

<p><b>INGEDE</b> <b>Method 11</b> <b>January 2018</b></p> <p>18 Pages</p>	<p><b>Assessment of print product recyclability</b></p> <p><b>– Deinkability test –</b></p>	 <p><b>INGEDE</b> International Association of the Deinking Industry</p>
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## **Introduction**

Good recyclability of print products is crucial for the sustainability of the graphic paper loop. It belongs to the focal work of INGEDE to safeguard and improve recyclability of the input to be used by the recycling paper mills.

One of the measures is to provide tools for the assessment of the recyclability in the two aspects:

- Deinkability
- Screenability of adhesive applications.

The industrial deinking process is complex since it has to cope with different types of inks, and it also has to remove impurities and unwanted materials of all kind. The key process steps for deinking are the detachment of the ink film from the paper, the ink fragmentation into a suitable size range and removal from the pulp slurry. This gives a sufficient and reliable indication on how a print product will perform in an industrial deinking plant. Flotation is the most widely used technology for ink removal in the paper recycling process.

Households are the main source of deinking grade paper for recycling (in many locations, small businesses are allowed to use the same collection systems as households) in deinking plants producing deinked pulp for newsprint, publication and other printing and writing papers. This material has a significant content of papers which are based on mechanical pulp. Such papers usually are pulped in an alkaline environment and the ink is removed by flotation. Typically, the ink collector is fatty acid based.

A second group of papers used in deinking are medium and high-quality grades, sourced from offices as well as printing and converting operations. If the raw material is mainly wood free, then near neutral or neutral process conditions, which are not within the scope of this method, are used. However, most print products end up in household collection or are mixed with this material and therefore have to be deinkable under alkaline conditions.

This method has been developed for the uniform assessment of the deinkability of individual print products. The method intentionally dispenses with additional or alternative process steps as dispersing, post-flotation, washing and bleaching. The deinkability of a printed paper product according to this simple method allows a good prediction of its suitability for deinking on an industrial scale even if the quality levels in a related assessment scheme, e. g. the Deinking Scorecard of the European Paper Recycling Council, are far less demanding than actual industrial quality requirements.

## **1 Scope**

This INGEDE Method describes a procedure to evaluate the deinkability of printed paper products under alkaline conditions by means of flotation deinking. It can be used for any kind of printed paper product.

## **2 References**

**INGEDE Method 1** – Test sheet preparation of pulps and filtrates from deinking processes

**INGEDE Method 2** – Measurement of optical characteristics of pulps and filtrates from deinking processes

**INGEDE Method 12** – Assessment of the recyclability of printed paper products – Testing of the fragmentation behaviour of adhesive applications

**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 4119** – Pulps – Determination of stock concentration

**ISO 5263-1** – Pulps – Laboratory wet disintegration

**ISO 5269-2** – Pulp – Preparation of laboratory sheets for physical testing. Part 2: Rapid-Köthen method

## **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 INGEDE Method 11

### **3.2 Undeinked pulp**

#### **UP**

pulp obtained from printed paper products, pulped with added deinking chemicals according to INGEDE Method 11, prior to flotation

### **3.3 Ink elimination**

#### **IE<sub>700</sub>**

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 Stock concentration**

ratio of the oven-dry mass of material, that can be filtered from a stock sample, to the mass of unfiltered sample in percent

### **3.5 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 in percent

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

### **3.6 Fibre yield**

ratio of the oven-dry mass of organic material in the deinked pulp to the mass of organic material before flotation in percent

## **4 Principle**

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

## **5 Equipment and auxiliaries**

### **5.1 Equipment**

- Drying oven, capable of maintaining the air temperature at  $105\text{ °C} \pm 2\text{ °C}$ , and suitably ventilate, according to ISO 638.
- Analytical balance up to 150 g with an accuracy of at least 0,001 g
- Balance up to 3000 g with an accuracy of at least 0,1 g
- Hobart pulper N50, available from Hobart GmbH. Use the type of stirrer and a comparable cover, shown in the following figures.



**Figure 1: Stirrer for the Hobart pulper**



**Figure 2: Cover for the Hobart pulper**

- Temperature-controlled water bath
- Heating plate equipped with magnetic stirrer, or commercial-grade hot-water heater
- Laboratory flotation cell (references: Voith Delta 25™, PTS cell)
- Plastic scraper (in case of PTS cell)
- Beakers
- Muffle furnace which can be adjusted to an incineration temperature of 525 °C
- pH measuring system with an accuracy of 0,1 points.

If different equipment is used, this has to be mentioned in the test report.

## **5.2 Chemicals**

- Sodium hydroxide (NaOH), pro analysis, CAS # 1310-73-2
- Sodium silicate 1,3–1,4 g/cm<sup>3</sup> (38–40 °Bé)
- Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), e.g. 35 %
- Oleic acid (C<sub>18</sub>H<sub>34</sub>O<sub>2</sub>), extra pure, CAS # 112-80-1, e.g. VWR Article No. 20447.293
- Calcium chloride dihydrate (CaCl<sub>2</sub> · 2 H<sub>2</sub>O), CAS # 10035-04-8.

## **6 Procedure**

### **6.1 General**

This laboratory scale INGEDE Method defines the essential steps of the flotation deinking process: pulping and flotation. In order to simulate the average age of paper collected 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 is shown in Figure 3.

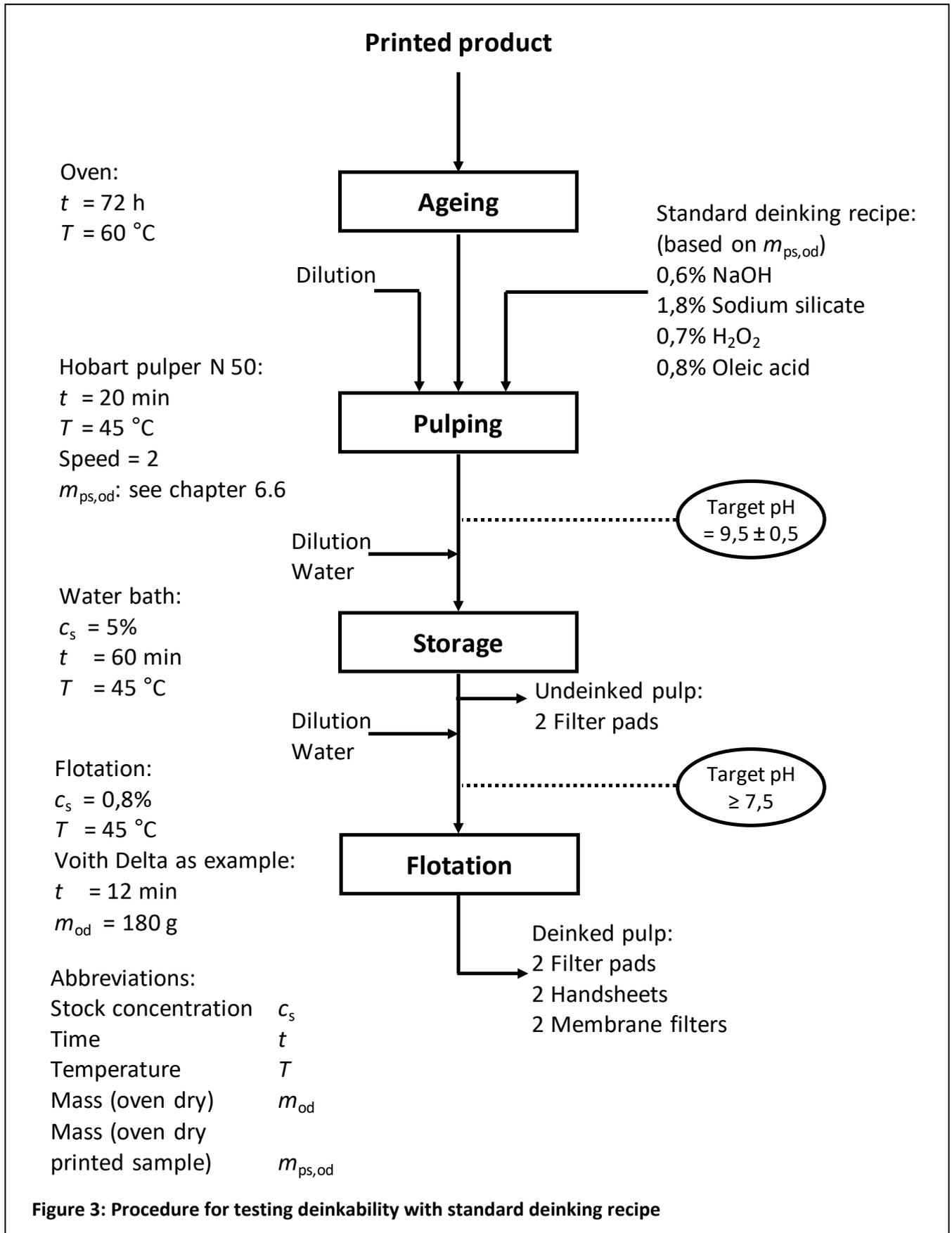
The deinkability is assessed by three quality parameters of the deinked pulp and two process parameters.

Quality parameters:

- Luminance
- Colour shade
- Dirt specks (in two different size categories).

Process parameters:

- Ink Elimination
- Filtrate darkening.



**Figure 3: Procedure for testing deinkability with standard deinking recipe**

## **6.2 Sampling and preparation**

### **6.2.1 General**

The printed samples used shall be representative of 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.

### **6.2.2 Identification**

Each print product is designated by its title, publishing house, issue date, product category, printing method and paper grade, if available.

Weigh the complete print product. After weighing, remove any loose paper-based inserts, which are not a constituent element of the print product, and any non-paper product materials from the print product to determine their share in the total mass of the product.

### **6.2.3 Loose and glued inserts / insertions**

Remove all inserts, glued-in inserts, stickers, sachets and similar items from the printed paper product.

### **6.2.4 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 paper product.

As an option, their removability can be tested separately by INGEDE Method 12.

### **6.2.5 Accelerated ageing**

Place the samples in a drying oven for accelerated ageing at  $60\text{ °C} \pm 3\text{ °C}$  for 72 h. Individual stacks should not contain more than 20 sheets.

**NOTE** Accelerated ageing of the samples is necessary because the storage of the papers for recycling can influence their deinkability. These accelerated ageing conditions correspond to 3 - 6 months of natural ageing.

### **6.2.6 Breaking up of samples**

Tear the accelerated aged samples into pieces of about  $2 \times 2\text{ cm}^2$  and allow them to equilibrate with the laboratory environment.

### **6.2.7 Moisture content**

A part of the equilibrated samples is used to determine the moisture content according to ISO 638 with at least one sample of about 50 g minimum. Based on the obtained results, calculate the appropriate air-dry weight of the samples which corresponds to the oven-dry weight prescribed.

**6.2.8 Ash content of print product**

Measure and calculate the ash contents 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 content, e. g. cover and interior part(s).

**6.2.9 Determination of the required amount of sample**

For print products with an ash content of 20% or lower, use a constant amount of 200 g oven dry sample mass. If the print product has an ash content higher than 20% a constant amount of 160 g organic material is used. The determination of the oven-dry sample mass for 160 g (oven-dry) fibre mass is calculated with equation (1):

$$m_{ps,od} = \begin{cases} 200 \text{ g} & \text{if } Ash_{ps} \leq 20\% \\ \frac{160 \text{ g} \times 100\%}{100\% - Ash_{ps}} & \text{if } Ash_{ps} > 20\% \end{cases} \quad (1)$$

where

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

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

The determination of the air-dry sample weight is calculated with equation (2):

$$m_{ps,ad} = m_{ps,od} \times \frac{(100\% + MC_{ps})}{100\%} \quad (2)$$

where

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

$MC_{ps}$  is the moisture content of the printed sample expressed as a percentage.

**6.2.10 Determination of the pulping concentration**

The viscosity of a pulp is – among other parameters – depending on its ash content. Low viscosity due to high ash content can result in insufficient disintegration of the sample and/or insufficient fragmentation of the ink particles. Therefore the pulping procedure is defined with an oven-dry fibre concentration of 12 % for all print products with an ash content of 20% or higher. Consequently, the total amount of those samples for the test is not constant but has to be calculated according to its moisture and its ash content. To be in line with previous versions of the method, the stock concentration during pulping is 15 % for print products with an ash content of or below 20%.

$$c_{ps} = \begin{cases} 15\% & \text{if } Ash_{ps} \leq 20\% \\ \frac{12\%}{100\% - Ash_{ps}} \times 100\% & \text{if } Ash_{ps} > 20\% \end{cases} \quad (3)$$

where

$c_{ps}$  is the pulping concentration expressed as a percentage.

### **6.3 Preparation of dilution water**

#### **6.3.1 General**

Make sure that the chemicals are dosed with a relative tolerance not exceeding  $\pm 1\%$ .

#### **6.3.2 Preparation of dilution water**

During laboratory treatment of the print products (6.4 to 6.8), use only water which has been treated to obtain the prescribed hardness values.

To obtain the desired water hardness, add 472 mg/l calcium chloride dehydrate ( $\text{CaCl}_2 \cdot 2 \text{H}_2\text{O}$ ) to deionised water.

If tap water is used instead of dilution water, this shall be mentioned in the test report indicating the respective hardness.

During sample preparation, a constant temperature of 45 °C should be maintained. The dilution water should therefore be stored in a water bath whose temperature can be controlled accordingly. It is also possible to heat part of the dilution water to a considerably higher temperature by means of a hot-water heater, and successively add cold dilution water until the desired temperature has been reached. It is not advisable to separately heat the individual stock solutions (dilution water, chemical stock solution, peroxide solution).

### 6.3.3 Preparation and dosing of chemicals

In order to avoid incorrect volume due to different temperatures, it is advised to prepare the chemicals by weight. The standard formulation is given in Table 1:

**Table 1: Standard deinking recipe**

Chemical	Dosage (related to oven-dry paper)
Sodium hydroxide	0,6% (100%)*
Sodium silicate	1,8% (1,3–1,4 g/cm <sup>3</sup> )*
Hydrogen peroxide	0,7% (100%)
Oleic acid	0,8% (extra pure)
*Only if the pH is either too low or too high after pulping or if it is too low before flotation, the dosages of sodium hydroxide and of sodium silicate have to be adapted (see 6.5)	

Prepare a total amount of 2000 g stock solution which will be sufficient for several tests. Dissolve 6 g sodium hydroxide in approximately 600 g deionised water, heat slowly to approximately 60 °C and proceed by adding 8 g oleic acid. Stir until the solution is clear, then add 18 g sodium silicate and fill up with deionised water to 2000 g. The formation of soap reduces the alkalinity. 0,114 % sodium hydroxide is needed to neutralise the oleic acid.

The required amount of stock solution and of hydrogen peroxide is dependent on the sample amount used for pulping and has to be calculated individually. Do not use a stock solution which is older than two weeks.

The determination of the required mass of stock solution is calculated with equation (4):

$$m_{St} = 2 \times m_{ps,od} \quad (4)$$

where

$m_{St}$  is the mass of stock solution in grams (g);

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

The determination of the required mass of peroxide solution is calculated with equation (5):

$$m_{\text{H}_2\text{O}_2} = 0,007 \times m_{\text{ps,od}} \quad (5)$$

where

$m_{\text{H}_2\text{O}_2}$  is the mass of hydrogen peroxide calculated with 100% concentration in grams (g);

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

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

#### 6.4 Pulping

The total mass of material – product sample, chemicals, water – is 1333 g. The amount of dilution water is therefore calculated with equation (6):

$$m_{\text{dw}} = 1333 \text{ g} - 100 \text{ g} - m_{\text{St}} - m_{\text{ps,ad}} \quad (6)$$

where

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

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

$m_{\text{St}}$  is the mass of stock solution in grams (g);

1333 g is the total mass of material in pulper;

100 g is the mass of hydrogen peroxide solution.

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

In very rare cases the required volume of the stock solution can be too high for the required fibre concentration. In those cases the amount of water during the preparation of the stock solution should be adapted to reach the required fibre concentration.

Fill the Hobart pulper with the prescribed sample quantity. Take the calculated amount  $m_{\text{St}}$  of stock solution and fill up with the calculated amount  $m_{\text{dw}}$  of appropriately heated dilution water. Start the rotor. After a few seconds 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 stay clean.

After the first stop, add the peroxide solution (100 ml). Immediately after, disintegrate the stock for 20 min at approx. 45 °C, using rotor speed 2.

To help maintaining the temperature and to avoid splashing losses, cover the vessel during pulping, e. g. with a suitably sized, tight-fitting lid (example see Figure 2). Optionally, a heating device may be used.

**6.5 pH value after pulping**

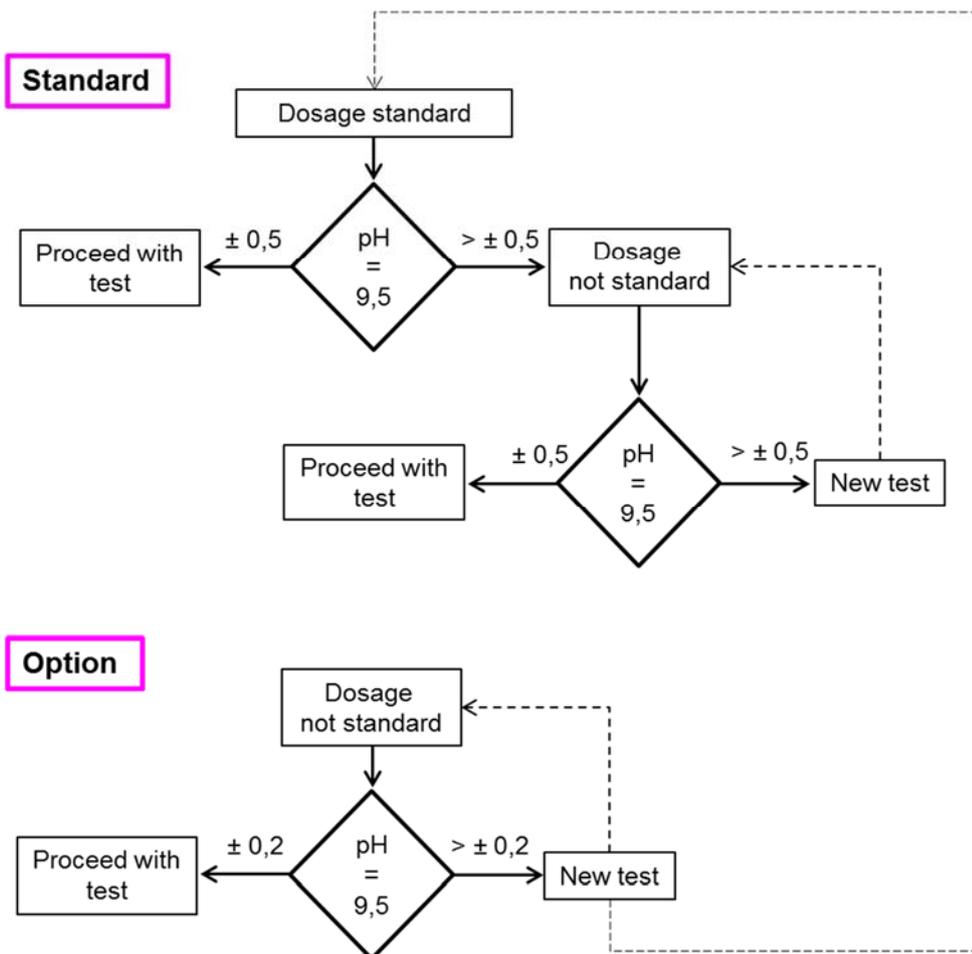
At the end of pulping, measure the pH. For a precise measurement of the pH after pulping it is necessary to create a small amount of filtrate by pressing out a pulp sample.

The target pH value is 9,5.

Using the standard formulation from chapter 6.3.3 the permitted range of pH is  $9,5 \pm 0,5$ . If the pH is beyond this range, the sample has to be discarded and the test repeated with an adapted dosage of chemicals. In case of too low pH after pulping the dosage of sodium hydroxide has to be increased. In case of too high pH, both sodium hydroxide and sodium silicate have to be reduced by the same ratio. The minimum dosage of sodium hydroxide is 0,2 %.

Beginning with a non-standard chemical formulation, while not proving to be in the range with the standard formulation, the accepted pH is  $9,5 \pm 0,2$ .

Figure 4 describes the procedure when starting with standard or non-standard chemical formulation.



**Figure 4: pH value tolerances**

ANNEX A describes a method to pre-test the pH after pulping with a smaller sample amount. It gives an idea whether a too low or too high pH has to be expected. This principle of pre-testing is applicable also with other chemical dosages, but does not compensate the original pulping with the full pulp amount. The requirements of pH tolerances must be fulfilled regardless the pre-test result.

### **6.6 Storage**

The amount of pulp needed for the subsequent treatment steps depends on the quantities required for the final handsheet and filter pad formation (see 6.9). Stock quantities of 12 g oven-dry undeinked (UP) and approx. 15 g oven-dry deinked pulp (DP) are needed in minimum. Stock losses will vary depending on the print products used and can amount up to 50% during flotation.

Store the amount of stock required for subsequent treatment for 60 min in a water bath at 45 °C and 5% concentration. Adjust the dilution water to a temperature of 45 °C and to the desired level of hardness.

Measure the pH before and after the storage time. The pH can be measured with reasonable accuracy in the pulp at storage concentration. However, it is recommended to measure the pH before and after the storage in a filtrate without fibres in order to increase the accuracy of the measurement. This filtrate can be generated by pressing a small cullender onto the surface of the pulp. The pH electrode can then be dipped into the filtrate which forms inside the cullender.

### **6.7 Dilution**

After storage the stock samples must be diluted with 45 °C warm water to terminate relevant chemical reactions before the treatment continues. Use tap water for the UP sample. For the pulp sample to be deinked, use the prepared dilution water that has been brought to a temperature of 45 °C. The concentration after this dilution should be around 1%.

Measure the pH. At flotation concentration it should be equal or higher than 7,5, provided that the defined range of the pH after pulping is met. If the pH before 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 UP specimens.

### **6.8 Flotation**

Heat up the cell with hot water. After some minutes pour out the heating water and fill in first some of the prepared dilution water of 45 °C to prevent the "concentrated" pulp from staying in dead corners later. Determine the quantity of diluted sample requested according to the flotation cell used (see Annex B) and add it into the flotation cell. Fill up with the proper amount of dilution water to achieve a stock concentration of 0,8%, and proceed as the instructions of the flotation cell describes. The starting point for the flotation time is when the air supply is started.

Parameters for some flotation deinking cells are available in Annex B. Set the flotation time accordingly. It is preferred to use Voith Delta 25™ which is described in Annex B1.

If the cell is not listed in Annex B, set the stock concentration to 0,8% at the beginning of the flotation, the temperature to approximately 45 °C and the flotation time to a value when the status of hyper-flotation is reached. The status of hyper-flotation is reached when the increase of luminance is lower than 0,3 points per minute. This has to 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 heatset offset process) and following the procedures described in this method.

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

### **6.9 Specimen preparation**

For undeinked pulp two filter pads and for deinked pulp two filter pads and two laboratory handsheets 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. INGEDE Method 1 is used to prepare the specimens.

### **6.10 Analysis**

The following optical characteristics of air conditioned filter pads, laboratory handsheets and filtrate filters are determined using INGEDE Method 2.

- Luminance  $Y$  of undeinked and deinked pulp
- $L^*$ ,  $a^*$ ,  $b^*$  colour coefficients of undeinked and deinked pulp
- Ink elimination  $IE_{700}$
- Filtrate Darkening  $\Delta Y$  of deinked pulp
- Dirt particle area  $A$  of deinked pulp.

Measure the stock concentration to maintain required conditions, e. g. for storage and flotation. Use the filter pads of stock concentration measurements to determine the ash content of undeinked and deinked pulp in accordance with ISO 1762.

In order to calculate yield values (overall yield and fibre yield) make sure to measure the feed and the overflow of the flotation. Maintain the correct amount of oven dry pulp for the flotation process.

The flotation yield is calculated as follows:

Yield (Overall yield):

$$Yield = \frac{(c_{UP} \times m_{UP}) - (c_{froth} \times m_{froth})}{(c_{UP} \times m_{UP})} \times 100 \% \quad (7)$$

Where:

$c_{UP}$  stock concentration of undeinked pulp expressed as a percentage;

$m_{UP}$  feed mass flotation, undeinked pulp in kilograms (kg);

$c_{froth}$  stock concentration of overflow expressed as a percentage;

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

The fibre yield is calculated on basis of the overall yield with the ash content determined at 525 °C of the undeinked and the deinked pulp samples as follows:

Fibre Yield:

$$Fibre Yield = Yield \frac{(100\% - Ash_{DP})}{(100\% - Ash_{UP})} \quad (8)$$

Where:

$Ash_{DP}$  Ash content of deinked pulp expressed as a percentage;

$Ash_{UP}$  Ash content of undeinked pulp expressed as a percentage.

## **7 Report**

The following shall be recorded in the test report:

- identification of print product as complete as possible – name, publishing company, date of print, product category (according to EPRC Assessment of Printed Product Recyclability – Deinkability Score), part of product, print process, paper quality (furnish, finishing, purpose, basis weight, brightness of unprinted paper and ash content);
- if the test result is used to assess deinkability, it might be necessary to classify the print product into the proper product category;
- date of test;

- a reference to this method;
- mass-related proportion of all inserts (removed as well as tested with the print product (when it is a constituent paper-based element of the print product), expressed as a percentage (for each single insert);
- indication which inserts were used in the test, if any;
- number and type of adhesive applications;
- pH after pulping, before and after storage and before flotation;
- chemical dosage for pulping;
- type of flotation cell;
- feed mass of undeinked pulp for flotation  $m_{UP}$ ;
- stock concentration of undeinked pulp  $c_{UP}$ ;
- flotation yield in % (calculated automatically if using INGEDE LabResult template);
- fibre yield in % (calculated automatically if using INGEDE LabResult template);
- flotation overflow mass  $m_{froth}$ ;
- flotation overflow stock concentration  $c_{froth}$ ;
- ash content of undeinked and deinked pulp;
- luminance  $Y$  of undeinked and deinked pulp, filtrate and control water;
- $L^*$ ,  $a^*$  and  $b^*$  of undeinked and deinked pulp, filtrate and control water;
- Brightness  $R_{457}$  of undeinked and deinked pulp, filtrate and control water;
- $R_{\infty 700}$  of undeinked and deinked pulp, filtrate and control water;
- Ink elimination  $IE_{700}$  in % (calculated automatically if using INGEDE LabResult template);
- alternatively to  $IE_{700}$ , the ink elimination using *ERIC* values ( $IE_{ERIC}$ ) may be determined;
- filtrate darkening  $\Delta Y$  of the deinked pulp sample filtrate (calculated automatically if using INGEDE LabResult template);
- dirt particle area of deinked pulp in  $mm^2/m^2$  in six size classes with the dirt particle area 50 to 100  $\mu m$ , 100 to 150  $\mu m$ , 150 to 200  $\mu m$ , 200 to 250  $\mu m$ , 250 to 500  $\mu m$  and  $>500 \mu m$  as well as the two categories  $> 50 \mu m$  and the dirt particle area  $> 250 \mu m$ . The two categories are calculated automatically if using INGEDE LabResult template;
- dirt particle quality of deinked pulp in  $1/m^2$  in six size classes with the dirt particle area 50 to 100  $\mu m$ , 100 to 150  $\mu m$ , 150 to 200  $\mu m$ , 200 to 250  $\mu m$ , 250 to 500  $\mu m$  and  $>500 \mu m$  as well as the total count (this is calculated automatically if using INGEDE LabResult template);
- technician and institute.

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

Any further optical characteristics of undeinked and deinked pulp yielded as well as their respective filtrate quality may also be noted in the test report.

## **8 Bibliography**

### **8.1 Literature and other related documents**

- European Paper Recycling Council, Assessment of Print Product Recyclability – Deinkability Score, January 2017, [www.paperforrecycling.eu](http://www.paperforrecycling.eu)

### **8.2 Sources**

This method has been published for the first time in 2001. A major revision was done in 2007 according to the definitions made in INGEDE Project 85 02 CTP/PMV/PTS – European Deinkability Method. In 2009 criteria for the pH after pulping and before flotation were added. After gaining some experiences, procedures related to the pH criteria were added to the version of 2012. The major change from that version is the dependence of the pulping concentration from the ash content of the print product.

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**Annex A:**

**Testing the pH of a smaller sample amount**

In case of not having a sufficient amount of sample paper for repeating the disintegration, test a small amount of your sample beforehand. Use 10% oven dry sample amount calculated according to equation (1) (Chapter 6.2.9). Prepare 10% of the required amount of stock solution according to equation (3) (Chapter 6.3.3) and 10 g of the peroxide solution by using 10% of the calculated mass of hydrogen peroxide according to equation (4) (Chapter 6.3.3). Use the calculated sample, pour in the necessary preheated standard chemical formulation and fill up to 157 g with preheated dilution water. Disintegrate the sample with a dispersing device (e.g. hand blender, laboratory dispersing machine), stop after some seconds and add the prepared peroxide solution. Then disintegrate until the sample is pulped. Store the pulp at 45 °C for 20 min and determine the pH.

**Annex B**

**Flotation cells**

**B1 Voith Delta 25™**

The air supply has to 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 °C, stock concentration 0,8% at the beginning with 180 g oven-dry pulp.

During the flotation process add the necessary amount of 45 °C of dilution water 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.

**B2 PTS flotation cell**

Use the following settings for flotation: air supply rate 60 l/h, stirrer speed in suspension 1 200 min<sup>-1</sup>, flotation period 10 min, suspension temperature 45 °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.