

ICS 85.060

**Papier und Faserstoff –
Prüfung der Deinkbarkeit für bedruckte Papierprodukte;
Text Englisch**

Paper and Pulp –
Deinkability test for printed paper products;
Text in English

Papier et pâte à papier –
Test de désencrabilité des produits papiers imprimés;
Texte en anglais

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Das vorliegende Dokument wurde nach den Verfahrensregeln einer PAS erstellt.

Gesamtumfang 31 Seiten

Dieses Dokument wurde durch die im Vorwort genannten Verfasser erarbeitet und verabschiedet.

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Foreword

This DIN SPEC has been developed according to the PAS (Publicly Available Specification) procedure. The development of a DIN SPEC according to the PAS procedure is carried out in workshops and does not require the participation of all stakeholders.

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A draft of this DIN SPEC has not been published.

DIN SPECS are not part of the body of German standards.

At present, there are no standards covering with this topic.

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Introduction

Deinking, the removal of ink, is an important step in reprocessing graphic paper for recycling to new paper. A good deinkability of printed paper products is a crucial feature for the sustainability of the graphic paper loop.

Therefore a method has been developed to simulate the common operating conditions of key process steps in an industrial deinking plant under standard conditions in a laboratory scale flotation. This allows the estimation of the relative challenge a printed paper product means to a deinking plant.

The industrial deinking process is complex since it has to cope with different types of inks, and it also has to remove impurities of all kind. The key process steps for deinking are ink detachment and removal from the pulp slurry. Deinking plants producing deinked pulp for newsprint, publication and other printing and writing papers predominantly use paper for recycling with a significant content of mechanical pulp based papers. These papers usually are pulped in an alkaline environment and the ink is removed by flotation. Typically, the ink collector is fatty acid based. The term “common operating conditions” encompasses these process settings. Other printed paper products collected from households together with newspapers and magazines are typically treated under these common operating conditions as well.

This method has been developed for the uniform assessment of the deinkability of individual printed paper products and is based on INGEDE Method 11. On purpose, the method dispenses with typical additional process steps as dispersing and post-flotation. The deinkability of a printed paper product according to this simple method allows a good prediction whether it will be suitable for deinking in industrial scale even if the quality levels in a related assessment scheme, e.g. the Deinking Scorecard of the European Recovered Paper Council, are far less demanding than industrial quality requirements.

1 Scope

This DIN SPEC specifies a basic laboratory test method to evaluate the deinkability of any kind of printed paper product by means of alkaline, single stage flotation deinking.

2 Normative References

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 and pulps — Determination of residue (ash) on ignition at 525 °C*

ISO 2469, *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 2471, *Paper and board — Determination of opacity (paper backing) — Diffuse reflectance method*

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

ISO 4119, *Pulps — Determination of stock concentration*

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°)*

ISO 9416:2009, *Paper — Determination of light scattering and absorption coefficients (using Kubelka-Munk theory)*

ISO 11664-4, *Colorimetry — Part 4: CIE 1976 $L^*a^*b^*$ Colour space*

3 Terms and definitions

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

3.1 deinked pulp

DP

pulp obtained from printed paper products, and deinked according to DIN SPEC 55700

3.2 undeinked pulp

UP

pulp obtained from printed paper products, disintegrated mechanically with added deinking chemicals according to DIN SPEC 55700, prior to flotation

3.3**ink elimination** IE_{700}

ratio of the difference of the light absorption coefficient k of the undeinked and deinked samples and the difference of the light absorption coefficient k of undeinked and unprinted samples, measured at a wavelength of 700 nm

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 unfiltered sample

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

3.5**fibre yield**

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

3.6**rate of filtration**

time in which a defined volume of a test fluid needs to pass a filter

4 Principle

Flotation deinking is the most widely used technology for ink removal in the paper recycling process. This method in a laboratory scale 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 aging 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 is shown in Figure 1.

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

Quality parameters:

- luminosity;
- colour shade;
- dirt specks.

Process parameters:

- ink elimination;
- filtrate darkening.

5 Equipment**5.1 General equipment**

5.1.1 Warming cabinet according to ISO 638.

5.1.2 Analytical balance up to 1 000 g with an accuracy of at least 0,001 g.

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

5.1.4 Beakers.

5.1.5 Muffle furnace which can be adjusted to an incineration temperature of 525 °C.

5.2 Equipment for preparation and flotation

5.2.1 Laboratory pulping device, capable to pulp about 200 g to 500 g of paper products under the conditions set in 7.5.1. 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 hot-water heater.

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

5.2.5 pH value measuring system 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 allows a pressure difference ≥ 60 kPa.

5.3.5 Filter paper: Grammage of 84 g/m², filtration time for distilled water 20 s, tested according to Annex C and wet burst strength > 30 kPa according to ISO 3689.

NOTE Filter paper Ahlstrom Munktel 1289¹⁾ meets these requirements.

5.3.6 Cellulose nitrate membrane filter: $\varnothing$ 50 mm, pore $\varnothing$ 0,45 μ m, white, without a grid.

5.3.7 Standard sheet former (model: Rapid-Köthen) with dryer (vacuum 95 kPa, 93 °C $\pm$ 4 °C), according to ISO 5269-2.

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

5.4 Equipment for Analysis

5.4.1 Flatbed scanner:

- a) Scanning area $\geq$ ISO A4;
- b) Optical scan resolution $\geq 2\,000$ dpi;
- c) Colour depth 48 bit;
- d) Optical density $D_{MAX} \geq 4,0$.

¹⁾ Ahlstrom Munktel 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 DIN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.4.2 Image analysis software.

5.4.3 Color-measuring equipment which meets 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 1,3–1,4 g/cm³ (38–40 °Bé).

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

6.4 Oleic acid²⁾ (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 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 (CaCl₂ · 2 H₂O), CAS # 10035-04-8.

6.6 Aluminium sulphate solution – Al₂(SO₄)₃.

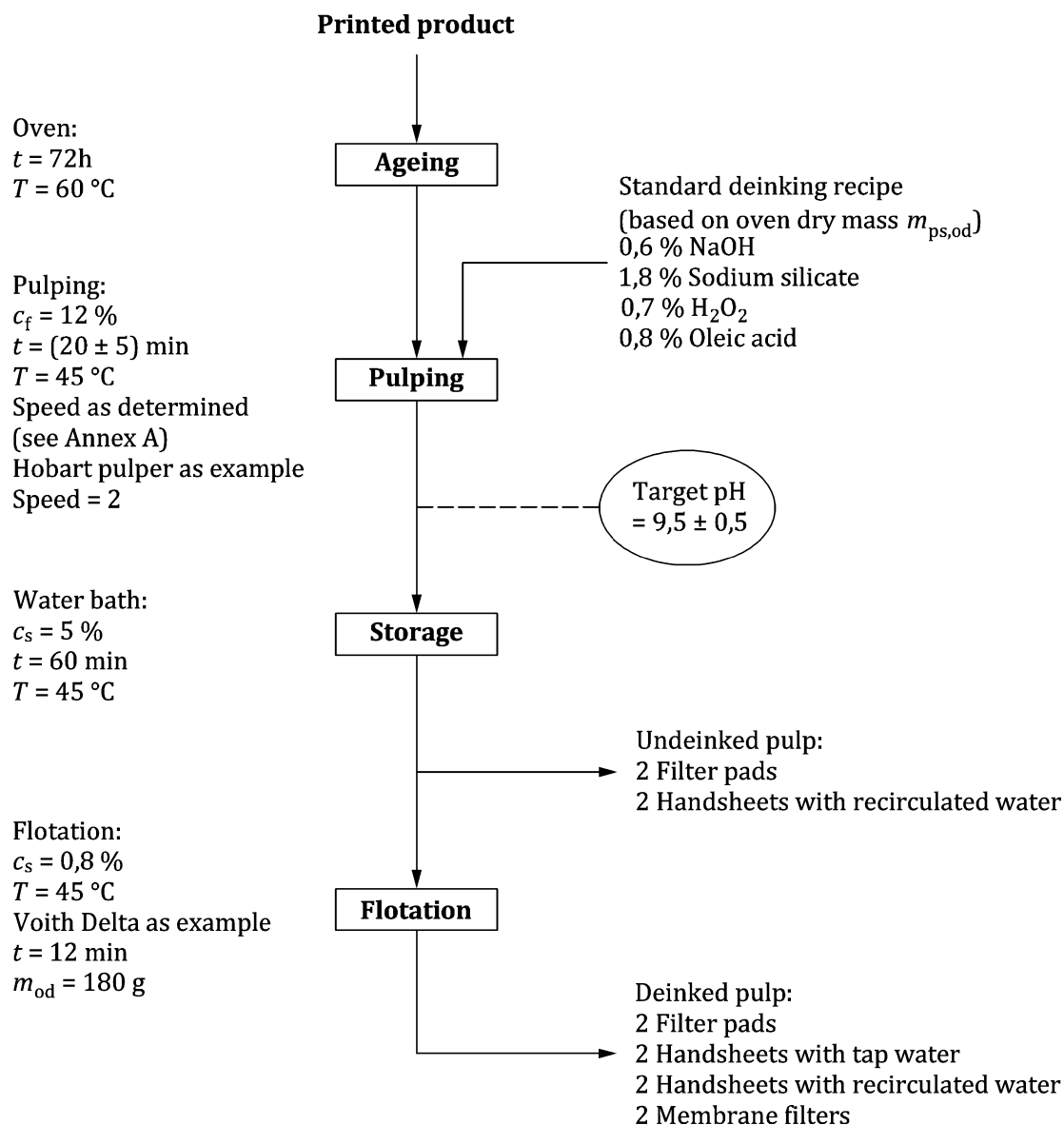
6.7 Water of calcium hardness of 18 °dH (see 7.4.1).

7 Procedure

7.1 General

The procedure is shown in Figure 1.

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 DIN of the product named. Equivalent products may be used if they can be shown to lead to the same results.



Legend

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

The printed samples used shall be representative for the tested product. The recommended amount of each printed sample is 1 000 g. If available, sample also some unprinted material and store separately for additional testing.