
**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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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, $\text{pH} = 6,8 \pm 0,2$;

- 1 % sodium hydrosulphite (expressed as pure product in relation to the dry pulp quantity to be adjusted depending on the purity of the product);
- 0,5 % DTPA (if needed, expressed as pure product in relation to the dry pulp quantity to be adjusted depending on the purity of the product).
- a) Weigh $(30 \pm 0,5)$ g or (60 ± 1) g OD of the pulp and place it in a glass jar that seals hermetically (a 1-litre or 2-litre preserving jar);
- b) Dilute the pulp with hot water (50 °C) to a concentration close to 3 % (leave some space for stirring and nitrogen bubbling);
- c) While stirring, adjust the pH with sulphuric acid and add the chelating agent if needed;
- d) While stirring, bubble nitrogen into the glass jar for 5 min to 10 min to remove the air present;
- e) Prepare a fresh solution of w/v 10 % of sodium hydrosulphite; sodium hydrosulphite breaks down very rapidly in air and water, so the product used shall be recent, dry, and have been properly stored;
- f) Pour the prepared hydrosulphite solution into the jar as quickly as possible, stop stirring, fill the glass jar to the brim with the rest of the water at 50 °C and close it;
- g) Place the jar carefully into a temperature controlled water bath at (50 ± 2) °C for 45 minutes, stirring from time to time;
- h) After 45 min, open the jar and proceed to make the pads; take the usual precautions when handling chemicals and using apparatus.

Sodium hydrosulphite titration is described in [Annex A](#).

9.4 Oxidative bleaching with hydrogen peroxide

9.4.1 Bleaching conditions

- Stock concentration: w/v 15 %;
- Temperature: 70 °C;
- Duration: 90 min;
- 1 % NaOH (expressed as pure product);
- 1 % H₂O₂ (expressed as pure product);
- 2,5 % sodium silicate (expressed as commercial product);
- 0,3 % DTPA-type chelating agent (expressed as commercial product).

9.4.2 Preparing the bleaching liquor

Put in a glass beaker, in the order indicated:

- a) Tap water at room temperature of (20 ± 2) °C;
- b) NaOH;
- c) Sodium silicate;
- d) DTPA chelating agent;
- e) H₂O₂.

9.4.3 Bleaching

- a) Weigh 50 g or 100 g of OD pulp. Given the concentration of the pulp (20 %), calculate the amount of bleaching liquor required to dilute the concentration of the pulp to w/v 15 % during bleaching;
- b) Pre-heat the pulp to 70 °C;
- c) Add the liquor to the pulp and mix manually to obtain a homogeneous pulp;
- d) Put the pulp in a strong plastic bag, remove as much air as possible, seal and put it carefully into a water bath temperature controlled at 70 °C;
- e) Agitate the pulp in the bag a few times during the 90 minutes of bleaching;
- f) When the bleaching is finished, remove all the pulp to homogenize it;
- g) If the residual peroxide content test is to be performed, this test shall be performed rapidly, extracting the water from this pulp;
- h) Dilute the pulp to approximately w/v 5 % with cold water to stop the reaction. Use this pulp to make pads.

Hydrogen peroxide titration is described in [Annex B](#).

10 Procedure for preparation of filter pads

Pads are made for the following stages:

- after re-slushing (unbleached pulp);
- after reductive bleaching with hydrosulphite (Y);
- after oxidative bleaching with hydrogen peroxide (P).

Prepare two pads after each stage as follows:

- a) Take the equivalent of 6 g to 8 g of dry pulp and note the mass sampled as is (M_1);
- b) Dilute the pulp to obtain a volume of approximately 700 ml;
- c) Stir the fibrous suspension;
- d) Adjust the pH to $7 \pm 0,1$ by gradually adding $\text{Al}_2(\text{SO}_4)_3$ solution;
- e) Add 5 ml of flocculant;
- f) Stop the stirrer and clean the stirrer blade;
- g) Filter the pulp on a previously weighed filter paper mass (M_2) to obtain a pad;
- h) Carefully detach half the filter without tearing the pad to facilitate detachment of the filter paper after the drying stage;
- i) Press the sample between 6 blotters using the roller (3 below – 3 above);
- j) Dry the filter pad for 15 min at 95 °C using heater plates or drying section of a Rapid-Köthen handsheet former;
- k) Weigh the pad with the filter (M_3);
- l) Calculate the mass concentration using [Formula \(1\)](#):

$$C = \frac{(M_3 - M_2)}{M_1} \times 100 \quad (1)$$

where

C is the concentration in %;

M_1 is the mass of dry pulp in grams (g);

M_2 is the mass of filter paper in grams (g);

M_3 is the total mass of the pad and filter in grams (g).

NOTE ISO 3688^[3] is not used in this document. ISO 3688 offers the possibility to make pads or handsheets that do not retain all the fines elements are present in paper for recycling. In addition, the pH is here adjusted to 7 to be representative of paper making conditions using deinked pulp and to avoid a partial dissolution of calcium carbonate filler that are often present in paper for recycling.

11 Conditioning of the filter pads for the measurements

Condition the manufactured pads according to ISO 187^[1] however, do not condition them at high temperatures, as this could modify the optical properties.

12 Optical measurements and calculating the decolouration index

12.1 Measurement conditions

Measure the optical properties of the pads using the CIE D65 illuminant setting, with 420 nm UV cut-off filter according to ISO 2496:2014, A.6.

12.2 Measurements

- a) Without touching the area of the pad which will be tested, follow the procedure for the reflectometer or spectrophotometer to obtain the CIE tristimulus values (X , Y , Z) of the first pad [or the CIELAB coordinates (L^* , a^* and b^*), if the apparatus is designed to give the results directly in these CIELAB coordinates]. Read and record the colorimetric components to an accuracy of 0,01 units.
- b) Perform two measurements on each side of each pad for each of the 2 bleaching processes (i.e. 8 measurements per processed pulp), as well as on the pads made from the unbleached pulp.

For each bleaching process and for the untreated pulp, calculate the mean values $\langle L^* \rangle$, $\langle a^* \rangle$ and $\langle b^* \rangle$ of the components L^* , a^* and b^* .

12.3 Calculating the decolouration index

Calculate a decolouration index (DRI) for each of the performed bleaching processes. The final result is composed of DRI values: one DRI after oxidative bleaching and one DRI after reductive bleaching.

The decolouration index (DRI) is calculated with reference to achromatic white (perfect white), based on the $L^*a^*b^*$ coordinates measured with a UV filter (perfect white is $L^*=100$, $a^*=0$ and $b^*=0$). The distance d in the colour space from a point P with coordinates (L^* , a^* , b^*) to achromatic perfect white is given by [Formula \(2\)](#):

$$d^2 = a^{*2} + b^{*2} + (100 - L^*)^2 \quad (2)$$

where

a^* is the colour position between red and green;

b^* is the colour position between yellow and blue;

L^* is the lightness of the colour.

The reduction in the distance in the colour space of an untreated pulp P_1 to a treated pulp P_2 which represents the colour removal, is defined as $-\Delta d^2$ according to [Formula \(3\)](#):

$$\Delta d^2 = d_2^2 - d_1^2 \quad (3)$$

where

Δd is the reduction in the distance in the colour space.

d_1 is the distance in the colour space of untreated pulp P_1 to achromatic white.

d_2 is the distance in the colour space of treated pulp P_2 to achromatic white.

according to [Formula \(4\)](#):

$$\Delta d^2 = (a_2^{*2} - a_1^{*2}) + (b_2^{*2} - b_1^{*2}) + [(100 - L_2^*)^2 - (100 - L_1^*)^2] \quad (4)$$

where

L_1^*, a_1^*, b_1^* are the tristimulus coordinates of the untreated pulp;

L_2^*, a_2^*, b_2^* are the tristimulus coordinates of the treated pulp.

The decolouration index DRI is defined according to [Formula \(5\)](#):

$$DRI = -100 \times \frac{\Delta d^2}{d_1^2} \quad (5)$$

where DRI is the decolouration index in %.

NOTE Index 1 corresponds to the measurements obtained on untreated pads and index 2 corresponds to the measurements obtained on pads having undergone a reductive or oxidative bleaching.

13 Test Report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 21896;
- b) the date and place of the test;
- c) all the information needed for complete sample identification;
- d) the type of instrument used;
- e) the temperature and relative humidity used for the test;
- f) for each bleaching, the number of test specimens tested and the number of measurements taken;
- g) for each bleaching, the arithmetic mean of the CIELAB coordinates and the decolouration indexes;
- i) any difference from the procedure described in this document that may affect the results.

Annex A (informative)

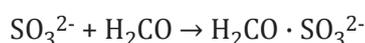
Sodium hydrosulphite titration

A.1 Principle and reaction

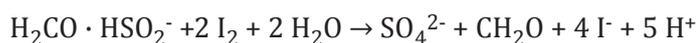
Hydrosulphite produces formaldehyde sulphonylate ions and formaldehyde sulphite ions in the presence of formaldehyde:



In the same conditions, sulphite produces formaldehyde sulphite ions.



In diluted acetic acid, formaldehyde sulphonylate salt reacts with iodine.



Hydrosulphite titration is based on this reaction.

A.2 Materials needed

250-ml Erlenmeyer flask, 2,5-ml and 10-ml pipettes, two 25 ml burettes.

A.3 Products used

- 20 % formaldehyde solution, pH adjusted to 10 with caustic soda;
- 10 % acetic acid solution;
- 1 % starch indicator, dissolve 1 g of starch in 100 ml of boiling water and cool;
- 0,1 N iodine;
- 0,01 N sodium thiosulphate;
- 1 % phenolphthalein in ethanol.

A.4 Solution to be analysed

For a solid: Weigh approximately 5 g of dithionite powder (note the exact mass introduced), dilute in 100 ml deionized water at 5 °C. Perform chemical titration as quickly as possible.

For a solution: Pour 50 ml of distilled water into a 250 ml Erlenmeyer flask.

A.5 Procedure

- Pour 50 ml of distilled water at 5°C in a 250 ml Erlenmeyer flask;
- Pipette 10 ml of formaldehyde, and put in the 250-ml Erlenmeyer flask prepared previously;

- Taking care to keep the tip of the pipette under the level of the liquid, add 2,5 ml of the dithionite solution;
- Add a few drops of phenolphthalein;
- Rinse with 5 °C distilled water;
- Add several drops of starch indicator;
- Add the acetic acid solution until the colour is removed and add a further 3 ml to 5 ml;
- Perform the titration with 0,1 N iodine until the colour is stable. Let this volume be V_1 ;
- Perform the titration of this mixture with 0,01 N sodium thiosulphate. Let this volume be V_2 .

A.6 Results

$$\% \text{ dithionite} = \frac{(0,1 \times V_1 - 0,01 \times V_2)}{\text{test portion volume}} \times 4,353;$$

$$\% \text{ dithionite in the solid} = \frac{\% \text{ dithionite}}{\text{mass}} \times 100.$$

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