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Paper and Pulp — Deinkability test for printed paper product mixtures

containing woodfree printed paper

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Foreword

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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 <u>www.iso.org/members.html</u>.

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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 printed papers are blends of a variety of individual products. Typical blends of graphic paper for recycling recovered from households contain a high content of mechanical-pulp based fibres and a minor amount 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 (mechanical-pulp based or woodfree). However, 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) often require specific treatments during recycling and/or repulping.

When the blends of paper for recycling are predominantly mechanical-pulp based offset printed papers, a simple single-loop alkaline flotation deinking step can sufficiently remove the ink colorants from the paper fibres. ISO 21993 describes a simplified test method that can be used to predict the deinkability of printed paper products under alkaline conditions. However, ISO 21993 specifies testing 100 % of a printed paper product of interest, whether mechanical-pulp based or woodfree, even though the proportion of woodfree paper in a deinking mill using alkaline conditions is typically small. This can possibly lead to misleading results in that a poor assessment by ISO 21993, and any related scorecard associated with it, does not necessarily mean that a particular print product cannot be deinked when present in a mixture of post-consumer papers for recycling^[1].

There is currently no test method which looks specifically at the case of woodfree printed paper as a small portion of the total blend, where the majority of the paper is mechanical-pulp based fibre^{[2][3][4][5]}. An alternative test method, which uses a specific mixture of mechanical-pulp based and woodfree printed paper grades, can help differentiate between inks, printed papers and ink/paper combinations.

As an adjunct to the method in ISO 21993, this document describes a method for testing the deinkability of woodfree printed papers when incorporated as a small amount in a blend with mechanical pulp based printed papers. A mixture of woodfree and mechanical pulp based printed papers is more representative of typical post-consumer recycled paper streams. This approach can also support recycled paper collection from preconsumer sources, where the amount of different grades can be controlled in the mill recipe, e.g., if woodfree printed paper is blended in at a small amount. The method described in this document, which uses a specified blend of mechanical pulp based and woodfree printed papers, has been developed to simulate the principal process steps for ink detachment and ink removal under standardised alkaline conditions at a laboratory scale. It gives an indication on how print product mixtures will perform in an industrial deinking operation where a high percentage of the printed paper for recycling comprises mechanical pulp-based fibres.

The method described in this document 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 can be more representative for printed papers consisting of mostly woodfree pulp fibres. However, the near-neutral or neutral flotation conditions are not within the scope of this document.

Paper and Pulp — Deinkability test for printed paper product mixtures containing woodfree printed paper

1 Scope

This document specifies a basic laboratory test method for deinkability of woodfree printed paper products as a mixture under alkaline conditions by means of single stage flotation deinking and fatty acid-based collector chemistry. The woodfree printed paper product of interest is tested as a mixture containing 10 % by weight of the woodfree printed paper with the balance comprising a mixture of printed wood-containing paper.

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 (all parts), Paper, board, pulps and cellulosic nanomaterials — Determination of dry matter content by oven-drying method

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

ISO 2469:2024, 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, 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 $(C/2^{\circ})$

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 terminology databases for use in standardization at the following addresses:

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

3.1

deinked pulp

pulp obtained from printed paper products, and deinked

3.2

un-deinked pulp

pulp obtained from printed paper products, pulped with added deinking chemicals, 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, 2.2, 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

3.7

mixture of printed wood-containing paper

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

Note 1 to entry: This definition is only applicable in the context of this document.

3.8

woodfree paper

freesheet paper

paper having, in principle, only chemical pulp in its fibre composition

Note 1 to entry: This definition can also apply to woodfree board: board having, in principle, only chemical pulp in its fibre composition.

Note 2 to entry: "chemical pulp" is defined in ISO 4046-4

Principle 4

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 in accordance with the ISO 638 series, capable of maintaining the air temperature at 105 °C ± 2 °C, and suitably ventilated.

- **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 <u>7.4.1</u>. Examples of suitable devices and operating conditions are listed in <u>Annex A</u>.

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 <u>7.6</u> and <u>Annex B</u>) and if applicable accessories.
- 5.2.5 pH meter with an accuracy of 0,1 points. ent Preview

5.3 Equipment for specimen preparation

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- **5.3.1**^S **Pulp distribution device** (volume: 10 l).^{124c088-e7f9-4459-9003-bf22b06ce27a/iso-fdis-12507}
- 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 in accordance with <u>Annex C</u> and wet burst strength > 30 kPa in accordance with 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 1289¹) meets these requirements.

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

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.

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

5.3.8 Paper cover sheets and carrier boards in accordance with ISO 5269-2.

5.4 Equipment for analysis

- **5.4.1** Flatbed scanner or camera with the following specifications:
- a) optical scan resolution \ge 600 dpi, equivalent to a pixel size of \le 42 μ m;
- b) colour depth 24 bit;
- c) optical density $D_{MAX} \ge 4,0$;
- d) with IT8 calibration (*.ICM-File) in accordance with ISO 12641-1 (see also Annex E IT8 7.2 calibration) and reach a mean grey value of 115 ± 2 for all fields of IT8 colour calibration sheet in accordance with Annex E.
- **5.4.2** Image analysis software, such as the ones described in <u>Annex E</u>.
- **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, CAS²) Registry Number[®] 1310-73-2.
- 6.2 Sodium silicate 1,3 g/cm³ to 1,4 g/cm³ (38° Bé to 40° Bé).

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6.3 Hydrogen peroxide (H₂O₂), e.g. 35 %.

6.4 Oleic acid³ (C₁₈H₃₄O₂), 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 acid³ (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 (CaCl₂ · 2 H₂O), CAS # 10035-04-8.

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

²⁾ CAS Registry Number® is a trademark of the American Chemical Society (ACS). 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.

³⁾ Oleic acid can be obtained at VWR Chemicals, 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.

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 printed paper recovered from households, an accelerated ageing step is part of the procedure. The whole laboratory procedure and the accepted chemicals are shown in Figure 1.

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

Quality parameters:

- luminance;
- pulp shade;
- dirt specks.

Process parameter:

filtrate darkening.

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