
ICC Headquarters, Marxergasse 2, 1030 Vienna, Austria
**Phone**: +43 1 70772020 **- Fax**: +43 1 7077202-300 - **E-mail**: office@icc.or.at

# ICC Methods Format Requirements

# INTERNATIONAL ASSOCIATION FOR CEREAL SCIENCE AND TECHNOLOGY

**ICC STANDARD No…**

Approval date
Revision date

## 1. Title

The title shall express the sample 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);
- the measurand(s) which can be determined by the method;
- the form in which measurand(s) is determined – speciation, total/available etc.;
- the sample matrix(es) within which those measurand(s) may be determined;
- a working range (measuring interval) over which the method may be used. This should refer to properties, e.g. concentrations, in the laboratory sample;
- known interferences which prevent or limit the use of the method;
- the instrumental technique used in the method;
- 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. Analytical 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.
Where appropriate to clarify the working of the method or calculations, include details of any relevant chemical reactions (for example, this may be relevant where derivatisation is involved, or in titrimetry). 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.”

## 6. Reactions

This clause shall indicate the essential reactions, if they are considered necessary for the comprehension of the text or the calculations. They 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.

## 7. Reagents and materials

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

- Chemical Abstract Service (CAS) Registry numbers (if available);
- details of any associated hazards including instructions for disposal;
- analytical grade or purity;
- need for calibration and 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 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. 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 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. 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. 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);
- 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. These 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, Describing the calibration procedure: preparation of calibrators, storage of calibrators, frequency of re-calibration etc....Consider appropriate metrological traceability of calibrants.

11. **Calculation and expression of Results**

Describe how the result(s) are calculated. Include information about the units in which the result and other quantities are to be expressed; the equation used for the calculation; the meanings of the algebraic symbols used in the equation; the numbe r of decimal places or significant figures to which the result is to be given.

**12. Precision**

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

**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 data, generated during analysis of samples using the validated method, into results which directly contribute to solving the customer'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 reasonable be attributed to the result. This uncertainty is established through the statistical distribution of results given by the interlaboratory test and characterised by 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"
- EURACHEM/CITAC Guide "Use of uncertainty information in compliance assessment".

**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. Annexes

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.

## 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