of the analytical methodology used for testing dairy produce and products was required.

Consequently MAF prepared Discussion Paper number 16, “MAF-Approved Test Methods” which included a draft Standard, MAF Standard D301, “MAF-Approved Dairy Test Methods”. Industry consultation was undertaken and based on the feedback received from industry MAF advised the Dairy Products Safety Advisory Council (DPSAC) that the draft Standard would not be progressed until there could be a better clearer definition of which methods would require approval and what validation would entail.

Further limited development of the draft Standard occurred in 2000 and after limited industry consultation in May 2000, MAF recommended to DPSAC that a DPSAC Working Group

“be established to resolve the issues relating to the approval of test methods by MAF and to reach agreement on the draft Standard prior to further industry consultation, DPSAC endorsement and promulgation.”

The DPSAC Working Group was formed in March 2001. This discussion paper has been developed by the Working Group at the same time as it drafted the Acceptable Criteria for the draft MAF Standard D301, “MAF Approved Test Methods” (see below).

4.2 CURRENT STATUS

A number of MAF Standards (D101, D108, D109, D115, D206) require the use of MAF-approved test methods. They reference the list of MAF-approved methods that will be available on the MAF website.

While international reference methods and methods specified by importing countries, such as the methods defined in the US FDA Memorandum of Understanding (MoU), are assumed to be approved, this information is not readily accessible to the industry. Related to this, a mechanism is required to enable MAF to approach importing countries to persuade them to accept the results of equivalent methods.

To assist the DPSAC Working Group (refer above), MAF has recently prepared a comprehensive document listing all the assurances given by MAF which would require MAF-approval of the test method used to verify conformance. These are assurances given either implicitly or explicitly via legislation, MAF Standards and official assurances.

Technically, the NZTM methods approved by MAF before 1995 or accepted by the MAF representative on the NZTM Working Groups are still MAF-approved provided they have not been modified and are used within their scope. Where these “approved” NZTM methods are not international reference methods, the methods’ characteristics and fitness for purpose may not be documented.

A number of MAF Circulars (20, 22, 25) providing unconditional approval of the NZTM test methods, although now obsolete, have not been formally revoked.

MAF registers dairy laboratories in accordance with regulations 25 to 35, 6(a) and 9(k) of the *Dairy Industry Regulations 1990*. MAF Standard D302.1, “Registration of Dairy Laboratories” outlines the acceptable criteria for laboratory registration. To be registered, the laboratory must be accredited/recognised by an accreditation/recognition body under the “Dairy Testing Programme”. This programme requires that for each laboratory, the accreditation/recognition body issues a certificate accompanied by a schedule detailing the tests, test methods and products for which they are accredited/recognised.

4.3 NEW ZEALAND’S LEGAL REQUIREMENTS

The Dairy Industry Act 1952, section 15(b), requires truth of labelling.

The Dairy Industry Regulations 1990, regulation 25, requires that tests conducted as part of a Product Safety Programme (PSP) or required by the *Dairy Industry Act 1952* (other than those carried out on raw milk or cream for payment purposes) be carried out in a registered laboratory.

Regulation 27 states that registered laboratories must have the equipment and procedures necessary to ensure that all testing carried out there for the purposes of these regulations will be carried out properly and competently.

In summary, the legislation requires that tests to establish truth of labelling and product safety must be carried out properly and competently.

Registered laboratories must meet the requirements of ISO Standard 17025, General Requirements for the Competence of Testing and Calibration Laboratories” (see below) or MRD-Stan 5, “General Requirements for the Competence of Category 2 Laboratories” and MAF Standard D302.1, ‘Registration of Dairy Laboratories’.

MRD-Stan 5, section 10, requires the laboratory to use appropriate methods and procedures. It says, “Where methods are not specified, the laboratory shall, wherever possible, select methods that have been published in international or national standards, those published by reputable technical organisations or in relevant scientific texts or journals.”

4.4 INTERNATIONAL STANDARDS AND GUIDELINES,

4.4.1 Codex Alimentarius

The Codex Draft Guidelines, “Design, Operation, Assessment and Accreditation of Food Import and Export Inspection and Certification Systems”, adopted in 1997, clauses 41 and 42 recommend that:

- validated analytical methods be used wherever possible; and
- laboratories apply the principles of internationally accepted quality control techniques to ensure the reliability of the analytical results.

4.4.2 ISO Standard 17025

ISO Standard 17025, section 5.4, requires the use of methods “which meet the needs of the client and which are appropriate, preferably those published as international, regional or national standards. Validated methods developed or adopted by the laboratory may also be used if they are appropriate for the intended use.”

4.5 IMPORTING COUNTRY REQUIREMENTS

4.5.1 European Union

4.5.1.1 EU/NZ Veterinary Agreement

Under the EU/NZ Veterinary Agreement 1996, milk products being exported to the EU must be certified by MAF as complying with the requirements of Directive 92/46 Annex C Chapter II.

Under the Agreement, the New Zealand regulatory system for laboratories has been judged as being equivalent to, and meeting, EU market access requirements.

As the Agreement does not provide equivalence for the existing raw milk national residue programme, Directive 96/23 applies for residue monitoring.

4.5.1.2 Milk Directive 92/46

The milk and milk products directive outlines test method requirements for milk and milk products. Article 20 provides for the establishment of reference methods and the criteria governing routine methods of analysis. Pending these decisions “any internationally accepted analysis and test methods shall be recognised as reference methods”.

Article 15 requires that examinations for residues must be carried out in accordance with proven methods which are scientifically recognised, and in particular those laid down at Community or international level. 96/23 (see below) contains an amendment to 92/46 Article 15 requiring residues to be monitored in accordance with 96/23.

4.5.1.3 EU Residue Directive 96/23

Article 15 of 96/23 provides for the establishment of detailed rules for the taking of official samples and the routine and reference methods to be employed for the analysis of official samples. The EU is currently preparing legislation setting out the performance criteria for the analytical methods used for 96/23.

4.5.1.4 Other EU legislation

In addition to the above, the EU has two pieces of legislation covering methods of analysis for raw milk and heat treated milk. These are Decisions 91/180 and 92/608 and include chemical, physical and microbiological methods for raw milk and milk products.

4.5.2 USA

The Memorandum of Understanding between MAF and the US FDA specifies the test methods to be used. From time to time the FDA approves alternative methods, either by application or recognition in the FDA “Bacteriological Analytical Manual for Foods” (BAM).

4.5.3 Other countries

Other importing countries may specify the test methods to be used. An assessment of these has been undertaken. Sri Lanka appears to be the only country other than the EU and USA that prescribes the test method to be used as the basis for assurances given by MAF.

5. Issues

5.1 WHY APPROVE TEST METHODS?

As described above, there is a history of MAF-approval of test methods. The Working Group considered whether test methods needed to be approved, and if so specifically by MAF.

5.1.1 Role of test methods in product conformance decisions

The performance of a test method does play a part in the quality of decisions regarding product conformance. The Working Group recognised that there are a number of other factors that are as least as important as the test method in the quality of these product conformance decisions. These factors include sampling error [process variation], the laboratory’s competence as well as the way the data is used to decide if produce/product is conforming.

The Working Group recognised that these other factors were managed by other MAF Standards such as D302.1, “Registration of Dairy Laboratories” and D109.1, “Dairy Product Conformance”.

5.1.2 What does test method approval provide?

The Working Group considered carefully what the MAF approval of a test method would and would not provide.

The approval of a method **would not provide** “correct” results as all results from any test method are subject to variation and uncertainty.

Method approval by itself **would not provide** total certainty of product conformance. As described above, product conformance is dependant on a number of factors, one of which is the test methodology. Even if a test method were exact, assessments of conformance are subject to error from other sources.

The Working Group agreed that the method approval by MAF **would provide** some level of confidence that the method would be appropriate for a specific purpose, provided the method was assessed and approved in the context of the use of the method and decisions made using the method’s results. A method’s appropriateness can only be judged when the purpose for which it is used is known.

The Working Group agreed that the method approval by MAF **would provide** some consistency and control in the way test methodology is used.

The Working Group also agreed that the method approval **would provide** MAF with some level of confidence in assessing produce/product conformance against a specific specification provided other factors, such as sampling plans, decision rules, laboratory accreditation and participation in interlaboratory comparison programmes (ILCPs), were also managed.

By comparison the absence of method approval could result in the use of methods that are flawed or not appropriate for a specific purpose. Consequently this could lead to incorrect product conformance decisions. By way of an example, the use of a microbiological test for pathogen detection that has a high level of false negatives could lead to unsafe product being sold and exported, simply because contamination was not detected.

MAF's credibility would be jeopardised if its assurances were found to be flawed and a matter of dispute. In situations of dispute, internationally recognised reference methods and methods of demonstrated and documented performance become the basis for deliberation and judgement.

On consideration, it was recognised that MAF requires confidence in the test methods being used for analysis, just as it requires confidence in product sampling, the laboratories doing the testing and the decisions being made using the data. Therefore it was agreed that it was appropriate for MAF to approve the methods on which its assurances were based. The Working Group recommended that MAF's role in approving methods continue.

5.2 WHICH TEST METHODS NEED TO BE APPROVED?

In the past, MAF approved test methods used for truth of labelling and MAF export product certification as well as product safety. The scope and the definitions of "truth of labelling" and "product/food safety" were considered. It proved to be difficult to provide boundaries, because truth of labelling relates to standard of identity, which is often enshrined in importing country legislation. It is rare for MAF to provide assurances relating to conformance with an importing country's standard of identity. Generally MAF Dairy and Plant Products states that it is the exporter's responsibility to identify and comply with all importing country requirements; non-compliance is at their commercial risk.

This scoping problem was then considered in the framework of the assurances MAF provides, either implicitly in the administration of the *Dairy Industry Act 1952* and *Dairy Industry Regulations 1990* or explicitly in the form of official assurances. This confirmed that test methods used as a basis for these assurances were the ones requiring approval by MAF, as MAF's credibility was at stake in these situations.

Based on this, MAF has prepared a comprehensive list of parameters/attributes requiring a MAF-approved test method. This list is available from MAF. In preparing this list, it was recognised that the list will continually evolve and have frequent updates, simply because regulatory requirement and the content of official assurances changes.

The administrators of the NZTM test methods manuals are currently using the list to assess which of the NZTM methods require MAF approval.

5.3 BASIS FOR TEST METHOD APPROVAL

One area of greatest concern to the dairy industry is the basis for method approval. In the previous proposal (Discussion Paper No. 16), method validation was proposed, but this was seen as an excessively high hurdle as well as being of an unknown quantity, as there is no single authoritative text on test method validation.

5.3.1 What is validation?

As mentioned earlier, validation is perceived to be “an unknown quantity” as there is no single authoritative text on test method validation. In some texts, validation measures some or all of a method’s characteristics, e.g. reproducibility. In others, validation also includes a step to confirm that the method is appropriate, i.e. fit for purpose. These differing interpretations create problems in communicating requirements for method approval.

To prevent confusion the Working Group specifically defined validation as:

“the process of establishing the performance characteristics and limitations of a test method. This includes the identification of factors that may affect these characteristics as well as the extent the method can determine the analyte in a range of matrices and the effect of interferences.”

To further clarify the requirements for test method approval, the Working Group has drafted the Standard to:

- specifically state which method characteristics are required; and
- describe how fitness for purpose is assessed.

5.3.2 Is validation required?

The Working Group agreed that validation (full characterisation) of methods including limitations, is not required for MAF approval of a method.

Guidance was taken from ISO Standard 17025 (see above). As a consequence two means are proposed to ensure that the method is appropriate.

Firstly, the Working Group proposed that MAF would approve international and national methods (refer section 5.3.3 below) unless there is some specific reason why it shouldn’t.

Secondly, to ensure MAF’s approval, it would need to be demonstrated that all other methods are appropriate for the use for which they are approved (fit for purpose) (refer section 5.3.4 below).

5.3.3 Internationally/nationally recognised methods

It was agreed that methods from the following sources be generally approved by MAF provided that they were used within their scope and unmodified:

- international standards, e.g. ISO, IDF, Codex; or
- methods published in reputable international texts, e.g. standard methods published by the American Public Health Association, AOAC Official Methods of Analysis, Pearsons; or

- national or regional standards or legislation, e.g. New Zealand Standards, Australian Standards, British Standards, Euronorm Standards, USA FDA “Bacteriological Analytical Manual for Foods” (BAM), EU legislation, etc.

When these methods are used outside of their scope or are modified, fitness for purpose must be demonstrated prior to approval.

It was recognised that in some instances methods from some of these sources may be outdated or have specific problems, so MAF need provision to decline approving such methods.

5.3.4 Other methods

5.3.4.1 Options considered

It was also recognised that there are a number of situations where methods other than international/national methods would require MAF approval. A number of options were considered as the basis for demonstrating that these other methods were appropriate. These options included:

- full characterisation;
- partial characterisation;
- demonstration of equivalence with reference method; and
- demonstration of fitness for purpose.

Full characterisation is the process of defining all of a method’s characteristics, i.e. the method’s performance. This is sometimes referred to as “validation” (see above). While full characterisation is valuable, it faces two difficulties. Firstly and most importantly a fully characterised method may still be used in applications where it is not fit for that purpose. It is important that a method be assessed in terms of its use, which includes the level of the parameter/ attribute in the produce and its variability, as well as the specification and criteria being used in the decision of conformance. Secondly consideration must be given to the time resource required to carry out a full characterisation.

Partial characterisation is the process of defining a few selected method characteristics. This has the advantage of providing some information on the method’s performance but also faces the problem that it does not resolve the problem of fitness for purpose.

Demonstration of equivalence with a reference method is the process of comparing a method’s characteristics with those of the reference method. Where the method’s performance is the same or better than those of the reference method, then the method is judged to be fit for purpose. This approach is used by a number of regulatory bodies throughout the world. This approach is based on the assumption that the reference method is fit for purpose. Therefore because the other method has no bias and the precision, limit of detection and range are the same or better than the reference method, it also is fit for purpose.

Fitness for purpose of test methods is the suitability of a test method for a particular application. Demonstration of fitness for purpose is the process of assessing a method in the context of the product, specification, and decision criteria, being used. This process requires:

- a knowledge of the method's characteristics;
- the level and variability of the attribute in the product;
- the confidence associated with sampling;
- the specification limits; and
- the rules being used for making the conformance decision which include the level of risk that the decision maker is prepared to accept that the product does not actually conform with the specification.

5.3.4.2 Approach agreed

Based on these four options the Working Group resolved that approval of "other methods" is based on a two step approach. The first step is to partially characterise the method (see section 5.3.5 below for further details). The second step is to assess the method's fitness for purpose using either demonstration of equivalence with the reference method or defining the effect of method performance on conformance assessment (see section 5.3.6 below for further details).

5.3.5 Method characteristics of interest

Test methods produce three different types of results: continuous, ordinal and nominal. MAF is specifically interested in continuous and nominal methods.

Continuous methods produce results which are expressed as numbers. Examples of continuous methods are butter moisture (example of result: 15.5% moisture), and aerobic plate count (example of result: 150 colony forming units per ml).

By comparison *nominal methods* only report the presence or absence of something. Examples of nominal methods are *Salmonella* detection (example of result: not detected per ml) and leakage of UHT containers (example of result: container leaks).

5.3.5.1 Method performance characteristics required for continuous methods

The following characteristics would be required for continuous methods:

- bias;
- precision (reproducibility or intermediate precision);
- limit of detection; and
- range.

Bias is the average difference between the test results and the accepted reference value. Bias is the total systematic error rather than random error, which is described by "precision".

Precision is an assessment of the closeness of agreement between independent test results from the same sample obtained under specified conditions. MAF is interested in a method's *reproducibility* (R) which is the precision of a method where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. In some instances there may be insufficient laboratories available to estimate the reproducibility with sufficient confidence. In this situation, intermediate precision can be provided. *Intermediate precision* is a measure of method precision due to changes in one or more of time, calibration, equipment, and operator in a single laboratory. Intermediate precision lies between the two extreme measures of precision repeatability and reproducibility.

The *limit of detection* is the concentration of analyte that leads to the conclusion, with a given probability of error, that the sample concentration exceeds the concentration in a blank.

The *range* is the concentrations of analyte lying between the limit of detection and the limit of linearity within which the method demonstrates a satisfactory relationship with the reference method.

5.3.5.2 Method characteristics required for nominal methods

The following characteristics would be required for nominal methods:

- specificity rate;
- sensitivity rate; and
- limit of detection.

The *sensitivity rate* (the true-positive detection rate) is the probability that the method will classify a test sample as positive, given that the sample is a “known” positive.

The *specificity rate* (the true-negative detection rate) is the probability that the method will classify a test sample as negative, given that the sample is a “known” negative.

The *limit of detection* is the concentration of analyte that leads to the conclusion, with a given probability of error, that the sample concentration exceeds the concentration in a blank.

5.3.6 Fitness for purpose

The Working Group agreed that it was critical to clarify the concept of a test method's “fitness for purpose”. A test method's fitness for purpose has been defined as:

“the suitability of a test method for a particular application. Fitness for purpose examines the effect of the test method's performance on assessments made using results generated by that method, e.g. in a sampling plan to determine whether a lot of product conforms.”

Fitness for purpose of a method is not a matter of whether decisions made are correct or incorrect; rather it is whether the increased risks, incurred due to the performance associated with the method, are acceptable. Fitness for purpose also depends on how the test results are used to make the decision and unacceptable risks that could occur if results are used in an inappropriate manner.

This assessment can be carried out in two different ways:

1. *Equivalence with a reference method.* If the method characteristics are equal to or better than the characteristics from the reference method for that particular parameter, then the method would be considered fit for purpose.
2. *Effect of method performance on conformance assessment.* If no reference method exists, then the method's fitness for purpose would be assessed as follows. The effect of selected method characteristics are assessed on the decisions made using the results generated by that method, e.g. in a sampling plan used to determine whether a lot is acceptable. One method may be fit for purpose in some situations but not fit in others. This becomes particularly important where methods with high variability are used to judge a lot's conformance to a specification with a narrow gap between the upper and lower specification limits or where the typical product analysis is "close" to the specification limit.

It is proposed that for these methods, the method's performance would be used to define some limits for the method and that these limits would be specified by MAF when they approve the method. The limits suggested are:

- For continuous methods; the precision is used to calculate a "buffer limit" for the test method which, when not exceeded will ensure that no more than 5 percent of the produce/product lies outside the specification for conformance. This buffer limit would be used by a laboratory to decide if the method is suitable for assessing conformance of lots of produce/products. In situations where the produce/product mean and standard deviation are in the "buffer zone", the produce/product is non-conforming. There are two options for corrective action, either another method (with better precision) is used or the process altered so the produce/product mean and standard deviation shift outside the buffer zone, i.e. the process altered so the produce/product becomes conforming.
- For nominal methods, the specificity and sensitivity rates are used to calculate the minimum number of samples required to be tested by the method to ensure that no more than 10 percent of the produce/product lies outside the specification for conformance.

5.4 NON-APPROVED METHODS

5.4.1 Customer methods

Only results from MAF-approved test methods will be used to verify conformance with regulatory requirements or official assurances provided by MAF. The Working Group proposed that where it is necessary to use methods that have not been approved, e.g. for a customer specification, a MAF-approved test method be run in parallel with the non-approved method.

5.4.2 Methods used for new products

Occasionally the industry needs to carry out an analysis for which there is no internationally/nationally recognised method, e.g. on a new product. In this case the method would require characterisation and demonstration of fitness for purpose.

5.4.3 Modified methods or those used outside of their scope

A modification is any change to a test method that changes the method's characteristics and/or fitness for purpose. If a method is modified or used outside the scope, characterisation and demonstration of fitness for purpose will be required before MAF will approve the method.

The Working Group proposed that in the absence of this approval, where modified methods are used or methods used outside their designated scope, a MAF-approved test method is run in parallel with the method.

5.5 IN-PROCESS TESTING

If results from in-process testing are being used to justify rationalised testing of finished product, then logic shows that this in-process testing is being used to verify conformance with regulatory requirements or official assurances provided by MAF. In this situation, in-process laboratories must be either an independently registered laboratory or registered as part of a Category 1 Laboratory (refer MAF Standard D302.1, "Registration of Dairy Laboratories"). Similarly the methods used by the laboratory for in-process testing must also be MAF-approved.

5.6 PROCESS FOR TEST METHOD APPROVAL

5.6.1 Overview of process

The Working Group considered and agreed the process by which a method would be approved. This proposed process is as follows:

- It is identified that a method requires approval.
- The data on the method is collected.
- An application is prepared.
- The application is provided to the accreditation body.
- The accreditation body assesses the method based on the data provided in the application.
- The accreditation body recommends to MAF whether the method be approved or not. In some instances the accreditation body may recommend approval of a method with conditions.
- MAF considers the application and recommendation and if satisfied approves the method and advises the applicant.
- MAF maintains a register of all approved methods and reviews approved methods every 5 years or more frequently if required.
- MAF provides the list of approved methods on the MAF website.

5.6.2 Data required for application

The Working Group agreed that the Standard would provide guidance in the information required in the application for approval.

5.6.2.1 International/national methods

It is proposed that the following information would be required for international/national methods:

- method title;
- reference (source of the method);
- copy of the method;

And from the MAF list of parameters/attributes requiring a MAF-approved test method, (refer section 5.2 above):

- the source, e.g. MAF Standard D107, “Dairy Product Safety”;
- the assurance, e.g. “The level of micro-organisms in a dairy product at the end of processing does not exceed the limits specified in Table A1.1 for that micro-organism in that product.”;
- the parameter/attribute, e.g. *E. coli*;
- specification, e.g. 100/g;
- scope (MAF classes or descriptions), e.g. Soft and semi soft cheese, firm and hard cheese, processed cheese, cottage cheese, cream cheese.

5.6.2.2 Other methods

For other methods, the information required for international/national methods (refer above) would be required as well as:

- reference of the standard or code used to demonstrate fitness for purpose;
- method type, e.g. continuous, nominal;
- the method characteristics (see section 5.3.5 above for the list of characteristics required);
- where equivalence with the reference methods is being sought, the characteristics of the reference method;
- the fitness for purpose assessment, either
 - assessment of equivalence with the reference method; or
 - the limits of the method for assessing produce/product conformance (for the effect of the method performance on conformance assessment),
- a report summarising the findings;
- copies of any other method(s) used in the report;
- all base data, i.e. test results etc;
- statistical analysis and calculations; and
- any other documentation necessary to support the application.

5.6.3 Assessment and recommendation by accreditation body

The accreditation body will receive applications for test method approval accompanied by the supporting information listed above. The accreditation body will assess the application for test method approval and recommend approval, approval with conditions or otherwise to MAF.

The Working Group agreed that to consistently assess these proposals, there needs to be a set of criteria by which the test method and the supporting information is judged to be satisfactory. The Working Group has undertaken some preliminary development of these criteria as follows:

- All required information is provided.
- All references (source material), support the application.
- For international/national methods:
 - the method provided is the same as the published method;
 - the method is adequately documented;
 - the method is current (not obsolete);
 - the method is based on sound scientific principles; and
 - there are no reports that suggest this method should not be approved. Where reports exist, there is sound scientific data that alleviates the reported concerns.
- For “other” methods:
 - the method provided is the same as the published method;
 - the method is adequately documented;
 - the method characteristics are determined correctly (this will require checking of the design, the raw data and the calculations of the characteristics);
 - for assessment of fitness for purpose:
 - + for equivalence with a reference method, the method has no bias and the other characteristics are better than or equal to the reference method characteristics and the method is appropriate for use in MAF-registered dairy laboratories.
 - + for the effect of the method performance on conformance assessment the limits of the method for assessing produce/product conformance are correctly calculated and the method is appropriate for use in MAF-registered dairy laboratories.

The Working Group also recommends that the accreditation body fully document the process for assessing test methods and the criteria used.

On completion of the assessment the accreditation body provides application, the assessment data and its recommendations, including conditions or restrictions on method use, to MAF.

5.7 STATUS OF CURRENTLY USED TEST METHODS

For those methods that fall within the scope of the MAF-approved test methods standard, it is likely that some of the methods currently used by the dairy industry will not be international/national methods generally approved by MAF. The NZTM methods are often unvalidated modifications of international methods. In addition, there are a number of customer specified tests that usually have no validation pedigree and are in some instances technically unsound. Thus these methods are usually not seen as accreditable tests by the accreditation body. In order for MAF to approve these NZTM methods, fitness for purpose must be demonstrated.

5.8 VERIFICATION OF TEST METHODS

Verification of a test method is the ongoing process of confirming that the laboratory is performing the test competently. Verification is one of the internationally accepted quality control techniques recommended by Codex. Verification is considered an internal laboratory function, which will be audited at a regular frequency by the accreditation body. For this reason, it is proposed that the verification of test methods not be included as part of the standard.

6. Implementation

6.1 MECHANISM FOR IMPLEMENTING

The changes proposed above would be implemented by preparing MAF D-series Standard for MAF-approval of dairy test methods. The Standard would outline the requirements for the proper and competent testing and acceptable criteria for the approval of test methods by MAF and their use by MAF-registered dairy laboratories.

6.2 CHANGES REQUIRED FOR IMPLEMENTATION

6.2.1 By MAF Dairy and Plant Products

- establishing business systems for processing recommendations from the accreditation body;
- providing resource to process applications and add approved test methods to the list published on the website;
- establishing business systems for maintaining and reviewing the list of approved test methods.

6.2.2 By registered laboratories

Fill out the form for test method approval attached to the Standard and approach the accreditation body. Where the test method does not fit the category of generally approved methods, a fitness for purpose exercise needs to be carried out.

6.2.3 By the accreditation body

Establish systems for processing applications for test method approval. These systems would include criteria by which a test method is judged to have satisfactorily demonstrated fitness for purpose.

6.3 PROPOSED IMPLEMENTATION PERIOD

Given the amount of work that will need to be completed in order to have some test methods approved, an 18-month implementation period is proposed for this Standard. Assuming that the Standard is issued on or about the 31 January 2002, then the Standard would come into force on 31 May 2003.

Any non-compliances after this period will be managed in a systematic manner using the existing procedures for the management of non-compliances.

It is envisaged that in some instances MAF may approve methods with restrictions. This restricted approval will restrict the use of the method and the data generated as well as setting an expiry date. The test method concerned would either be fully approved or withdrawn from use by the expiry date.

7. Proposal

With regards to the approval of test methods by MAF, the following is proposed. That:

- A MAF Standard relating to the approval of test methods (D301.1) be drafted and provided for industry consultation.
- For testing dairy produce or products' conformance with the *Dairy Industry Act 1952*, *Dairy Industry Regulations 1990*, MAF Standards, or official assurances provided by MAF, either in the form of memoranda of understanding/ agreements with other countries or export certificates this Standard requires that:
 - MAF only accept test results from tests conducted in MAF-registered laboratories;
 - MAF-approval is required for the test method used;
 - test methods from international standards, methods published in reputable international texts or national or regional standards, be approved by MAF provided they are used within their scope and are unmodified;
 - other methods be approved by MAF provided selected method characteristics are determined and the method is demonstrated to be fit for purpose;
 - where a test method is not approved, a MAF-approved method be run alongside the unapproved method.
- This Standard outlines the process for approval of test methods.
- A database of MAF-approved test methods be maintained and a list of approved methods be published on the MAF website.
- Assuming that the Standard is issued on or about 31 January 2002, that it come into force 18 months later.

8. Draft Standard

Draft MAF Standard D301.1, "MAF-approved Dairy Test Methods", has been drafted based on the findings and recommendations of this discussion document. Copies of the draft Standard can be obtained from MAF's website www.maf.govt.nz or by contacting MAF Food.; Dairy and Plant Products.

9. Consultation

The draft Standard is of concern to all MAF-registered laboratories and their accreditation/recognition bodies in the New Zealand dairy industry. Internal and external consultation will be conducted according to the MAF Food: Dairy & Plants Consultation Policy. The deadline for submissions is **2 November 2001**. Instructions for making submissions are provided with the draft Standard.

10. References

- The *Dairy Industry Act 1952*
- The *Dairy Industry Regulations 1990*
- MAF Circular number 13, "Development of Validation of Laboratory Methods Code of Practice"
- MAF Circular number 17, "Microbiological Methods and Chemical Methods"
- MAF Circular number 19, "Physical Methods Manual"
- MAF Circular number 20, "Functional Methods Manual; Sensory Methods Manual; *Salmonella* and *Listeria* Testing Code of Practice"
- MAF Circular number 22, "Chemical Methods Manual"
- MAF Circular number 25, "NZTM 2: Microbiological Methods Manual"
- MAF Discussion Paper 16, "MAF-Approved Dairy Test Methods"
- MAF Standard D101.2, "Product Safety Programmes"
- MAF Standard D108.1, "Non-conforming Dairy Produce"
- MAF Standard D109.1, "Dairy Product Conformance"
- MAF Standard D115.1, "Raw Milk Acceptance"
- MAF Standard D206.2, "Dairy Export and Related Sanitary Certification"
- MAF Standard D302.1, "Registration of Dairy Laboratories"
- MAF Standard MRD-Stan 5, "General Requirements for the Competence of Category 2 Laboratories"
- MQD 10, "Standard Procedures for Milk Appraisal"
- MQD 12, "Market Milk Code of Practice"
- Dairy Products Safe and True. MAF Food Group, 1999. World Wide Web address: <http://www.maf.govt.nz/Dairy>
- ISO Standard 17025, "General Requirements for the Competence of Testing and Calibration Laboratories"
- Alinorm 97/30A, Appendix II, "Guidelines for the Design, Operation, Assessment and Accreditation of Food Import and Export Inspection and Certification Systems," clauses 41 and 42. Codex Alimentarius Commission, 1997

- European Communities Commission Decision laying down certain methods of analysis and testing of raw milk and heat-treated milk. 91/180/EEC, 14 February 1991
- European Communities Council Decision laying down methods for the analysis and testing of heat- treated milk for direct human consumption. 92/608/EEC, 14 November 1992
- European Communities Council Directive laying down the health rules for the production and placing on the market of raw milk, heat treated milk and milk based products. 92/46/EEC, 16 June 1992
- European Communities Council Directive on measures to monitor certain substances and residues thereof in live animals and animal products and repealing Directives 85/358/EEC and 86/469/EEC and Decisions 89/187/EEC and 91/664/EEC. 96/23/EC, 29 April 1996.