INTERNATIONAL STANDARD ISO 3688

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Pulps — Measurement of diffuse blue reflectance factor (ISO brightness)

Pâtes — Mesurage du facteur de réflectance diffuse dans le bleu (degré de blancheur ISO)

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Descriptors : paper pulps, determination, reflectance factor, brightness, ISO brightness.

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3688 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*, and was circulated to the member bodies in April 1975.

It has been approved by the member bodies of the following countries :

Australia	Israel	South Africa, Rep. of
Belgium	Italy	Spain
Czechoslovakia	Mexico	Sweden
Egypt, Arab Rep. of	Netherlands	Switzerland
Finland	New Zealand	Turkey
France	Norway	United Kingdom
Germany	Pakistan	U.S.A.
Hungary	Poland	U.S.S.R.
Iran	Romania	

No member body expressed disapproval of the document.

This International Standard contains an annex which was submitted directly to the ISO Council in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

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Pulp — Measurement of diffuse blue reflectance factor (ISO brightness)

AMENDMENT 1

Amendment 1 to the International Standard ISO 3688-1977 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*, and was circulated to the member bodies in October 1979.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Romania
Belgium	Hungary	South Africa, Rep. of
Brazil	India	Spain
Canada	Italy	Sweden
Chile	Kenya	Switzerland
Czechoslovakia	Korea, Rep. of	United Kingdom
Egypt, Arab, Rep. of	Netherlands	USA
Finland	Norway	USSR
France	Poland	

No member body expressed disapproval of the document.

Page 1

Clause **2, References**

Replace the second reference by the following :

"ISO 5263, *Pulps — Laboratory wet disintegration*" and delete footnote 1).

Page 2

Sub-clause **5.4 Standard disintegrator** specified in ISO . . .

Replace by the following :

"**Standard disintegrator** specified in ISO 5263."

Sub-clause **7.1.1** *Pulp in sheets or compressed slabs*

Replace the existing text by the following :

"Split the sheets or slabs and tear them into 20 to 30 mm pieces. Weigh out the appropriate quantity of the pulp and disintegrate in water containing 0,5 ml of EDTA solution (4.1) per gram of pulp in the disintegration apparatus (5.4). The minimum number of disintegrator revolutions to achieve dispersal of the pulp, free of visible fibre clots or bundles, shall be used for the test. The number of revolutions given in ISO 5263 should not normally be exceeded. Dilute the suspension to 6 litres with water.

NOTE — Dry pulp shall be soaked for 0,5 h in water containing EDTA solution (4.1) before disintegration, so as to facilitate the separation of the fibres."

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Pulps — Measurement of diffuse blue reflectance factor (ISO brightness)

0 INTRODUCTION

The reflectance factor depends on the conditions of measurement, particularly the spectral and geometric characteristics of the instrument used. This International Standard should be read in conjunction with ISO 2469.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for measuring the diffuse blue reflectance factor (ISO brightness) of pulp.

This method is applicable to all wood pulps and to most other types of pulp. Very long-fibred pulps, such as those from unshortened cotton, flax and similar materials, should be reduced to suitable fibre length before testing by this method. This method is not applicable to pulps that contain added fluorescent materials.

NOTE — Addition of fluorescent materials to pulps is very rare; however, the lignin in some sulphite pulps exhibits a weak fluorescence. This is of no importance when measuring blue reflectance factor and can be tolerated.

2 REFERENCES

ISO 2469, *Paper, board and pulps — Measurement of diffuse reflectance factor.*

ISO . . ., *Pulps — Laboratory wet disintegration.*[1)]

3 DEFINITIONS

For the purpose of this International Standard, the following definitions apply.

3.1 reflectance factor, *R* : The ratio, expressed as a percentage, of the radiation reflected by a body to that reflected by a perfect reflecting diffuser under the same conditions.

3.2 intrinsic reflectance factor, R_∞ : The reflectance of a layer or pad of the material thick enough to be opaque.

3.3 diffuse blue reflectance factor (ISO brightness) : The intrinsic reflectance factor measured at an effective wavelength of 457 nm with a reflectometer having the characteristics specified in ISO 2469.

4 REAGENTS AND AUXILIARY MATERIALS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from colouring matter and from iron and copper ions.

4.1 EDTA (*di*sodium salt), 5 g/l solution

Dissolve 5 g of (ethylenedinitrilo)tetraacetic acid *di*sodium salt, dihydrate, (EDTA *di*sodium salt), $(C_{10}H_{14}O_8N_2Na_2.2H_2O)$ in 1 l of water.

NOTE — Some varieties of unbleached sulphite pulp have a pronounced tendency to redden when certain metal ions, such as copper or iron, are present. EDTA (*di*sodium salt) forms complexes with these ions and in most cases prevents the reddening, but does not diminish any reddening that may already have occurred. Hence, by adding this chemical to the distilled water before disintegrating the pulp, the brightness of the pulp in the condition in which it was received can be evaluated even though traces of discolouring metal ions are present.

4.2 Sodium hydroxide, approximately 1 N solution, containing 40 g of sodium hydroxide (NaOH) per litre.

4.3 Sulphuric acid, approximately 1 N solution, containing 28 ml of sulphuric acid (H_2SO_4, ρ 1,84 g/ml) per litre.

4.4 Thick filter paper, medium hard, fast filtering with a diameter of 110 mm, and free from fluorescent materials and soluble impurities.

4.5 Thin filter paper, very hard, slow filtering with a minimum diameter of 125 mm, and free from fluorescent materials and soluble impurities.

4.6 Blotters, suitable for interleaving and for absorbing water pressed from the test sheets, grammage approximately 250 g/m², and free from fluorescent materials and soluble impurities.

4.7 Disks made from chromium-plated metal, stainless steel or inflexible plastics [for example of poly(methyl methacrylate)], with a diameter of not less than 125 mm and a thickness of 1 to 1,5 mm.

1) In preparation.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Reflectometer calibrated against the reference instrument specified in ISO 2469 and equipped for the measurement of blue reflectance factor.

5.2 Filter, that, in conjunction with the spectral characteristics of the basic instrument, gives an overall effective wavelength of 457 ± 0,5 nm, and a bandwidth at half-height of 44 nm.

5.3 Two working standards calibrated against ISO reference standards of level 3 supplied by the authorized laboratory for blue reflectance factor standardization purposes.

Details of the calibration of the working standards together with cleaning precautions and use are given in ISO 2469. The working standards shall be calibrated by using ISO reference standards of level 3. In each case, recently calibrated reference standards intended for the calibration of the instrument for diffuse blue reflectance factor (ISO brightness) of pulp measurements shall be used at suitable intervals to ensure agreement with the reference instrument.

5.4 Standard disintegrator specified in ISO . . .

NOTE — All equipment with which the pulp comes into contact shall be of non-corrodible material, for example glass, porcelain, plastics, chromium-plated or stainless steel. Iron, copper, brass and bronze particularly shall be avoided, since iron and copper ions have a strong tendency to cause colour reversion in pulp. The distilled water, too, shall be free from iron and copper ions.

5.5 Büchner funnel of non-corrodible material (see the note in 5.4), the perforated bottom of which shall be flat, and having an internal diameter of 115 mm and a volume above the bottom of at least 500 ml.

5.6 Hydraulic disk-press.

5.7 pH meter.

6 SAMPLING

The sample from which the test pieces are taken shall be representative, and precautions shall be taken, during storage, to protect it from heat, light and change in moisture content.

A quantity of pulp sufficient for at least four test sheets having a grammage of approximately 200 g/m² is required. This corresponds to a pulp quantity of 2 g per test sheet (calculated on an oven-dry basis).

7 PREPARATION OF TEST SHEETS

7.1 Pretreatment of pulp

7.1.1 *Pulp in sheets or compressed slabs*

Split the sheets or slabs and tear them into 20 to 30 mm pieces. Weigh out the appropriate quantity of the pulp and disintegrate in water containing 1 ml of the EDTA (*di*sodium salt) solution (4.1) per test sheet, in the disintegration apparatus (5.4) as specified in ISO . . . Dilute the suspension to 6 l with water.

NOTE — Dry pulp shall be soaked for 0,5 h in water containing EDTA (*di*sodium salt) solution before disintegration, so as to facilitate the separation of the fibres.

7.1.2 *Slush pulp*

Determine the pulp concentration, measure out the appropriate volume, and add 1 ml of the EDTA (*di*sodium salt) solution (4.1) per test sheet.

7.2 Production of sheets

Using the pH meter (5.7), check that the pH of the pulp suspension obtained from the pretreatment is between 4,0 and 5,5. If not, adjust the pH to this interval with the sodium hydroxide solution (4.2) or the sulphuric acid solution (4.3).

NOTE — The brightness of unbleached pulps is affected by pH. For this reason, the measurement of ISO brightness shall be made on test sheets made from suspensions having a pH between 4,0 and 5,5.

Stir the suspension well and divide it into portions, each portion containing 2 g of pulp (oven-dry basis). Place a thick filter paper (4.4) in the Büchner funnel (5.5) and wet it with water. Place the funnel so that its bottom is horizontal and pour one portion of the pulp suspension into the funnel. Let the water drain under suction. Avoid drawing any appreciable amount of air through the sheet that is formed. Remove the sheet by turning the funnel upside down and by blowing into the stem and catching the sheet on a thin filter paper (4.5). Gently remove the thick filter paper and return it to protect the test sheet. Mark the top side of the sheet.

Proceed in the same way with at least three more portions of the pulp suspension. The number of test sheets shall be such that the ISO brightness of the pad of test sheets will not be changed by increasing its thickness. Four sheets will be sufficient for most types of pulp.

Arrange the disks (4.7), blotters (4.6) and test sheets for pressing in the following sequence, commencing from the bottom :

a) one metal or plastics disk;

b) two dry blotters;

c) the test sheet covered by the filter paper;

d) two dry blotters;

e) one metal or plastics disk;

f) two dry blotters;

g) the next test sheet covered by filter paper, etc.

Press the pack thus formed in the disk press (5.6) for 1 min so that a pressure of approximately 300 kPa is applied to the sheets (often different from the pressure gauge reading), having made certain they are centralized on the pressure platen before applying the pressure.

After pressing, loosen the filter papers from the test sheets, but leave them to protect the sheets. Dry the test sheet for 2,5 to 4 h to a moisture content of 5 to 15 % at room temperature by hanging them with the filter papers in a current of dust-free air.

Press the dried test sheets, protected by the filter papers, in the disk press (5.6) (300 to 500 kPa) for 30 s to make them as flat as possible.

Protect the test sheets from contamination and from unnecessary exposure to light or heat. Measure the ISO brightness of the test sheets immediately and in any case not more than 4 h after drying has been completed.

8 PROCEDURE

Remove the protecting filter papers and assemble the test sheets in a pad, with the top side uppermost. Check that the correct filters are in the light beams of the instrument. Without touching the test area, use the procedure appropriate to the instrument, and the working standards (5.3), to measure the intrinsic reflectance factor of the top side of the test sheet pad. Read and record the value of the reflectance factor to the nearest 0,1 %. Move the uppermost test sheet to the bottom of the pad and determine the diffuse blue reflectance factor to the next and similarly for the following test sheets until all the test sheets have been measured.

9 EXPRESSION OF RESULTS

Report the mean intrinsic reflectance factor as the diffuse blue reflectance factor (ISO brightness) of the pulp, in per cent, to the nearest 0,5 % reflectance factor.

10 PRECISION

At the present time, no information about the precision of the test can be given.

11 TEST REPORT

The test report shall include the following details :

a) precise identification of the sample;

b) a reference to this International Standard;

c) the results and the form in which they are expressed;

d) any particular points observed in the course of the test;

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

ANNEX

GENERAL INFORMATION ON ISO BRIGHTNESS AND REFLECTANCE FACTOR STANDARDS FOR PAPERS, BOARDS AND PULPS

(Not part of the standard)

In ISO 2469, ISO 2470, ISO 2471 and ISO 3688, which deal with measurements of diffuse reflectance factors, ISO brightness and opacity and opacity (paper backing), a sequence of reference standards of three different levels is mentioned in which, for diffuse reflectance factor measurements, the ultimate reference standard (the ISO standard of level 1) is the "perfect reflecting diffuser". The use of this ideal uniform diffuser, with a reflectance equal to 1,0, constitutes a deviation from the older practice of using smoked magnesium oxide as ultimate reference. However, 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) which replaced smoked magnesium oxide by the perfect reflecting diffuser in 1969.

It appears that with this change a reference standard which is difficult to produce (magnesium oxide) is now replaced by a reference standard which probably can never be physically materialized. However, there are good reasons for this regulation. The preparation of a smoked magnesium oxide surface is a slow and tedious process which produces reference standards of low precision. A survey of the literature shows that the reflectances of magnesium oxide surfaces prepared in different laboratories vary by about 2 %. Such uncertainty in the ultimate reference cannot be tolerated if instruments are available which can measure relative reflectance factors with a precision of the order of 0,1 %. Reference to the perfect reflecting diffuser is equivalent to absolute measurements of reflectance factors and the techniques of such measurements have been improved in recent years to an accuracy which is of the order of ± 0,3 % and better[1]. Consequently it is possible to calibrate material standards in such absolute reflectometers to an accuracy which is far superior to the accuracy of smoked magnesium oxide standards[2].

For the implementation of this ultimate reference standard or "ISO reference standard of level 1" = IR 1 and the reference standards of levels 2 and 3, ISO proposes the following procedure.

Certain laboratories, which are equipped for absolute reflectance factor measurements, are appointed by ISO/TC 6 as "standardizing laboratories". These laboratories issue "ISO reference standards of level 2" = IR 2 to certain "authorized laboratories" for calibrating their "reference instruments". These authorized laboratories, which are also appointed by ISO/TC 6, then issue "ISO reference standards of level 3" = IR 3 on demand to industrial laboratories which are advised to use the IR 3 only for the purpose of calibrating their working standards periodically.*

The standardizing laboratories are requested to exchange samples from time to time so that agreement between their measurements is maintained. The same holds for the authorized laboratories. It is expected that this procedure, which is specified in certain ISO documents, will achieve those accuracies which are suggested in the "Expression of results" clause in the above-mentioned International Standards.

It should be mentioned that barium sulphate powders for pressing tablets are commercially available for which the absolute spectral reflectance factors are given on the container. These values are determined with care but they are valid only if the procedure of pressing the tablets is very close to that of the laboratory which determined these values.

One consequence of this conversion in the ultimate reference is that diffuse reflectance factors, for example the ISO brightness, when referred to the perfect reflecting diffuser, are lower by about 1,0 to 1,5 % than those referred to smoked magnesium oxide. It is very important that this fact be borne in mind, not only in commercial transactions, but in general whenever various measurements on one sample are to be compared. Measurements according to the above International Standards are always referred to the perfect reflecting diffuser. Consequently, "ISO brightness" can only be an absolute value, never relative to smoked magnesium oxide. However, if reflectance factors are given without the ISO prefix, it is advisable to mention the reference by a qualifying "absolute" or "MgO = 100".

Opacity measurements are, of course, almost unaffected by the change in the ultimate reference.

Two aspects of these International Standards must be kept in mind :

1) The term "diffuse" refers to a diffuse illumination on the sample which is achieved by means of an integrating sphere. It is important to recognize that other specifications, such as TAPPI 452, use a different geometry and that, in general, another geometry will yield different values.

2) The instruments described in these International Standards are equipped with a "gloss trap" so that the specularly reflected component is excluded. It is important to observe this condition because, for samples exhibiting gloss, the introduction of the gloss trap may cause an additional decrease of the value of the reflectance factor by up to 1 %.

* The up-to-date lists of standardizing and authorized laboratories are available from the Secretariat of ISO/TC 6 (AFNOR) or from the ISO Central Secretariat.

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