



INTERNATIONAL ASSOCIATION FOR
CEREAL SCIENCE AND TECHNOLOGY

"Food **quality, safety** and **security** for the **health** and well-being of **all people**."

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ICC Standard Method Validation and Publication

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1. Introduction:

Reliable analytical methods are required for compliance with national and international regulations in all areas of analysis. ICC's primary objective is the development of internationally approved and accepted standard testing procedures for cereals and flour that serve international trade, national and international legislation, industry standards such as ISO and CEN, and as guidelines for food manufacturers and control laboratories. The testing methods may include but are not limited to qualitative analysis, quantitative analysis, screening analysis, confirmatory analysis, limit tests.

Methods may be validated for one or more measurand, one or more matrices, and one or more instruments or platforms.

2. Objectives:

The main purpose of validating a method of analysis is to determine that the method is fit for its intended purpose.

The protocol or method of analysis is the set of permanent instructions for the conduct of the method of analysis. Method validation is a distinct phase from method development/ optimisation and should be performed subsequently to method development. The method developer validates a method by conducting experiments to determine the specific performance characteristics that serve to define and quantify method performance.

The method of analysis that is finally used should be the same as the one that was studied and revised as a result of research, optimisation, and ruggedness trials.

3. **Scope:**

ICC method validation is required for:

- Submission of a new or original method.
- Expansion of the scope of an existing method to include additional measurands.
- Expansion of the scope of an existing method to include additional matrices.
- Changes in the intended use of an existing method.
- Modifications to a method that may alter its performance specifications (e.g., changes to the fundamental science of an existing method, significant changes to reagents, apparatus, instrumental parameters, sample preparation and/or extraction, or modification of a method's range beyond validated levels.)

4. **Procedural guidance for method validation:**

4.1 Reference standard method and standard method:

A reference method is a method by which the performance of an alternate or new method (potential standard method) may be measured or evaluated for a specific measurand.

4.2 Existing protocols, standards, and guides:

A number of protocols and guidelines on method validation and measurement uncertainty have been prepared through International harmonisation projects and can be used as reference:

- Eurachem “the fitness for purpose of analytical methods: a laboratory guide to method validation & related topics, second edition 2014.
- IUPAC “Harmonised guidelines for single laboratory validation of methods of analysis. Adopted by Codex alimentarius (*CAC/GL 49-2003*)
- IUPAC “Protocol for the design, conduct and interpretation of method-performance studies”.
- ISO 5725-1:1994. Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions (and Corrigendum 1:1998).
- ISO 5725-2:1994. Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method (and Technical Corrigendum 1:2002).
- ISO 5725-3:1994. Accuracy (trueness and precision) of measurement methods and results – Part 3: Intermediate measures of the precision of a standard measurement method (and Corrigendum 1:2001).
- ISO 5725-4:1994. Accuracy (trueness and precision) of measurement methods and results – Part 4: Basic methods for the determination of the trueness of a standard measurement method.
- ISO 5725-5:1998. Accuracy (trueness and precision) of measurement methods and results –

Part 5: Alternative methods for the determination of the precision of a standard measurement method (and Corrigendum 1: 2005).

- ISO 5725-6:1994. Accuracy (trueness and precision) of measurement methods and results –

Part 6: Use in practice of accuracy values (and Corrigendum 1: 2001).

- AOAC Official Methods of Analysis (2002), Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis.

- AOAC Official Methods of Analysis (2012), Appendix F: guidelines for standard method performance requirements.

-FAO/IAEA Food and Nutrition paper 68: validation of analytical methods for food control, 1997.

ICC guidelines “validation and publication of standard methods” brings together the essential scientific principles of the above documents to provide information acknowledged internationally and, more importantly, to point the way forward for best practice in method validation.

4.3 Documentation of validated methods (required method format, precision and performance data):

In principle ICC validation studies / ring trials follow the requirements of the IUPAC / AOAC / ISO international harmonised protocol for collaborative trials (W. Horwitz, Pure and Applied Chemistry, 67 (1995): 331-343). However, the ICC protocol also accounts for several issues that have been discussed at Codex Alimentarius level more recently (e.g. recovery, recovery correction, measurement uncertainty, etc.) ISO Guide 35:2006 gives statistical principles to assist in the understanding and development of valid methods to assign values to properties of a reference material, including the evaluation of their associated uncertainty, and establish their metrological traceability.

The requirements for any ICC validation study are given below and may serve as guideline for organisers and evaluators of ring trials within the ICC standardisation program.

4.3.1 Requirements of the validation study

-The submitting organisation / institution and the coordinating institution / laboratory must be an ICC Member.

-A minimum of 8 useful results from min. 8 laboratories, respectively (after elimination of outliers and non-compliant laboratories). ICC recommends including at least 12 laboratories and blind duplicate samples

-Participating laboratories from ICC Member Countries will be preferentially selected, however trial participants from non-member countries may be invited upon the request of the organiser.

-To be counted as an international ring trial, a minimum of 3 countries needs to be involved.

-Regionally used methods may have their justified place in the ICC Standards Collection, however, ring trials not meeting the international scope need to highlight this limitation in the scope.

- No more than a maximum of 50% of the participating laboratories may show an organisational relationship (e.g. laboratories in various locations, but belonging to the same company or distribution network).
- A detailed description of the ring trial setup and a full report including a copy of all original data and statistical calculations must be provided to ICC for review by the ICC Technical Director and final discussion and approval by the ICC Technical Committee.
- It is advisable to discuss the planned trial with the ICC Technical Director, before undertaking the study. If a study was not planned with the ICC Technical Director it can be submitted to ICC Headquarters for approval / acceptance, but ICC reserves the right to turn down the certification as ICC Standard Method down based on non-compliance to ICC rules.
- Joint validation and/or joint submission/publication of Standards in collaboration with related SODs (Standardisation Organisations) are recommended.
- If a method / appliance /test kit or reference material is jointly validated with other SODs a mutual recognition / equivalence needs to be stated in the published methods, respectively.
- For final analysis of the results the statistical methods used must be clearly described.
- Statistical evaluation of the method must include the following parameters:
 - Description of the method / appliance /test kit / reference material used.
 - Description of the "measurand".
 - Description of the "sample" / reference material.
 - Homogeneity test results of the sample material (usually performed by the organising laboratory and / or confirmed by 1-2 additional laboratories, e.g. F-test, ANOVA on 10 replicate samples tested taken from the same lot).
 - Detailed protocol.
 - Details of participating laboratories including name of operator, operator's function.
 - Copy of original results / data including lot number, serial number, etc.
 - Repeatability.
 - Reproducibility.
 - Limit of Detection (LOD) and Limit of Quantitation (LOQ), where applicable.
 - Measurement uncertainty.
 - Traceability to SI units, if applicable.

4.3.2. Samples

Any material used as a sample needs to be well described (origin, ingredients, concentration, etc.).

Homogeneity and stability needs to be tested, documented and monitored throughout the ring trial.

According to the IUPAC / AOAC / ISO international harmonised protocol for collaborative trials at least 5 different samples or 2 different matrix samples at 4 different concentration levels (including a zero / blank sample) shall be used.

The samples must be representative for the range of variation of the measurand in the matrix. The samples shall be selected to cover the relevant range of application of the method with concentrations distributed across the whole application range.

4.3.3 Required method format

ICC's method format takes into account the requirement of:

- ISO 78-2:1999 Chemistry - Layouts for standards - Part 2: Methods of chemical analysis.
- ISO 78-3:1999 Chemistry - Layouts for standards - Part 3: Standard for molecular absorption spectrometry.
- EURACHEM guide for "The fitness for purpose of analytical methods" -second edition 2014.

Whilst it is desirable that all methods should have the same document format, it should also be recognised that not all methods warrant the same degree of detail and frequently it will be appropriate to omit some of the recommended sections from the documentation.

The format specified in the technical instruction (TI/ICC/01) is for reference as a suitable layout. It is for guidance only and could be adapted to suit any special requirements.

5. **Costs for proprietary methods**

The costs for validation studies can vary depending on each case. For more details contact office@icc.or.at

6. **Publication**

After approval of an analytical method or a reference material, it is published as ICC Draft Standard Method, until a second review after 2 years of first issue.

ICC Draft Standards may become regular ICC Standard Methods by approval after trial period of two years as Draft Standard.

ICC Standard Methods can be purchased either as single method standard or as part of the ICC Standard Method Collection. The method provider and organiser of the ring trial shall receive on free copy of the ICC Standard.

ICC requires the publication of the obtained results of the validation study in a peer-reviewed journal. The report shall be written and submitted for publication by the organiser of the ring trial or the method provider.

7.0 Ownership

ICC Standard Methods are owned by ICC and thus are protected by ICC copyright. ICC Standard Methods are sold by ICC Services GmbH and authorised distributors contracted by ICC. Any submission of ICC Standards to CEN, ISO, Codex Alimentarius by any national / regional government or other institution needs to be approved by ICC and the authorship of ICC needs to be referred to in the publication / submission form.

8.0 Liability

ICC is only liable for the content and the performance data at the time of evaluation and publication. ICC is not responsible for deviations of performance quality due to manufacturing or non-compliant method application.

Any changes and/or additions to ICC validated methods need to be communicated to the ICC Headquarters at the earliest.

9.0 Revisions

ICC will revise its ICC Standard Methods at a minimum of 10-year intervals. ICC reserves the right to request method revisions in certain intervals, if the advancements and method developments in a certain field of analysis demand a more rapid turnover.

ICC reserves the right to ask for revision or a withdrawal of an ICC Standard Method in ease of doubt. The method provider is obliged to inform ICC, if changes or additions have been made to a published method. When changes have been made to an existing ICC Standard, ICC reserves the right to request a new ring trial for the modified / improved method for continued use and circulation as ICC Standard. Failure to provide updating of information for a modified method / appliance will result in the withdrawal of the method as ICC Standard and informing its users and collaborating SDOs about the withdrawal.

When an ICC Standard Method is changed by international committees, such as ISO, CEN, Codex Alimentarius, ICC requests to be informed about the proposed changes by the submitting agency (i.e. the International committee or the method provider).

10.0 Application for Standardisation

Applications to have as method validated/standardised according to ICC requirements must be submitted to the ICC Headquarters, by filling the "Application Questionnaire" form (PO/ICC/01).

The application must include a short description of the principle of the method, its application, its current significance and potential future use. The ownership of the method must be clearly stated (generic or proprietary).

Contact details of the method provider and the organiser of the ring trial must be provided to ICC Headquarters.

Once the submitted method is considered for ICC standardisation, the payment for handling and publication must be received prior to validation/standardisation and is non-refundable irrespective of the results of the validation study.