BS ISO 2469:2014

BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of ISO 2469:2014. It supersedes BS ISO 2469:2007 which is withdrawn.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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

Papier, carton et pâtes — Mesurage du facteur de luminance énergétique diffuse (facteur de réflectance diffuse)

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Foreword

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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. www.iso.org/directives

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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, board and pulps*.

This fifth edition cancels and replaces the fourth edition (ISO 2469:2007), which has been technically revised.

Introduction

The radiance factor depends on the conditions of measurement, particularly the spectral and geometric characteristics of the instrument used. The diffuse radiance factor as defined by this International Standard is determined using instruments having the characteristics given in Annex A and calibrated according to the procedure specified in Annex B.

The diffuse radiance factor is the sum of the reflected radiance factor and the luminescent radiance factor, and the luminescent radiance factor of a luminescent (fluorescent) object is dependent on the spectral power distribution of the illumination. If adequately accurate measurements are to be carried out on fluorescent objects, the UV-content of the instrument illumination must therefore be adjusted to produce the same amount of fluorescence for a fluorescent reference standard as the selected CIE illuminant. The preparation of fluorescent reference standards to enable this adjustment to be made is described in Annex C. The use of these fluorescent reference standards is described in detail in the International Standards describing the measurement of the properties of the materials containing fluorescent whitening agents.

The spectral diffuse radiance factor or the weighted diffuse radiance factor applicable to one or several specified wavelength bands is often used to characterize the properties of pulp, paper and board. Examples of diffuse radiance factors associated with specified wavelength bands are the ISO brightness (diffuse blue radiance factor) and the luminance factor.

The diffuse radiance factor or diffuse reflectance factor is also used as the basis for calculating optical properties, such as opacity, colour, whiteness and the Kubelka-Munk scattering and absorption coefficients. These various properties are described in detail in specific International Standards, and for all of these, ISO 2469 is the primary normative reference.

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

1 Scope

This International Standard describes the general procedure for measuring the diffuse radiance factor of all types of pulp, paper and board. More particularly, it specifies in detail in Annex A the characteristics of the equipment to be used for such measurements, and in Annex B the procedures to be used for calibrating that equipment.

This International Standard may be used to measure the diffuse radiance factors and related properties of materials containing fluorescent whitening agents, provided that the UV-content of the instrument illumination has been adjusted to give the same level of fluorescence as a fluorescent reference standard for a selected CIE illuminant, in accordance with the specific International Standard describing the measurement of the property in question.

This International Standard describes in Annex C the preparation of fluorescent reference standards, although the procedures for using these standards are not included, since their use is described in detail in the specific International Standards describing the measurement of the properties of materials containing fluorescent whitening agents.

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 4094, *Paper, board and pulps — International calibration of testing apparatus — Nomination and acceptance of standardizing and authorized laboratories*

ASTM E308-06, *Standard Practice for Computing the Colors of Objects by Using the CIE System*

3 Terms and definitions

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

NOTE It is intended that these terms and definitions and their symbols be included in ISO/TR 10688, in order to have a single and common reference document for International Standards for measurement of optical properties of paper, board and pulps.

3.1
radiance factor
β
ratio of the radiance of a surface element of a body in the direction delimited by a given cone with its apex at the surface element to that of the perfect reflecting diffuser under the same conditions of illumination

Note 1 to entry: For luminescent (fluorescent) materials, the total radiance factor, β, is the sum of two portions, the reflected radiance factor, β_S, and the luminescent radiance factor, β_L, so that

$$\beta = \beta_S + \beta_L$$

For non-fluorescent materials, the reflected radiance factor, β_S, is numerically equal to the reflectance factor, R.

3.2
diffuse radiance factor
R
ratio of the radiation reflected and emitted from a body to that reflected from the perfect reflecting diffuser under the same conditions of diffuse illumination and normal detection

Note 1 to entry: The ratio is often expressed as a percentage.

Note 2 to entry: This International Standard prescribes diffuse illumination and normal detection in an instrument constructed and calibrated in accordance with the provisions of this standard. The term “diffuse radiance factor” is used here both for bidirectional and sphere geometries.

3.3
intrinsic diffuse radiance factor
R_∞
diffuse radiance factor of a layer or pad of material thick enough to be opaque, i.e. such that increasing the thickness of the pad by doubling the number of sheets results in no change in the measured radiance factor

Note 1 to entry: The radiance factor of a single non-opaque sheet is dependent on the background and is not a material property.

3.4
reflectance factor
ratio of the radiation reflected by a surface element of a body in the direction delimited by a given cone with its apex at the surface element to that reflected by the perfect reflecting diffuser under the same conditions of illumination

Note 1 to entry: The ratio is often expressed as a percentage.

Note 2 to entry: This term may be used only when it is known that the test material exhibits no luminescence (fluorescence).

3.5
diffuse reflectance factor
R
ratio of the reflection from a body to that from the perfect reflecting diffuser under the same conditions of diffuse illumination and normal detection

Note 1 to entry: The ratio is often expressed as a percentage.

Note 2 to entry: This International Standard specifies diffuse illumination and normal detection in an instrument constructed and calibrated in accordance with the provisions of this standard.

3.6
intrinsic diffuse reflectance factor
R_∞
diffuse reflectance factor of a layer or pad of material thick enough to be opaque, i.e. such that increasing the thickness of the pad by doubling the number of sheets results in no change in the measured reflectance factor

Note 1 to entry: The reflectance factor of a single non-opaque sheet is dependent on the background and is not a material property.

3.7
international reference standard of level 1
IR1
perfect reflecting diffuser (see CIE publication 17.4, No 845.04.54), ideal spectrally uniform isotropic Lambertian diffuser with a reflectance equal to 1 at all wavelengths

Note 1 to entry: Reflectance is defined as the ratio of the reflected to the incident radiation, see Annex E.

3.8
international reference standard of level 2
IR2
standard whose radiance (reflectance) factors have been determined by a standardizing laboratory in relation to the IR1 as defined by ISO 4094

Note 1 to entry: This International Standard refers to two types of IR2:

A non-fluorescent IR2 whose spectral reflectance factors have been determined by a standardizing laboratory in relation to the IR1. A non-fluorescent IR2 is used to calibrate the photometric scale of an authorized laboratory's reference instrument.

A white fluorescent IR2 whose spectral radiance factors corresponding to a specified CIE illuminant have been determined by a standardizing laboratory. A fluorescent IR2 standard is used to adjust the UV level of an authorized laboratory's reference instrument.

3.9
international reference standard of level 3
IR3
reference standard
standard whose radiance factors have been determined by an authorized laboratory in relation to an IR2, as defined by ISO 4094

Note 1 to entry: This International Standard refers to two types of IR3:

A non-fluorescent IR3 whose spectral reflectance factors have been determined by an authorized laboratory in relation to the IR2. A non-fluorescent IR3 is used to calibrate the photometric scale of a testing laboratory's reference instrument.

A white fluorescent IR3 whose calibration values have been determined by an authorized laboratory in relation to the IR2. A testing laboratory uses a fluorescent IR3 to adjust the relative amount of UV radiation incident on the sample to a specified level.

3.10
working standard
physical standard whose radiance (reflectance) factors have been determined by calibration with a suitable international reference standard (IR3) for subsequent use on a single instrument that conforms to this International Standard

3.11
primary working standard
working standard which is used routinely to validate and calibrate a given measuring instrument for its intended use

Note 1 to entry: The calibrated radiance (reflectance) factors of the primary working standard may not be transferred to a different instrument, even of the same type (see 3.10). However, it is possible to use a primary working standard for validation purposes only on instruments of the same type.

3.12
control plate
secondary working standard which is used on an infrequent basis to monitor and validate the performance of a given primary working standard

Note 1 to entry: When one or more control plates give anomalous results on a given instrument, it may be necessary to re-calibrate the primary working standard used with that instrument with an appropriate international reference standard (IR3).

4 Principle

A test piece is irradiated diffusely in a standard instrument and the light reflected (and emitted as a result of fluorescence) in a direction normal to the surface is passed to a detection system. This detection system may consist either of a defined optical filter and photodetector or of an array of photodetectors where each detector responds to a specific effective wavelength. The desired radiance factors are determined directly from the output from the photodetector in the former case or by calculation from the detector array outputs using appropriate weighting functions in the latter case.

5 Apparatus

5.1 Reflectometer, having the geometric, spectral and photometric characteristics described in Annex A.

5.2 Reference standards. For photometric calibration of the instrument and its working standards, a non-fluorescent reference standard issued by an authorized laboratory and fulfilling the requirements for an International reference standard of level 3 (see 3.9) as specified in Annex B.

Use reference standards sufficiently frequently to ensure satisfactory calibration.

NOTE If fluorescent materials are to be measured, a fluorescent reference standard issued by an authorized laboratory is required to enable the UV-content of the instrument illumination to be adjusted to produce the same amount of fluorescence as the selected CIE illuminant. This UV adjustment procedure is described in detail in Annex C. The use of these fluorescent reference standards is described in the International Standards for the determination of specific optical properties.

5.3 Working standards. For measurements on non-fluorescent materials, two working standards of opal glass, ceramic or other suitable material with flat surfaces.

NOTE In some instruments, the function of the primary working standard (see 6.3) may be fulfilled by a built-in internal standard.

For measurements on white fluorescent materials, stable fluorescent working standards of plastic or other material incorporating a fluorescent whitening agent are required. These working standards are described in the relevant International Standards.

5.4 Black cavity, for calibration or validation of the low end of the photometric scale. This black cavity shall have a radiance factor which does not differ from its nominal value by more than 0,2 percentage points at all wavelengths. The black cavity should be stored upside-down in a dust-free environment or with a protective cover. During calibration, the instrument shall be adjusted to the nominal value of the black cavity.

It is not yet possible to institute a system of reference standards to enable testing laboratories to check the reflectance factor of the black cavity. At the time of delivery, the level should be guaranteed by the instrument maker. Questions concerning the use and condition of the black cavity should be resolved by contacting the instrument maker.

6 Photometric calibration of the instrument and its working standards

6.1 Calibration of the instrument

Using the procedure appropriate to the instrument, calibrate the photometric scale of the instrument with an IR3 and, when the measurements are to be made on fluorescent materials, carry out a UV-calibration with a fluorescent IR3. Make a measurement on the IR3 in order to check that the calibration is satisfactory. The deviation between the measured and the assigned brightness and/or tristimulus values of the IR3 used for the primary calibration should not exceed 0,05 .

NOTE Although barium sulfate powders for pressing tablets are commercially available for which the absolute spectral radiance factors are given on the container, these values are not considered to be traceable according to the principles of modern metrology, and tablets based on barium sulfate powder are not considered to be suitable for use as an IR3 as required by this International Standard.

All calibrations are thus related to the IR1 through a calibration chain comprising an IR2 and an IR3 to which absolute values have been assigned respectively by a standardizing laboratory and by an authorized laboratory using an instrument conforming to this International Standard.

Handle each IR3 carefully and protect the test area from contamination. Store it in darkness, when not in use.

6.2 Calibration of the working standards for its intended use

Clean the working standards (see 6.4) and measure their radiance factors using the IR3 and read off and record the values to the nearest 0,01 percentage point. This calibration of the working standard is instrument-specific, for given conditions of measurement. The working standard shall only be used for subsequent calibration on the same instrument and for the same instrument conditions that it was originally calibrated.

NOTE In order to achieve agreement with the reference instrument, a working standard may be assigned multiple calibration values, depending upon the working level and the purpose of the measurement. This applies, for example, if the working standard is translucent or glossy and if the linearity of the instrument scale is poor so, in this case, the calibration is both sample and instrument specific.

6.3 Use of working standards

Use one plate as a primary working standard for checking and calibrating a given instrument, and use the other much less frequently as a control plate for checking the primary working standard. The frequency with which the instrument needs to be calibrated depends on the type of instrument. Frequent calibration of the instrument tends to introduce undesirable fluctuations in the instrument, and the instrument should be recalibrated only when a check with the primary working standard indicates that calibration is necessary. Check the primary working standard periodically against the control plate. If any change in the radiance factor is noticed, clean the primary working standard by the procedure described in 6.4. If the change persists, clean and recalibrate both working standards against an appropriate IR3 reference standard.

The primary working standard should be checked against the control plate sufficiently often to ensure that any change in the primary working standard is discovered before an error is introduced into the calibration.

6.4 Cleaning the working standards

Handle with care. If cleaning is necessary, follow the manufacturer's instructions. In the case of working standards of opal glass or ceramic material, rinse with distilled water and detergent free from fluorescent ingredients while rubbing with a soft brush. Rinse thoroughly in distilled water and dry in the air in a dust-free environment without allowing anything to touch the surface. Leave them in a desiccator until they are optically stable.

NOTE In the case of ceramic material standards, it is recommended to avoid getting water onto the back of the material, as the backing of a ceramic is very porous and may require days of drying in a dessicator to restore the optical properties.

7 Sampling

If the tests are being made to evaluate a lot, the sample should be selected in accordance with ISO 186.[1] If the tests are made on another type of sample, make sure that the test pieces taken are representative of the sample received.

8 Preparation of the test pieces

Prepare the test pieces according to the instructions given in the relevant International Standard for the determination of radiance factors or optical properties based on the measurement of radiance factors.

If it is desired simply to measure the radiance factor, rather than some other optical property defined by another International Standard, follow the following procedure.

Avoiding watermarks, dirt and obvious defects, cut rectangular test pieces approximately 75 mm × 150 mm, taking care to avoid touching the future test area.

If it is desired to measure the intrinsic radiance factor, assemble test pieces in a pad with their top sides uppermost; the number should be such that doubling the number of test pieces does not alter the radiance factor. Protect the pad by placing an additional sheet on both the top and bottom of the pad; avoid contamination and unnecessary exposure to light or heat. Mark the top test piece in one corner to identify the sample and its top side.

NOTE If the top side can be distinguished from the wire side, it should be uppermost; if not, as may be the case for papers manufactured on double wire machines, ensure that the same side of the sheet is uppermost throughout the pad.

If sufficient sheets are not available or if it is desired to measure a background-dependent radiance factor, select a suitable background and include a description of this background in the report.

9 Procedure

Determine the radiance factor as specified in the relevant International Standard for the determination of radiance factors or optical properties based on the measurement of radiance factors.

If it is desired simply to measure the radiance factor, rather than some other optical property defined by another International Standard, follow the following procedure.

9.1 Verification of calibration

Check the calibration of the instrument using a non-fluorescent working standard calibrated in relation to an IR3 (5.3). Recalibrate the instrument if necessary.

If the instrument is of the spectrophotometer type, and if the material to be measured contains or may contain a fluorescent component, the UV content of the illumination must be adjusted to match the fluorescence produced by the selected CIE illuminant using the fluorescent (5.2) and non-fluorescent (see 5.2) international level 3 reference standards in an iterative procedure. The procedure for UV-adjustment to match the CIE standard illuminant D65 is given in ISO 11475 and for UV-adjustment to match the CIE illuminant C in ISO 2470-1.

9.2 Measurement

Remove the protecting sheets from the test piece pad. Without touching the test area, use the procedure appropriate to the instrument, and the working standard, to measure the desired radiance factor. Read and record the value to the nearest 0,01 percentage points or better.

10 Calculation and expression of results

Express the radiance factor results with the number of decimals appropriate to the uncertainty and reproducibility of the procedure.

Calculate the results as required in the relevant International Standard for the determination of radiance factors or optical properties based on the measurement of radiance factors, e.g. ISO 2470-1,[2] ISO 2470-2,[3] ISO 2471,[4] ISO 5631-1,[5] ISO 5631-2,[6] ISO 5631-3,[7] ISO 9416,[8] ISO 11475,[9] ISO 11476.[10]

NOTE Some informative comments on the definition and calculation of the measurement uncertainty are given in Annex D.

11 Precision

Data relating to the precision of results obtained according to the procedure described in this International Standard are given in the relevant test method for the determination of radiance factors or optical properties based on the measurement of radiance factors. (See also Annex D).

12 Test report

The test report shall include the following details:

a) date and place of testing;

b) precise identification of the sample;

c) a reference to this International Standard;

d) the test results;

e) the wavelength range, pitch and bandwidth if a spectrophotometer is used, or the type of filter if a filter instrument is used;

f) the illuminant to which the UV-content of the illumination of the instrument is adjusted;

g) the number of test pieces and the procedure adopted to calculate the reported results;

h) the type of instrument used;

i) any departure from this International Standard or any circumstances or influences that may have affected the results.

Annex A
(normative)

Instruments for the measurement of radiance factor

The geometric, photometric and spectral characteristics of the instruments to which this International Standard applies are defined as follows:

A.1 Geometric characteristics

A.1.1 The test piece and reference area shall be subjected to diffuse illumination which is achieved by means of an integrating sphere (see Reference [14], 845.05.24) with an internal spectrally non-selective white diffusing surface and an internal diameter of (150 ± 3) mm.

A.1.2 The sphere shall be constructed as a dual-beam instrument so that a measurement can be made on a test piece, and a reference measurement can be made simultaneously on a reference region of the inner surface of the sphere.

A.1.3 The sphere shall be constructed or equipped with screens (baffles) to ensure that neither the test piece nor the reference region is directly illuminated by the light source.

A.1.4 The total area of the apertures and other non-reflecting areas in the sphere shall not exceed 13 % of the area of the inner surface of the sphere.

A.1.5 The receptor aperture shall be surrounded by a black annulus subtending a half-angle of (15,8 ± 0,8)° at the centre of the test piece aperture. This black annulus serves as a "gloss trap" so that specularly reflected light from the test piece does not reach the receptor. The black annulus shall be matt and shall have a radiance factor of less than 4 %, at all wavelengths within the visible region.

A.1.6 The test piece aperture shall be designed so that the test piece itself is essentially a continuation of the internal wall of the sphere. The rim of the test piece aperture shall have a thickness of (1,0 ± 0,5) mm including the thickness of the internal coating.

A.1.7 The measured test area on the test piece shall be circular with a diameter of (28 ± 3) mm.

NOTE It is expected that the use of the smaller area aperture will eliminate edge effects which can lead to a pseudo-nonlinearity, and that this will lead to a higher reproducibility between instruments.

A.1.8 The diameter of the aperture shall be larger than that of the test area (34,0 ± 0,5) mm, to ensure that no light reflected from the rim of the test piece aperture or from the test piece within a distance of 1 mm from the rim of the aperture can reach the detector.

A.1.9 The test piece shall be viewed normally, i.e. at an angle of (0 ± 1)° to the normal. Only reflected rays within a cone, the vertex of which is centred in the test piece aperture and the half-angle of which is not greater than 4°, shall fall on the receptor.

A.2 Photometric linearity

The photometric accuracy of the instrument shall be such that the residual departure from photometric linearity after calibration does not give rise to systematic errors exceeding 0,3 % radiance factor.

For the measurement of fluorescent papers, photometric linearity up to a total radiance factor value of at least 200 % is necessary in the wavelength region corresponding to the fluorescence emission.

A.3 Spectral characteristics

There are two main types of instrument which conform to this standard, known respectively as filter colorimeters and abridged spectrophotometers.

In the case of filter colorimeters, the spectral characteristics are determined by the filters inserted into the light beams in combination with the characteristics of the receptor, the sphere lining, the lamps and other optical parts of the instrument, or by a set of individual optical filters with different specific wavelengths. The filters shall be chosen so that the overall characteristics of the instrument agree with the spectral functions specified in the test methods relating to the determination of specific optical properties. The CIE recommended methods for characterizing these filter colorimeters are given in CIE Publication 179:2007.

In the case of abridged spectrophotometers, the spectral characteristics are determined by the accuracy to which the individual receptors represent the nominal wavelengths assigned to them, the band-width associated with each receptor, and the values given to the mathematical functions used in the subsequent calculations. For colorimetry, the instrument shall incorporate not less than 16 receptors uniformly spaced over at least the range from 400 to 700 nm.

In instruments providing spectral data, the manufacturer shall indicate the optical bandpass of the instrument. Ideally the colorimetric data shall be computed only from spectral data measured at wavelength intervals equal to the instrument's optical bandpass-width. The centroid wavelength of each band shall not differ from its nominal wavelength by more than ± 0,5 nm. However, in practical applications, it may be necessary to carry out the calculations using predicted rather than measured data at a wavelength interval of 10 or 20 nm depending upon the nominal instrument bandpass.

The spectral characteristics can be checked using suitable coloured reference standards.

A.4 Computational procedures

To calculate tristimulus values as specified by the CIE illuminant and standard observer functions (1931 or 1964), the appropriate tables of weighting factors presented in ASTM E308-06 [1)] for measurement at e.g. 10 nm or 20 nm intervals, shall be used. The tristimulus values shall be calculated by direct summation using these tabulated values with no attempt at interpolation using e.g. a cubic spline function. The actual tables of values to be used are given in the relevant test methods for determining specific optical properties.

The ASTM E308-06 tables to be used are those which assume that the spectral bandpass of the instrument used to obtain the data is equal to the measurement interval and is triangular in shape. These tables are to be used together with the data for which the manufacturer has identified the instrument bandpass as previously mentioned.

The instructions given in ASTM E308-06 shall be followed with regard to the summation of the tabulated values below 400 nm or above 700 nm if the measurement data do not cover the full extent of the tables.

If data are from an instrument with a triangular lineshape operating with a bandpass-width of 5 nm, this 5-nm data shall be convolved with a 10-nm or 20-nm triangular bandpass function to give data at 10-nm or 20-nm, respectively. If data from an instrument operating with a bandpass-width of 10 nm are to be presented and used at 20 nm intervals, the 10 nm data shall not be converted merely by taking the

1) Reprinted, with permission, from E 308-06 Standard Practice for Computing the Colors of Objects by Using the CIE System, copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. A copy of the complete standard may be obtained from ASTM (www.astm.org).

10 nm data at 20 nm intervals. If the original data are from an instrument with a triangular lineshape, the convolved data should preferably be calculated using a 3-point equation

$$R_{20}(\lambda_i) = \frac{1}{4} R_{10}(\lambda_i - \Delta\lambda) + \frac{1}{2} R_{10}(\lambda_i) + \frac{1}{4} R_{10}(\lambda_i + \Delta\lambda) \tag{A.1}$$

where $\Delta\lambda$ = 10 nm.

It should be noted that such a computation may still be only approximate. This is presented as a guideline to the procedure which is preferred but it should not be interpreted as a recommendation within the scope of this International Standard.

NOTE If data are from an instrument that does not have a triangular lineshape, then a 3-point convolution equation is not necessarily a good choice. For example, for data from an instrument using a two-monochromator method for the measurement of fluorescent IR2 standards, the instrument lineshape is nearly Gaussian and a 5-point equation may give better inter-instrument agreement.

A.5 UV-adjustment

For the measurement of materials containing fluorescent whitening agents, some means of setting the spectral power distribution of the incident radiation upon the test piece is required. The incident radiation must be set to a specified UV-content within the spectral range defined by the CIE. There must be a means to maintain this level or of mathematically simulating such a power distribution.

For this purpose, a filter having a half-peak cut-off wavelength of 395 nm shall be used or an equivalent procedure having the same impact shall be employed. If the filter is movable, it shall be mounted in a device which permits its position to be identified and maintained, and reproducibly reset.

NOTE The relative spectral power distributions of the CIE illuminants C, D50, and D65 are defined only for wavelengths longer than 300 nm.

A.6 Fluorescence elimination

For the measurement of radiance factors with the fluorescence effect eliminated, the instrument shall be equipped with a sharp cut-off UV-absorbing filter having a transmittance not exceeding 5 % at and below a wavelength of 410 nm and exceeding 50 % at a wavelength of 420 nm (i.e. a half-peak cut-off wavelength of 420 nm), or shall employ an equivalent procedure.

The cut-off filter shall have characteristics such that a reliable radiance factor value is obtained at 420 nm. This value shall be repeated at all lower wavelengths to provide adequate data for the colorimetric computations, provided that the International Standard for the quantity concerned does not include other instructions.

NOTE This procedure is equivalent to the ASTM E308–06 instruction to add the weighting functions if data for certain wavelengths are missing.

Instrument makers should recognize the need to provide the means to maintain at least three interchangeable and easily accessible calibrated situations, UV(D65) corresponding to the CIE standard illuminant D65, UV(C) corresponding to the CIE illuminant C, and UVex(420) corresponding to a fluorescence-eliminated (420 nm cut-off) situation.

Annex B
(normative)

Calibration service — Photometric calibration

In this International Standard, a sequence of non-fluorescent reference standards of three different levels is mentioned, the ultimate reference standard (the international reference standard of level 1) being the "perfect reflecting diffuser". The use of the perfect reflecting diffuser as ultimate reference is in full agreement with a recommendation made by the prime authority on optical properties, the *Commission Internationale de l'Éclairage* (CIE), in 1969.

To permit working instruments to relate their radiance factor measurements to this ultimate reference standard, the following procedure is specified.

B.1 Structure of the service

This calibration procedure involves two stages for fundamental reasons. The standardizing laboratory provides a transfer standard of level 2 with assigned radiance factor values directly *traceable to the perfect reflecting diffuser*. The authorized laboratories take this transfer standard and, with the help of a reference instrument conforming to this International Standard, mediate to industrial laboratories a reference standard of level 3 with assigned radiance factor values which are traceable to the perfect reflecting diffuser *through an instrument having the prescribed geometric characteristics*. Calibration at the industrial laboratory level with a transfer standard of level 2 is not in accordance with this standard.

B.2 Standardizing laboratories

Certain laboratories equipped for absolute radiance factor measurements are appointed as "standardizing laboratories" in accordance with the provisions of ISO 4094. The standardizing laboratories issue International reference standards of level 2 (IR2) to the authorized laboratories.

The standardizing laboratories are required to exchange reference standards of level 2 at intervals of no longer than five years, so that the level of agreement between their measurements is monitored and maintained.

B.3 Authorized laboratories

Laboratories having the necessary technical competence and maintaining reference instruments having the characteristics specified in Annex A of this International Standard are appointed as "authorized laboratories" in accordance with the provisions of ISO 4094. Each authorized laboratory maintains an instrument conforming to the requirements of Annex A as a reference instrument which is calibrated using a reference standard of level 2. The authorized laboratories then issue International reference standards of level 3 (IR3) on request to industrial laboratories which use the IR3 for the purpose of calibrating their instruments and working standards periodically.

The authorized laboratories are required to exchange reference standards of level 3 at intervals of no longer than two years. It is expected that this procedure will achieve those accuracies which are suggested in the "Expression of results" clause in the International Standards dealing with the determination of specific optical characteristics.

B.4 IR3 Non-fluorescent standards

The IR3 non-fluorescent reference standards shall have the following properties:

a) when properly cared for, their reflectance factors shall not change, within the accuracy of the instrument, over a reasonable period of time;

b) they shall be clean, opaque, and uniform in reflectance factor;

c) they shall be flat and shall have a smooth matte surface;

d) they shall be free from fluorescence.

B.5 Assignment of calibration values to IR3s

For the calibration of abridged spectrophotometers, the authorized laboratory shall provide reference standards having assigned reflectance factors obtained by direct measurement of the IR3 in the calibrated reference instrument.

For the calibration of three-filter colorimeters, the authorized laboratory shall provide reference standards having assigned R_x, R_y and R_z values. These shall be calculated for C/2° illuminant/observer conditions as follows (ISO/TR 10688):

$$R_x = (X - 0{,}167\,07\,Z)/78{,}321$$

$$R_y = Y/100 \qquad \text{(B.1)}$$

$$R_z = Z/118{,}232$$

where X, Y and Z are the tristimulus values, calculated according to A.4.

If a reference standard is required for the calibration of a three-filter colorimeter for measurement under D65/10° illuminant/observer conditions, the assigned $R_{x.10}$, $R_{y.10}$ and $R_{z.10}$ values shall be calculated as follows:

$$R_{x.10} = (X_{10} - 0{,}167\,47\,Z_{10})/76{,}841$$

$$R_{y.10} = Y_{10}/100 \qquad \text{(B.2)}$$

$$R_{z.10} = Z_{10}/107{,}304$$

where X_{10}, Y_{10} and Z_{10} are the D65/10° tristimulus values, calculated according to A.4.

Annex C
(normative)

Calibration service — UV-adjustment

For the measurement of fluorescent materials, special fluorescent reference standards are required to enable the relative UV-content in the illumination falling on the test piece to be adjusted to conform to the specified illuminant.

To enable this to be done, the following procedure is established.

C.1 Standardizing laboratories

A laboratory or laboratories equipped to make primary spectrofluorimetric measurements using the two-monochromator method are appointed as "standardizing laboratories" in accordance with the provisions of ISO 4094. This laboratory issues fluorescent International reference standards of level 2 (IR2) to the authorized laboratories. Such reference standards shall be assigned spectral total radiance factor data for the required illuminant.

C.2 Authorized laboratories

C.2.1 Laboratories having the necessary technical competence and maintaining reference instruments having the characteristics specified in Annex A of this International Standard are appointed as "authorized laboratories" in accordance with the provisions of ISO 4094.

NOTE It is anticipated that these authorized laboratories will be the same as those authorized in accordance with Annex B of this International Standard, but the standardizing laboratories will not necessarily be the same as those appointed according to Annex B since different equipment is required.

C.2.2 The authorized laboratory shall make any necessary adjustment to correct for differences in the basic photometric level between the instrument at the standardizing laboratory and the photometric level established at the authorized laboratory by the procedure described in Annex B, before calculating the appropriate property value for the IR2 and using this value to adjust the UV-content of the reference instrument. The calculations shall be carried out using 10 nm data and the weighting functions given in ASTM E308-06.

NOTE The property value required depends on the type of UV-adjustment being made, i.e. whether the adjustment is to conform to the CIE illuminant C or to the CIE illuminant D65. The exact details are given in the relevant International Standard.

C.2.3 The authorized laboratory shall take steps to ensure that directional effects in the IR2 which may affect the measurements at the standardizing laboratory are recognized and taken into account when determining the value to be used when transferring this calibration to an instrument providing diffuse illumination.

C.2.4 The authorized laboratories shall prepare International reference standards of level 3 (IR3) and shall supply these to industrial laboratories for the adjustment of the UV-level in their instruments.

C.2.5 The authorized laboratories are required to exchange reference standards of level 3 at intervals of no longer than two years, so that the level of agreement between their measurements is monitored and maintained.

C.3 IR3 fluorescent reference standards

C.3.1 The IR3 fluorescent reference standards shall consist of white paper uniform in radiance factor and aged for a sufficient time to give the paper an optical stability of 4-6 months without any deterioration greater than that permitted by the relevant International Standard.

C.3.2 The standards shall be prepared in the form of opaque pads and shall have a smooth non-glossy surface. The pad shall be covered with a suitable protective cover. The fluorescent reference standards shall be issued by an AL with assigned calibration values that correspond to the optical quantities of the relevant International Standard.

NOTE Fluorescent tablets and tiles are suitable local working standards but they have been shown not to be suitable for use as transfer standards for this procedure which is specific for white papers.

Annex D
(informative)

Measurement uncertainty

D.1 General

The presentation of the result of a measurement or test is not considered to be complete unless the numerical result is accompanied by a statement of the uncertainty associated with the result. This is particularly important in connection with ISO certification or accreditation under the terms of, e.g. ISO/IEC 17025, and this means that it is necessary to consider carefully what is meant by “uncertainty” and what information is required for the calculation of a statement of uncertainty.

If it is desired to state a measurement uncertainty of a reflectance factor value in relation to the perfect reflecting diffuser, it is necessary to calculate the cumulative effect of the uncertainties associated with each stage in the calibration and measurement procedure, starting from the declared uncertainty given by the standardizing laboratory. This information is however seldom required.

A major purpose of this International Standard is to reduce to a minimum the differences between the results of measurements made in different industrial laboratories by

a) providing a rigid specification of the geometrical and optical characteristics of the instrument to be used, and

b) specifying a routine for the calibration of such instruments.

It is implicit throughout this International Standard that the major need of the pulp and paper industry is for a procedure which gives not the greatest accuracy but the best possible inter-laboratory agreement, i.e. the best possible precision.

To achieve such an agreement, a number of authorized laboratories have been appointed to maintain designated reference instruments conforming to the requirements of this standard and calibrated with traceability to the perfect reflecting diffuser by transfer from one of the appointed standardizing laboratories. The specification that measurement in accordance with this International Standard requires calibration with an IR3 provided by such an authorized laboratory means that an industrial laboratory is *de facto* primarily concerned with an assessment of its measurement uncertainty in relation to the level provided by the authorized laboratory to which it is traceable, taking into consideration the level of agreement between the different authorized laboratories.

D.2 Measures of uncertainty

The uncertainty associated with a result is not a simple unambiguous concept. The term “uncertainty” may mean different things to different people in different circumstances. Among the different types of uncertainty which it is possible to define, the following merit consideration:

a) the uncertainty associated with the fact that the test material varies within itself, which is expressed with reference to the standard deviation of the measurements within a test and the confidence interval associated with the mean;

b) the uncertainty associated with the stability of the measurement device and test conditions, which is usually expressed with reference to the repeatability;

c) the uncertainty associated with the fact that another laboratory with a different instrument would be expected to give a different result, which is expressed with reference to the reproducibility;

d) the uncertainty associated with the probable deviation of the reported value from the true value, which is usually expressed with reference to the accuracy or trueness of the method.

The uncertainties a), b) and c) are all measures of the *precision* of the method.

D.3 Accepted reference value

In each case, the uncertainty is expressed in relation to the particular physical or hypothetical reference value that is accepted as being applicable to the particular circumstances which apply.

ISO 5725-1 defines and exemplifies the concept of the accepted reference value, as follows:

accepted reference value: value that serves as an agreed-upon reference for comparison, and which is derived as:

a) a theoretical or established value, based on scientific principles;

b) an assigned or certified value, based on experimental work of some national or international organization;

c) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group;

d) when a), b) and c) are not available, the expectation of the (measurable) quantity, i.e. the mean of a specified population of measurements.

It may be important to realize that in inter-laboratory transactions, the accepted reference value is not necessarily the perfect reflecting diffuser. It is more probably a level defined in relation to the mean level associated with the IR3's distributed by the authorized laboratories or in relation to the mean of some other specified population of measurements.

D.4 Role of the authorized laboratory

The optical calibration service described in Annex B of this International Standard is based on the concept of the *perfect reflecting diffuser* as the primary absolute standard, related to natural phenomena. A standardizing laboratory (SL) provides IR2s with assigned reflectance values and declares an uncertainty based on the combination of its estimated metrological errors. If an authorized laboratory (AL) is accredited in accordance with ISO/IEC 17025, it is obliged to give an uncertainty statement relating to the physical quantity reported, and this uncertainty shall include the uncertainty of the IR2 data and the uncertainty associated with the transfer procedure.

The optical calibration service described in Annex B nevertheless seeks to establish a system which not only transfers absolute data but also provides a potential for achieving a degree of precision and reproducibility among mill laboratories which is better than the SL uncertainty.

Experience has shown that the level of the values assigned by an SL is extremely stable, and that it shows a variation with time that is an order of magnitude less than the reported uncertainty in the measurement. In other words, the statement of uncertainty is an estimate of an unknown possible systematic error and is not in any sense an indication of an uncontrolled variability in the measurement.

Experience has also shown that a slight change in the level of the data provided by an SL, and in turn by an AL, can lead to large problems within the pulp and paper industries, even if such a change is well within the stated uncertainty of the measurements. In fact, the pulp and paper industries have come to consider the data supplied to them by the AL as having an absolute status.

This means that it is important that each AL shall not only report the total uncertainty in relation to the perfect reflecting diffuser, but also its own within-laboratory reproducibility. In addition, in order to make it possible for each industrial laboratory to assess its own uncertainty in terms of the expected between-industrial laboratory deviations, it is important that the AL's continually report the results of their inter-laboratory comparisons in terms of the between-AL variance. This can then be introduced as a component in each industrial laboratory's uncertainty calculation.

D.5 The calculation of uncertainties by industrial laboratories

The general model for an uncertainty statement is that the expanded uncertainty, U, is expressed as

$$U = \pm ks \tag{D.1}$$

where s is a standard deviation and k is a constant, usually equal to 2. This is essentially a statement that, with 95 % probability, the reported value is not expected to deviate by more than U from the accepted reference value. It also means that two independent measurements are not expected to deviate from each other by more than $\sqrt{2}U$.

If there are several different uncorrelated independent sources of error, the standard deviation s is calculated as the root mean square sum of the standard deviations of the various independent components.

In general, this means that an industrial laboratory which, for example, wishes to calculate the uncertainty associated with the value which it assigns to a product, should consider the following components:

a) the uncertainty of the IR3 standard provided by the AL;

b) the uncertainty in transferring the calibration;

c) the uncertainty within the laboratory associated with the repeatability and stability of the instrument and its calibration and the reproducibility between different operators on different occasions;

d) the uncertainty associated with the sampling procedure;

e) the uncertainty associated with variations within the sample tested;

f) the uncertainty due to laboratory conditions, and the instrument performance, etc.

It is not the ambition of this annex to provide information as to how these different components can be assessed through internal controls, inter-laboratory comparisons, etc.

D.6 Spectral reflectance factor values and weighted optical parameters

The calibration data and uncertainty data provided by the ALs are based on spectral reflectance factor values.

An industrial mill does not, however, usually specify the quality of its products in terms of spectral data. It is more usual to specify the value of a property such as ISO brightness or CIE whiteness which is a weighted mean of the spectral data. This means that it may be difficult to decide how to calculate uncertainties.

In fact, experience shows that, in a series of measurements on a single test piece, the repeatability of a weighted value such as an ISO brightness value or a tristimulus Y(C/2°) value is usually slightly better than the repeatability of the spectral reflectance factor values on which it is based. The variations in the individual reflectance factor values at different wavelengths are to some extent independent of each other so that the weighted value is more stable than its components.

More advanced mathematical techniques may be developed in the future for calculating uncertainties, but it should be remembered that in any trade context, it is important that both buyer and seller understand exactly what is meant by the uncertainty statement, and that it should be possible for both buyer and seller to agree upon the type of calculation to be employed.

Annex E
(informative)

Radiance and reflectance

In this edition of this International Standard it has been considered advisable to introduce the term radiance factor rather than reflectance factor into the title, because the increasing use of fluorescent whitening agents in papermaking means that the measurement is seldom limited to reflectance.

The radiance and the reflectance are not however defined in the same manner. The radiance is defined as the energy radiated from unit area of the material in a unit solid angle, whereas the reflectance is defined as the ratio of the reflected energy to the incident energy. The radiance has the units $cd \cdot m^{-2} \cdot sr^{-1}$, whereas the reflectance is dimensionless.

The radiance factor and the reflectance factor are, however, defined in an analogous manner. They are defined as the ratios of the radiation radiated or reflected respectively from the test material to that reflected by the perfect reflecting diffuser under the same conditions of illumination and detection. From a single measurement the instrument cannot distinguish between the two factors. For this reason, the same symbol, R, is here used for both properties.

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