TECHNICAL REPORT

ISO/TR 24498

Second edition
2019-01

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

Papiers, cartons et pâtes — Estimation de l'incertitude pour les méthodes d'essai par comparaisons interlaboratoires

Reference number
ISO/TR 24498:2019(E)

© ISO 2019



COPYRIGHT PROTECTED DOCUMENT

© ISO 2019

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

 © ISO 2019 – All rights reserved

Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms and definitions ... **1**
4 Procedure ... **2**
5 Preparation of an interlaboratory study ... **3**
5.1 Laboratories ... 3
5.1.1 Qualification of laboratories ... 3
5.1.2 Number of participating laboratories ... 3
5.2 Sample preparation and distribution ... 3
5.2.1 Number and type of material ... 3
5.2.2 Selection of the material ... 3
5.2.3 Identification and packaging ... 4
5.2.4 Additional and specific care ... 5
5.3 Documentation for the interlaboratory study ... 5
6 Recommendations for testing ... **5**
7 Analysis of the results ... **6**
7.1 Calculations ... 6
7.2 Case of interlaboratory study organised by the working group ... 6
7.2.1 General ... 6
7.2.2 Consistency evaluation ... 7
7.2.3 Handling of outlying data ... 7
7.3 Case of data provided from proficiency testing services ... 7
8 Re-evaluation of the uncertainty ... **8**
9 Report ... **8**
10 Archiving raw data ... **8**
Annex A (informative) **Tappi and ISO codifications** ... **9**
Annex B (informative) **Example of a recommended form to be distributed to participating laboratories for the collation of results for analysis** ... **10**
Annex C (informative) **Example of precision statements for physical properties** ... **11**
Bibliography ... **12**

© ISO 2019 – All rights reserved

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 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).

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 www.iso.org/iso/foreword.html.

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.

— Clauses 4, 5 and 6 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 www.iso.org/members.html.

© ISO 2019 – All rights reserved

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][4]).

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.

© ISO 2019 – All rights reserved

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 https://www.iso.org/obp

— IEC Electropedia: available at http://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 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.]

© ISO 2019 – All rights reserved

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 conditions
conditions where the test results are obtained with the same method on test pieces from a sample in different laboratories with different operators using different equipment

[SOURCE: ISO 3534-2:2006]

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

[SOURCE: JCGM 200:2012, 2.26, modified]

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.

And if neither of the two first options is possible, bibliographic data should be reported.

© ISO 2019 – All rights reserved