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# Paper, board and pulps — Estimation of uncertainty for test methods by interlaboratory comparisons



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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.



This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.



This first edition cancels and replaces the second edition of ISO/TR 24498:2019, which has been technically revised.

The main changes are as follows:

— ISO/TR 24498 has been changed into ISO/TS 24498 adding normative language

— Lignins and kraft liquors have been introduced in the scope of the document, and a subclause on the sampling of these materials in 5.2 has been added

— Subclause 7.3 has been updated.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Introduction

One step in the development of any new standard test method is to estimate the uncertainty of the method. After such a procedure, a "Precision statement" is usually included in ISO test methods for pulp, paper and board and is recommended by ISO/TC 6 for all new and revised ISO/TC 6 standards. This is normally performed in a precision experiment, in which samples are sent to a number of laboratories and the results are compared. Such a precision experiment is often referred to as "interlaboratory comparative testing".

The procedures for conducting a precision statement are outlined in the ISO 5725 series[1], which is general and does not cover the special conditions that apply in the testing of pulp, paper, board and cellulosic nanomaterials (this is the reason why some countries have published national standards or test methods dedicated to pulp, paper and board[2][3]).

For example, paper and board materials as well as cellulosic nanomaterials are very sensitive to changes in relative humidity and temperature. Changes in the environmental conditions may induce significant moisture content variations in paper and board, which may induce changes in physical and mechanical properties.

Due to product heterogeneity, randomisation of the samples and /or test pieces is essential to minimize the impact of such variability. For the same reason, the variation in the properties can increase drastically when the test piece size decreases, for example when measuring grammage or Cobb water absorptiveness.

These reasons make it necessary to have special instructions for precision experiments for pulp, paper, board and cellulosic nanomaterials.

One effect of the heterogeneity of the product is that a large number of measurements is required in order to achieve sufficient precision. Most standardized test methods are therefore based on 10 or more measurements. The result is generally the average of these measurements.

Uncertainty has multiple components including a random component and a systematic component. This document focuses on the random component, defined by a repeatability and reproducibility of the measurements.

There are four main purposes for testing:

— Research, where the main question is whether there is an expected maximum difference between two samples, for instance, papers produced using different pulp mixtures.

— Verification of conformance with a specification. This can be at the production central testing laboratory site or in an independent laboratory.

— Evaluation of a new test method, where the aim is to verify that the precision of the test method is acceptable.

— Determination of a precision statement for an existing test method either where one does not exist or where it requires revision.

When the uncertainty of a test method is to be expressed, the following aspects should be considered.

— The conditions for the tests. Are the conditions as similar as possible, or as different as possible?

— The uncertainty can be expressed in different statistical measures, as a standard deviation or as a confidence interval.

— The uncertainty can be expressed either as a variation in the test results themselves, or as the difference between two test results.

# Paper, board and pulps — Estimation of uncertainty for test methods by interlaboratory comparisons

## 1 Scope

This document presents guidelines for a methodology for the estimation of the uncertainty of methods for testing lignins and kraft liquors, pulps, paper, board, cellulosic nanomaterials, as well as products thereof containing any portion of recycled material or material intended for recycling.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

## 3 Terms and definitions



For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp



— IEC Electropedia: available at https://www.electropedia.org/

**3.1**
**interlaboratory comparison**
organization, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions

[SOURCE: ISO 13528:2015, 3.1]

**3.2**
**interlaboratory test**
series of measurements of one or more quantities performed independently by a number of laboratories on samples of a given material

[SOURCE: ISO 11459:1997, 3.16]

**3.3**
**repeatability condition of measurement**
repeatability condition
condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time

Note 1 to entry: A condition of measurement is a repeatability condition only with respect to a specified set of repeatability conditions.

Note 2 to entry: In chemistry, the term “intra-serial precision condition of measurement” is sometimes used to designate this concept.

[SOURCE: JCGM 200:2012, 2.20]

**3.4**
**repeatability limit**
*r*
value less than or equal to which the absolute difference between two test results obtained under repeatability conditions is expected to be with a probability of 95 %

[SOURCE: ISO 3534-2:2006]

**3.5**
**repeatability standard deviation**
standard deviation of test results obtained under repeatability test conditions

[SOURCE: ISO 3534-2:2006]

**3.6**
**reproducibility condition of measurement**
reproducibility condition
condition of measurement, out of a set of conditions that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects

Note 1 to entry: The different measuring systems may use different measurement procedures.

Note 2 to entry: A specification should give the conditions changed and unchanged, to the extent practical.

[SOURCE: JCGM 200:2012, 2.24]



**3.7**
**reproducibility limit**
*R*
value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions is expected to be with a probability of 95 %



[SOURCE: ISO 3534-2:2006]

**3.8**
**reproducibility standard deviation**
standard deviation of test results obtained under reproducibility test conditions

[SOURCE: ISO 3534-2:2006]

**3.9**
**uncertainty**
<measurement> non-negative parameter which characterizes the variability in the values obtained from measurements

## 4 Procedure

The preferred procedure is for an expert from the working group responsible for developing an ISO Standard to organize the interlaboratory testing while the standard is being developed.

In these conditions, tests are performed with commercially available materials, as uniform and stable as possible, utilizing test instruments which are also available on the market and in the participating laboratories. A call for participation of laboratories outside of the working group may be permitted.

In the case where an interlaboratory test cannot be implemented by the working group, the use of comparative testing services data (for example from pulp and paper, collaborative testing services round robins, or CEPI Comparative Testing Service) are recommended. In this case, the most recent data should be provided and recalculated in the format used in the ISO/TC 6 standards.

If neither of the two first options are possible, bibliographic data should be reported.

# 5 Preparation of an interlaboratory study

## 5.1 Laboratories

### 5.1.1 Qualification of laboratories

Any laboratory that would be considered qualified to run the test is permitted and encouraged to participate in the interlaboratory study.

Laboratories shall be properly equipped to follow all details of the procedure, including climate conditions when specified, and be willing to assign the work to a skilled operator on a timely basis with competent personnel having knowledge of the materials and of the property to be tested.

In many situations it is preferable that participating laboratories meet the requirements of ISO/IEC 17025[4] or equivalent, or that at least they participate in a comparative testing service and have been shown to be competent in the test for which the precision data is being obtained.

The decision on permitting a laboratory to participate should be based on information provided to the working group, including information as to the required time for calibrating the apparatus and for testing all of the materials.



### 5.1.2 Number of participating laboratories

It is recommended to include at least eight laboratories to obtain a valid estimate of the uncertainty associated with the test method.

No interlaboratory round robin test should be performed with less than five laboratories.



## 5.2 Sample preparation and distribution

### 5.2.1 Number and type of material

The number and types of materials to be included in the interlaboratory study should cover the range of the values of the property being measured and be representative of the number of types or classes of materials to which the test method is to be applied. It also should cover each scale of the instrument (e.g. Scott Bond) if applicable.

If the interlaboratory study is restricted in any of these areas, the omitted information should be reported in the precision statement.

### 5.2.2 Selection of the material

#### 5.2.2.1 General

The sampling procedure shall be appropriate to the property to be assessed and the type of material (pulp, paper, board or cellulosic nanomaterial).

It is up to the person responsible for the interlaboratory study to check if the material selected is suitable or not. If not, the material shall be changed.

It is also up to the person responsible for the interlaboratory study to check if the property is normally distributed. When normality of distribution cannot be proven, it is advised to group the data. One can also use comparability techniques, i.e. to compare the average mean differences between laboratories, once the consistency of data coming from these laboratories is also proven graphically.

Each selected product shall be sampled so that the variability among the specimens of that material will be minimized. In this way, several basic considerations shall be kept in mind. There are always cross-machine variations in properties, and the rate of change on the edges of the web may be significantly greater than in the middle. The changes in the machine direction are usually less severe. Since paper is anisotropic and two-sided, care shall be taken when the reel is split into sheets to ensure that the machine direction is differentiated from the cross direction and that the two sides of the paper are identified if relevant.

This can be achieved for example by cutting the sheets with different dimensions in the machine and cross directions and drawing a line on the edge of each sheet, on the same side.

Sampling for chemical analysis and for some physical tests such as burst does not require distinguishing between machine and cross direction or sheet sidedness.

Samples shall be rendered homogeneous by appropriate randomization.

Enough test pieces or specimens from each sample shall be prepared to provide the required test material for all participating laboratories and a significant number of additional test pieces or specimens for replacement of any lost or spoiled items.

With paper and board, sampling may be performed from rolls or from reams. In each case, specified procedures should be followed. Rolls are preferred to reams because rolls are more homogeneous.

Since the sampling procedure may vary depending on the type of sample and on the particular testing method, follow the specified sampling procedures and test methods.



#### 5.2.2.2 Sampling from rolls

If sampling is performed on rolls, all specimens should be taken from a small area of a single roll, avoiding the edges of the roll. Usually, specimens taken adjacent to each other in the machine direction are more similar than those adjacent in the cross direction.



#### 5.2.2.3 Sampling from reams

Reams are usually constituted from sheets issued from four to six mother rolls. If sampling is performed on reams of papers or boards, measurements should be performed on at least 25 consecutive top sheets from each ream in order to eliminate from the sample sheets showing deviation.

If one pack does not contain enough material, additional reams shall be sampled. In this case, it shall be verified that all the reams are from the same production, and that their characteristics are close enough to ensure a uniform statistical population.

Randomization shall be performed after the selection of all the necessary reams, and not ream by ream.

#### 5.2.2.4 Sampling of cellulosic nanomaterials

Rolls or reams, pulp-like samples, sheets or other samples resembling pulp, paper or board shall be sampled using the appropriate sampling technique described herein or in the corresponding ISO standard.

Powders, granules, flakes or other dry forms shall be rendered homogeneous by appropriate randomization and representative samples taken using the appropriate sampling technique.

Slurries or suspensions of cellulosic nanomaterials shall be rendered homogeneous by stirring or other appropriate methods and representative samples taken from the homogeneous mixture.

#### 5.2.2.5 Sampling of lignins and kraft liquors

Lignins shall be rendered homogeneous by stirring or other appropriate methods and representative samples taken from the homogeneous mixture.

### 5.2.3 Identification and packaging

Samples should be identified by codes by the person responsible for the round-robin in order to be tested anonymously by the participants.

The side to be tested and/or machine or cross direction shall be clearly identified if relevant.

Where relevant (depending on the test) samples shall be pre-conditioned and conditioned according to ISO 187 before being packed.

Samples should be packaged in moisture barrier, light-proof packages to ensure that no changes in physical, optical or chemical properties occur.

### 5.2.4 Additional and specific care

Specific care shall be taken when sampling and handling microbiological samples.

Whenever possible, submit samples to the testing laboratories in the original unopened containers. Otherwise transfer the representative sample to a sterile plastic bag or pack it in household aluminium foil, under aseptic conditions. Identify each packed sample with a properly marked strip of masking tape, excluding any solvent markers and adhesives.

## 5.3 Documentation for the interlaboratory study

Adequate documentation for the participants in the study shall be prepared by the person responsible for the study, and shall include at least the following information:



— a description of the samples to be tested;

— the procedure to be implemented to perform the measurement. In principle, the draft or existing ISO standard is the procedure to be used;

— when important, a required day or week for the analysis (for microbiological tests for example, analysis should be ideally performed by the different laboratories within two months because the microbe number could change);

— conditioning or ageing of the samples;

— a request for additional information such as relative humidity, temperature, type of instrument, date of analysis (important parameters for microbiological tests for example);

— a request for comments on the measurements;

— a deadline for returning the results.

A template shall be distributed for recording:

— all the replicate measurements (an electronic spreadsheet is in most cases suitable), including the number of decimal places to be used, and calculations for the average, standard deviation, coefficient of variation or other parameters depending on the specifications of the draft standard;

— additional information such as relative humidity, temperature, type of instrument;

— comments on the measurements.

One file per laboratory shall be used, containing one sheet per sample assessed. One example of a template is given in Annex B.