



*Pulp, paper and board*

Updated lists of methods/standards in Annexes B and C:  
 Withdrawn – ~~SCAN-C-XX~~  
 New method – **ISO XXX**

# Uncertainty of results from physical testing

## 0 Introduction

This SCAN-test Guideline has been developed to indicate how to assign an uncertainty to the result of a test of a physical property of pulp or paper.

Published methods, e.g. Guide to the Expression of Uncertainty in Measurement, GUM (9.1), exist for the evaluation and expression of this kind of uncertainty. Due to the large variations in the material and since the testing is mostly destructive, this SCAN-test Guideline for the pulp and paper industry has been considered necessary.

This Guideline is based on a statistical approach, which is applicable for most properties. A consequence of this and the fact that tests are destructive is that it is not possible to distinguish between material variations and short-term measurement variations.

For some properties it is possible to make an error analysis relating to the physical definition of the property. This type of analysis is not however covered in this document.

## 1 Scope

This Guideline provides a basis for the evaluation and expression of an uncertainty to a result from the physical testing of a pulp or paper property. It covers however only the uncertainty components that can be controlled within the laboratory and it disregards effects that arise when testing does not follow standard procedures.

A prerequisite for the application of the method in this Guideline is that data are available from comparative testing of the property. Depending on the situation, this comparative testing can be either a comparison between different occasions in the same laboratory or between different laboratories.

The special problems associated with testing of pulps can be handled using the procedure described in this Guideline.

For a number of common properties, typical values for the uncertainty between tests and between laboratories are included.

Chemical testing of pulp and paper is not included in this Guideline.

## 2 Reference

SCAN-G 2:63 Statistical treatment of test results

## 3 Definitions

For the purpose of this Guideline, the following definitions apply:

3.1 *Test* – A complete procedure including preparation of the test material, performing the number of measurements required, and making the necessary calculations.

*Note 1* – For paper, a complete procedure includes conditioning of the samples, preparation of the test pieces, performing the number of measurements required and making the necessary calculations.

*Note 2* – For pulp testing, test portions are prepared, or a whole sequence can be included in the test, e.g. beating, preparation and testing of laboratory sheets or parts thereof.

3.2 *Measurement* – The processing of a single test piece or test portion. The number of measurements for one test is stated in the appropriate method.

3.3 *The result of a measurement* – The value of the property obtained by one single measurement.

3.4 *The result of a test* – A value (e.g. average, interpolated or indexed value), reported from the test, which is calculated based on the measured values.

3.5 *Repeatability conditions* – Conditions where independent test results are obtained with the same method in the same laboratory by the same operator using the same equipment within short intervals of time.

*Note 3* – This definition differs from that given in ISO 5725, clause 3.14 (9.2), which includes the condition “on identical test items”. This condition cannot however be met in a destructive test.

## 4 Basic assumptions

### 4.1 Components of uncertainty

The uncertainty associated with a result from a laboratory testing is divided into the following three components:

- 1 the uncertainty due to variations within the test, both material variations and testing variations;
- 2 the uncertainty due to variations between tests within the same laboratory;
- 3 the uncertainty due to variations between different laboratories.

*Note* – Physical testing of paper and pulp is performed on samples from lots having different sizes. This lot can for instance be a delivery from a producer to a customer, a laboratory-produced sample or material from the continuous production. This Guideline deals with the uncertainties within a laboratory that strictly follows the procedures in the standard. In addition, there is uncertainty associated with the sampling of the lot. This uncertainty can in many cases be a significant part of the total uncertainty.

*More information regarding the selected approach is given in Annex F.*

### 4.2 Data from comparative testing

An assumption for the application of the procedure described in this Guideline is that comparative testing data are available for the property in question. This can be either or both of the following:

- Measurements within the laboratory at irregular intervals over a long time period of sets of test pieces

taken from a stable, homogenised batch of material. *Examples of such measurements are given in Annex B.*

- Measurements at a sufficiently large number of different laboratories on sets of test pieces taken from a stable, homogenised batch of material. *Estimated values obtained from such measurements for paper and board are given in Annex C. Some examples for pulp are given in Annex D.*

When a comparison is made with other laboratories on the basis of interlaboratory comparative testing, the laboratory shall have taken part in that comparative testing. If the laboratory’s result is outside action limits defined by the comparative testing, necessary action must be taken before the comparison can be made.

### 4.3 Types of uncertainty statement

This Guideline describes the procedure which makes it possible for a laboratory to make statements such as the following regarding a test result from the laboratory:

"The reported result has an uncertainty of

- *xx* in relation to the average result expected if the test were carried out at this laboratory on other occasions;
- *yy* in relation to the average result expected if the test were carried out by other laboratories performing the test in full agreement with the standard".

The result of the test shall be reported together with an expanded uncertainty (see 7.1).

## 5 Principle

The complete error model is described by the following equation:

$$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2 \quad [1]$$

where

- $s_{tot}$  is the standard deviation associated with the test result;
- $s_{bm}$  is the standard deviation between measurements made under repeatability conditions;

*Note* – This standard deviation combines material variations and short term measurement variations.

- $n$  is the number of measurements in a test;
- $s_{bt}$  is the standard deviation associated with the variability between test occasions in the same laboratory;
- $s_{bl}$  is the standard deviation associated with the variability between laboratories.

This total model makes it possible to assign uncertainties to the test results, either for comparison with other test results from the same laboratory or for comparison with results from other laboratories.

In this model it is assumed that the various sources of variation are mutually independent.

The uncertainty is expressed as an expanded uncertainty, which defines an interval estimated to have a defined level of confidence.

The expanded uncertainty,  $U$ , is given by:

$$U = \pm k \cdot s \quad [2]$$

where

$s$  is the standard deviation for the situation concerned;

$k$  is the coverage factor corresponding to the selected level of confidence.

A presumption for estimating and expressing the uncertainty in this manner is that the variables are normally distributed. As most of the test results are computed as averages of a number of measurements this will apply for many of the methods within pulp and paper (9.3).

### 5.1 Paper testing

Physical tests on paper normally involve making a number of measurements and taking the average as the result of the test. (Index calculations are another type of result from physical testing of paper and they require special care for the estimation of the uncertainty. See 7.5.)

The variation between measurements ( $s_{bm}$ ) is due to both material and testing variations and is estimated on each test occasion, since the material variation can differ significantly between different products and different producers.

The variation between replicated tests ( $s_{bt}$ ) in the same laboratory is determined by internal laboratory control of the testing procedure over a long period of time.

The variation between different laboratories ( $s_{bl}$ ) is determined from interlaboratory comparative testing.

### 5.2 Pulp testing

The testing of pulps is generally much more complex than the testing of paper. It often comprises a whole sequence of operations: refining, preparation and testing of laboratory sheets and finally interpolation to some property value. This means that the standard deviation between measurements cannot be used for uncertainty estimations, as earlier steps can introduce a systematic error into all samples.

In order to assign an uncertainty to the results from physical pulp testing, an internal laboratory control or interlaboratory comparative testing, whose conditions correspond closely to those of the whole test sequence, is required. Depending on the type of comparative testing available, it is then possible to assign an

uncertainty for comparison either with the same laboratory or with other laboratories.

## 6 Procedure

This section describes how different components of the total variation are estimated. Numerical examples are given in Annex A. The section describes primarily the procedures for paper testing. Pulp testing is partly covered in Annex D.

### 6.1 Estimation of variation between measurements

A numerical example for this section is given in Annex A.2.

Calculate the standard deviation between measurements,

$s_{bm}$  in the model  $s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2$ , for each test. If it

can be assumed that the standard deviation is the same for a number of tests, it is recommended that a pooled average for those tests be used. The square of a standard deviation is called the variance, and the pooled average standard deviation is calculated as the square root of the mean of those test variances.

The variation between measurements has two separate causes:

- variations in the paper material between test pieces in the sample;
- variations due to the measurement equipment and measurement procedure within a test.

The effects of these types of variations on the uncertainty are however identical, which means that they can be handled together.

The impact of the standard deviation between measurements,  $s_{bm}$ , on the total variance,  $s_{tot}^2$ , is  $\frac{s_{bm}^2}{n}$

because  $s_{tot}^2$  refers to the average of  $n$  measurements (see GUM, section 4.2 (9.1)).

### 6.2 Estimation of variation between tests

A numerical example for this section is given in Annex A.3.

Calculate the standard deviation within the laboratory,

$s_{bt}$ , in the equation  $s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2$ , from an

internal control of the stability of the test method. This control should cover equipment, personnel and other procedures. It is assumed that the necessary tests will be performed a number of times each year. The interval should preferably be irregular, to avoid introducing any correlation. It should thus cover:

- different operators
- different times of the day
- different weekdays
- etc.

See Annex E for selection and treatment of reference material.

This internal test can either:

- consist of a number of separate measurements, giving an average and a standard deviation (typical for paper testing) or;
- be just a single number (typical for interpolated data from pulp testing).

Prior to the calculations, apply normal statistical considerations, at least:

- to eliminate outliers;
- to identify trends.

The procedure for identifying outliers is described in SCAN-G 2.

If there is a trend, decide (based on investigations or experience) whether it is a consequence of material ageing, equipment ageing or just a coincidence. Take the necessary action according to the decision reached.

Calculate the standard deviation between the results of the tests,  $s_{btr}$ , from this internal control.

In cases where the result from each test consists of a single number, then this  $s_{btr}$  is the estimate of the standard deviation between tests. The standard deviation between measurements,  $s_{bm}$ , should not be used in this case.

In cases where each test result is an average of a number of measurements, the observed variation between the results,  $s_{btr}$ , can be divided into two causes:

- variations between measurements within the same test;
- variations between tests.

This leads to the following error model:

$$s_{btr}^2 = s_{bt}^2 + \frac{1}{n \cdot m} \sum_{i=1}^m s_{bm.i}^2 \quad [3]$$

Calculate the variation between tests using the following formula:

$$s_{bt}^2 = s_{btr}^2 - \frac{1}{n \cdot m} \sum_{i=1}^m s_{bm.i}^2 \quad [4]$$

where

- $s_{bt}$  is the standard deviation between tests in the laboratory;
- $s_{btr}$  is the standard deviation between the results of the tests;
- $s_{bm.i}$  is the standard deviation between measurements in test number  $i$ ;
- $n$  is the number of measurements in each test;
- $m$  is the number of tests performed.

This estimate of the variation between tests is the difference between two variances. It must be remembered that the uncertainty when estimating a variance is fairly large. This means that the difference between two variances will have an even larger uncertainty, especially expressed as percentage. A consequence is that the calculated difference can in some cases be negative. In these cases, the conclusion is that there is no variation between tests and that  $s_{bt} = 0$ .

### 6.3 Estimation of variation between laboratories

A numerical example for this section is given in Annex A.4.

The variation between laboratories is estimated by interlaboratory comparative testing.

This covers equipment, personnel and other procedures and thus includes all components of variation between tests.

This test can, as in the internal test, either:

- consist of a number of separate measurements, giving an average and a standard deviation as result (typical for paper testing) or;
- give just a single number (typical for interpolated data from pulp testing).

See Annex E for selection and treatment of reference material.

Prior to the calculations, apply normal statistical considerations, at least to eliminate outliers, as described in SCAN-G 2.

Calculate the standard deviation,  $s_{blr}$ , between the results of the tests from the different laboratories.

In cases where the results from each test consists of just a single number, then this  $s_{blr}$  is the estimate of the standard deviation between laboratories. The standard deviation between measurements,  $s_{bm}$ , should not be used in this case.

In cases where each test result is an average of a number of measurements, the observed variation between the results,  $s_{blr}$ , can be divided into three causes:

- variations between measurements within the same test  $\frac{s_{bm}^2}{n}$ ;
- variations between tests in a single laboratory;
- variations between the laboratories.

From an interlaboratory study in which each laboratory carries out only one test on a single set of test pieces, it is only possible to estimate the combination of  $s_{bt}^2$  and  $s_{blr}^2$  in the equation:

$$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2 \quad [5]$$

This combination is denoted as the laboratory standard deviation,  $s_l$ .

$$s_l^2 = s_{bt}^2 + s_{bl}^2 \quad [6]$$

The data from the analysis of variance of the interlaboratory data are represented by the equation:

$$s_{blr}^2 = s_l^2 + \frac{1}{n \cdot m} \sum_{i=1}^m s_{bm.i}^2 \quad [7]$$

Calculate the laboratory variance  $s_l^2$  as:

$$s_l^2 = s_{blr}^2 - \frac{1}{n \cdot m} \sum_{i=1}^m s_{bm.i}^2 \quad [8]$$

where

- $s_l$  is the standard deviation between laboratories (including the variation between tests  $s_{bt}$ );
- $s_{blr}$  is the standard deviation between the averages from the laboratories;
- $s_{bm.i}$  is the standard deviation between measurements in laboratory number  $i$ ;
- $n$  is the number of measurements in each laboratory;
- $m$  is the number of laboratories participating.

A consequence of this is that all laboratories participating in the same interlaboratory test will have the same between-laboratory standard deviation. *Estimated values from such measurements are given in Annex C for paper and board and in Annex D for pulp.*

*Note* – If it is necessary to separate the standard deviation between tests,  $s_{bt}$ , from the standard deviation between laboratories,  $s_{bl}$ , a number of interlaboratory tests performed on the same test material is required. In this case the procedure in chapter 6.2 for estimating the standard deviation between tests,  $s_{bt}$ , should be applied to each laboratory. The variance between tests can then be estimated, and this can in turn be subtracted from the laboratory variance,  $s_l^2$ , to yield the standard deviation between laboratories,  $s_{bl}$ , as given in equation [6].

## 7 Calculation and expression of uncertainty

This section describes how different uncertainty estimates are calculated. *Numerical examples are given in Annex A for paper and board and in Annex D for pulp.*

### 7.1 Calculation of expanded uncertainty

The uncertainty for a result from physical testing of pulp and paper shall be expressed as an expanded uncertainty:

$$U = \pm k \cdot s \quad [9]$$

where

$k$  is the coverage factor .

In order to simplify the calculations it is suggested that a standard coverage factor of 2,3 be used. The coverage factor shall always be included in the report.

*Note* – A coverage factor of 2,3 corresponds closely to the t-value in the Student distribution for 95 % confidence level for 9 degrees of freedom, which complies with 10 repetitive measurements which is the procedure mostly used within the pulp and paper industry.

The result of a test shall thus be reported as

$$y \pm U \text{ or } y \pm k \cdot s \quad [10]$$

where

$y$  is the result of the test;  
 $U$  or  $k \cdot s$  is the expanded uncertainty.

This defines an interval estimated to have a level of confidence of 95 %.

### 7.2 Different levels for a property

The standard deviations between tests and between laboratories are estimated for some specific levels of the properties. When actual measurements are made, the results are seldom exactly at those specific levels, and some kind of interpolation thus has to be made. For the sake of simplicity it is suggested that the standard deviation is either considered to be constant (i.e. independent of level) or considered to be proportional to the level (i.e. a constant coefficient of variation). A decision must be made individually for each type of test. If information concerning the standard deviation is available at only one level of a given property, it is recommended that a constant standard deviation be assumed at levels lower than the available level and a constant coefficient of variation at levels higher than the available level. (Examples are given in Annex C).

**7.3 Uncertainty relative to results from the same laboratory**

A numerical example for this section is given in Annex A.5.1.

Calculate the expanded uncertainty,  $U_{bt}$ , for a result, in relation to other tests from the same laboratory according to the equation:

$$U_{bt} = 2,3 \cdot \sqrt{\frac{s_{bm}^2}{n} + s_{bt}^2} \quad [11]$$

where

- $s_{bm}$  is the standard deviation between measurements in the currently performed test;
- $n$  is the number of measurements in the test;
- $s_{bt}$  is the standard deviation between tests in the laboratory.

For the type of testing, where  $s_{bm}$  is not available (typical for pulp testing) only  $s_{bt}$  is utilised.

**7.4 Uncertainty relative to results from other laboratories**

A numerical example for this section is given in Annex A.5.2.

Calculate the expanded uncertainty,  $U_{bl}$ , for a result, in relation to results from tests in other laboratories, according to the following equation:

$$U_{bl} = 2,3 \cdot \sqrt{\frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2} \quad [12]$$

or

$$U_{bl} = 2,3 \cdot \sqrt{\frac{s_{bm}^2}{n} + s_l^2} \quad [13]$$

where

- $s_{bm}$  is the standard deviation between measurements in the currently performed test;
- $n$  is the number of measurements in the test;
- $s_{bt}$  is the standard deviation between tests in the laboratory;
- $s_{bl}$  is the standard deviation between laboratories (excluding standard deviation between tests in the laboratory);
- $s_l$  is the standard deviation between laboratories (including standard deviation between tests in the laboratory).

For the type of testing, where  $s_{bm}$  is not available (typical for pulp testing) only  $s_{bt}$  and  $s_{bl}$  or  $s_l$  shall be utilised.

**7.5 Uncertainty of indexed properties**

In some cases, the test result is expressed as an index where data are normalised with respect to grammage. In those cases where the uncertainties of the individual properties are available, it is possible to calculate an estimate for the index from the data for the separate properties. Indexes are often used in pulp testing. In those cases, however, it is not normally possible to estimate the individual uncertainties. The uncertainties must then be estimated from the indexed data.

The theory for how to combine uncertainties (GUM section 5.1 (9.1)) is related to the basic statistical theory of the additivity of variances.

If an index  $XI$  is calculated as

$$XI = \frac{X^m}{W^n} \quad [14]$$

where both  $X$  and  $W$  have associated uncertainties, and are mutually independent, the combined uncertainty is given as a first approximation as:

$$\left(\frac{u_{XI}}{XI}\right)^2 = m \cdot \left(\frac{u_X}{X}\right)^2 + n \cdot \left(\frac{u_W}{W}\right)^2 \quad [15]$$

where

- $u_{XI}$  is the uncertainty associated with  $XI$ ;
- $u_X$  is the uncertainty associated with  $X$ ;
- $u_W$  is the uncertainty associated with  $W$ .

This is based on a first order Taylor approximation. A numerical example for this section is given in Annex A.7.

**8 Reporting uncertainty**

There are two alternative ways of reporting the uncertainty:

- For comparison with results of tests performed in the same laboratory;
- For comparisons with results of tests performed at any other laboratory.

Both alternatives can be used in the same report. The uncertainty statement shall include a coverage factor or a confidence limit.

The alternative to be used shall be decided with due regard to the availability of data and the preferences of the client. A numerical example is given in Annex A.6.

**9 Literature**

9.1 GUM: Guide to the Expression of Uncertainty in Measurement. ISBN 92-67-10188-9 1995

9.2 ISO 5725-1:1994 Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions

9.3 G. Casella and R. L. Berger, "Statistical inference", Duxbury 1990, pp. 216-220, ISBN 0-534-11958-1

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## Annex A

### Example of estimation of uncertainty of a test – paper and board

#### A.1 Background

A client delivers a sample to the laboratory and requests that the value of a specific property be determined. The client also requests that the value be reported together with statements of the uncertainty both for comparison with other results from this laboratory and for comparison with other laboratories.

#### A.2 Test of client material

The test procedure requires that 20 test pieces be measured. The result of the testing is given in the table below:

	Value
Measurement 1	107,5
Measurement 2	110,9
Measurement 3	105,5
Measurement 4	101,1
Measurement 5	112,8
Measurement 6	108,5
Measurement 7	113,1
Measurement 8	104,8
Measurement 9	102,6
Measurement 10	107,8
Measurement 11	102,6
Measurement 12	107,8
Measurement 13	112,8
Measurement 14	102,0
Measurement 15	104,8
Measurement 16	113,1
Measurement 17	110,9
Measurement 18	108,5
Measurement 19	105,5
Measurement 20	114,1
<b>Average</b>	<b>107,8</b>
<b>Standard deviation (Std dev)</b>	<b>4,15</b>

It is now possible to evaluate the first term in the equation:

$$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2 \quad [A.1]$$

simply by calculating

$$\frac{s_{bm}^2}{n} = \frac{4,15^2}{20} = 0,86 \quad [A.2]$$

#### A.3 Standard deviation for comparison with results from this laboratory

In order to be able to report the uncertainty with respect to results from this laboratory the term  $s_{bt}^2$  in the equation  $s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2$  has to be estimated. The basis for this term is the data relating from the internal control of the measurement instrument and procedure.

Assume that the internal control has given the following result for the same homogenised material (20 measurements are made per test):

Date	Test average	Standard deviation between measurements	Variance between measurements
Dec 96	115	2,3	5,3
Jan 97	116	4,5	20,3
April 97	114	4,6	21,2
May 97	107	6,1	37,2
July 97	115	3,3	10,9
Sept 97	117	3,9	15,2
Nov 97	115	3,3	10,9
Jan 98	111	3,5	12,3
Feb 98	104	4,2	17,6
April 98	112	4,9	24,0
Jun 98	114	6,3	39,7
Sept 98	110	3,2	10,2
Oct 98	117	7,2	51,8
Jan 99	119	6,7	44,9
<b>Average</b>	<b>113,3</b>		<b>23,0</b>
<b>Std. dev.</b>	<b>4,12</b>		

From these data it is possible to calculate further terms in the model  $s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2$ , viz.:



- $s_{tot}^2 = 4,12^2 = 17,0$
- $s_{bm}^2 = 23,0$
- $n = 20$
- $s_{bl}^2 = 0$  as the tests are made in the same laboratory

The standard deviation,  $s_{bt}$ , between tests can thus be calculated as:

$$s_{bt}^2 = s_{tot}^2 - \frac{s_{bm}^2}{n} = 17,0 - \frac{23,0}{20} = 15,8 \quad [A.3]$$

$$s_{bt} = 3,97$$

We assume here that it has been shown for this property that the best approximation is to consider the size of the between-measurement variation as constant, independent of the level. (In fact the mean of our internal control data is very similar to the mean of our client's material).

The standard deviation associated with our client's data for comparisons with results from this laboratory can thus be calculated according to the equation:

$$s_{tot}^2 = s_{bt}^2 + \frac{s_{bm}^2}{n} = 15,8 + \frac{4,15^2}{20} = 16,7 \quad [A.4]$$

$$s_{tot} = 4,09$$

Note that, in this calculation, the between-test variance within the laboratory is combined with the between-measurement variance  $s_{bm}^2$  for the client's material.

#### A.4 Standard deviation for comparison with results from other laboratories

In order to be able to report the uncertainty for comparison with results from other laboratories the sum of the terms  $s_{bt}^2 + s_{bl}^2$  and in the equation

$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2$  has to be estimated. The basis for calculating this sum is data obtained from an interlaboratory comparative testing of the measurement instrument and procedure.

Assume that this interlaboratory comparative testing has given the following result for the same homogenised material (20 measurements have been made per test):

	Average	Standard deviation	Variance
Lab A	103,3	7,1	50,41
Lab B	100,0	3,1	9,61
Lab C	101,3	5,9	34,81
Lab D	92,8	3,0	9,0
Lab E	91,0	4,4	19,36
Lab F	103,3	3,4	11,56
Lab G	100,0	3,7	13,69
Lab H	104,2	5,0	25,0
Lab I	104,2	3,4	11,56
Lab J	97,8	6,2	38,44
Lab K	104,2	8,1	65,61
Lab L	106,3	5,6	31,36
Lab M	94,5	5,8	33,64
Lab N	103,3	4,7	22,09
Lab O	100,0	4,9	24,01
Lab P	90,2	2,8	7,84
Lab Q	89,8	1,6	2,56
Lab R	94,8	5,8	33,64
Lab S	105,1	5,4	29,16
<b>Mean</b>	<b>99,3</b>		<b>24,9</b>
<b>Std. dev.</b>	<b>5,47</b>		

From these data it is possible to calculate two of the terms (not separately, but added together) in the model

$$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2, \text{ viz.}:$$

- $s_{tot}^2 = 5,47^2 = 29,9$
- $s_{bm}^2 = 24,9$
- $n = 20$

The variance between laboratories (including the variance between tests) can thus be calculated as

$$s_{bt}^2 + s_{bl}^2 = s_{tot}^2 - \frac{s_{bm}^2}{n} = 29,9 - \frac{24,9}{20} = 28,6 \quad [A.5]$$

The standard deviation for comparisons with results from other laboratories can thus be calculated according to the equation:

$$s_{tot}^2 = (s_{bt}^2 + s_{bl}^2) + \frac{s_{bm}^2}{n} = 28,6 + \frac{4,15^2}{20} = 29,5 \quad [A.6]$$

$$s_{tot} = 5,48$$

Note that when calculating the standard deviation used for uncertainty statements relating to the client's result, the between-measurement variance,  $s_{bm}^2$ , for the client's material is used.

**A.5 Uncertainty of a test result**

**A.5.1 Uncertainty relative to the same laboratory**

The uncertainty with respect to results of tests from the same laboratory is thus given by:

$$U = 2,3 \times 4,09 = 9,4 \quad [A.7]$$

**A.5.2 Uncertainty relative to other laboratories**

The uncertainty with respect to results from any other laboratory is given by:

$$U = 2,3 \times 5,48 = 12,6 \quad [A.8]$$

**A.6 Expression of the test result incl. uncertainty**

The test result can be expressed as:

- The average value for the property is 107,8 with an uncertainty of  $\pm 9,4$  at approximately 95 % level of confidence with respect to other results from this laboratory. A coverage factor of 2,3 has been used.
- The average value for the property is 107,8 with an uncertainty of  $\pm 12,6$  at approximately 95 % level of confidence with respect to results from any other laboratory. A coverage factor of 2,3 has been used.

If the sample submitted by the client has been taken from a lot of paper, the uncertainties are valid for the average of the lot, provided that the samples have been randomly taken from the whole lot.

**A.7 Uncertainty of indexed properties**

It is common practice, especially in pulp testing, to express the result of a test as an index, where data are normalised to for instance grammage.

Examples:

The burst index,  $B^w$ , is calculated as:

$$B^w = \frac{B}{w} \quad [A.9]$$

where

$B$  is the bursting strength;  
 $w$  is the grammage.

and the bending stiffness index,  $S^w$ , is calculated as:

$$S^w = \frac{S^b}{w^3} \quad [A.10]$$

where

$S^b$  is the bending stiffness;  
 $w$  is the grammage.

In GUM, section 5.1 (9.1), the theory for how to combine uncertainties is described. These rules are related to the basic statistical theory of the additivity of variances.

If these rules are applied to the bending stiffness index, the uncertainty  $u_{S^w}$  is given by:

$$\left(\frac{u_{S^w}}{S^w}\right)^2 = \left(\frac{u_{S^b}}{S^b}\right)^2 + 3 \cdot \left(\frac{u_w}{w}\right)^2 \quad [A.11]$$

where

$S^w$  is the bending stiffness index;  
 $u_{S^b}$  is the uncertainty for the bending stiffness;  
 $S^b$  is the bending stiffness;  
 $u_w$  is the uncertainty for the grammage;  
 $w$  is the grammage.

This formula can be interpreted as indicating that the square of the coefficient of variance of the index is equal to the sum of the squares of the coefficients of variance for the components. The grammage term is multiplied by 3 in this case because grammage in equation [A.10] is raised to the third power.

Assume the following numerical values for the variables:

$$\begin{aligned} u_{S^b} &= 1,1 \text{ mNm} \\ S^b &= 21,2 \text{ mNm} \\ u_w &= 4,2 \text{ g/m}^2 \\ w &= 231 \text{ g/m}^2 \end{aligned}$$

These values lead to:

$$\begin{aligned} \frac{u_{S^b}}{S^b} &= 5,15 \% \\ \frac{u_w}{w} &= 1,82 \% \end{aligned}$$

The uncertainty for bending stiffness index expressed as percentage is thus:

$$\sqrt{5,15^2 + 3 \cdot 1,82^2} = 6,07 \quad [A.12]$$

## Annex B

### Examples of the between-test component of the variation – paper and board

This Annex shows examples of data obtained for a number of different properties by a number of different laboratories by carrying out repeated tests on the same homogenised material over relatively long periods of time.

An estimate of the standard deviation between tests,  $s_{bt}$ , in the same laboratory has been calculated using the expression:

$$s_{bt}^2 = s_{btr}^2 - \frac{1}{n \cdot m} \sum_{i=1}^m s_{bm.i}^2 \quad [B.1]$$

The results from these estimations are presented in the table as the average level and coefficient of variation using the equation:

$$CoV_{bt} = \frac{s_{bt}}{Mean} \cdot 100 \quad [B.2]$$

where

$CoV_{bt}$  is the coefficient of variation presented in the table below;

$s_{bt}$  is the standard deviation calculated by the equation B.1;

$Mean$  is the average value for the property.

Property	Standard method	Lab 1		Lab 2		Lab 3		Lab 4	
		Mean	CoV,%	Mean	CoV,%	Mean	CoV,%	Mean	CoV,%
Tensile strength, kN/m 0,20 alt 0,36 mm/s	SCAN P 38:80	2,61	1,3	6,09	1,2	4,72	4,1	4,62	2,5
	ISO 1924-2:1994	7,20	1,4	11,1	0,6	18,4	1,5	7,19	0,8
1,7 mm/s	SCAN P 67:93					4,73	1,7		
	ISO 1924-3:2005					23,5	1,2		
Stretch, % 0,20 alt 0,36 mm/s	SCAN P 38:80	1,0	3,8	1,8	2,8	2,3	1,3	2,2	2,5
	ISO 1924-2:1994	2,2	3,3	4,0	2,6	7,5	1,7	7,5	1,3
1,7 mm/s	SCAN P 67:93					2,5	1,1		
	ISO 1924-3:2005					7,4	1,5		
Tearing resistance, mN	SCAN P 11:96	188	1,8	500	2,5	872	2,1	752	2,3
	ISO 1974:1990	1330	3,6			1230	2,5	2070	1,8
Interfiber bond, J/m <sup>2</sup> Scott	TAPPI T 833 pm-94	176	3,0			329	3,0	350,5	3,0
		274	7,8						
		389	2,8						
Roughness, ml/min Bendtsen	SCAN P 21:67	75	5,3	110	5,2	290	6,2	50	5,7
	ISO 8791-2:1990					1200	3,4		
Roughness, µm PPS at 1 MPa	SCAN P 76:95	1,0	4,7			0,95	3,1	3,4	1,1
	ISO 8791-4:1992	5,2	3,5			3,4	2,5		
Thickness, µm	SCAN P 7:96			282	0,8	121	0,5	71	1,3
	ISO 534:1988					518	0,9	226	0,5
CIE-whiteness (D65/10 °)	SCAN P 66:93 ISO 11475:1999			140	0,2	135	0,6	136	0,7

## Annex C

### Estimation of uncertainty between laboratories – paper and board

This Annex provides the best available estimates of the standard deviation between laboratories ( $s_l$ ) for a large number of the methods which are in use for the testing of paper and board.

These values have been derived from data made available by the CEPI-Comparative Testing Service (CEPI-CTS), and they are recommended for use by laboratories which participate in that service. Laboratories participating in other comparative studies should derive similar data from their own test material.

The values presented in Table C.1 have been derived from the results of the CEPI-CTS pretest round in which a number (usually about 15) of qualified laboratories undertake to carry out the test strictly according to the standardised test method. After elimination of any statistical outliers, the CEPI-CTS pretest report presents, for each property, a value for the root mean square standard deviation ( $s_{bm}$ ) within a single laboratory, and a value for the total reproducibility standard deviation ( $s_R$ ) based on the data for all the qualified laboratories.

From these data, the standard deviation between laboratories has here been computed from the CEPI-CTS pretest data according to the expression:

$$s_l = \sqrt{s_R^2 - \frac{s_{bm}^2}{n}} \quad [C.1]$$

where

$n$  is the number of test pieces in the set of pieces tested.

The data have been found to be remarkably stable. The data presented in Table C.1 for each level are the root mean square standard deviations for five different pretest rounds during 1997–1999, together with recommended values for use in the estimation of uncertainties.

For each property, data for several different levels are given. A study of these data indicates that for some properties, e.g. thickness of paper, the standard deviation increases with increasing level but the coefficient of variation is roughly constant for the different levels. In these cases, a recommended value is indicated in a shaded rectangle in the final column.

For other properties, e.g. inter-fibre bond, the coefficient of variation decreases with increasing level and the standard deviation is roughly constant for the different levels. In these cases, a recommended mean standard deviation is indicated in a shaded rectangle. The ISO brightness data also fall into this category, except perhaps for very low brightness values.

In other cases, such as gloss, the behaviour is intermediate between these two extreme cases and it is not possible to recommend any mean value.

Table C.1

Property	Standard method	Level	<i>s<sub>l</sub></i>	CoV <sub>1</sub> , %
Tensile strength, kN/m	ISO 1924-2 (SCAN-P 38)	1	0,07	6,3
		7	0,29	4,1
		12	0,57	5,0
				<b>5,1</b>
Stretch at break, %	ISO 1924-2 (SCAN-P 38)	1	0,1	8,4
		2	0,2	9,2
		4	0,3	7,2
				<b>8,3</b>
Tearing resistance, mN	ISO 1974 (SCAN-P 11)	350	16,1	4,6
		1000	43,6	4,3
		1700	61,0	3,6
		2700	11,48	4,3
				<b>4,2</b>
Bursting strength, kPa (paper)	ISO 2758 (SCAN-P 24)	150	7,0	4,6
		250	10,9	4,3
		450	24,6	5,5
		950	58,5	6,2
				<b>5,2</b>
Bursting strength, kPa (board)	ISO 2759 (SCAN-P 25)	285	36,4	12,7
		475	36,5	7,7
		950	43,9	4,7
		1400	139,4	10,0
				<b>8,8</b>
Bursting strength, kPa (corrugated board)	ISO 2759 (SCAN-P 25)	1200	64,5	5,5
		2100	127,6	6,0
				<b>5,8</b>
Inter-fibre bond, J/m <sup>2</sup> Scott	TAPPI T 833	240	20,2	8,4
		420	22,6	5,4
		650	25,4	3,9
				<b>22,7</b>
Roughness, ml/min Bendtsen	ISO 8791-2 (SCAN-P 21)	60	8,6	15,3
		250	18,2	8,0
		1300	106,5	8,4
				<b>10,6</b>
Roughness, µm PPS, at 1MPa	ISO 8791-4 (SCAN-P 76)	4	0,14	3,7
		8	0,27	4,7
				<b>4,2</b>
Air permeance, ml/min Bendtsen	ISO 5636-3 (SCAN-P 60)	40	3,9	9,7
		130	13,2	10,1
		500	42,7	8,4
		1400	124,3	8,9
				<b>9,3</b>
Air resistance, s (per 100 ml) Gurley	ISO 5636-5 (SCAN-P 19)	45	3,2	6,7
		95	4,7	5,0
		510	37,0	7,2
				<b>6,3</b>

Property	Standard method	Level	<i>s<sub>l</sub></i>	CoV <sub>1</sub> , %
Picking resistance, m/s IGT	ISO 3783 (SCAN-P 63)	0,5	0,09	27,8
		1,0	0,16	18,0
Thickness, µm (paper)	ISO 534 (SCAN-P 7)	50	1,8	3,5
		100	1,4	1,6
		250	3,2	1,3
		500	17,4	3,6
				<b>2,5</b>
Thickness, mm (corrugated board)	ISO 3034 (SCAN-P 31)	4	0,04	1,0
		7	0,03	0,5
			<b>0,04</b>	
ISO brightness, %	ISO 2470 (SCAN-P 3)	90	0,32	0,36
		85	0,29	0,34
		75	0,30	0,41
		60	0,33	0,55
		50	0,39	0,76
		5	(0,20)	5,12
			<b>0,33</b>	
Gloss (75 °)	ISO 8254-1	50	2,8	5,6
		80	1,7	2,1
Bending resistance, mN 15 °/10	ISO 2493 (SCAN-P 29)	30	2,3	8,1
		55	3,3	5,9
				<b>7,0</b>
Short span compression, kN/m	ISO 9895 (SCAN-P 46)	3	0,15	4,9
		6	0,24	4,3
		10	0,59	5,5
				<b>4,9</b>
Bending stiffness, mNm Resonance method	ISO 5629 (SCAN-P 64)	1	0,04	9,6
		6	0,26	4,1
		25	1,14	4,3
		375	20,3	5,4
				<b>5,9</b>
Folding endurance, N log (Köhler-Mohlin)	ISO 5626 (SCAN-P 17)	2,5	0,18	8,0
		3,5	0,04	1,1
Grammage, g/m <sup>2</sup>	ISO 536 (SCAN-P 6)	50	0,35	0,7
		100	0,43	0,4
		250	1,62	0,7
				<b>0,6</b>
Water absorption, g/m <sup>2</sup> Cobb	ISO 535 (SCAN-P 12)	20	0,9	4,2

## Annex D

### Examples of uncertainty between laboratories – pulp

#### D.0 Introduction

Interlaboratory testing of pulps is not so well established as for paper and board. This Annex presents examples of available data for the between-laboratory standard deviation for chemical pulp. For mechanical pulp, there is practically no data available from interlaboratory testing.

The normal routine when chemical pulps are tested is that the testing includes preparation of the test material, that is: disintegration, beating, consistency measurement, determination of SR-number, preparation of laboratory sheets (forming, pressing and drying) and the testing of the sheets.

In the normal procedure, the sample of pulp is divided into test portions that are beaten to different degrees (SR-number). The procedure above is completed for each beating degree. Interpolations are made within a drawn beating curve to obtain for instance tensile index at a given SR-number.

For each property a number of these operations will contribute to the between-laboratory uncertainty. For most properties the assumptions have to be specified (for instance SR-number, that can be measured without beating or after beating).

The term  $s_{bm}$  is practically never applicable in pulp testing.

$$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2 \quad [D.1]$$

The consequence is that it is either  $s_{bt}$  from internal control or  $s_l$  from interlaboratory comparisons that is available for estimation of uncertainty for pulp testing.

$$s_l^2 = s_{bt}^2 + s_{bl}^2 \quad [D.2]$$

In the tables below a number of examples are given for  $s_l$  and these will be the base for the total uncertainty,  $U_l = k \cdot s_l$ .

#### D.1 Example for SR-number

The data presented in Table D.1 are derived from CEPI-CTS data for five pre-test rounds between 1997 and 1999 (see Annex C).

In this case, laboratories have received samples of dry pulp and have slushed these pulps and determined the SR-number without any further treatment of the pulp. Each laboratory has carried out 5 tests, which means that the standard deviation and coefficient of variation presented in Table D.1 are valid for the average of 5 measurements.

Table D.1 Analysis of SR-number

Property	Level	$s_l$	CoV, %
SR-number	20	2,0	9,4
	30	4,3	14,4
	50	5,2	10,7
<b>Average</b>			<b>11,5</b>

#### D.2 Example for pulp properties

Table D.2 presents data from three rounds of the STFI comparative testing based on the PFI beating of two different pulps, one hardwood pulp and one softwood pulp. The sheets have been tested after the pulp has been beaten for a defined number of revolutions of the PFI-mill (hardwood = 3000 rev; softwood = 6000 rev).

Table D.2 Analysis of pulp properties

Property	Level	Pulp	$s_l$	CoV, %
Density, kg/m <sup>3</sup>	700	Softwood	17	2,3
	800	Hardwood	20	2,5
	Average			2,4
Tensile index, Nm/g	90	Softwood	7,0	7,8
	95	Hardwood	5,6	5,9
	Average			6,9
Tear index, mNm <sup>2</sup> /g	8	Softwood	0,51	6,4
	10	Hardwood	0,66	6,6
	Average			6,5

Table D.3 presents data for interpolated values based on a series of test portions beaten to different degrees. In some cases, the values are interpolated to a given SR number and in other cases to a given density.

Table D.3 Analysis of interpolated properties

Property	Level	Pulp	$s_l$	CoV, %
Density	710	Hardwood	27	3,8
	720	Softwood	13	1,8
Tensile index at 25 SR	60	Hardwood	8,0	13,3
	90	Softwood	5,8	6,4
Tear index at 25 SR	8	Hardwood	0,49	6,1
	12	Softwood	0,68	5,7
Tensile index at density 800	85	Hardwood	6,6	7,8
Tensile index at density 750	100	Softwood	5,4	5,4

## Annex E

# Reference material

### E.0 Introduction

This Annex provides recommendations concerning how to select, homogenise and randomise material for internal control within the laboratory and for inter-laboratory comparative testing.

### E.1 Requirements

In order to be able to estimate the long-term between-test variation in the measurement of a given property within a single laboratory, data are required within the laboratory from repeated tests on homogeneous material over a relatively long period of time (internal reference material). Similarly, in order to be able to estimate the between-laboratory variation in the measurement of a given property, data from tests on homogeneous material at a number of different laboratories are required (inter-laboratory comparative testing).

The reference material for each property should be selected according to two principles:

- the grade or grades shall represent the most frequently tested grades and property levels within the laboratory;
- the variations within the material should be as small as possible.

*Note* – To obtain data for paper grades which are liable to deteriorate, it may be better to choose a more permanent paper having the values of the properties concerned at the same level.

### E.2 Selection of homogeneous reference material

The following procedures are recommended to achieve as small variations as possible within the material.

#### *E.2.1 Paper reference material taken from a paper machine*

Make sure that the process is running under stable conditions. Select the reference material from a position in the middle of the paper machine web to avoid cross machine (CD) variations. Take a sufficient amount of paper for the number of properties and the time period that is needed.

Normally the paper is produced during such a short period of time that it can be considered to be homogeneous in the machine direction (MD). Test the relevant properties to make sure that they are at the

desired levels. Cut the paper into sheets of a suitable size. Store the material as described in E.4.2.

#### *E.2.2 Paper reference material taken in sheet form*

Sometimes it is necessary to take paper from commercial packages. This paper is normally packed in reams cut from several reels, and it is not certain that reels originate from the same jumbo roll. In such a case it is necessary to make a check to see whether any reel deviates from the others. Number e.g. the first 20 sheets and make measurements at a suitable number of test positions on each sheet. Examine the results to see whether there is a pattern, and identify any reel or reels which clearly differ from the others (this is a question of identifying major differences and not calculating statistical significances). Such a pattern will be evident in a four-sheet, five-sheet or six-sheet cycle, depending on the number of reels which have been cut simultaneously. Go carefully through the ream and reject all the sheets which come from the deviating reel(s). Store the material as described in E.4.2.

#### *E.2.3 Pulp reference material*

Make sure that the process is running under stable conditions. Take out a sufficient amount of pulp for the number of properties and the time period that is needed. Test the relevant properties to make sure that they are at the desired levels. Store the material as described in E.4.2.

### E.3 Randomisation of test material

Although the reference material selected according to E.2 is as homogeneous as it is possible to achieve by such procedures, further homogenisation by a procedure for statistical randomisation is necessary.

Take the selected reference material and prepare test material as described in the standardized method for the property concerned. (This may involve cutting test pieces, but it may in some cases involve cutting of raw pieces from which the actual test pieces are cut on the occasion when the test is to be carried out). Randomise all the test material in an appropriate manner to create a single statistical population. A detailed instruction of how this is to be done is outside the scope of this document, but care should be taken to ensure that the method adopted does indeed give a randomised test material.



#### **E.4 Stabilisation and storage of reference material**

##### ***E.4.1 Stabilisation of reference material***

Before use, the reference material must be stored in order to stabilise its physical properties. Test the material over a time period until the properties are stable. The time needed depends on the pulp or paper grade to be used. A detailed instruction of how this is to be done is outside the scope of this document.

##### ***E.4.2 Storage of reference material***

The reference material has to be kept at conditions where the influence on the properties over time is as little as possible. Preferably keep the reference material in a dark cupboard or a dark room.

The best atmosphere for storage of paper is the preconditioning climate. At no time should the humidity exceed 52 % RH, nor should the temperature be higher than 24 °C.

Pulp shall be stored in a cool room, if necessary in a dark plastic bag.

## Annex F

### Background to the selected approach

#### F.1 Error model

The recommendations given in “Guide to the expression of Uncertainty in Measurement”, reference 1 (9.1) are based on a model, which assumes that it is possible to express a relationship between the result of a test and all the input factors on which the result depends. In order to utilise this model it is thus necessary to be able to identify and measure the effects of all the factors which contribute to the result. It is also assumed that it is easier to estimate the individual contributions of these effects on the final results than to vary all the quantities on which the result of a measurement depends and determine the effect (GUM, section 3.4.1 (9.1)).

There are two particular features of the pulp and paper industry which merit comment. Firstly, many of the different methods provide results which are defined by the design characteristics of the instrument and the procedure for carrying out the test, so that it is impossible to identify a chain of factors linking the result to any primary standard. Secondly there is a long tradition of interlaboratory comparative testing and internal laboratory control routines which provide data that makes it possible to estimate uncertainty. A pre-requisite is that it is possible and inexpensive to store material in a manner, which conserves most properties and makes it possible and meaningful to repeat tests.

These considerations have led to the conclusion that it is more convenient to use this type of available data to estimate test uncertainty than to attempt to undertake the task of estimating the size and impact on the result of all conceivable disturbances for a large number of properties.

It is assumed that if this analysis shows large uncertainties, a special investigation is carried out in order to find the main contributors.

#### F.2 Selection of precision statement

ISO 5725 considers the two extreme conditions of variation between measurements, viz.: repeatability conditions and reproducibility conditions. It is however noted that intermediate conditions between these two extremes exist and that these can be of interest. (Section 0.4 in ISO 5725-1:1994(9.2)).

In the testing of pulp and paper it has been considered extremely important to be able to compare test results from different occasions from the same laboratory as well as from different laboratories. This is the background to the error model used in this Guideline.

The error model in ISO 5725 expresses the total variance,  $s_{Tot}^2$ , in term of two components as follows:

$$s_{Tot}^2 = s_r^2 + s_L^2 \quad [F.1]$$

where

- $s_r$  is the standard deviation between measurements under repeatability conditions;
- $s_L$  is the standard deviation between laboratories under reproducibility conditions.

This error model makes it possible to assign uncertainties to results for comparison with test results from other laboratories and for comparison with test results from the same laboratory performed under repeatability conditions.

The error model in this Guideline has the following components:

$$s_{tot}^2 = \frac{s_{bm}^2}{n} + s_{bt}^2 + s_{bl}^2 \quad [F.2]$$

where

- $s_{tot}$  is the standard deviation associated with the test result;
- $s_{bm}$  is the standard deviation between measurements made under repeatability conditions, as defined in this Guideline;
- $n$  is the number of measurements in a test,
- $s_{bt}$  is the standard deviation associated with the uncertainty between test occasions in the same laboratory;
- $s_{bl}$  is the standard deviation associated with the uncertainty between laboratories.

This error model makes it possible to assign uncertainties to results for comparison with test results from other laboratories, and also for comparison with test results from the same laboratory which have not been performed under repeatability conditions.

There are at least two reasons for adopting the type of error model used in this Guideline:

- The error components in the model are possible to estimate from internal control programs carried out within the laboratory.
- Comparison of client test result is often made between tests from the same laboratory performed on different occasions i.e. not under repeatability conditions.

**F.3 Comments on the definition of a test result**

The material variations within paper are large, and for this reason a number of test pieces are usually measured in one test. In this Guideline, the result of a test is defined as the average of these repeated measurements.

This means that the standard deviation between measurements is not an estimate of the repeatability of the test. The repeatability must be based on the differences between *test* results.

According to this definition, the repeatability is estimated by

$$\frac{s_{bm}}{\sqrt{n}}$$

where

$s_{bm}$  is the standard deviation between measurements;

$n$  is the number of repeated measurements in a single test.