INTERNATIONAL STANDARD ISO 2469

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

Paper, board and pulps — Measurement of diffuse reflectance factor

Papier, carton et pâtes — Mesurage du facteur de réflectance diffuse

Second edition — 1977-02-15

UDC 676.017 **Ref. No. ISO 2469-1977 (E)**

Descriptors : paper, paperboards, pulps, tests, optical tests, reflectance, diffuse reflection.

Price based on 7 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 2469 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*, and circulated to the member bodies in September 1971.

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

Australia	India	South Africa, Rep. of
Austria	Iran	Spain
Belgium	Ireland	Sweden
Bulgaria	Israel	Switzerland
Canada	Italy	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
Finland	Norway	U.S.A.
France	Poland	U.S.S.R.
Germany	Portugal	
Hungary	Romania	

No member body expressed disapproval of the document.

This second edition, incorporating a new annex D which was submitted directly to the ISO Council in accordance with clause 6.12.1 of the Directives for the technical work of ISO, cancels and replaces the first edition (ISO 2469-1973).

Printed in Switzerland

Paper, board and pulps — Measurement of diffuse reflectance factor

0 INTRODUCTION

The reflectance factor depends on the conditions of measurement, particularly the spectral and geometric characteristics of the instrument used. The diffuse reflectance factor is determined using instruments having the characteristics given in annex A.

Measurements of reflectance factor need to be made to a high degree of accuracy. The only practical means of achieving this is by calibration using ISO reference standards of level 3. It is, therefore, essential that for each country, or group of countries, there shall be appointed a standardizing laboratory that will issue ISO reference standards of level 2 at prescribed regular intervals (see annex D). The standardizing laboratories shall, by means of the interchange of ISO reference standards of level 2, check their results and agree upon the values to be assigned to these reference standards with respect to the ISO reference standard of level 1. This method is entirely dependent on these arrangements being established within and between countries.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the equipment and preliminary procedures for measuring the diffuse reflectance factor of pulp, paper and board.

Measurements of diffuse reflectance factor can be used for the evaluation of optical properties such as diffuse blue reflectance factor (ISO brightness), the scattering coefficient of pulp, opacity, whiteness, luminous reflectance factor, the chromaticity co-ordinates of paper and the intrinsic reflectance factor of non-fibrous materials. By means of filters or other equipment the spectral characteristics of the instruments are adjusted according to the properties to be measured. Numerical values optically characterizing the materials are calculated specifically for each property determined.

2 DEFINITIONS

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

2.1 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 condition.

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

2.3 ISO *reference standard of* level 1 (IR 1)[1] : The perfect reflecting diffuser (CIE 45-20-195)[2]. Ideal uniform diffuser with a reflectance equal to 1.

2.4 ISO reference standard of level 2 (IR 2) : Standard whose reflectance factor has been determined by a standardizing laboratory in relation to the IR 1. These standards are used by the authorized laboratories for the calibration of their reference instrument.

2.5 ISO reference standard of level 3 (IR 3) : Standard measured by an authorized laboratory against an IR 2. These standards are used by the working laboratories for the calibration of their instruments.

3 PREREQUISITES

3.1 Reference instruments

To obtain reproducible values of the reflectance factor it is essential that the reference instruments used by the authorized laboratories shall be maintained in perfect working order. Their characteristics are given in annex A. It is vital that these reference instruments shall be kept in calibration by the interchange of ISO reference standards of level 3.

1) *The ISO reference standard nomenclature given in 2.3 to 2.5 is consistent with the present state of International Standard ISO . . ., which is still under study.*

2) CIE (Commission Internationale de l'Éclairage), *International Lighting Vocabulary*, 3rd Edition, for CIE definitions.

3.2 ISO reference standards of level 2

To set the upper end of the scale of the reference instruments plane, ISO reference standards of level 2 with known intrinsic reflectance factors are required. For these reference standards materials such as freshly prepared tablets of barium sulphate or plates of high reflectance, high opacity opal glass may be used.

The preparation of pressed tablets of barium sulphate is described in annex C.

3.3 ISO reference standards of level 3

The working instruments shall be calibrated against a reference instrument. For this purpose ISO reference standards of level 3 are distributed by authorized laboratories (see annex B). These reference standards shall cover various working ranges and spectral conditions of the instruments.

3.4 Working standards

These may be ground surface opal glass plates that can be used for the routine adjustment of working instruments.

4 APPARATUS

4.1 Reflectometer having the same essential geometric, spectral and photometric characteristics as the reference instruments described in annex A.

4.2 Two opal glass plates with ground surfaces, calibrated with respect to ISO reference standards of level 3, or of level 2 and level 3, for use as working standards. To ensure the highest degree of accuracy the reflectance factors of each IR 3 should be kept within the general range of samples to be measured; the opal glass plates may be assigned different values depending upon the working region and the purpose of the measurement.

4.2.1 *Calibration of the working standards*

The calibration of the working standards is performed with either ISO reference standards of level 3, or level 2 and level 3.

4.2.1.1 CALIBRATION WITH RESPECT TO ISO REFERENCE STANDARDS OF LEVEL 3 ALONE

All calibrations are basically referred to ISO reference standards of level 3 which have been measured by an authorized laboratory against an IR 2 in relation to an IR 1.

The filters prescribed for the measurement are inserted into the light beams. Using the procedure appropriate to the instrument, read off and record to the nearest 0,1 % reflectance factor of one of the working standards with respect to each IR 3. Calculate the mean of these readings. The range of such a set of values shall not exceed 0,3 % reflectance factor. Repeat this procedure for the second working standard.

Calibrate the cleaned working standards against the ISO reference standards of level 3 and use recently calibrated reference standards frequently enough to ensure satisfactory calibration.

NOTE – Handle each IR 3 carefully and protect the test area from any contamination. Keep them in darkness, preferably in plastics envelopes.

4.2.1.2 CALIBRATION WITH RESPECT TO AN ISO REFERENCE STANDARD OF LEVEL 2 AND ISO REFERENCE STANDARDS OF LEVEL 3

The filters prescribed for the measurement are inserted into the light beams. Using the procedure appropriate to the instrument, measure the reflectance factor of both working standards against the IR 2. Read and record the reflectance factor of the working standard to the nearest 0,1 % reflectance factor.

When removing a barium sulphate standard from the test piece aperture, remove with a clean dry cloth any powder adhering to the contact surface. Otherwise it may be transferred to the working standard subsequently applied.

At suitable intervals check with respect to IR 3 reference standards, that the instrument has remained in calibration with the reference instrument.

4.2.2 *Use of working standards*

Use one plate as a working standard and the other as a control plate for the working standard. Check the working standard daily against the control plate. It is of the utmost importance that the opal glass plates used as working standards shall be cleaned before the daily check. If there is any change in the reflectance factor, clean it by the procedure described in 4.2.3. If the change persists, recalibrate both plates against IR 3 reference standards.

4.2.3 *Cleaning the working standards*

Rinse with distilled water, rubbing with a soft brush (with synthetic fibre bristles) and detergent free from fluorescent ingredients. Rinse thoroughly in distilled water and dry by blotting with filter paper. Leave in a desiccator until stable.

5 SAMPLING

The sampling procedure, which will depend on the purpose of the measurement, shall be agreed between the parties concerned.

6 PREPARATION OF TEST PIECES

Instructions for the preparation of test pieces are given in the relevant test methods for determining optical properties, based on measurements of reflectance factor.

7 PROCEDURE

Determine the reflectance factor as specified in the relevant test methods for the determination of optical properties, based on measurements of reflectance factor.

8 EXPRESSION OF RESULTS

Calculate and report the results as stated in the relevant test methods for optical properties, based on measurement of reflectance factor. [See :

– ISO 2470, *Paper and board – Measurement of diffuse blue reflectance factor (ISO brightness);*

– ISO 2471, *Paper and board – Determination of opacity (paper backing) – Diffuse reflectance method;*

– ISO 3688, *Pulps – Measurement of diffuse blue reflectance factor (ISO brightness).*]

ANNEX A

THE REFERENCE INSTRUMENTS FOR THE MEASUREMENT OF REFLECTANCE FACTOR[1)]

The responsibility for maintaining and operating the reference instrument shall, by agreement with ISO/TC 6, be delegated by each country, or group of countries, to an authorized laboratory. The authorized laboratories shall keep their reference instruments in calibration with one another by the exchange of IR 3 reference standards. The essential geometric, photometric and spectral characteristics of these instruments are defined as follows :

A.1 GEOMETRIC CHARACTERISTICS

Diffuse illumination of the sample and reference is effected by means of an integrating sphere (CIE 45-30-135).[2)]

The sum total of the areas of the apertures in the sphere does not exceed 10 % of the area of the sphere.

The receptor aperture is surrounded by a black annulus of external diameter subtending a half-angle of 15 1/2 ± 1/2° at the centre of the test piece aperture,

No light reflected from the rim of the test piece aperture shall reach the receptor.

The measured test area on the test pieces is circular with a diameter not less than 30 mm.

The sample is viewed normally. Only reflected rays within a solid cone, whose vertex is in the test piece aperture and of half-angle not greater than 4°, should fall on the receptor.

A.2 PHOTOMETRIC CHARACTERISTICS

The accuracy of the photometer, whether mechanical or electronic, is such that the residual departure from photometric linearity after calibration does not give rise to systematic errors exceeding 0,3 % reflectance factor.

A.3 SPECTRAL CHARACTERISTICS

The spectral characteristics are determined mainly by the filters inserted into the light beams. They are also modified by the characteristics of the receptor, the sphere lining and other parts of the instrument. Since the filters to be used are specified in the relevant test methods for testing optical properties based on measurements of reflectance factor, the overall characteristics are not given here.

A check of the spectral characteristics can be made with suitable coloured IR 3 reference standards or filters. IR 3 reference standards containing suitable fluorescent dyes can be used to check the spectral condition of the sphere lining which shall be renewed when necessary.

NOTE – Since the ultra-violet content of the spectral power distribution of the radiation incident on the test piece is less than that of daylight, fluorescent dyestuffs – if incorporated in the paper – may not be fully excited. The method may be used to evaluate such papers, but will not necessarily agree with assessments under different conditions of illumination.

ANNEX B

CALIBRATION SERVICE

The authorized laboratories will, upon agreement, distribute ISO reference standards of level 3 with reflectance factors accurately established with the reference instrument(s).

The IR 3 reference standards shall have the following properties :

a) when properly cared for, their reflectance factor 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 have a smooth surface, which, except for special applications, shall not have a high gloss;

d) they shall be as free as possible from fluorescence, except for those standards specifically chosen to check the spectral characteristics.

1) A suitable instrument is that manufactured by Carl Zeiss, Oberkochen, Federal Republic of Germany, under the trade name Elrepho. This **must be provided** with a black annulus.

2) CIE (Commission Internationale de l'Éclairage), *International Lighting Vocabulary*, 3rd Edition, for CIE definitions.

ANNEX C

THE BARIUM SULPHATE ISO REFERENCE STANDARD OF LEVEL 2

All standardization based on barium sulphate shall be carried out with compressed barium sulphate tablets made from powder having a certified spectral reflectance factor for an optical geometry corresponding to that given in clause A.1. The certificate shall be issued by a standardizing laboratory (see annex D).

C.1 APPARATUS AND MATERIALS

C.1.1 Barium sulphate. It is essential that the barium sulphate shall be of the highest purity, and suitable for the purpose. Its reflectance factor shall be checked between standardizing laboratories.

C.1.2 Powder press[1], capable of forming approximately 12 g of barium sulphate into a compressed tablet, approximately 45 mm in diameter and 5 mm thick. The tablets shall be supported in suitable holders and the working surface formed in contact with a glass plate having a matt surface.

The glass plate must be etched with hydrofluoric acid after matt polishing. The design of the powder press shall be such that the surfaces in contact with the tablet do not rotate during its formation and that after compression the tablet in its holder can be removed undamaged from the press.

C.1.3 Porcelain spoon.

C.1.4 Soft brush with synthetic fibre bristles.

C.1.5 Synthetic detergent, free from fluorescent ingredients.

C.1.6 Acetone, analytical grade.

C.2 PREPARATION OF THE BARIUM SULPHATE TABLET

Clean meticulously and dry all parts of the powder press and accessories. Use the porcelain spoon when handling the powder. Determine the number of spoonsful required to give the correct thickness intially by weighing; subsequent weighing is then unnecessary.

The tablet is formed in the powder press as determined by the press design. When the tablet in its holder has been removed from the press and the working surface exposed by removing the glass plate against which it is formed, the condition of the tablet is checked under grazing incident light. The tablet shall be completely smooth and have no glossy areas, depressions or elevations.

Prepare two or three such tablets. It is important that the various parts of the device be cleaned after the preparation of each tablet. This applies particularly to the matt glass surface. Use synthetic detergent in distilled water. Brush, then rinse carefully and thoroughly in distilled water and finally flush with acetone, and air dry. The brush shall be used solely for this purpose and shall be protected from contamination when it is not in actual use.

It is of the utmost importance to keep the barium sulphate stock bottle closed except during removal of powder. Powder once removed from the bottle shall not be put back.

The reflectance factor of tablets that are visually satisfactory shall be checked for uniformity. Comparison of the reflectance factors of the tablets shall show that they agree within 0,1 % reflectance factor; a greater range indicates unsatisfactory preparation.

C.3 PROPERTIES OF BARIUM SULPHATE TABLETS

C.3.1 Reflectance factors of tablets correctly made from a single stock bottle of barium sulphate shall not differ from one another by more than 0,1 % reflectance factor.

C.3.2 Reflectance factors of tablets from a stock bottle of barium sulphate shall not differ from the value stated on the bottle.

C.3.3 Tablets shall show no detectable change in reflectance factor within 2 weeks of production, when protected from contamination by dust, etc. by storing for instance in a Petri dish.

C.3.4 The reflectance factors of barium sulphate tablets shall not change with moderate changes in atmospheric humidity.

NOTE – Correctly prepared tablets have spectral reflectance factor characteristics as stated by the standardizing laboratory in relation to the IR 1 reference standard over the visible range of 400 to 700 nm.

1) A suitable powder press is manufactured by Carl Zeiss, Oberkochen, Federal Republic of Germany, under the trade name Powder Press 45.

ANNEX D

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 %.

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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