### TECHNICAL REPORT

ISO/TR 24498

Second edition 2019-01

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

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Reference number ISO/TR 24498:2019(E)



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Published in Switzerland

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#### **Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

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

This second edition cancels and replaces the first edition (ISO/TR 24498:2006), which has been technically revised. The following changes were made:

- The scope has been modified as the revised document presents guidelines for a methodology for the estimation of the uncertainty of methods for testing the properties of pulp, paper, board and cellulosic nanomaterials.
- 2.1 and 2.2 have been moved into the Introduction.
- 2.3 has been moved into the Bibliography.
- Terms and definitions have been updated.
- <u>Clauses 4</u>, <u>5</u> and <u>6</u> have been entirely revised.
- New information has been introduced regarding procedure, preparation of an interlaboratory study, including information on the participating laboratories, sample preparation and distribution and documentation for the interlaboratory study.
- Specifications for cellulosic nanomaterials and for microbiological tests have been introduced.
- 7.3, Clauses 8, 9, 10 and Annexes A and C have 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 <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

#### 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<sup>[1]</sup>, 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<sup>[2][3][4]</sup>).

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.

Furthermore, many tests carried out on pulp, paper, board or cellulosic nanomaterials are destructive. In such cases, it is impossible to achieve a true repeatability measurement.

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.

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.

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## 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 pulps, paper, board, cellulosic nanomaterials, and lignins, as well as products thereof containing any portion of recycled material or material intended for recycling.

#### 2 Normative references

There are no normative references in this document.

#### 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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <a href="http://www.electropedia.org/">http://www.electropedia.org/</a>

#### 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 conditions

observation conditions where independent test results are obtained with the same method on test pieces from the same specimen in the same laboratory by the same operator using the same equipment within a short interval of time

Note 1 to entry: Repeatability conditions are not fulfilled in the procedure described in this document. It is assessed by the average standard deviation within laboratories instead of a strict repeatability study.

[SOURCE: ISO 3534-2:2006, 3.3.6, modified — "identical test/measurement items in the same test or measuring facility" has been replaced with "test pieces from the same specimen in the same laboratory"; the Note has been deleted and Note 1 to entry has been added.]