TECHNICAL REPORT

ISO/TR 24498

First edition
2006-10-01

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

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

Reference number
ISO/TR 24498:2006(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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.

ISO/TR 24498 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

Introduction

One step in the development of any new standard test method is to estimate the uncertainty of the method. 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 refered to as "interlaboratory comparative testing".

The procedures for conducting a precision statement are outlined in ISO 5725 (all parts), which is general and does not cover the special conditions that apply in the testing of pulp, paper and board.

There are, however, a number of different standards and publications available for the estimation of precision in pulp, paper and board testing. The focuses of these standards differ, depending on the purpose of the test.

There are three main purposes identified for testing:

- Research, where the main question is whether there is a difference between two samples, for instance, papers produced using different pulp mixtures.
- Testing in order to verify compliance with a specification. This can be both at the production site and 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.

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

1 Scope

This Technical Report presents guidelines for the selection of the best method for the estimation of the precision of methods for testing pulp, paper and board.

2 Background information

2.1 General

A "Precision statement" is included in most of the ISO test methods for pulp, paper and board.

Such a "Precision statement" is usually based on collaborative interlaboratory experiments.

ISO 5725 (all parts) describes the procedure for generating the numbers for conducting such collaborative interlaboratory experiments. However, there are specific conditions in the testing of pulp, paper and board which are not covered by ISO 5725.

2.2 Special conditions in the testing of pulp, paper and board

Paper and board materials are very sensitive to humidity and temperature. In addition, the variation in the properties increases drastically when the sample size decreases. The coefficient of variation for grammage (mass per area), for instance, can increase from 1 % to 10 % if the averaging area is decreased from 1 m^2 to 1 mm^2.

In addition, many tests carried out on pulp, paper and board are destructive.

For many pulp, paper and board properties, it is also a reality that there is no "absolute" true value. A simple example is thickness, which is dependent on such factors as measurement pressure, speed of applying this pressure, air humidity and for how long the sample has been in the measurement climate. This means that only the thickness of paper measured under specific circumstances can be determined. Due to the complexity of paper, all the variable circumstances are not taken into account in this Technical Report. Consequently, there is no absolute true thickness value for a paper.

One effect of the large product variations between small areas is that a fairly large number of samples is, in practice, always required in order to achieve sufficient precision. Most test methods are therefore based on 10 or more measurements. The result of such a test is generally the average of these measurements.

Another specific feature of paper is that, not only are the surface properties of paper often very important, for instance for the result of printing, but there is a risk that they can be modified by merely handling the paper.

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

2.3 Available publications for estimation of precision

Three different standards and publications have been found that address the task of estimating the precision in testing of pulp, paper and board.

These are:

- TAPPI T 1200, Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility [5];
- SCAN-G 6:00, Pulp, paper and board — Uncertainty of results from physical testing [6];
- AS/NZS 1301.460s:1998, Methods of test for pulp and paper. Method 460s: Statistical concepts used in pulp and paper testing [7].

TAPPI T 1200 describes the procedure for conducting a collaborative interlaboratory experiment.

SCAN-G 6:00 has been written to support testing laboratories in providing estimations of the uncertainty associated with tests performed in the laboratory. This estimation is a necessity for laboratories that have an ISO/IEC 17025 [4] accreditation.

AS/NZS 1301.460s:1998 covers statistics in general for pulp and paper, but it also contains a section on the "Organization of an interlaboratory experiment".

3 Terms and definitions

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

3.1
variance
moment of order r where r equals 2 in the centred probability distribution of the random variable

[ISO 3534-1:2006, 2.36 [3]]

3.2
standard deviation
positive square root of the variance

[ISO 3534-1:2006, 2.37 [3]]

3.3
two-sided confidence interval
when T_1 and T_2 are two functions of the observed values such that, θ being a population parameter to be estimated, the probability $P_r(T_1 < \theta < T_2)$ is at least equal to $(1 - \alpha)$ [where $(1 - \alpha)$ is a fixed number, positive and less than 1], the interval between T_1 and T_2 is a two-sided $(1 - \alpha)$ confidence interval for θ

3.4
repeatability conditions
conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within a short interval of time

NOTE Adapted from ISO 3534-1:2006, definition 3.3.6.

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

NOTE Adapted from ISO 3534-2:2006, definition 3.3.7.

3.6
repeatability limit
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 %

NOTE Adapted from ISO 3534-2:2006, definition 3.3.9.

3.7
reproducibility conditions
conditions where the test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

NOTE Adapted from ISO 3534-2:2006, definition 3.3.11.

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

NOTE Adapted from ISO 3534-2:2006, definition 3.3.12.

3.9
reproducibility limit
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 %

NOTE Adapted from ISO 3534-2:2006, definition 3.3.14.

4 Discussion of the definitions

When the uncertainty of a test method is to be expressed, the following aspects have to 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 absolute difference between two test results.

The conditions for the test are chosen to be the same as in ISO 3534-1, i.e. repeatability and reproducibility conditions.

Standard deviation is the most fundamental expression of a dispersion. The *confidence interval*, which is derived from the standard deviation (provided that the variables have a normal distribution) is the fundamental expression of an uncertainty. The *confidence interval* is incomplete unless it is accompanied by a probability. This probability is often selected to be 95 %, as, for instance, in the definition of *repeatability* and *reproducibility limits*. 19 values out of 20 are expected to be inside a 95 % *confidence interval*. The 95 % confidence interval is $\pm 1{,}96\sigma$, where σ is the estimated standard deviation. The assumptions are that the variable has a normal distribution and that the standard deviation is known, or that it is estimated on the basis of a sufficient number of values that the uncertainty in the standard deviation can be neglected.

The uncertainty of a test method can either be expressed as the variation in the test results themselves, or as the absolute value of the expected difference between test results.

Provided that the test results have a normal distribution, the difference between two test results will also have a normal distribution, with zero as the expected average, and the standard deviation is increased by a factor of $\sqrt{2}$ = 1,41, compared to the standard deviation of the original test results.

The *repeatability* and *reproducibility limits* can thus be calculated as $\pm 1{,}96 \times 1{,}41\sigma = \pm 2{,}77\sigma$, for test results that have a normal distribution and when the standard deviation σ is based on a large number of tests.

5 Analysis

The purpose of a precision statement in a test standard is primarily to show that the method is sufficiently developed to become a standard method. There is also a secondary intention to serve as a first approximation of what can be expected if the test method is used in different testing laboratories.

In ISO 5725-1, *repeatability* and *reproducibility conditions* are the selected conditions for the evaluation of the precision of test methods. ISO 5725-2 recommends that the results of collaborative interlaboratory experiments should be presented as the *standard deviations* under *repeatability* and *reproducibility conditions*. The *repeatability* and *reproducibility limits* are not calculated.

Both TAPPI T 1200 and AS/NZS 1301.460s:1998 describe how to conduct a collaborative interlaboratory experiment applied to the special needs of paper and board products, and they both use the same conditions as those defined in ISO 5725-1, i. e. *repeatability* and *reproducibility conditions*.

Both TAPPI T 1200 and AS/NZS 1301.460s:1998, however, recommend using the *repeatability* and *reproducibility limits* as the final reported result. This means that the standard deviation is multiplied by a factor. The factor is 2,77 in TAPPI T 1200 and is rounded off to 2,8 in AS/NZS 1301.460s:1998.

At the time of publication of this Technical Report, the current version of TAPPI T 1200 was more comprehensive than AS/NZS 1301.460s:1998.

SCAN-G 6:00 is an instruction for individual laboratories indicating how to estimate the uncertainties in the delivered test results. The uncertainties can be either for comparison with other results from the same laboratory, or for comparison with results from other laboratories. The estimations are based on collaborative interlaboratory experiments, but SCAN-G 6:00 does not contain any instructions as to how such experiments should be performed.

TAPPI T 1200 contains the following information.

- Definitions complying with ISO 3534-1, although the exact nomenclature differs in some cases, for instance "TAPPI repeatability" instead of "repeatability limit".
- A nomenclature for the multitude of measurements that are often required to obtain a test result for paper and board: specimens, samples, observations, test determinations, test result.
- Planning of the interlaboratory study: test methods and selection of laboratories.
- Conducting the interlaboratory study: selection of materials, preparation of samples and specimens.
- Calculation of the results for repeatability and reproducibility conditions, including standard deviation and repeatability and reproducibility limits.
- Consistency evaluation to determine whether the data show inconsistent values, general trends or unusual patterns.
- A description of how to write the "Statement of the precision of the test method".
- Examples.
- Instructions for conducting an intra-laboratory test.

6 Recommendation

It is recommended that the current version of TAPPI T 1200 be used for the development of precision statements within ISO/TC 6.

The entire content of TAPPI T 1200 is applicable, with the following exceptions.

- The proper ISO nomenclature shall be used in the precision statement. This means that TAPPI repeatability and reproducibility shall be reported as *repeatability and reproducibility limits*.

- The explanation suggested in TAPPI T 1200 shall also be included and it will then read: "The repeatability and reproducibility limits reported are estimates of the maximum difference which should be expected in 19 of 20 instances, when comparing two test results for material similar to those described under similar test conditions. These estimates may not be valid for different materials or different testing conditions."

- The explanation that "*Repeatability and reproducibility limits* are calculated by multiplying the *repeatability and reproducibility standard deviations* by 2,77" should also be added, with a note explaining that $2{,}77 = 1{,}96\sqrt{2}$.

- In order to produce a precision statement covering both *repeatability and reproducibility conditions*, a collaborative interlaboratory experiment is required; so that the parts covering intra-laboratory testing in TAPPI T 1200 are of secondary interest.

Bibliography

[1] ISO 3534-1:2006, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

[2] ISO 3534-2:2006, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

[3] ISO 5725 (Parts 1 to 6), *Accuracy (trueness and precision) of measurement methods and results*

[4] ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

[5] TAPPI T 1200, *Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility*

[6] SCAN-G 6:00, *Pulp, paper and board — Uncertainty of results from physical testing*

[7] AS/NZS 1301.460s:1998, *Methods of test for pulp and paper. Method 460s: Statistical concepts used in pulp and paper testing*

ICS 85.040; 85.060

Price based on 6 pages