INGEDE Method 11	Assessment of Print Product Recyclability – Deinkability Test –	INGEDE International Association of the Deinking Industry
July 2012 13 Pages		

Introduction

The good recyclability of printed products is a crucial feature for the sustainability of the graphic paper loop. It belongs to the focal work of INGEDE to safeguard and improve recyclability.

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

- Deinkability
- Screenability of adhesive applications.

Therefore a set of methods has been developed to simulate the common operating conditions of relevant process steps in an industrial deinking plant under standard conditions in a laboratory scale. This allows estimating the relative challenge a printed product means to a deinking plant. 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 grades. These papers usually are deinked in an alkaline environment. This is meant by the term “common operating conditions”. Printed paper products recovered by household collection together with newspapers and magazines are also treated under these common operating conditions.

This method has been developed for the assessment of the deinkability of individual printed products.

1 Scope

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

2 Terms and definitions

Deinked Pulp (DP):

- Pulp consisting of printed products deinked according to this method.

Undeinked Pulp (UP):

- Pulp consisting of printed products disintegrated mechanically with added deinking chemicals, prior to flotation.

3 Principle

Flotation is the most widely used technology for ink removal in the paper recycling process. This INGEDE 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 3.

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

Quality parameters:

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

Process parameters:

- Ink Elimination
- Filtrate darkening.

4 Equipment and auxiliaries

4.1 Equipment

- Warming cabinet with free or forced ventilation or with air turbulence according to ISO 287.
- Analytical balance up to 1 000 g with an accuracy of at least 0,001 g
- Analytical balance up to 3 000 g with an accuracy of at least 0,1 g
- Hobart pulper N 50, available from Hobart GmbH. Use the type of stirrer and a comparable cover, shown in the following figures. Additionally, it is possible to install a revolution counter, which stops the device automatically.

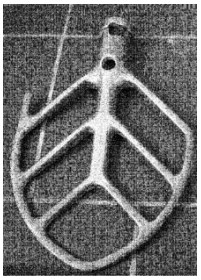

Figure 1: Stirrer for the Hobart pulper

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: PTS cell, Voith Delta 25™)
- 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.

4.2 Chemicals

- Sodium hydroxide (NaOH), pro analysis, CAS # 1310-73-2
- Sodium silicate 1,3–1,4 g/cm³ (38–40 °Bé)
- Hydrogen peroxide (H_2O_2), e.g. 35 %
- Oleic acid ($C_{18}H_{34}O_2$), extra pure, CAS # 112-80-1, e.g. Merck Article No. 1.00471
- Calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$), CAS # 10035-04-8

5 Procedure

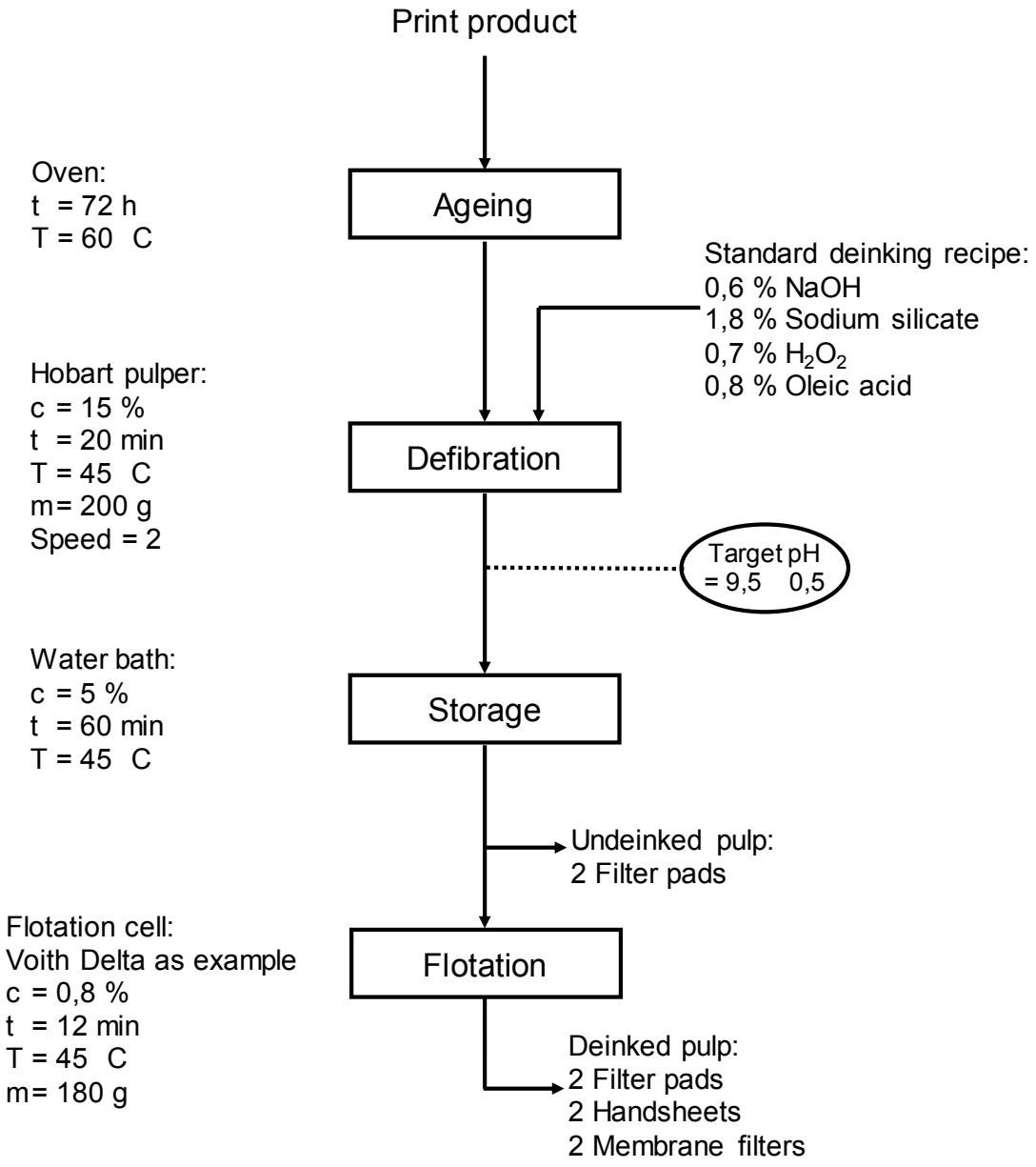

Figure 3: Procedure for testing deinkability with standard deinking recipe

5.1 Sampling

The printed samples used in the tests must not be split up. The minimum amount of each printed sample is 250 g oven-dry.

5.2 Identification

Each print product is designated by its title, publishing house, issue date, product category, printing method and paper grade, if available. Determine the ash content of the paper sample.

Weigh the complete printed product. After weighing, remove any inserts and non-paper components from the printed product to determine their share in the total mass of the product.

5.3 Separation of adhesive applications

To allow the sticky-forming potential of the printed product to be assessed independently, separate all evident adhesive applications from the paper, mark them according to their use, and store them separately.

Glued backs of magazines or catalogues shall be separated according to INGEDE Method 12.

5.4 Accelerated ageing

Place the samples in a warming cabinet for accelerated ageing at 60±3 °C for 72 hours

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.

5.5 Breaking up of samples

Accelerated aged samples are torn into pieces of about 2×2 cm^2 and acclimatised. A part of the acclimatised samples is used to determine the moisture content according to ISO 287 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.

5.6 Weighing the samples

After homogenising the samples by hand, weigh out samples of 200 g oven-dry.

5.7 Preparation of dilution water

During laboratory treatment of the printed products (5.9 to 5.13), use only water which has been treated to obtain the prescribed hardness values.

To obtain the desired water hardness, add calcium chloride dihydrate ($CaCl_2 \cdot 2\,H_2O$) in deionised water until the concentration of calcium ions reaches 3,21 mmol/l, equivalent to 472 mg/l. This is equivalent to 128 mg Ca^{2+}/l.

If tap water is used, 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).

5.8 Preparation and dosing of chemicals

The standard formulation is as follows:

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^3)*
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 5.10).

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

It is useful to prepare a total amount of 2 l stock solution which will be sufficient for 5 tests. Dissolve 6 g sodium hydroxide in deionised water, heat slightly to approx. 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 2 litres. The formation of soap reduces the alkalinity. 0,114 % sodium hydroxide is needed to neutralise the oleic acid.

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

5.9 Defibration

Fill the Hobart pulper with the prescribed sample quantity (200 g oven-dry). Take 400 ml of chemicals solution and fill up to a total volume of 1233 ml with appropriately heated dilution water. Add this deinking liquor into the vessel and run the Hobart pulper for some seconds. Then stop it, brush down any scrap of paper from the vessel wall. Repeat this step as often as necessary.

After the first stop, add the peroxide solution (100 ml). The stock consistency is now 15 %. Immediately after, disintegrate the stock for 20 min at approx. 45 °C, using rotor speed 2.

To maintain a constant temperature and avoid splashing losses, cover the vessel during disintegration, for example with a suitably sized, tight-fitting plastic lid (see Figure 2).

5.10 pH after defibration

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 is 9,5.

Using the standard formulation from chapter 5.8 the permitted range of pH is 9,5±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. If the pH is too low 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±0,2.

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

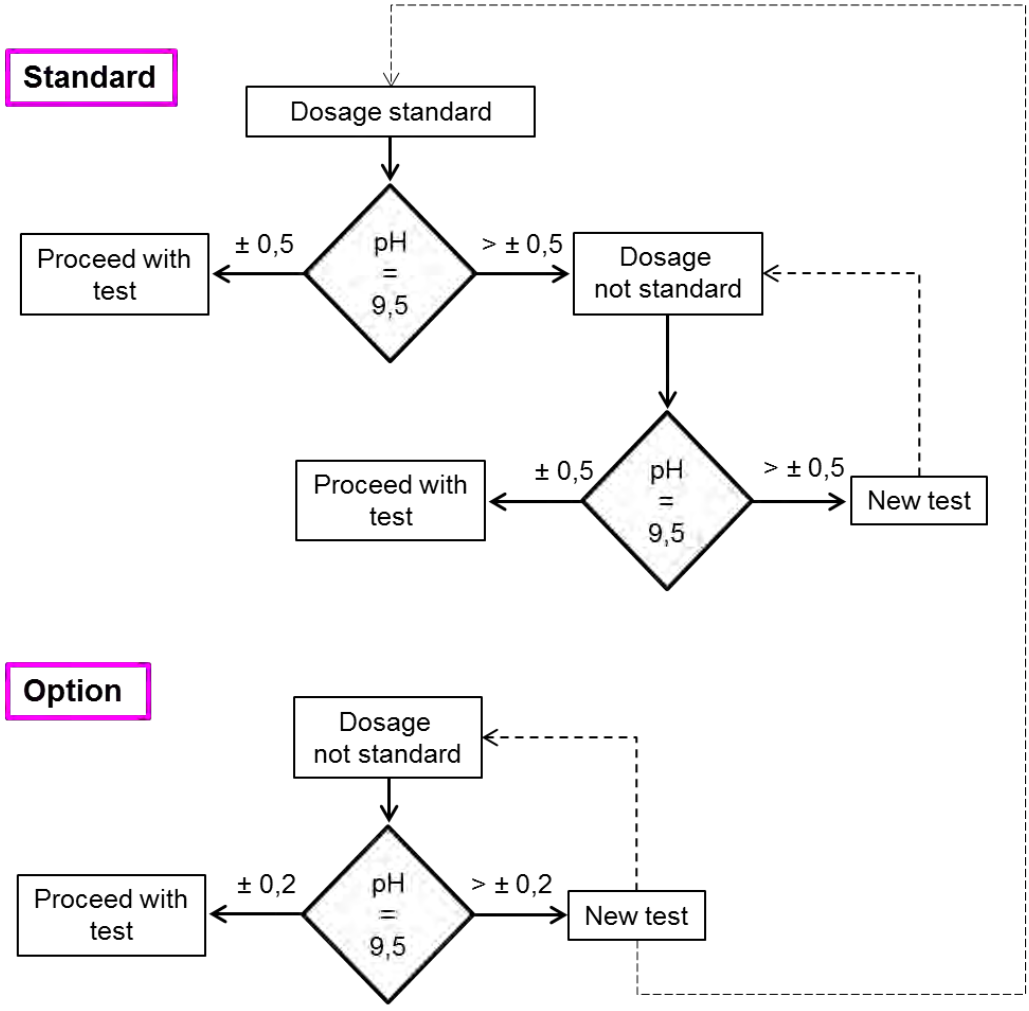

Figure 4: pH tolerances

ANNEX A describes a method to pre-test the pH after storage 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 defibration with 200 g oven-dry pulp. The requirements of pH tolerances must be fulfilled regardless the pre-test result.

5.11 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 5.14). 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 % consistency. The dilution water has been brought 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 consistency. 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.

5.12 Dilution

After storage the stock samples must be diluted with 45 °C warm water to terminate any chemical reaction before the treatment continues. Use tap water for the UP sample. For the pulp sample to be deinked, use water that has been brought to a temperature of 45 °C and to the desired level of hardness. The consistency after this dilution should be around 1 %; it can be the consistency required for flotation.

Measure the pH. At flotation consistency 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.

5.13 Flotation

Heat up the cell with hot water if the cell has big metal parts. 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. Add the diluted sample into the flotation cell. Fill up with dilution water, and proceed as the instructions of the flotation cell describes. The starting point for the flotation time is when the air supply is started. The process time is set in the following instructions for the recommended flotation cells. For other cells, the process should run until the status of hyperflotation is reached.

5.13.1 PTS flotation cell

Use the following settings for flotation: air supply rate 60 l/h, stirrer speed in suspension 1200 min^{-1}, flotation period 10 min, suspension temperature approx. 45 °C, consistency approx. 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. Determine the amount of the overflow oven-dry according to ISO 4119 and use this amount to calculate the flotation yield.

5.13.2 Voith Delta 25TM

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

During the flotation process add the necessary amount of 45 °C warm 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. Determine the amount of the overflow oven-dry according to ISO 4119, and use this amount to calculate the flotation yield.

5.13.3 Other laboratory flotation cells

Use flotation parameters and conditions similar to the standard conditions applied during the laboratory treatment of deinked recycled pulps.

The flotation should run until the status of hyperflotation is reached. Set the flotation time in order to get maximum luminosity and ink elimination.

5.14 Specimen preparation

For undeinked pulp two filter pads and for deinked pulp two filter pads and two laboratory hand-sheets 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.

5.15 Analysis

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

- Luminosity Y of deinked pulp
- L*, a*, b* colour coefficients of deinked pulp
- Ink elimination IE_{700} and/or IE_{ERIC}
- Filtrate Darkening ΔY of deinked pulp
- Dirt particle area A of deinked pulp.

Measure the stock consistency to maintain required conditions, e.g. for storage and flotation. Use the filter pads of stock consistency measurements to determine the ash content of un-deinked 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):

$$Yield = \frac{(c_{UP} \cdot m_{UP}) - (c_{froth} \cdot m_{froth})}{(c_{UP} \cdot m_{UP})} \cdot 100\,\%$$

Where:

c_{UP} in g/kg	stock consistency of undeinked pulp
m_{UP} in kg	feed amount flotation, undeinked pulp
c_{froth} in g/kg	stock consistency of overflow
m_{froth} in kg	overflow mass

Fibre Yield:

$$Fibre\,Yield = Yield \cdot \frac{(1 - Ash_{DP})}{(1 - Ash_{UP})}$$

Where:

Ash_{DP}	Ash content of deinked pulp in decimal
Ash_{UP}	Ash content of undeinked pulp in decimal (e. g. 0.03)

6 Report

The following should be recorded in the test report:

- Identification of print product as to name, publishing company, date of issue, product category, print process and paper quality, ash content
- Mass-related proportion of supplements and non-paper material in %
- Number and type of adhesive applications
- pH after pulping, before and after storage and before flotation
- Chemical dosage for pulping
- Ash content of undeinked and deinked pulp
- Flotation yield in %
- Fibre yield in %
- Overflow mass m_{froth}
- Overflow stock consistency c_{froth}
- Luminosity Y of deinked pulp.
- L*, a* and b* of deinked pulp
- Ink elimination IE_{700} in %, $R_{\infty,UP}$, $R_{\infty,DP}$ at 700 nm
- Alternatively to IE_{700}, the ink elimination using ERIC values (IE_{ERIC}) may be determined
- Filtrate darkening ΔY of the deinked pulp sample filtrate
- Dirt particle area of deinked pulp in mm²/m² in two categories with the dirt particle area > 50 µm and the dirt particle area > 250 µm.

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.

7 References

7.1 Cited Standards and methods

- 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 287 – Paper and board – Determination of moisture content of a lot – 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

7.2 Literature and other related documents

- European Recovered Paper Council, Assessment of Print Product Recyclability – Deinkability Score – User's Manual, March 2009, www.paperforrecycling.eu/

7.3 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 experience, procedures related to the pH criteria were added to this version.

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 20 g oven dry sample, pour in 40 ml of the pre-heated standard chemical formulation and fill up to 123 ml with preheated dilution water. Prepare 10 ml of the peroxide solution. 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 minutes and determine the pH.

Contact:
INGEDE e. V. (International Association of the Deinking Industry)
Office
Gerokstr 40
74321 Bietigheim-Bissingen, Germany
Tel. +49 7142 7742-81
Fax +49 7142 7742-80
E-Mail office@ingede.org
www.ingede.org