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Paper and pulp — Deinkability test for printed paper products

Papier et pâte à papier — Essai de désencrabilité des produits en papier imprimés

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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 (see www.iso.org/directives).

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For an explanation of 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 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

The types and sources of paper for recycling are manifold. The most significant grades by volume are packaging products from industry, trade and households, followed by graphic papers from households and to a lesser extent from offices. These papers are blends of a variety of individual products. Typical blends of graphic paper for recycling recovered from households contain many different products printed on papers with a high content of wood-containing pulp fibres and a lesser share of woodfree pulp fibres. Graphic paper for recycling originating from printing and converting operations is typically rather pure and may contain just one type of paper (wood-containing or woodfree). Paper for recycling from printing and converting, as well as special grades, constitute only a minor share of the total volume of paper for recycling. Special grades (e.g. liquid packaging or label stock release liners) sometimes require specific treatments during recycling.

Deinking, the removal of ink from the substrate, is an important step in reprocessing graphic paper for recycling to new paper. A wide variety of papers are produced entirely or partially from deinked pulp and these include:

— graphic papers (of different quality levels);

— hygienic papers (such as toilet paper, hand and kitchen towels);

— white top layers of packaging paper and board.

Good deinkability of printed paper products is crucial for the sustainability of the graphic paper loop. The key process steps for deinking are the detachment of the ink film from the paper, ink fragmentation into a suitable size range and removal from the pulp slurry. Flotation deinking under alkaline conditions is the most widely used technology for ink removal in the paper recycling process. A wider range of the process pH may be utilised for separately collected printed products on predominantly woodfree substrates.

A simplified method herein has been developed to simulate the principle process steps for ink detachment and ink removal under standardised alkaline conditions at a laboratory scale. This gives an indication on how print products will perform in an industrial deinking operation. The method defined in this document is based on INGEDE Method 11. When the first version of INGEDE Method 11 was published, the deinking industry was predominantly using wood-containing raw material. INGEDE Method 11 is widely used by the paper industry and by many stakeholders in the paper value chain. The method is not designed to model additional or alternative process steps, such as dispersing, post-flotation, washing and bleaching. Cleaning and screening stages, which are designed to remove impurities and unwanted materials in the industrial process, are also not included in this method. An alternative deinking test method with near-neutral or neutral flotation conditions may be suitable for paper products mainly consisting of woodfree pulp fibres. However, the near-neutral or neutral flotation conditions are not within the scope of this document.

In most cases, the industrial flotation deinking process is designed and operated to remove a variety of inks and toners. Alkaline pulping conditions and fatty acid based collectors are widely used. However, fatty acid based collector chemistry is not singly used in industrial deinking processes in soft water areas. Assessments based on this laboratory scale method give an indication of how the tested print product will perform in a full-scale alkaline flotation deinking plant, but it will not necessarily provide the same absolute result. An example of this type of relation is given by INGEDE Method 11[3] and the Deinking Scorecard of the European Paper Recycling Council[4].

Paper and pulp — Deinkability test for printed paper products

1 Scope

This document specifies a basic laboratory test method for deinkability, applicable to any kind of printed paper product, under alkaline conditions by means of single stage flotation deinking and fatty acid-based collector chemistry.

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 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

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

ISO 1762, *Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 525 °C*

ISO 2469:2014, *Paper, board and pulps — Measurement of diffuse radiance factor (diffuse reflectance factor)*

ISO 2470-1, *Paper, board and pulps — Measurement of diffuse blue reflectance factor — Part 1: Indoor daylight conditions (ISO brightness)*

ISO 3689, *Paper and board — Determination of bursting strength after immersion in water*

ISO 4119:1995, *Pulps — Determination of stock concentration*

ISO 5269-1, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method*

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

ISO 5631-1, *Paper and board — Determination of colour by diffuse reflectance — Part 1: Indoor daylight conditions (C/2 degrees)*

ISO 12641-1:2016, *Graphic technology — Prepress digital data exchange — Colour targets for input scanner calibration — Part 1: Colour targets for input scanner calibration*

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
deinked pulp
pulp obtained from printed paper products, and deinked according to this document

3.2
un-deinked pulp
pulp obtained from printed paper products, pulped with added deinking chemicals according to this document, prior to flotation

3.3
stock concentration
ratio of the oven-dry organic and inorganic mass of material that can be filtered from a stock sample, to the mass of the unfiltered sample

[SOURCE: ISO 4119:1995, modified — Part of the sentence "when determined as specified in this International Standard" and Note 1 were removed.]

3.4
fibre concentration
ratio of the oven-dry mass of organic material, that can be filtered from a stock sample, to the mass of the unfiltered sample

Note 1 to entry: Organic material is the total material, less its ash.

Note 2 to entry: The organic material mainly consists of cellulosic fibres and fines.

3.5
fibre yield
ratio of the oven-dry mass of organic material after flotation to the oven-dry mass of organic material before flotation

Note 1 to entry: Organic material is the total material, reduced by the oven-dry mass of its ash.

Note 2 to entry: The organic material mainly consists of cellulosic fibres and fines.

3.6
rate of filtration
time taken for a defined volume of a test fluid to pass a filter

4 Principle

Printed papers are subjected to accelerated ageing and then pulping followed by flotation deinking under defined conditions. Pulp samples from each stage are taken and converted to dry state for characterization.

5 Equipment

5.1 General equipment

5.1.1 Drying oven, according to ISO 638.

5.1.2 Analytical balance, up to 150 g with an accuracy of at least 0,001 g.

5.1.3 Balance, up to 3 000 g with an accuracy of at least 0,1 g.

5.1.4 Beakers.

5.1.5 Muffle furnace, according to ISO 1762.

5.2 Equipment for preparation and flotation

5.2.1 Laboratory pulping device, capable of pulping about 150 g to 500 g of paper products under the conditions set in 7.4.1. Examples of suitable devices and operating conditions are listed in Annex A.

5.2.2 Temperature-controlled water bath.

5.2.3 Heating plate, equipped with magnetic stirrer, or commercially available water heater.

5.2.4 Laboratory flotation deinking cell (see 7.6 and Annex B) and – if applicable – accessories.

5.2.5 pH meter, with an accuracy of 0,1 points.

5.3 Equipment for specimen preparation

5.3.1 Pulp distribution device (volume: 10 l).

5.3.2 Büchner funnel.

5.3.3 Vacuum filtration unit for membrane filtration, with 39 mm bottom inner diameter of the funnel.

5.3.4 Vacuum device, that can produce a pressure difference ≥60 kPa.

5.3.5 Filter paper: Grammage of (84 ± 4) g/m², filtration time for deionized water (20 ± 4) s, tested according to Annex C and wet burst strength ≥30 kPa according to ISO 3689.

NOTE 1 The definition of the filter paper is much stricter than in ISO 3688, because the filtrate is used for further analysis (filtrate darkening).

NOTE 2 For example, the filter paper Ahlstrom Munktell 1289[1)] meets these requirements.

5.3.6 Cellulose nitrate membrane filter: nominal ø 50 mm, pore ø 0,45 µm, white, without a grid.

5.3.7 Standard sheet former (model: Rapid-Köthen) with dryer, according to ISO 5269-2 or conventional sheet former according to ISO 5269-1.

5.3.8 Paper cover sheets and carrier boards, according to ISO 5269-2.

5.4 Equipment for analysis

5.4.1 Flatbed scanner or **camera**:

a) Optical scan resolution ≥ 600 dpi, equivalent to a pixel size of ≤42 µm;

b) Colour depth 24 bit;

c) Optical density, D_{MAX} ≥ 4,0;

1) Ahlstrom Munktell 1289 can be obtained at Ahlstrom Germany GmbH, Bärenstein Plant, Niederschlag 1, 09471 Bärenstein, Germany. 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.

d) with the IT8 calibration (*.ICM-File) according to ISO 12641-1 (see also Annex E IT8 7.2 calibration) and reach a mean grey value of 115 ± 2 for all fields of the IT8 colour calibration sheet according to Annex E.

5.4.2 Image analysis software, such as the ones described in Annex E.

5.4.3 Colour-measuring equipment, which meets the requirements of ISO 2470-1 and ISO 5631-1.

6 Chemicals

6.1 Sodium hydroxide (NaOH), pro analysis, CAS # 1310-73-2.

6.2 Sodium silicate 1,3 g/cm^3 to 1,4 g/cm^3 (38 °Bé to 40 °Bé).

6.3 Hydrogen peroxide (H_2O_2), e.g. 35 %.

6.4 Oleic acid[2] ($C_{18}H_{34}O_2$), purified, CAS # 112-80-1, with the following specifications:

a) acid number: 198 to 240;

b) iodine number: 92 to 100;

c) linoleic acid (C18:2): max. 18 %;

d) oleic acid (C18:1): min. 72 %;

e) palmitic acid (C16:0): max. 8 %;

f) palmitoleic acid (C16:1): max. 1 %;

g) stearic acid (C18:0): max. 4 %.

6.5 Calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$), CAS # 10035-04-8.

6.6 Saturated aluminium sulphate solution $Al_2(SO_4)_3$.

NOTE A concentration of 330 g/l is considered as a saturated aluminium sulphate solution.

7 Procedure

7.1 General

This laboratory scale method defines the essential steps of the flotation deinking process: pulping and flotation. In order to simulate the average age of paper recovered from households, an accelerated ageing step is part of the procedure. Special care was taken to define a procedure without the need to test unprinted paper. The whole laboratory procedure and the required chemicals are shown in Figure 1.

The deinkability is assessed by three main quality parameters of the deinked pulp and one important process parameter.

2) Oleic acid can be obtained at VWR Chemicals, Prolabo, oleic acid, purified, article no. 20447.293. 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.

Quality parameters:

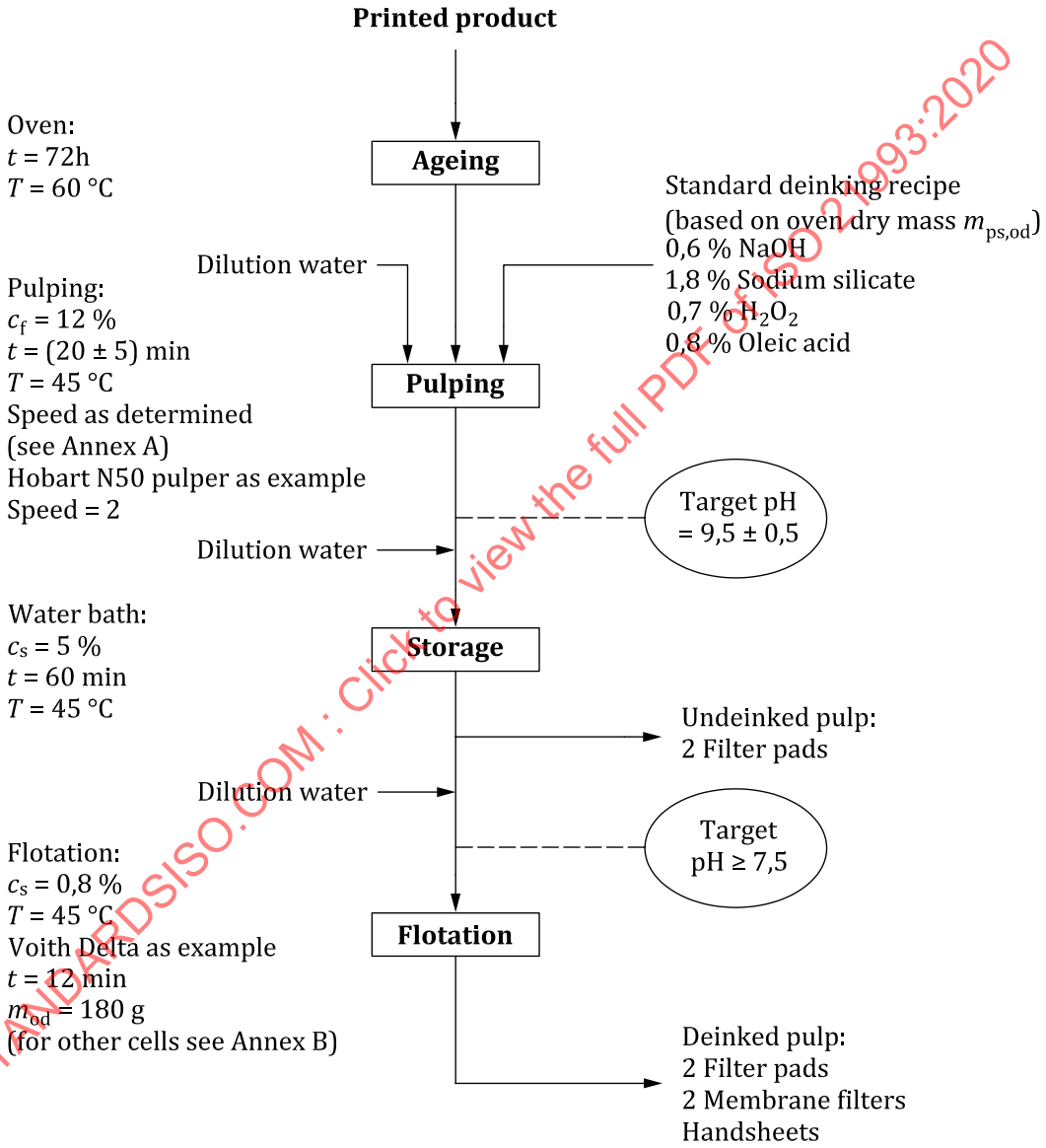
— luminance;

— pulp shade;

— dirt specks.

Process parameter:

— filtrate darkening.



Key

c_f	fibre concentration
c_s	stock concentration
t	time
T	temperature
m_{od}	mass (oven dry)
$m_{ps,od}$	mass of the oven-dry printed sample

Figure 1 — Procedure for testing deinkability with standard deinking recipe

7.2 Sampling and sample preparation

7.2.1 General

Take a sample which is representative of the printed material to be tested. The recommended amount of each printed sample is 1 000 g. If available, sample also some unprinted material and store separately for additional testing.

7.2.2 Identification

Describe, if possible, full details of each paper in the printed material to be tested. If available, the following shall be included:

a) identification of the printed paper product as to title, publishing company, date of issue, product category;

b) the print process, printing and drying/curing parameters and press settings;

c) the name and exact identification of inks or toner and of varnishes (if applicable);

d) any pre- or post-treatment applied (if any);

e) the paper grade, manufacturer and brand name, ash content of the printed paper sample.

Specify whether the printed sample contains inserts and/or supplements.

7.2.3 Non-paper material/loose and glued inserts/insertions

Remove any non-paper material, all inserts, glued-in inserts, stickers, sachets and similar items from the printed sample.

7.2.4 Adhesive applications

In order to avoid any interference with the test procedure and the results, remove any visible adhesive material – i.e. adhesives used in inserts, stickers, spines and similar – from the printed sample.

7.2.5 Accelerated ageing

Place the printed sample in a drying oven for accelerated ageing at (60 ± 3) °C for 72 h. Individual stacks should not contain more than 20 sheets.

NOTE 1 Accelerated ageing is necessary because the age of printed paper products can influence their deinkability. These accelerated ageing conditions correspond to 3 months to 6 months of natural ageing.

NOTE 2 If the age of the printed product is three months or higher, the accelerated ageing can be omitted. This is to be noted in the report as deviation of the procedure.

7.2.6 Breaking up of samples

Tear the accelerated aged printed sample into pieces of about 2 cm × 2 cm and allow them to equilibrate in the laboratory environment.

7.2.7 Measurement of moisture

Determine the moisture content of the air-dry sample by testing a portion according to ISO 638. Based on the results obtained, calculate the appropriate air-dry mass of the printed sample which corresponds to the oven-dry mass prescribed.

7.2.8 Measurement of ash content

Determine the ash content of the printed sample according to ISO 1762. Take special care to take a representative portion of the sample if it is comprised of different paper grades with different ash contents, e.g. cover and interior part(s).

7.2.9 Determination of the required amount of sample

The viscosity of a pulp slurry is – among other parameters – dependent on its ash content. Low viscosity due to high ash content can result in insufficient disintegration of the sample. Therefore, the pulping procedure is defined with an oven-dry fibre concentration of 12 %. Consequently, the total amount of the printed sample for the test is not constant but has to be calculated according to its ash content (see 7.2.8) and moisture content (see 7.2.7).

Determination of the oven-dry sample mass for 200 g (oven-dry) fibre mass is calculated using Formula (1):

$$m_{\mathrm{ps,od}} = \frac{100\%}{100\% - C_{\mathrm{a,ps}}} \times 200 \tag{1}$$

where

$m_{\mathrm{ps,od}}$ is the mass of the oven-dry printed sample in grams (g);

$C_{\mathrm{a,ps}}$ is the ash content of the printed sample expressed as a percentage.

Determination of the air-dry sample mass is calculated using Formula (2):

$$m_{\mathrm{ps,ad}} = m_{\mathrm{ps,od}} \times \frac{\left(100\% + C_{\mathrm{m,ps}}\right)}{100\%} \tag{2}$$

where

$m_{\mathrm{ps,ad}}$ is the mass of the air-dry printed sample in grams (g);

$m_{\mathrm{ps,od}}$ is the mass of the oven-dry printed sample in grams (g);

$C_{\mathrm{m,ps}}$ is the moisture content of the printed sample expressed as a percentage.

7.3 Preparation of dilution water and chemicals

7.3.1 General

All chemicals shall be dosed with a relative tolerance not exceeding ±1 %.

7.3.2 Preparation of dilution water

During laboratory treatment of the printed sample (see 7.4 to 7.5), use only deionized water adjusted to a calcium hardness of 3,21 mmol/l, equivalent to 128 mg Ca^{2+}/l.

To obtain the desired water hardness, add 472 mg/l calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$) to deionized water. This is equivalent to 128 mg Ca^{2+}/l.

During pulp preparation and flotation deinking, a constant temperature of (45 ± 2) °C shall be maintained. The dilution water should therefore be stored in a temperature-controlled water bath. It is also possible to heat part of the dilution water to a considerably higher temperature by means of a water heater, successively adding cold dilution water until the desired temperature has been reached.

7.3.3 Preparation of chemicals

It is advisable to prepare the chemicals by mass as volume is affected by temperature. The standard deinking recipe is given in Table 1.

Table 1 — Standard deinking recipe

Chemical	Dosage (relative to oven-dry paper)
Sodium hydroxide	0,6 % (100 %)[a]
Sodium silicate	1,8 % (1,3 g/cm^3 to 1,4 g/cm^3)[a]
Hydrogen peroxide	0,7 % (100 %)
Oleic acid	0,8 % (100 %)

[a] The dosages of sodium hydroxide and of sodium silicate shall be adjusted, if the pH is either too low or too high after pulping or if it is too low before flotation (see 7.4.2).

Prepare 2 000 g of deinking solution which will be sufficient for several tests. Dissolve 6 g of sodium hydroxide in approximately 600 g of deionised water, heat slowly to approximately 60 °C and proceed by adding 8 g of oleic acid. Stir until the solution is clear, then add 18 g of sodium silicate and fill up with deionised water to 2 000 g. The formation of soap reduces the alkalinity. 0,114 % of sodium hydroxide is needed to neutralise the oleic acid.

In addition, prepare 100 g of hydrogen peroxide solution for each test using deionised cold water.

The required amount of deinking solution and of hydrogen peroxide is dependent on the sample amount used for pulping and shall be calculated individually. Do not use a deinking solution which is older than one week.

Determine of the required mass of deinking solution using Formula (3):

$$m_{ds} = 2 \times m_{ps,od} \tag{3}$$

where

m_{ds} is the required mass of deinking solution in grams (g);

$m_{ps,od}$ is the mass of the oven-dry printed sample in grams (g).

Determine the required mass of hydrogen peroxide solution is calculated using Formula (4):

$$m_{H_2O_2} = 0{,}007 \times m_{ps,od} \tag{4}$$

where

$m_{H_2O_2}$ is the mass of hydrogen peroxide calculated with 100 % concentration in grams (g);

$m_{ps,od}$ is the mass of the oven-dry printed sample in grams (g).

7.4 Pulp preparation

7.4.1 Pulping

The total mass of material – product sample, chemicals, water – is 1 667 g. Therefore, determine the mass of dilution water using Formula (5):

$$m_{dw} = 1667 - 100 - m_{ds} - m_{ps,ad} \tag{5}$$

where

m_{dw} is the mass of dilution water in grams (g);

$m_{ps,ad}$ is the mass of the air-dry printed sample in grams (g);

m_{ds} is the mass of deinking solution in grams (g);

1 667 is the total mass of material in pulper in grams (g);

100 is the mass of hydrogen peroxide solution in grams (g).

Preheat the pulper with hot water in order to achieve an initial pulping temperature of (45 ± 2) °C. Discard the water after the vessel has reached the desired temperature.

In very rare cases the required volume of the deinking solution can be too high for the required fibre concentration. In these cases, the amount of water added during the preparation of the deinking solution should be modified to achieve the required fibre concentration.

Add the prescribed mass of printed sample to the pulper (200 g oven-dry fibres). Add the calculated amount m_{dw} of dilution water, heated to the required temperature, to the calculated amount m_{ds} of deinking solution. Add this deinking liquor into the vessel and run the pulper for a few seconds at low speed. Then stop it, brush down any scrap of paper from the vessel wall. Repeat this step as often as necessary until the sides of the vessel remain clean.

After the first stop, add the hydrogen peroxide solution (100 g). The fibre concentration is now 12 %.

Immediately afterwards, pulp the stock according to the instructions in Annex A.

To help maintaining the required temperature and to avoid splashing losses, cover the vessel during pulping, for example with a suitably sized, tight-fitting plastic lid. In addition, a heating device may be used to maintain the required temperature.

7.4.2 pH requirement

At the end of pulping, measure the pH. For a precise measurement of pH after pulping it is necessary to create a small amount of filtrate by pressing a pulp sample.

The target pH is 9,5.

Using the standard deinking recipe described in 7.3.3 (Table 1), the permitted range of pH is 9,5 ± 0,5. If the pH is outside this range, the sample shall be discarded and the test repeated with a modified dosage of chemicals. If the pH is too low after pulping, the dosage of the sodium hydroxide shall be increased. If the pH is too high, both sodium hydroxide and sodium silicate dosages shall be reduced by the same factor. The minimum dosage of sodium hydroxide is 0,2 %.

Beginning with an optional deinking recipe, while not proving to be in the range with the standard deinking recipe, the accepted pH is 9,5 ± 0,2.

Figure 2 describes the procedure when starting with standard and optional deinking recipe.

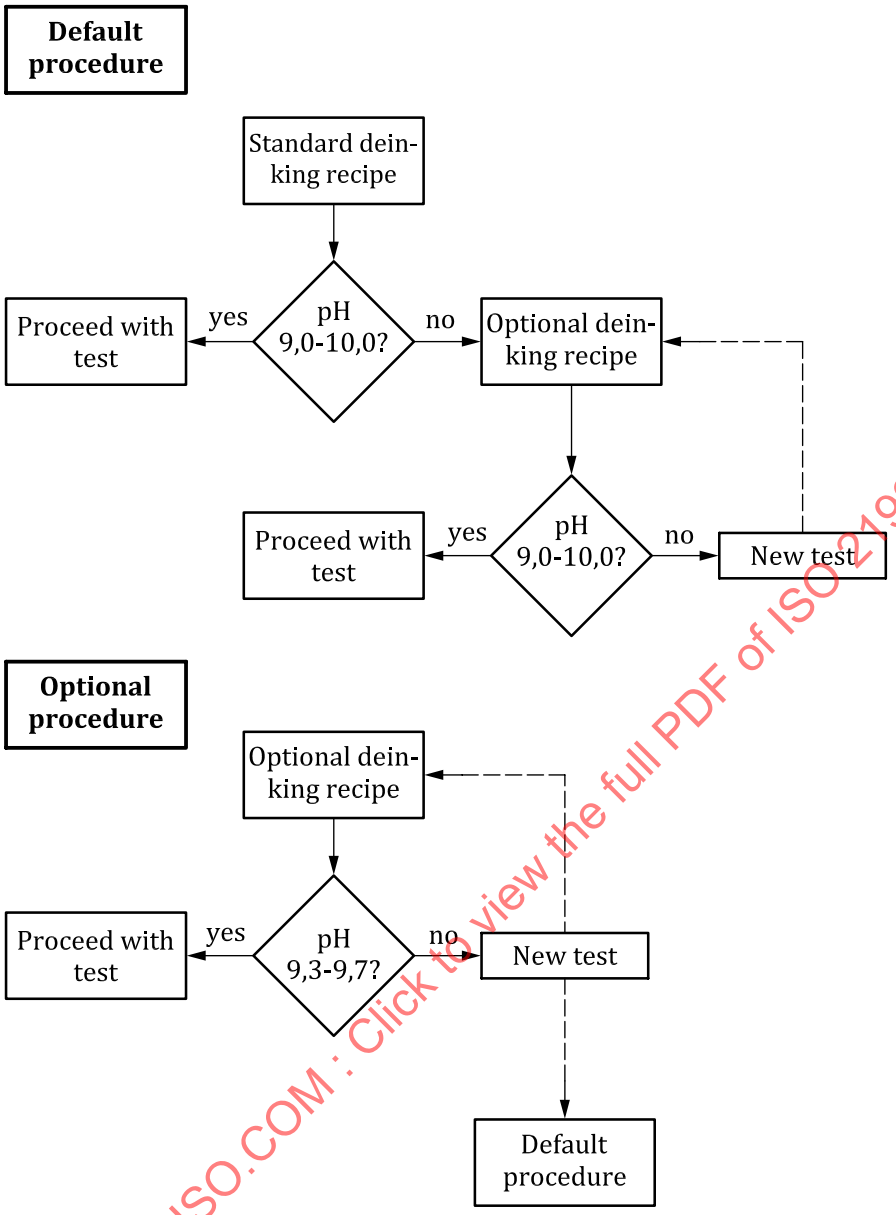


Figure 2 — Default and optional procedures with pH requirements and tolerances

Annex D describes a method to pre-test the pH after storage with a smaller sample amount. It gives an indication whether too low or too high pH will be expected. The requirements of pH tolerances shall be fulfilled regardless of the pre-test result.

7.4.3 Storage

Reduce the concentration of the amount of stock required for deinking to 5 % using dilution water (7.3.2), at a temperature of (45 ± 2) °C, and store it for 60 min in a water bath at (45 ± 2) °C.

The amount of stock needed for the subsequent treatment steps depends on the quantities required for flotation as well as for the final handsheet and filter pad formation (see 8.2 to 8.4). Minimum stock quantities of approximately 12 g oven-dry un-deinked pulp and approximately 15 g oven-dry deinked pulp are needed.

Measure the pH at the beginning and end of the storage time. The pH can be measured with reasonable accuracy in the pulp at storage concentration. However, it is recommended that the pH at the beginning and end of the storage is measured using filtrate, without fibres in order to increase the accuracy of the

measurement. This filtrate can be generated by pressing a small sieve onto the surface of the pulp. The pH electrode can then be dipped into the filtrate which forms inside the sieve.

7.4.4 Dilution

After storage the stock samples shall be diluted to a stock concentration of about 1 % to terminate any relevant chemical reaction before the treatment continues. For dilution of the un-deinked pulp sample use tap water. For dilution of the pulp sample to be deinked, use dilution water (7.3.2) with a temperature of (45 ± 2) °C.

Measure the pH. At flotation concentration it should be equal to or higher than 7,5, provided that the defined range of the pH after pulping is met. If the pH before the flotation is below 7,5, discard the sample and repeat the test with a higher dosage of sodium hydroxide.

Start the flotation before preparing the un-deinked pulp specimens.

7.5 Flotation

Heat up the cell with hot water. Pre-heat the cell with hot water close to the process temperature and then discard the water used for heating and add some of the prepared dilution water (7.3.2) of (45 ± 2) °C to prevent the "concentrated" pulp from subsequently staying in dead corners. Determine the quantity of diluted sample required for the flotation cell used (see Annex B) and add it into the flotation cell. Add the required amount of dilution water to achieve a stock concentration of 0,8 % and proceed according to the manufacturer's instructions. The starting point for the flotation time is at which the air is introduced.

Parameters for some flotation deinking cells are available in Annex B. Set the flotation time accordingly.

If the cell is not listed in Annex B, set the stock concentration to 0,8 % at the beginning of the flotation, set the temperature to (45 ± 2) °C and the flotation time to a value when the status of hyper-flotation is reached. The state of hyper-flotation is defined by a maximum increase of luminance of 0,3 points per minute. This shall be determined for each cell type by using a mix of printed paper products (50 % newspapers, printed with coldset offset, 25 % magazines on SC paper and 25 % magazines on LWC paper, both printed with the heatset offset process) and following the procedures described in this document.

Determine the amount of the oven-dry overflow according to ISO 4119, and use this amount to calculate the overall yield and the fibre yield of the flotation. If the fibre yield is below 65 %, repeat the test with a shorter flotation time.

7.6 Yield

In order to calculate yield values (overall yield and fibre yield) of the flotation, make sure to measure the mass and concentration of the feed and the overflow of the flotation and the ash content of the deinked pulp.

The overall yield is calculated using Formula (6):

$$y_o = \frac{(c_{UP} \times m_{UP}) - (c_{froth} \times m_{froth})}{(c_{UP} \times m_{UP})} \times 100\% \quad (6)$$

where

y_o is the overall yield;

c_{UP} is the stock concentration of undeinked pulp in %;

m_{UP} is the feed mass flotation of undeinked pulp in kilograms (kg);

c_{froth} is the stock concentration of overflow in %;

m_{froth} is the overflow mass in kilograms (kg).

The fibre yield is calculated using Formula (7):

$$y_f = y_o \times \frac{\left(100 - C_{a,dp}\right)}{\left(100 - C_{a,ps}\right)} \quad (7)$$

where

y_f is the fibre yield;

y_o is the overall yield;

$C_{a,dp}$ is the ash content of deinked pulp expressed as a percentage;

$C_{a,ps}$ is the ash content of the printed sample expressed as a percentage.

8 Specimen preparation

8.1 General

The brightness R_{457}, the CIELab colour coordinates (L^*, a^*, b^* values) and the luminance Y are measured from the samples. The dirt specks particle area A is analysed using a scanner-based image analysis system. Table 2 provides an overview of sample types, parameters to be measured and corresponding subclauses where more details can be found.

Table 2 — Overview of sample types, parameters to be measured and corresponding subclause

Sample	Specimen preparation according to subclause	Parameters
Filter pads (from pulp)	8.2	R_{457}, L^*, a^*, b^*, Y
Membrane filters (from filtrate)	8.3	Y, ΔY, R_{457}, L^*, a^*, b^*
Handsheet (from deinked pulp)	8.4	Dirt particle area A

For un-deinked pulp two filter pads and for deinked pulp two filter pads as well as at least two handsheets with tap water are required to permit an optical evaluation. In addition, two membrane filter specimens are prepared from the filter pad filtrate of the deinked pulp so as to be able to assess filtrate quality.

8.2 Filter pads

At least two filter pads are prepared of the pulp samples respectively.

NOTE The procedure described here deviates from ISO 3688 because ISO 3688 allows too many degrees of freedom, which have an impact on the result.

The filter pad is formed using a Büchner funnel which has been covered by a moistened filter paper. The prepared filter pads shall have a basis mass of 225 g/m^2. A filter paper diameter of 150 mm and a Büchner funnel diameter of 160 mm are recommended. In this case, 4,0 g oven-dry pulp material is used, and the suspension is topped up with tap water to a volume of 1 l.

Other filter diameters may be used according to Table 3. The diameter of the Büchner funnel corresponds to the filter diameter and should not exceed the maximum value in Table 3. Usually, Büchner funnels are purchased by their nominal diameter that is identical with the filter diameter.

If a differing size of Büchner funnel and filter paper are used, the sample volume shall be adapted according to Table 3. The concentration of the pulp sample remains 0,4 %.

Table 3 — Pulp volumes for Büchner funnel filtration

Diameter Büchner funnel mm	**Diameter filter paper** mm	**Oven-dry material** g	**Sample volume at 0,4 % stock concentration** ml
120	110	2,15	538
135	125	2,75	688
160	150	4,00	1 000
195	185	6,10	1 525

For optical assessment of the filtrate quality according to 8.3 and 9.3 collect the filtrate.

Experience has shown that the support of a thin wire made of plastic helps to avoid marks during dewatering. For this purpose use a plastic wire with a mesh width of about 140 µm and a mesh diagonal of about 190 µm and place it under the filter paper. This option is allowed when preparing the filter pads, but not if the filtrate is analysed for filtrate darkening, because the wire might cause a small leak at the edge of the filter paper. For the determination of the optical properties of the filter pad, the difference of the two ways to prepare the pads is negligible.

After filtering and carefully removing the filter paper, the wet filter pad is laid between two new paper cover sheets before drying. The paper cover sheets should not be removed from the filter pad until immediately prior to measuring the optical properties. If a Rapid-Köthen device is available (according to ISO 5269-2), dry the filter pads in the integrated dryer for 10 min. If a Rapid-Köthen device is not available, dry the filter pads on a suitable hot-pressing device (e.g. photo dryer or similar) to minimize roughness of the finished filter pads. The drying temperature and time should be as low as possible to minimize yellowing effects.

8.3 Membrane filters

The complete filtrate obtained by dewatering the pulp for one filter pad is homogenized by shaking. 100 ml filtrate is completely drained using a cellulose nitrate membrane filter in a vacuum filtration unit. Any fibrous material found on the membrane filter may indicate that some pulp bypassed the filter paper when preparing the filter pad. In that case, discard the membrane filter as well as the filtrate and prepare a new filter pad and filtrate as described in 8.2.

The filtrate of two filter pads (see 8.2) is filtered according to this procedure. Generally, the filtration is done without any retention aids. The result of this filtration shall be an uncoloured, clear liquid.

Exception:

If there is still a coloured filtrate after membrane filtration, repeat the procedure with a new sample (100 ml). Add a saturated aluminium sulphate solution before membrane filtration to a pH of 6,0 ± 0,5. State in the report whether the membrane filtrate was coloured and if aluminium sulphate was used.

The membrane filters are removed from the filtration unit and dried in a desiccator.

Reference membrane filters are made in the same way but using exclusively 100 ml of tap water without pulp. Prepare a membrane filter for each test series or on at least a daily basis.

8.4 Handsheets

An appropriate volume of material should be taken from the pulp distribution device for each handsheet. After standard laboratory handsheet formation, dry the handsheet in such a way that the surface is as smooth as possible. In the case of Rapid-Köthen (according to ISO 5269-2), dry the handsheet in the integrated dryer between carrier board and a cover sheet for 7 min. In the case of a conventional sheet former, dry the handsheet according to ISO 5269-1.

The cumulative area of multiple handsheets shall be a minimum of 0,1 m^2 and a maximum of 0,5 m^2. Within these limits, prepare enough handsheets to be able to measure 150 dirt specks or more. The oven dry grammage should amount to (42,6 ± 1,6) g/m^2. In the case of Rapid-Köthen prepare two laboratory handsheets with a mass of (1,35 ± 0,05) g, related to oven-dry substance.

Protect the sheets by placing an additional sheet of paper or board on both the top and bottom of the sheet.

9 Analysis

9.1 General

The samples shall be measured with C/2° conditions while using the edge filter of 420 nm (UV filter). This applies to all reflectance measurements. The calibration for reflectance measurements shall be done according to ISO 2469.

9.2 Reflectance measurements

9.2.1 General

Prior to optical properties assessment, the prepared specimens shall be conditioned according to ISO 187.

Both sides of filter pads should be measured. Avoid measurements too near to edges, kinks or on visible non-uniformities of the filter pads.

Two samples should be measured with four measurements on each side of the un-deinked pulp and deinked pulp filter pads. Just one measurement is made on the top side of each membrane filter sample.

9.2.2 Reflectance factors

Brightness is measured on the opaque filter pad according to the method described in ISO 2470-1.

The luminance Y and the CIELab colour coordinates L^*, a^* and b^* shall be determined according to the method described in ISO 5631-1 with UV cut off according to ISO 2469:2014, Annex A.6.

NOTE As an option, further reflectance factors such as R_{700} (see ISO 2469) and ERIC (see ISO 22754) can be measured.

9.3 Filtrate darkening

The filtrate darkening ΔY is the difference in luminance $Y_{filtrate}$ of membrane filters prepared with filter pad filtrate and $Y_{reference}$ of membrane filters prepared with tap water.

The preparation of the membrane filters is described in 8.3.

The luminance Y is determined (according to 9.2.2) as the average value of two membrane filters each. By subtracting $Y_{filtrate}$ from $Y_{reference}$, ($\Delta Y = Y_{reference} - Y_{filtrate}$), all factors affecting filtrate quality and not attributable to the pulp are eliminated.

9.4 Dirt particle measurement

9.4.1 Scanner

For the determination of the dirt particle area *A*, a scanner-based image analysis system is needed for optical analysis. The scanner shall be calibrated to ensure reproducibility of the measurements.

Requirements on measuring accuracy of flatbed scanner after warm-up period (see scanner manual) and under scanning conditions (see 9.4.2) are described in Annex E.

Repeatability of mean grey value (8 ± 1) bit: An ISO A4 sample shall be scanned 10 times without any movement of the sample. All mean grey value of total sample area should be within two grey values.

Deviation of colour value (RGB-8 bit) ≤5 (after calibration a scanned image of IT8-Target should not have more deviation to associated reference file than ±5 values in every colour channel – RGB).

NOTE For suitable scanners see Annex E.

9.4.2 Procedure for dirt particle measurement

The top and the bottom side of at least two laboratory handsheets per specimen shall be assessed by the image analysis system. The arithmetic mean of min. four measured values shall be calculated. This mean value shall be taken as the dirt specks area *A*. The results shall show at least the dirt speck area *A* above 50 µm circle equivalent diameter and above 250 µm circle equivalent diameter.

The sheets should be free of crinkles and waves to lie flat on the scanner. The sheets shall be scanned individually. As background an opaque batch of woodfree copy paper (min. five sheets with a luminance of Y = 84 ± 2 measured with illumination C/2° and 420 nm edge filter) should be used. Every handsheet should be scanned one time from top and from the bottom with 8-bit grey modus, 600 dpi and reflective light.

If the scanner is idle for more than 15 min a blank scan shall be made in advance of any new measurement.

NOTE For image analysis software parameterisation see Annex E; for values of the threshold and size classification see Annex F.

10 Test report

The test report shall include the following information [for items c) to f) include all information which is available]:

a) a reference to this document, i.e. ISO 21993:2020;

b) the identification of printed paper product as to title, publishing company, date of issue, product category;

c) the print process, printing and drying/curing parameters and press settings;

d) the name and exact identification of inks or toner and of varnishes (if applicable);

e) any pre- or post-treatment applied (if any);

f) the paper grade, manufacturer and brand name, ash content of the printed sample;

g) the exact designation of the laboratory testing equipment used – pulper, flotation cell, image analysis;

h) the pH after pulping, before and after storage and before flotation;

i) the chemical dosage for pulping;

j) the flotation time;

k) the ash content of deinked pulp;

l) the overall yield of the flotation in %;

m) the fibre yield of the flotation in %;

n) the overflow mass m_{froth};

o) the overflow stock concentration c_{froth};

p) the luminance Y of deinked pulp.

q) L^*, a^* and b^* of deinked pulp;

r) the filtrate darkening ΔY of the deinked pulp sample filtrate;

s) the dirt particle area of deinked pulp in mm^2/m^2 in two categories with the dirt particle area >50 µm and the dirt particle area >250 µm circle equivalent diameter;

t) any deviations from the conditions stipulated for this test method, if applicable (e.g. pulping device, specification of the laboratory flotation cell, conditions of flotation);

u) any further optical characteristics of un-deinked and deinked pulp yielded as well as their respective filtrate quality (e.g. R_{700} and/or ERIC).

NOTE For an example of a test report see Annex F.

Annex A
(normative)

Pulping devices

A.1 Determination of pulper operating conditions

In order to determine the pulping conditions, run a pulping test with pure eucalyptus sulphate pulp instead of a printed paper product. The eucalyptus sulphate pulp shall have a Schopper-Riegler-value of (18 ± 2) SR, a medium fibre length of (0,7 ± 0,1) mm (arithmetic average) and a kappa number of <5. Use 200 g of oven-dry pulp and fill it with water to a total mass of 1 667 g. Record the energy consumption for 20 min pulping time. Also, determine the no-load consumption of the equipment by operating it empty. Subtract the no-load consumption from the consumption with pulp in order to obtain the net power consumption for pulping. Set the speed to a value that the pulping process needs a net power consumption of (18 ± 3) Wh, in (20 ± 5) min at (45 ± 2) °C. Priority shall be given to meeting the net power consumption target as closely as possible. If the speed adjustment of the pulper allows, the target pulping time shall also be achieved as closely as possible.

Use these settings for pulping of the printed product.

A.2 Hobart pulper N 50

Hobart pulper N 50[3)].

Use rotor speed 2 and B-flat beater as well as a cover to prevent splashing. Additionally, it is possible to install a revolution counter, which stops the device automatically.

A.3 Kenwood pulper KMM 020

Kenwood Major Titanium KMM 020[4)].

Typically, the pulping time is 20 min at rotor speed 3,5.

Optional heating should be used to maintain (45 ± 2) °C during pulping.

3) Hobart pulper N 50 is the trade name of a product and can be obtained at Hobart Hobart, 701 South Ridge Ave., Troy, Ohio 45374, USA. 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.

4) Kenwood Major Titanium KMM 020 can be obtained at De'Longhi Appliances S.r.l. - Via L. Seitz, 47, 31100 Treviso, Italy. 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.

Annex B
(informative)

Examples of flotation cells

B.1 Voith Delta 25™

Voith Delta 25™[5)]

The air supply shall be set to 7 l/min. Use the supplier's calibration sheet for the air supply to find the corresponding point on the scale. The other parameters are: flotation period 12 min, suspension temperature (45 ± 2) °C, stock concentration 0,8 % at the beginning with 180 g oven-dry pulp.

During the flotation process add the necessary amount of dilution water heated up to (45 ± 2) °C several times in order to maintain the level of the aerated suspension in the cell. In case of low foaming tendency, increase the level in order to guarantee the overflow of foam.

After the flotation period switch off the air supply. Use dilution water to bring down any rejects from the overflow into the collecting tank, and then dewater the froth.

B.2 PTS Flotation cell

PTS Flotation cell[6)]

Use the following settings for flotation: air supply rate 60 l/h, stirrer speed in suspension 1 200 min^{-1}, flotation period 10 min, suspension temperature (45 ± 2) °C, stock concentration 0,8 % at the beginning with 12 g oven-dry pulp.

During the entire flotation process, use the scraper to remove the froth without stock, if possible. Collect the skimmed-off flotation rejects in a tank. Continually add dilution water to compensate for the drainage, keeping the suspension level constantly up at the edge of the overflow for the duration of the flotation.

After a flotation period of 10 min switch off the air supply and the stirrer. Use dilution water to bring down any rejects from the overflow into the collecting tank and then dewater the froth.

5) Voith Delta 25™ can be obtained at Voith Paper Fiber Systems GmbH & Co. KG, Escher-Wyss-Strasse 25, 88212 Ravensburg, Germany. 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.

6) PTS Flotation Cell can be obtained at Papiertechnische Stiftung, Pirnaer Strasse 37, 01809 Heidenau, Germany. 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.