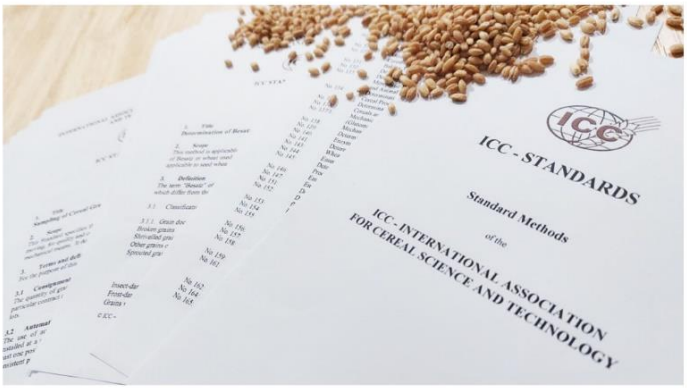
ICC STANDARD METHOD VALIDATION
FORMAT REQUIREMENTS



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Approved by TC/202311

Note:

In addition to the instructions of the Format Requirements document, it is recommended that the draft submitted to ICC for evaluation be readable and graphically well structured. In particular, are recommended:

- sufficient and uniform font size and line spacing
- uniform alignment of paragraphs
- sufficient image size, so that essential details of figures can be appreciated
- proper alignment of figures and tables with their titles and with the text
- tables should not be pasted as figures, but as Word or Excel tables, with a readable font size, clear column alignment and line spacing

If the draft is poorly readable, ICC will ask for improvement before evaluation.

INTERNATIONAL ASSOCIATION FOR CEREAL SCIENCE AND TECHNOLOGY

ICC STANDARD No…

Approval date
Revision date

1. Title

The title shall express the matrix types to which the test method applies, the measurand or the characteristic to be determined and the principle of the determination. It should be limited, wherever possible, to the following information. Preferred format: Determination of A{measurand} (in the presence of B{interference}) in C {matrix} using D {principle}.

2. Scope and field of application

This section enables a potential user to see quickly whether the method is likely to be appropriate for the desired application, or whether limitations exist. The following details should be covered:

- a description of the underlying problem (why the method is needed);
- a short description of the application area and exceptions of the application area;
- the measurand(s) which can be determined by the method;
- the form in which measurand(s) is determined – speciation, total/available etc.;
- the matrix(es) within which those measurand(s) may be determined;
- the instrumental technique used in the method;
- a working range (measuring interval) over which the method may be used. This should refer to properties, e.g. concentrations;
- known interferences which prevent or limit the use of the method;
- indicate comparability with other methods (ISO/CEN/IUPAC/AOAC and others) ;
- the minimum sample size.

3. References

This clause shall give a list of those documents which are necessary for the application of the method (such as other standard methods, for example: sampling methods, moisture determination methods etc.). Documents which have merely served as references in the preparation of the method shall be indicated in a bibliography at the end of the document.

4. Definitions

Give any definitions of terms used in the text that may be necessary for its complete understanding. Quote sources. Chemical structures can be included here if relevant.

5. Principle

Outline the essential steps of the method, the principle by which the analytical technique operates. A flow chart or cause-and-effect diagram may help. This section should be written so as to allow an at-a glance summary of how the method works. Include an explanation on the principle of the calculation.

6. Reactions

This clause shall indicate the essential reactions, if they are considered necessary for the comprehension of the text or the calculations. They must justify the calculations made from the data obtained in the determinations and may lead to a better understanding of the method, especially if several successive changes occur in the state of oxidation of the element being determined. When titrations are involved, they are particularly useful in indicating the number of equivalents in each mole of reactant. E.g. "The concentration is derived from a 6-point calibration curve by reading off the concentration, corresponding to the sample absorbance, corrected for the blank value, and multiplying it by the concentration factor."

7. Reagents and materials

List all reagents and materials required for the analytical process, together with their essential characteristics (concentration, density, etc., as needed) and numbered for later reference. List:

- Chemical Abstract Service (CAS) Registry numbers (if available);
- details of any associated hazards including instructions for disposal, attention is drawn to the regulations governing the handling of hazardous substances are referred to. **Technical, organizational and personal protective measures must be considered, the user of an ICC method is responsible to inform him/her in advance about the current hazard warnings and safety advice for handling the reagents listed in the method.**
- analytical grade or purity, in no case adjectives like conc., diluted. etc. are used;
- need for calibration and quality control (QC) materials to come from independent batches;
- details of preparation, including need to prepare in advance;
- containment and storage requirements;
- shelf life of raw material and prepared reagents;
- required composition with notes of type of concentration or other quantity;
- labelling requirements.

8. Apparatus

Describe individual equipment required for executing the procedure and how they are connected in sufficient detail to enable unambiguous set-up. Number the items for later reference. Diagrams and flowcharts may assist clarity. Especially for special apparatus it may be useful to explain the instrument more in detail, e.g. by giving the dimensions in millimetres necessary for reproduction. Any checking of the functioning of the assembled apparatus shall be described in the "Procedure" clause in a sub clause headed "Preliminary test" or "Check test" (see 10).

List minimum performance requirements and verification requirements and any relevant instrument manuals. If appropriate, refer to international standards or other internationally acceptable documents concerning laboratory glassware and related apparatus.

Include environmental requirements (fume cupboards, temperature requirements during operation etc.).

9. Sampling

Wherever possible refer to published sampling guidelines or standards.

The sampling includes both the sampling to obtain the laboratory sample and the subsampling in the laboratory to obtain the test sample from which the test portion will be drawn. If sampling for the preparation of the laboratory sample is independent of the chemical analysis as such, it is generally sufficient to refer informatively to the relevant procedure dealing specifically with this question. If no such relevant procedure exists, the sampling clause may include a sampling plan and sampling procedure, giving guidance on how to avoid alteration of the product and taking into account requirements concerning the application of statistical methods.

The sampling clause should give all the information necessary for the preparation of the test sample from the laboratory sample.

Include storage, conditioning/pre-treatment and disposal details. If this stage is particularly complicated, a separate document describing individual steps may be justified.

10. Procedure

Describe each sequence of operations as detailed as necessary so that a user not familiar with the method can understand the different steps. If the method to be described is already given in another standard, the phrase "use the method specified in…" or "use one of the methods specified in…" shall be used, with an indication of any modification, if necessary. Mention operations for which special safety precautions are necessary. **If necessary, the 'Procedure' clause shall be introduced**

with references to possible safety risks. It may be useful to have someone unfamiliar with the method perform the method using the method description prior to a ring test to identify pitfalls. The 'Procedure' clause shall normally include sub clauses on the following:

- test portion (its preparation from the test sample or laboratory sample and the required mass or volume given with appropriate units);
- the permissible tolerance of the specified size of the test portion is accurately given e.g. "ca. 5 g accurately to within 0.001 g" or "precisely 5 g ± 0.001 g";
- temperatures are given with a tolerance, e.g. 25 ± 2°C;
- blank tests (conditions and limitations);
- preliminary test or check test (e.g. to verify the performance of a measuring instrument);
- **determination(s) or test(s):** This includes mentioning the number of measurements or tests (e.g. duplicate) and detailed description of all steps;
- **calibration**: Identify the critical parts of the analytical process, which will have to be controlled by careful operation and calibration. Cross-reference to the relevant sections above. Include calibration of equipment – what needs to be calibrated, and curve fitting.
- describing the calibration procedure: preparation of calibrators, storage of calibrators, frequency of re-calibration etc....Consider appropriate metrological traceability of calibrants.

If it is necessary to carry out the method or parts of the method within a certain time, this should be mentioned.

11. Calculation and expression of Results

Describe how the result(s) are calculated. This clause includes:

- all information about the units in which the result and other quantities are to be expressed;
- the required number of significant figures
- the equation used for the calculation together with the meanings of the algebraic symbols used in the equation
- the number of significant figures or significant figures to which the result is to be given.

12. Precision

For methods that have been subjected to an interlaboratory comparison trial (collaborative test), the precision data (i.e. the repeatability and reproducibility) shall be indicated. The precision data shall be calculated, and has to be also published, in accordance with the relevant part of ISO 5725 or in accordance with another suitable International Standard (which shall be referenced). It is necessary to clearly state whether the precision values are expressed in absolute or relative terms, or as precision limits.

The following wording shall be used:

> The precision of the method is the result of a collaborative test organized by …… (organizer) on an international basis*. The study was carried out in ….. (give year e.g. 2023) on …..samples (give number of samples). The results are given in table 1.
>
> The precision data are listed as a table as follows:

Sample	**A**	**B**	**C**	**D**	**E**
Number of participating laboratories (N)					
Number of laboratories retained after eliminating outliers (n)					
Number of individual test results of all laboratories on each sample (z)					
Mean Value (m)*					
Repeatability standard deviation (S_r)					
Repeatability relative standard					

deviation (RSD_r)*					
Repeatability limit (r) (2.9 S_r)*					
Reproducibility standard deviation (s_R)*					
Reproducibility relative standard deviation (RSD_R)*					
Reproducibility limit (2.8 S_R)					

*The appropriate dimension is added here.

13. Performance data

In order to utilise a result to decide whether it indicates compliance or non-compliance with a specification, it is necessary to take into account the measurement uncertainty**.** It is important that the analyst is able to translate the datagenerated during analysis of samples using the validated method, into results which directly contribute to solving the client's problem. The performance characteristics established during the validation process help to do this. Issues such as measurement uncertainty need to be treated carefully in certain circumstances, for example in legal contexts. It may be better to be open about the existence of uncertainty attached to measurements and be prepared to justify decisions made in the light of knowing that uncertainty.

Where a statement of uncertainty is required with the result, it may be appropriate to quote an expanded uncertainty by applying a suitable coverage factor. For example, a coverage factor of 2 corresponds to an interval with a level of confidence of approximately 95 %.

Uncertainty, Ux, is a parameter characterising the dispersion of values that can reasonably be attributed to the result. This uncertainty is established through the statistical distribution of results given by the interlaboratory test and characterised by the experimental relative standard deviation

Ux=k*SR

Where:

SR: the relative standard deviation of reproducibility

k: a coverage factor corresponds to a level of confidence

k:2 corresponds to a level of confidence 95%.

For well-established ICC methods that are due for revision the uncertainty for the past decade can be easily compiled using statistical data from proficiency testing bodies.

Some guidance is already available on such issues, such as the

- EURACHEM/CITAC Guide CG4 "Quantifying Uncertainty in Analytical Measurements"; 3rd Edition 2012,
- EURACHEM/CITAC Guide "Use of Uncertainty Information in Compliance Assessment"; 2nd Edition 2021.

14. Test report

This clause should specify the information to be given in the test report. The following aspects of the test should normally be included:

- a reference to the method used;
- the result(s) and an indication of the associated quality (precision, specified uncertainty; confidence interval) if applicable, including a reference to the "Calculation" clause;
- any deviations from the procedure;
- any unusual features observed;
- the date of the test.

15. Annex

To improve readability, some information is more conveniently presented in an annex. It shall be clearly stated whether the annex is normative or informative. Examples of information which can be annexed are data from the method validation work, risk analysis and uncertainty calculations. For the latter, the major sources of uncertainty relating to the method should be identified and the assigned values listed. Insignificant contributions not used in the final calculation should be mentioned. The combined standard uncertainty and/or the expanded uncertainty should be listed together with an explanation of how it was derived. A more detailed treatment may be in a cross-referenced file.

In the annex all further information can be included e.g. typical chromatograms, titration curves or illustrations of specific devices. It is also possible to add specific settings for devices e.g. titration parameters necessary for the appropriate run of the method.
It is also useful to include information necessary for the assessment of the results.

16. Bibliography

If informative references are considered necessary, these may be given at the point in the text at which they are referred to or, if there are several, in a bibliography at the end of the document.

17. Remarks

18. Acknowledgments