#### PD ISO/TS 20460:2015



### **BSI Standards Publication**

Paper and board — Automated on-line testing — Metrological comparability between standardized measurements and output of on-line gauges



#### **National foreword**

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## TECHNICAL SPECIFICATION

ISO/TS 20460

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# Paper and board — Automated on-line testing — Metrological comparability between standardized measurements and output of on-line gauges

Papiers et cartons — Essais en ligne automatisés — Comparabilité métrologique entre mesures normalisées et résultats de jauges en continu



## PD ISO/TS 20460:2015 **ISO/TS 20460:2015(E)**



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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 6, *Paper, boards and pulps*.

#### Introduction

There are two concepts discussed in this Technical Specification, calibration and correlation. The calibration process compares the output of an instrument to primary standards having known measurement characteristics. Correlation, in the context of this Technical Specification, is the degree of association between the quality control laboratory and the online sensor. [20]

Online gauge users are looking to evaluate the capability (in a SPC "Statistical Process Control" sense) of their equipment. This is done usually through a measuring process which mixes a calibration process of the gauge itself, as presented in 4.1, and a correlation process with laboratory equipment, as presented in 4.2.

The requirements for online measuring equipment and its measurement process in the context of paper and board manufacturing is discussed in <u>4.1</u>. Usual acceptable tolerances for the instrument itself and for the process to be measured are given.

The gauge itself may regularly be verified automatically through a so-called "automatic standardization" or "internal standardization". A "static calibration" often refers to an operation during which the gauge is removed from the moving web. A "dynamic calibration" often refers to an operation on the moving web, either in a fixed position or traversing.

The requirements for periodic calibration procedures and for decision-making are given in <u>4.2</u>. This type of verification is a correlation or a comparison between online and off-line measuring system.

Results of actions of either 4.1, or 4.2, or both, may lead to a physical "calibration adjustment" of the gauge sensor and constitutes the metrological comparability.

Calculations of uncertainties are widely described in several ISO documents<sup>[18]</sup> and uncertainties linked to equipment are not within the scope of this Technical Specification.

Properties such as formation, fibre orientation, optical roughness and air permeance are measured widely with online gauges. For these properties, ISO standards for laboratory equipment do not exist or international reference materials are not available, and therefore testing of these properties are out of the scope of this document. However, it is recommended to use methodology requirements of  $\underline{4.1}$  and calibration procedure principles of  $\underline{4.2.1}$ .

## Paper and board — Automated on-line testing — Metrological comparability between standardized measurements and output of on-line gauges

#### 1 Scope

This Technical Specification establishes guidelines to link and, where applicable, calibrate the online gauge, following laboratory measurement for a given paper and board property.

Paper and board online measuring equipment is mostly based on different technology to that of laboratory equipment. Therefore, this Technical Specification specifies the International Standards to be chosen for the determination of physical properties of paper and board when measured online. It is applicable to all kind of paper and board.

In case of dispute, the usual reference is the laboratory testing but the parties may decide that the online measurements are valid based on the application of this International Standard.

#### 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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

ISO 287, Paper and board — Determination of moisture content of a lot — Oven-drying method

ISO 534, Paper and board — Determination of thickness, density and specific volume

ISO 536, Paper and board — Determination of grammage

ISO 1762, Paper, board and pulps — Determination of residue (ash) on ignition at 525 degrees C

ISO 2469, Paper, board and pulps — Measurement of diffuse radiance factor (diffuse reflectance factor)

ISO 8254-1, Paper and board — Measurement of specular gloss — Part 1: 75 degree gloss with a converging beam, TAPPI method

ISO 15397, Graphic technology — Communication of graphic paper properties

ISO 22514-1, Statistical methods in process management — Capability and performance — Part 1: General principles and concepts

ISO 22514-7, Statistical methods in process management — Capability and performance — Part 7: Capability of measurement processes

#### 3 Terms and definitions

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

#### ISO/TS 20460:2015(E)

#### 3.1

#### calibration

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with *measurement uncertainties* (3.7) provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a *measurement result* (3.5) from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called "self-calibration", nor with *verification* (3.15) of calibration.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99:2007, 2.39]

#### 3.2

## certified reference material CRM

reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, obtained using valid procedures

EXAMPLE Calibration service for Photometric calibration described in ISO 2469:2014, Annex A for the measurement of diffuse radiance factor (diffuse reflectance factor).

Note 1 to entry: "Documentation" is given in the form of a "certificate" (see ISO Guide 31:2000).

Note 2 to entry: Procedures for the production and certification of certified reference materials are given, e.g. in ISO Guide 33 and ISO Guide 35.

Note 3 to entry: In this definition, "uncertainty" covers both 'measurement uncertainty' and 'uncertainty associated with the value of a nominal property', such as for identity and sequence. "Traceability" covers both "metrological traceability (3.9) of a quantity value" and "traceability of a nominal property value".

Note 4 to entry: Specified quantity values of certified reference materials require metrological traceability with associated measurement uncertainty (Accredited Quality Assurance, 2006).[21]

Note 5 to entry: ISO/REMCO has an analogous definition (Accredited Quality Assurance, 2006) but uses the modifiers "metrological" and "metrologically" to refer to both quantity and nominal properties.

[SOURCE: ISO/IEC Guide 99:2007, 5.14]

#### 3.3

#### control chart

chart on which some statistical measure of a series of samples is plotted in a particular order to steer the process with respect to that measure and to control and reduce variation

Note 1 to entry: The particular order is usually based on time or sample number.

Note 2 to entry: The control chart operates most effectively when the measure is a process variable which is correlated with an ultimate product or service characteristic.

[SOURCE: ISO 3534-2:2006, 2.3.1]

#### 3.4

#### cumulative sum control chart CUSUM chart

control chart where the cumulative sum of deviations of successive sample values from a reference value is plotted to detect shifts in the level of the measure plotted

Note 1 to entry: The ordinate of each plotted point represents the algebraic sum of the previous ordinate and the most recent deviation from the reference, target or control value.

Note 2 to entry: The best discrimination of changes in level is achieved when the reference value is equal to the overall average value.

Note 3 to entry: The chart can be used in control, diagnostic or predictive mode.

Note 4 to entry: When used in control mode it can be interpreted graphically by a mask (e.g. V-mask) superimposed on the graph. A signal occurs if the path of the CUSUM intersects or touches the boundary of the mask.

[SOURCE: ISO 3534-2:2006, 2.3.5]

#### 3.5

#### measurement result

#### result of measurement

set of quantity values being attributed to a measureand together with any other available relevant information

Note 1 to entry: A measurement result generally contains "relevant information" about the set of quantity values, such that some may be more representative of the measurand than others. This may be expressed in the form of a probability density function (PDF).

Note 2 to entry: A measurement result is generally expressed as a single measured quantity value and a *measurement uncertainty* (3.7). If the measurement uncertainty is considered to be negligible for some purpose, the measurement result may be expressed as a single measured quantity value. In many fields, this is the common way of expressing a measurement result.

Note 3 to entry: In the traditional literature and in the previous edition of the VIM, measurement result was defined as a value attributed to a measurand and explained to mean an indication, or an uncorrected result, or a corrected result, according to the context.

[SOURCE: ISO/IEC Guide 99:2007, 2.9]

#### 3.6

#### measuring system

set of one or more measuring instruments and often other devices, including any reagent and material, assembled and adapted to give information used to generate measured quantity values within specified intervals for quantities of specified kinds

Note 1 to entry: A measuring system may consist of only one measuring instrument.

[SOURCE: ISO/IEC Guide 99:2007, 3.2]

#### 3.7

## measurement uncertainty uncertainty of measurement uncertainty

non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

Note 1 to entry: Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned quantity values of measurement standards, as well as the definitional uncertainty. Sometimes estimated systematic effects are not corrected for but, instead, associated measurement uncertainty components are incorporated.

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Note 2 to entry: The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a specified multiple of it), or the half-width of an interval, having a stated coverage probability.

Note 3 to entry: Measurement uncertainty comprises, in general, many components. Some of these may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the quantity values from series of measurements and can be characterized by standard deviations. The other components, which may be evaluated by Type B evaluation of measurement uncertainty, can also be characterized by standard deviations, evaluated from probability density functions based on experience or other information.

Note 4 to entry: In general, for a given set of information, it is understood that the measurement uncertainty is associated with a stated quantity value attributed to the measurand. A modification of this value results in a modification of the associated uncertainty.

[SOURCE: ISO/IEC Guide 99:2007, 2.26]

#### 3.8

## metrological comparability of measurement results metrological comparability

comparability of measurement results, for quantities of a given kind, that are metrologically traceable to the same reference

EXAMPLE Measurement results, for the distances between the earth and the moon, and between Paris and London, are metrologically comparable when they are both metrologically traceable to the same measurement unit, for instance the metre.

Note 1 to entry: See Note 1 to 3.9.

Note 2 to entry: Metrological comparability of measurement results does not necessitate that the measured quantity values and associated *measurement uncertainties* (3.7) compared be of the same order of magnitude.

[SOURCE: ISO/IEC Guide 99:2007, 2.46]

#### 3.9

#### metrological traceability

property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty

Note 1 to entry: For this definition, a "reference" can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

Note 2 to entry: Metrological traceability requires an established calibration hierarchy.

Note 3 to entry: Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed.

Note 4 to entry: For measurements with more than one input quantity in the measurement model, each of the input quantity values should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network. The effort involved in establishing metrological traceability for each input quantity value should be commensurate with its relative contribution to the measurement result.

Note 5 to entry: Metrological traceability of a measurement result does not ensure that the measurement uncertainty is adequate for a given purpose or that there is an absence of mistakes.

Note 6 to entry: A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and, if necessary, correct the quantity value and measurement uncertainty attributed to one of the measurement standards.

Note 7 to entry: The ILAC considers the elements for confirming metrological traceability to be an unbroken metrological traceability chain to an international measurement standard or a national measurement standard, a documented measurement uncertainty, a documented measurement procedure, accredited technical competence, metrological traceability to the SI, and calibration intervals (see ILAC P-10:2002).

Note 8 to entry: The abbreviated term "traceability" is sometimes used to mean "metrological traceability" as well as other concepts, such as "sample traceability" or "document traceability" or "instrument traceability" or "material traceability", where the history ("trace") of an item is meant. Therefore, the full term of "metrological traceability" is preferred if there is any risk of confusion.

[SOURCE: ISO/IEC Guide 99:2007, 2.41]

#### 3.10

#### reference material

#### RM

material, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties

Note 1 to entry: Examination of a nominal property provides a nominal property value and associated uncertainty. This uncertainty is not a *measurement uncertainty* (3.7).

Note 2 to entry: Reference materials with or without assigned quantity values can be used for measurement precision control whereas only reference materials with assigned quantity values can be used for *calibration* (3.1) or measurement trueness control.

Note 3 to entry: "Reference material" comprises materials embodying quantities as well as nominal properties.

EXAMPLE 1 Colour chart indicating one or more specified colours.

Note 4 to entry: A reference material is sometimes incorporated into a specially fabricated device.

EXAMPLE 2 Substance of known triple-point in a triple-point cell.

EXAMPLE 3 Glass of known optical density in a transmission filter holder.

EXAMPLE 4 Spheres of uniform size mounted on a microscope slide.

Note 5 to entry: In a given measurement, a given reference material can only be used for either calibration or quality assurance.

Note 6 to entry: The specifications of a reference material should include its material traceability, indicating its origin and processing (Accredited Quality Assurance, 2006).

[SOURCE: ISO/IEC Guide 99:2007, 5.13]

#### 3.11

#### repeatability conditions

observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in the same test or measurement facility by the same operator using the same equipment within short intervals of time

Note 1 to entry: Repeatability conditions include the following:

- the same measurement procedure or test procedure;
- the same operator;
- the same measuring or test equipment used under the same conditions;
- the same location;
- repetition over a short period of time.

[SOURCE: ISO 3534-2:2006, 3.3.6]

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#### 3.12

#### reproducibility conditions

observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in different test or measurement facilities with different operators using different equipment

[SOURCE: ISO 3534-2:2006, 3.3.11]

#### 3.13

### secondary reference standard secondary standard

measurement standard established through calibration with respect to a primary measurement standard for a quantity of the same kind

Note 1 to entry: Calibration may be obtained directly between a primary measurement standard and a secondary measurement standard, or involve an intermediate measuring system calibrated by the primary measurement standard and assigning a measurement result to the secondary measurement standard.

Note 2 to entry: A measurement standard having its quantity value assigned by a ratio primary reference measurement procedure is a secondary measurement standard.

[SOURCE: ISO/IEC Guide 99:2007, 5.5]

#### 3.14

#### **International System of Units**

#### SI

#### **System of units**

name adopted by the 11th General Conference on Weights and Measures (1960) for the recommended practical system of units of measurement; the base units are a choice of seven well-defined units: the metre, the kilogram, the second, the ampere, the Kelvin, the mole, and the candela

[SOURCE: ISO/IEC Guide 99:2007, 1.16]

#### 3.15

#### verification

provision of objective evidence that a given item fulfils specified requirements

Note 1 to entry: (CALA/AIHA-LAP, LLC) A procedure normally associated with the acquisition of data regarding an instrument to provide some indication as to whether it is operating within expected tolerances. For example, weights may be placed on a balance and the reading can provide some indication as to whether the balance is operating within expected tolerances. This operation should not be confused with *calibration* (3.1). Verification does not establish traceability. Verification seeks only to determine whether or not the instrument is operating within its expected tolerances. It is not a method of establishing the expanded uncertainty, which is the core issue in a calibration

Note 2 to entry: Be aware that manufacturer's tolerances, as provided in data sheets and instrument manuals, will use the same method of expression as an uncertainty, such as  $\pm 3\%$  or  $\pm 4$  grams. These are still only *tolerances* and should not be confused with the expanded *uncertainties* associated with the measurement result

EXAMPLE 1 Confirmation that a given *reference material* (3.10) as claimed is homogeneous for the quantity value and measurement procedure concerned, down to a measurement portion having a mass of 10 mg.

EXAMPLE 2 Confirmation that performance properties or legal requirements of a *measuring system* (3.6) are achieved.

EXAMPLE 3 Confirmation that a target measurement uncertainty can be met.

Note 3 to entry: When applicable, *measurement uncertainty* (3.7) should be taken into consideration.

Note 4 to entry: The item may be, e.g. a process, measurement procedure, material, compound, or measuring system.

Note 5 to entry: The specified requirements may be, e.g. that a manufacturer's specifications are met.

Note 6 to entry: Verification in legal metrology, as defined in VIML<sup>[22]</sup> and in conformity assessment in general, pertains to the examination and marking and/or issuing of a verification certificate for a measuring system.

Note 7 to entry: Verification should not be confused with *calibration* (3.1). Not every verification is a validation.

Note 8 to entry: In chemistry, verification of the identity of the entity involved, or of activity, require a description of the structure or properties of that entity or activity.

[SOURCE: ISO/IEC Guide 99:2007, 2.44]

#### 3.16

## working measurement standard working standard

measurement standard that is used routinely to calibrate or verify measuring instruments or measuring systems

Note 1 to entry: A working measurement standard is usually calibrated with respect to a reference measurement standard.

Note 2 to entry: In relation to *verification* (3.15), the terms "check standard" or "control standard" are also sometimes used.

[SOURCE: ISO/IEC Guide 99:2007, 5.7]

#### 4 Methodology requirements

#### 4.1 Requirements for the measuring equipment and its measurement process

The laboratory off-line equipment and associated ISO standard shall be considered the reference.

#### 4.1.1 General requirements for measuring systems

An organization should

- determine the monitoring and measurement to be undertaken and the monitoring and measuring equipment needed to provide evidence of conformity of product to determined requirements, and
- establish processes to ensure that monitoring and measurement can be carried out and are carried out in a manner that is consistent with the monitoring and measurement requirements.

NOTE 1 See for example ISO 9001:2008, 7.6.

When reporting measurement results in accordance with ISO 22514-1, it is important to include indicators about the quality of said result. Uncertainties in measuring the characteristics of interest should be evaluated and be at an acceptable level in relation to the specified acceptable range for the level of the property. This means that the employed measurement equipment should have sufficient metrological characteristics for the measurement task.

NOTE 2 A simplified procedure to estimate the uncertainty in measurements is to calculate the measurement process capability index  $C_{MP}$  as described in ISO 22514-7. Process capability index, noted  $C_{MP}$  describes the specification interval (upper specification limit minus lower specification limit) divided by the reference interval 6 $\sigma$  for a process in statistical control. The requirement for the measurement process capability index is a  $C_{MP}$  value not less than 1,33. A measurement process giving under 1,33 is usually regarded as unsatisfactory and should not be used in its current condition, as it is likely to disguise the process variation.

#### 4.1.2 Consistency of the online equipment itself

The online measuring equipment

- a) should be calibrated or verified, or both, at specified intervals or prior to use, against international or national measurement standards. Where no such standards exist, the basis used for calibration or verification shall be recorded, the stand-alone equipment being taken as the basis;
- b) shall be calibrated or verified, or both, at specified intervals or prior to use, against internal measurement standards; results shall be maintained,
- c) shall be adjusted or readjusted as necessary,
- d) shall have identification in order to display its calibration status, and
- e) shall, as a minimum, fulfil the repeatability of the standalone measuring system.

The capability ratio,  $Q_{MS}$ , for the measuring system is described in ISO 22514-7.  $Q_{MS}$  is defined as the expanded uncertainty  $4\sigma$  of the measuring system divided by the production process interval (upper limit minus lower limit). According to ISO 22514-7, it is recommended that the capability ratio of a measuring system based on the calculation of repeatability does not exceed 15 %.

#### 4.1.3 Consistency of the online measurement process

The following requirements for evaluation of the consistency of the measurement process shall be fulfilled.

a) Report graphically the results of the gauge reading of reference material.

These reference material measurements, consolidated versus time or sample number, may be understood as an evaluation of reproducibility. ISO 22514-7 describes the capability ratio  $Q_{MP}$  for the measuring process.  $Q_{MP}$  is defined as the expanded uncertainty  $4\sigma$  of measuring process divided by the production process interval (upper limit minus lower limit). According to ISO 22514-7, it is recommended that the capability ratio of a measurement process does not exceed 30 %. A study of the consistency of a measurement process requires a minimum of five samples and 30 measurements, and includes confirmation of the long term stability.

Another approach to evaluating reproducibility is through comparison of the results of the measurement process with information and data from other sources. As an example, through comparison of grammage results from an online gauge with those obtained from the weight of a jumbo reel.

- b) Draw an SPC (Statistical Process Control) control chart. A simplified CUSUM (cumulative sum) control chart should be used, as described in <u>Annex B</u>.
  - NOTE ISO 7870-4:2011 provides statistical procedures for setting up CUSUM schemes for process and quality control using variables (measured) and attribute data. It describes general-purpose methods of decision-making using CUSUM techniques for monitoring, control and retrospective analysis. [9]
- c) Set a maximum acceptable level for the deviation of the results of the measurement process and, if necessary, make adjustments to the online gauge.
- d) Possibly review the sampling size and intervals.

#### 4.2 Requirements for comparison of online and off-line equipment

#### 4.2.1 Calibration procedure principles

Metrological traceability for online gauges is not always possible, whereas it is the case for most standalone equipment. This section describes how to establish and document the comparability between the gauge and the stand-alone equipment. In practice a correlation is obtained between the online equipment

and the stand-alone laboratory equipment. This operation constitutes a metrological comparability and is described as a periodic calibration procedure being the best attempt to reach calibration.

The objectives of the procedure are the following:

- to improve the estimation of the deviation between a reference value and the value obtained using the online equipment, and the uncertainty in this deviation, at the time the instrument is actually used;
- to confirm the uncertainty of measurements made with the online equipment;
- to assess whether or not there has been any alteration of the online equipment which could introduce doubt about the results delivered in the elapsed period.

In this process, there are three independent types of errors: online equipment errors, laboratory equipment errors and sampling errors. An effective estimate of laboratory and sampling errors should therefore be conducted. A large sample set, which reduces the effect of random laboratory and sampling errors, should be used.

#### 4.2.2 Calibration procedure requirements

The following steps shall be fulfilled:

- a) position the gauge at a given location on the web, so that the gauge measures the same portion of paper as the sample which will be sent to laboratory;
- b) gather the relevant data given by the gauge;
- c) collect sample for off-line testing;
- d) measure the paper sample on the reference laboratory equipment;
- e) report graphically the difference between the online and laboratory equipment readings. An SPC (Statistical Process Control) control chart shall be used and a simplified CUSUM (cumulative sum) control chart should be used, as described in Annex B.
  - NOTE ISO 7870-4:2011 provides statistical procedures for setting up CUSUM schemes for process and quality control using variables (measured) and attribute data. It describes general-purpose methods of decision-making using CUSUM techniques for monitoring, control and retrospective analysis. [9]
- f) set a maximum acceptable level for the deviation of the results from the online and laboratory equipment and, if necessary, make adjustments to the online gauge;
- g) possibly review the sampling size and intervals.

#### 5 Methods to evaluate comparability

#### 5.1 Sampling operations, conditioning and stability

Sampling can be performed using hand or motor driven cutters. Before taking the sample, remove all damaged layers from the outside of the reel. Care shall be taken in handling samples as contact with bare hands can appreciably affect the chemical, physical, optical, surface or other characteristics of the paper or board. Use of cotton gloves is recommended during the sampling procedure. Samples shall be free from wrinkles, folds and dirt and protected from exposure to conditions which may change the relevant properties. Avoid contact with the surface that is to be tested and place the sample on a clean table if necessary.

Laboratory conditioning shall be set according to ISO 187 requirements. In case of automated testing, for quick feedback for the production line it is usually impracticable to condition according to ISO 187. Locate the automated off-line tester in the conditioned test room and allow sufficient time for the sample to partially condition before testing of properties sensitive to moisture content is carried out. The time required will depend on equipment available for rapid conditioning and the grammage of

the sample. It is desirable for preconditioning to be carried out as the first step. Usually the testing is started within 3-15 min from sampling in the machine hall. See also Reference [17].

Stability of the gauge is determined off-sheet by measuring secondary or transfer standards specific to the gauge. Charting these data shows whether or not the sensor is remaining stable.

#### 5.2 Basis weight

ISO 536 describes the procedure for laboratory measurement, which should be used as reference.

A usual correlation method is the template method, which uses a template to cut samples from the roll at reel turn-up. The samples are then weighed in the laboratory. An alternative method uses the actual weight of the reel core and the gross weight of the reel (core weight plus product) to compare it to the gross reel weight calculated by the Basis Weight sensor.

#### 5.3 Thickness

Thickness (caliper) is a measure of sheet thickness under specific conditions which include measurement head size and pressure.

ISO 534 describes the procedure for laboratory measurement, which should be used as reference.

Online thickness measurement cannot achieve the specifications in ISO 534.

Taking a sample whose thickness has been measured on laboratory equipment and placing it in the online gauge may be considered. There are problems with this approach. The moisture content will probably change when moving the sample from the laboratory to the online gauge. When measuring a moving web, various drag forces are applied to caliper planes, which are not present when measuring static samples. Therefore, metrological comparability and correlation is thus the only way to simulate a calibration.

#### **5.4** Moisture content

ISO 287 describes the procedure for laboratory measurement, which should be used as reference. Percent moisture determination is done by gravimetric method. Errors in laboratory determination of the moisture content of samples when taken from the paper machine can result from changes in moisture content during transportation to the laboratory and the pickup of moisture by the oven-dried sample before measurement of its mass.

Verification of moisture sensors is usually performed using a secondary or transfer standard. Glass-encapsulated samples of known moisture content can be used.

#### 5.5 Brightness, colour, whiteness, k and s

ISO 2469 describes the measurement principles for laboratory determination, which should be used as reference.

The geometry of online sensors is different to that of laboratory equipment used in papermaking. The ultraviolet content of the illuminant in the online sensor may also be different. Therefore, specific attention shall be paid to fluorescence calibration as described in the relevant ISO standards.

Metrological comparability and correlation is thus the only way to simulate a calibration.

Brightness measurements are described in ISO 2470-1 and ISO 2470-2.

Colour measurements are described in ISO 5631-1, ISO 5631-2, and ISO 5631-3.

Whiteness measurements are described in ISO 11475 and ISO 11476.

Basic calculations of k and s coefficients are described in ISO 9416.

ISO 15397 describes how to choose the best optical standard for a particular end-use and should be used as a reference.

NOTE 1 Equipment conforming to ISO 13655 has similar measurement geometry to that of online sensors.

NOTE 2 Optical properties of samples containing fluorophores will vary with the amount of fluorescence detected by the measuring system. Online emission measurements are directional and probe only a small solid angle of the total hemisphere, which is probed by laboratory instruments conforming to ISO 2469. The spatial distribution of radiation is expected to be different for the reflected part and the fluorescent part of the total radiance. Thus while response of an online instruments can be adjusted to a laboratory instrument using a particular sample, this adjustment may not be valid for a different sample.

Temperature and moisture content of the samples can be very different during online and off-line testing. This will have an influence on the amount of fluorescence emission at equal excitation. The magnitude of this effect is sample dependent. Again, any adjustment between online and off-line instruments is only valid for the samples and conditions used for adjusting.

#### 5.6 Ash content

ISO 1762 describes the procedure for laboratory measurement, which should be used as a reference.

Online ash sensors measure the inorganic content of a paper web or the inorganic coating applied to a paper web. The term "ash" relates to the laboratory method used to measure it. Ash sensors use X-rays or isotopic gamma rays to sense inorganic additives. When running a new grade that has a different mineral concentration, it may be necessary to recalibrate the sensor.

#### **5.7 Gloss**

ISO 8254-1 describes the procedure for laboratory measurement, which should be used as a reference. ISO 8254-2 and ISO 8254-3 may be used in specific cases.

Paper web generally exhibits a systematic difference between gloss measurements in the Machine Direction and the Cross Direction. The best practice here is to choose an online sensor, which matches the orientation of the paper mill's laboratory technique.

#### Annex A

(informative)

#### **Typical tolerances**

#### A.1 Grammage

Typical tolerances acceptable for in-house calibrations/verification:  $\pm 0.3$  g/m<sup>2</sup>.

Acceptable variation for process measurement to be compared with process variations:  $\pm 0.3$  g/m<sup>2</sup> in the range 50 g/m<sup>2</sup> to 120 g/m<sup>2</sup>.

Comparability below ±1 % is typical. If greater than this value, take action.

#### A.2 Thickness

Comparability of profiles of gauge versus laboratory is recommended.

For example, on a stack of 10 reel layers taken from the whole width web, test 40 points on width profile and compare graphically lab results and gauge results. Calculate averages of gauge profile and lab profile. Superimpose both curves. Accept a deviation outside the  $\pm 1~\mu$ . Count the number of deviations and accept below 5 points outside the range  $\pm 1~\mu$ m and act if above.

#### A.3 Moisture content

Comparability below ±0,6 % (absolute value) is typical. If greater than this value, take action.

#### A.4 Brightness, colour, whiteness

Brightness comparability below ±1 is typical. If greater than this value, take action.

L\*, a\*, b\* comparability below  $\pm 1$ ,  $\pm 0.2$  and  $\pm 0.5$ , respectively, is typical. If greater than these values, take action.

Whiteness comparability below ±2 is typical. If greater than this value, take action.

#### A.5 Ashes

Comparability below 1 % (absolute value) is typical. If greater than this value, take action.

#### A.6 Gloss

Comparability of profiles is recommended.

Comparability below ±2 is typical. If greater than this value, take action.

#### Annex B

(informative)

#### **Examples of graphical evaluations**

#### **B.1 Simplified CUSUM chart**

Calculate  $X_n$  as difference between gauge and targeted lab value. Usually, this difference is supposed to be 0.

Report graphically the cumulative sum, namely:  $X_n$ ,  $X_n + X_{n+1}$ ,  $X_n + X_{n+1} + X_{n+2}$ , etc.

This method is an efficient visual assessment of possible bias and decision is made easier.

This is a simplified method compared to traditional SPC CUSUM chart. Bilateral CUSUM charts give best results.

#### **B.2** XmR control chart

Calculate X as difference between gauge and targeted Lab value and report graphically X versus time.

Calculate mR (moving Range) being range within a subgroup of 2 consecutive values of X and report graphically mR versus time.

Make decisions based on usual SPC control chart limits rules.

Below rules (known as Western Electric rules) may be used to detect inconsistency:

- 1 point outside control limit (±3 s), s being standard deviation or control limits;
- 2/3 points outside 2 s;
- 4/5 points outside 1 s;
- 8 points at the same side of the central line.

#### **B.3** Profile range chart

Principle: Compare the thickness profile online to the profile from laboratory, measured on several sheets.

Sample a profile of 10 sheets on the full web width. Mark the sheet so that 40 measures are taken at equal distance from each other. The 10 sheets are meant to reduce the impact of MD variation.

The gauge profile is the average of 8 last gauge travelling.

Measure thickness of the 10 sheets plies. Divide by 10 for one sheet thickness.

Compare to the gauge data. Take the average thickness of the gauge and set it as zero. The average is rarely similar to the one from the lab, therefore only the profile is compared.

The target is to get as close as possible similar profile from gauge and laboratory. If differences are  $\pm 1~\mu m$ , count it for one significant difference (being above gauge uncertainty evaluated as  $\pm 1~\mu m$ ).

If the number of differences is above a given level, determined from previous statistics, consider the profile as out of control.

#### **Annex C**

(informative)

## Online gauges principles, limitations to correlations, sources of errors

#### C.1 General

This annex presents the technical limitations of the online gauges which complicate correlations and thus motivate use of comparability procedures that are described in this standard.

The sources of errors, linked to the principles on which gauges operate and to other possible errors, are also presented. This will help the analysis to reduce variations when using comparability procedures.

#### C.2 Online gauges principles

Online sensors have to be designed to withstand demanding conditions on the paper machine. The measurement repeatability and reproducibility of the sensor's (internal) calibration (often called the sensor's standardization) are the key factors which identify its measurement performance. Other constraints on the design of online gauges relate to the desire to maximize measurement performance, including the signal-to-noise ratio (SNR), for the property being measured and the particular local paper path and process conditions on the paper machine.

Generally the online sensors are installed in scanning heads mounted on C-frames or O-frames to measure the fast moving web at various locations on the paper machine. Most often the online sensors are located close to the reeling operation, before machine calendaring, if installed. For base paper measurements, the location is before the coating operation, where the web moisture may be extremely low, approaching  $1\,\%$ , and the web temperature very high, even approaching  $110\,\%$ C.

Many of the properties of the paper, such as moisture content and basis weight, are and can be related to those of a single sheet of paper. However optical properties as defined in International Standards, require measurements on a pile of sheets, theoretically an infinitely thick pile. This represents enduser conditions for paper in books and magazines. Stand-alone optical instruments can generally make measurements conforming to ISO standards as they can test a thick stack of sheets. Optical instruments in the form of online gauges, or installed in automatic laboratory instrument lines often cannot make measurements which conform to ISO standards as they are restricted to testing single paper sheets and estimating the properties of a stack of sheets using a non-standard method.

To obtain a correlation between an online sensor and an off-line instrument, the online sensor should have

— a method of self-calibration (also called a sensor's standardization) which often includes a mathematical method to adjust the measured value to a target value, and

NOTE 1 This commonly takes place hourly.

 a method of calibration to adjust the value obtained by the online sensor to the value of a traceable standard.

NOTE 2 This is done so that the value obtained conforms to those which would be obtained using an off-line, standalone instrument meeting the requirements of the relevant International Standard.

## C.3 Limitations of off-sheet calibration/static calibration/internal standardization

Online sensor measurements are correlated with those of off-line instruments perfectly in the ideal case. This is referred to as off-line correlation or static cross correlation between online and off-line measurements. For example, for moisture content glass encapsulated samples of known moisture content can be used. However the spectral properties of the glass may bias the measurement.

Unfortunately, for various reasons commonly used standards cannot be used for the calibration of online sensors. This is due to, factors restricting the selection or use of transfer standards.

The following are some factors that restrict the selection or use of transfer standards:

- legally required scanner safety interlocks as a result of there being a radiation source in the sensor;
- mechanical design limitations due to a restriction on the size of the sensor;
- the measurement principle of the online sensor which may require the sheet to be moving while the
  off-line laboratory measurement is made on a non-moving sheet;
- restrictions created by the paper path required to stabilize the paper and/or by the required vertical location of the sheet in the measurement gap of the scanner head;
- ambient conditions (moisture, temperature) in the paper machine hall which may differ greatly from those in the quality control laboratory.

Especially for optical measurements, there may be cases where off-line correlation (static cross correlation) has no meaningful metrological significance. The reason may be the following:

- the internal working standard(s) used in the online optical sensor is smaller than the diameter of the instrument's aperture required by the ISO standard;
- the thickness, translucency and/or opacity of the transfer standard differs considerably from that of the moving, single layer web.

#### C.4 Sources of errors during on-sheet calibration/dynamic (cross) correlation

As described in this Technical Specification, the correlation between online and off-line measurements is based on statistical online comparability. It is often called dynamic cross correlation. Here are some often overlooked issues which can affect this correlation:

- the effect of machine calendering or some other operation(s) after the online measurement is made but before sampling takes place;
- the structural properties of the paper;
- a high sheet temperature (50 °C up to 110 °C) versus the temperature in the quality laboratory (23 °C);
- the effectiveness/performance of Fluorescent Whitening Agents;
- thermochromic colourants;
- the moisture content of the paper. The moisture content of the paper in the atmosphere of the quality control laboratory is often about 6 % to 7 %, while for copy paper, for example, the online moisture content may be below 5 %;
- scattering within and on the top surface of the paper.

#### **C.5** Other common sources of errors

The following are other common sources of errors:

- sampling errors;
- a machine direction (MD) or cross direction (CD) offset;
- an unstable paper machine process;
- measurement errors in the online or off-line instrument;
- mishandling of calibration standards;
- laboratory errors;
- sample preconditioning;
- sample preparation;
- handling of sheets, the number of sheets and differences between the top and bottom sides of sheets.

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