
**Paper, pulp, and recycling —
Decolouration test of dye coloured
paper products and paper products
printed using dye inks**

*Papier, pâte et recyclage — Essai de décoloration des produits papier
colorés en masse et des produits papier imprimés au moyen d'encre à
colorants*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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This document was prepared by Technical Committee ISO/TC 6, *Paper, Board and Pulps*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Large scale recycling of paper and board is now used worldwide since recycled fibres make up approximately half of the fibre resources needed for global production.

Graphic paper products, such as newspapers, magazines, office paper, etc., are approximately one third of the paper recycled in Europe, being about to 15 million tons. They are normally recycled by using a deinking process, for removal of printing inks. The basic feature is the detachment of the ink film from the substrate, its fragmentation into a suitable particle size and its removal from the pulp slurry. The latter is done predominantly by flotation, in special cases substituted by a washing process. For higher qualities of the deinked pulps, deinking is complemented by oxidative and/or reductive bleaching in order to further enhance the optical properties.

The quality of the pulp obtained is characterized by its brightness/colour, cleanliness, mechanical properties and ash content. It depends both on the type of paper and board for recycling being recycled and the treatment applied.

A very small percentage of graphic paper products is produced using dye inks and dyed papers, which are typically not able to be removed by deinking, but can be brightened and decoloured in the bleaching stage(s).

This document provides a laboratory test method for the decolouration capability of products, thus allows assessing a dyed paper or a graphic product printed with dye inks. On industrial scale, bleaching can act as a supplement to deinking but not as a substitute. The major reason is that raw material for deinking is nearly always a mix of paper grades and printing as well as converting technologies. Bleaching is only effective for a small portion of it.

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Paper, pulp, and recycling — Decolouration test of dye coloured paper products and paper products printed using dye inks

WARNING — Use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with their use. It is the responsibility of the user of this document to take appropriate measures to ensure the safety and health of personnel prior to application of the document and to determine the applicability of any restrictions for this purpose.

1 Scope

This document defines a method for measuring the decolouration capability of commercial products, produced using paper and board dyed in mass and/or printed with dye inks.

This document does not apply to paper printed with pigment inks.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 186, *Paper and board — Sampling to determine average quality*

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

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

ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method*

ISO 5635, *Paper — Measurement of dimensional change after immersion in water*

ISO 5637, *Paper and board — Determination of water absorption after immersion in water*

ISO 8787, *Paper and board — Determination of capillary rise — Klemm method*

ISO/TR 10688, *Paper, board and pulps — Basic terms and equations for optical properties*

ISO 11475, *Paper and board — Determination of CIE whiteness, D65/10 degrees (outdoor daylight)*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

**3.1
bleaching**

removal or modification, to a greater or lesser extent, of coloured components of pulp with a view to increasing its brightness (blue reflectance factor)

[SOURCE: ISO 4046-2:2016, 2.9]

**3.2
decolouration**

chemical destruction of colour

**3.3
pad**

pulp deposited on a filter paper and dried on laboratory apparatus in order to obtain an opaque layer

**3.4
DRI decolouration index**

value indicating the efficiency of the *decolouration* (3.2) action performed, %

**3.5
trimulus values**

X, Y, Z

X_{10}, Y_{10}, Z_{10}

amounts of the three reference colour stimuli, in a given chromatic system, required to match the stimulus considered

Note 1 to entry: Depending on the observer conditions, the tristimulus values are represented differently.

Note 2 to entry: In ISO 5631-1^[7], the CIE 1931 (2°) standard observer and the CIE illuminant C are used to define the trichromatic system.

Note 3 to entry: In ISO 5631-2^[8], the CIE 1964 (10°) standard observer and the CIE standard illuminant D65 are used to define the trichromatic system.

Note 4 to entry: In ISO 5631-3^[9], the CIE 1931 (2°) standard observer and the CIE illuminant D50 are used to define the trichromatic system.

Note 5 to entry: The tristimulus values (X, Y, Z) without subscript are used for the CIE 1931 (2°) standard observer. The subscript 10 is used for the CIE 1964 (10°) standard observer

[SOURCE: ISO/TR 10688:2015, 2.32]

**3.6
CIELAB colour space**

three-dimensional approximately uniform colour space, produced by plotting in rectangular coordinates L^* , a^* , b^* quantities defined by the formulae given in ISO/TR 10688:2015, 3.7

Note 1 to entry: The quantity L^* is a measure of the lightness of the test piece, where $L^* = 0$ corresponds to black and $L^* = 100$ is defined by the perfect reflecting diffuser. Visually, the quantities a^* and b^* represent respectively the red-green and yellow-blue axes in colour space, such that

- + a^* is a measure of the degree of redness of the test piece,
- a^* is a measure of the degree of greenness of the test piece,
- + b^* is a measure of the degree of yellowness of the test piece, and
- b^* is a measure of the degree of blueness of the test piece.

[SOURCE: ISO/TR 10688:2015, 2.5, modified — "If both a^* and b^* are equal to zero, the test piece is grey" removed.]

4 Symbols and abbreviations

DRI (%): decolouration index in percent.

DTPA: pentetic acid or diethylene triamine pentaacetic acid.

5 Principle

After re-slushing coloured and/or printed paper according to a specified method, the pulp obtained is bleached using two processes and pads are made. A decolouration index is determined by optical measurements on the pads obtained by each of the bleaching processes specified in this document.

6 Apparatus

6.1 Pulp preparation material

Mixer with a recommended capacity of 5 l able to re-slush paper for recycling at a high stock concentration (ca. w/v 20 %).

NOTE A Hobart N50¹⁾ type apparatus is suitable.

6.2 Reductive bleaching with sodium hydrosulphite

- Balance, with an accuracy of 0,1 g;
- pH meter;
- Glass jars, which can be sealed hermetically ("preserving jar" type);
- Water bath heating to (50 ± 2) °C.

6.3 Oxidative bleaching with hydrogen peroxide

- Balance, with an accuracy of 0,1 g;
- pH meter;
- Strong plastic bag;
- Water bath heating to (70 ± 2) °C.

6.4 Preparation of pads

- Büchner-type filtration equipment with a 150-mm diameter and filtration flask;
- 1-litre beakers;
- Heater plate (95 ± 2) °C or drying section of a Rapid-Köthen handsheet former as specified in ISO 5269-2;
- Pre-weighed, to an accuracy of 0,1 g, 150-mm diameter filter paper. The filter shall be capable of retaining particles with a circle equivalent diameter greater than or equal to 25 µm.

1) Hobart pulper N 50 is the trade name of a product and can be obtained at Hobart GmbH, Robert-Bosch-Straße 17, 77656 Offenburg. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

NOTE Whatman 589/1²⁾ or equivalent filter papers are suitable.

- Blotting paper: manufactured from 100 % bleached chemical pulp, neutral pH, and containing no bonding agent, chemical additive, impurity or fluorescent contamination. It shall be at least 3 mm larger than the filter paper in each direction. The grammage shall be (250 ± 25) g/m²; the water absorption capacity, determined according to ISO 8787, shall be (70 ± 20) mm and the change in dimensions after immersion, measured according to ISO 5635, shall not exceed 3 % in each direction. The grammage after immersion, determined following the procedure described in ISO 5637, shall be (450 ± 50) g/m²;
- Agitator equipped with turbine stirrer able to operate at 2 000 rpm;
- pH meter;
- Roller for pressing the pads, with a roll width of approximately 20 cm and mass of between 10 kg and 15 kg (a Cobb roller, defined in ISO 535^[2] may be used for this purpose).

6.5 Optical measurements

Reflectometer or **spectrophotometer**, with the geometrical, spectral and photometric specifications described in ISO 2469 and calibrated according to ISO 2469 and ISO 11475.

7 Reagents

7.1 Reductive bleaching with sodium hydrosulphite

- Sodium hydrosulphite (= dithionite) as powder;
- 4 N · H₂SO₄;
- Chelating agent (some commercial dithionite products already contain a stabilizer. In this case, no chelating agent is needed), DTPA type (i. e. solution with a specified concentration of 40 % of commercial product);
- Nitrogen;
- Tap water at room temperature.

7.2 Oxidative bleaching with hydrogen peroxide

- Hydrogen peroxide H₂O₂, the peroxide concentration shall not exceed 40 g/l (unstable above this limit);
- DTPA-type chelating agent;
- NaOH (caustic soda) in tablet form or a solution at a concentration of 20 %;
- standard sodium silicate;
- Tap water at room temperature.

7.3 Preparation of pads

- High molecular weight, low cationic charge polyacrylamide (flocculant) prepared at 1 g/l (powder dissolved in water);

2) Whatman 589/1 is the trade name of a product and can be obtained at www.analytix-shop.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

- Bulk solution of $\text{Al}_2(\text{SO}_4)_3$ at 330 g/l in water;
- Tap water at room temperature.

8 Sampling

If tests are performed to evaluate a lot, the sample shall be chosen according to ISO 186. If tests are performed on another type of sample, make sure that the test specimen taken is representative of the sample of recycled paper containing dye received.

In all cases precautions shall be made to avoid any change in moisture content of the material that will be tested.

9 Procedure for preparation of pulp for optical measurements

9.1 Pulp preparation

Conditions for re-slushing are as follows:

- Stock concentration: w/v 20 %;
 - Temperature: 50 °C;
 - Pulping duration: 30 min.
- a) Determine the moisture content of the specimen according to ISO 638;
 - b) Weigh approximately 200 g OD of paper, note the exact mass and cut into pieces of approximately 2 cm × 2 cm;
 - c) Preheat the pulper to (50 ± 2) °C with hot water;
 - d) Re-slush thoroughly at a concentration of w/v 20 % by adding water at (50 ± 2) °C;

NOTE For example, the ISO 21993^[6] protocol can be followed, increasing the re-suspension time but without adding chemicals.

- e) After 30 minutes, set aside part of the pulp for testing prior to bleaching (approximately 30 g dry weight) and the other part for bleaching.

9.2 Bleaching the pulp

The pulp prepared in this way is then separated into two lots and each batch is bleached according to one of the two following procedures:

- Reductive bleaching with sodium hydrosulphite, follow the procedure described in [9.3](#);
- Oxidative bleaching with hydrogen peroxide, follow the procedure described in [9.4](#).

9.3 Reductive bleaching with sodium hydrosulphite

Bleaching conditions are as follows:

- Stock concentration: w/v 3 %;
- Temperature: 50 °C;
- Duration: 45 min;
- pH before bleaching process, $\text{pH} = 6,8 \pm 0,2$;