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Harmonised European laboratory test method to generate parameters enabling the assessment of the recyclability of paper and board products in recycling mills with conventional process (Part I)

Paper and Board – Recyclability Laboratory Test Method – Part I – Recycling mill with Conventional process

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1. INTRODUCTION

The paper and paper board value chain is an example for circularity, displaying very high recycling rates. In addition, technical innovations are leading to new products made from paper and cardboard as well as other cellulose fibre-based products, which are increasingly replacing other traditional packaging materials.

To maintain and further increase the sustainability and circularity of the paper and board value chain and to help EU Member States and other European countries meet high recycling targets[1], it is important to ensure that paper and board-based materials and other cellulose fibre-based products (e.g. moulded fibre products) are recyclable by the paper industry. The paper manufacturing and converting industry has issued joint guidance on paper-based packaging recyclability[2] at national and European levels. To confirm recyclability, it is necessary to define a harmonised test method as a basis for assessing the general recyclability of these materials and products.

The harmonised test method emulates the most common phases of the industrial processes to measure the main parameters of recyclability of paper and board-based materials and other cellulose fibre-based products based on current knowledge and technology.

This makes it possible to:

- Supplement the evaluation of recyclability required by EN 13430 with regard to paper and board-based materials and other cellulose fibre-based products that are sent for recycling in the paper industry.
- Guide eco-design, in terms of recyclability, of paper and board-based materials and other cellulose fibre-based products currently in use, as well as new materials under development and additives used in the converting phase that can affect the recyclability of the final product.
- Support declarations related to the recyclability of materials or products based on grading systems developed by third-party organizations.

2. SCOPE

This document describes a laboratory scale method for determining the key parameters for evaluating the level of recyclability of paper and board-based materials and other cellulose fibre-based products, e.g., moulded fibre products, emulating the relevant phases of conventional paper and board recycling mills without flotation-deinking technology[3] or other special features to recycle paper for producing new paper and board.

This method enables analysing both process parameters (coarse reject, fine reject, dissolved and colloidal substances and sticky particles with a diameter smaller than 2 mm) and quality parameters (sheet formation and interfering materials like adhesiveness and visual impurities). This document considers only the minimum characteristics of paper and board products that can be generally recycled with conventional technologies. Therefore, it does not take into consideration additional specifications necessary to valorise the paper and board products using flotation-deinking technologies. It also does not include parameters of recyclability in mills with specialised processing technology.

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[1] E.g. directives 2018/851/EU, 2018/852/EU set high recycling targets for municipal waste and paper-based packaging (85% by 2025, 90% by 2030).
[2] Cepi, FEFCO, Citpa, ACE: Paper-based packaging recyclability guidelines. 2019. Confederation of Paper Industries: Paper and Board Packaging Recyclability Guidelines,Revision One, Published January 2020.
[3] EPRC: Assessment of printed product recyclability: Deinkability score User's manual, www.paperforrecycling.eu

3. NORMATIVE REFERENCES

This document incorporates, by way of dated or undated references, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed below. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated into it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

DIN 38409-1
German standard methods for the examination of water, wastewater and sludge; parameters characterizing effects and substances (group H); determination of total dry residue, filtrate dry residue and residue on ignition (H 1)

UNI 11 743
Paper and board - Determination of parameters of recyclability of cellulose-based materials and products

EN 643
Paper and board - European list of standard grades of paper and board for recycling

TAPPI/ANSI T 275
Screening of pulp (Somerville-type equipment)

EN 12231-11
Food processing machinery - Mincing machines - Safety and hygiene requirements

EN 13430
Packaging – Requirements for packaging recoverable by material recycling

ISO 638-1
Paper, board and pulps — Determination of dry matter content by oven drying method — Part 1: Solid materials

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

ISO 4046
Paper, board, pulps and related terms – Vocabulary

ISO 4119
Pulps – Determination of stock concentration

ISO 5263-1
Pulps - Laboratory wet disintegration - Part 1: Disintegration of chemical pulps

ISO 5269-2
Pulps - Preparation of laboratory sheets for physical testing Part 2: Rapid-Koethen method

ISO 15360-2
Recycled pulps — Estimation of Stickies and Plastics — Part 2: Image analysis method

ISO 15705
Water quality - Determination of the chemical oxygen demand index (ST-COD) - Small-scale sealed-tube method

4. TERMS AND DEFINITIONS

While the term “paper” is defined by ISO 4046, the following terms and definitions are applicable for the purpose of this standard (note: the scope of the standard includes paper and board-based materials and other cellulose fibre-based products like moulded fibre products.

Paper and board: a web comprising substantially (at least 50 %) of cellulosic fibres forming hydrogen bridge bonds, which may contain fillers and coatings. Moulded fibres are also included in this definition.

Cellulose fibre-based products: Finished objects (such as packaging, printed materials, articles for domestic use, etc.) comprised of over 50 % (in weight) of paper and board.

Paper and board for recycling (EN 643): Natural fibre-based paper and board suitable for recycling and consisting of:

- paper and board in any shape.
- products made predominately from paper and board, which may include other constituents that cannot be removed by dry sorting, such as coatings, laminates, spiral bindings, etc.

Use of paper and board for recycling: This refers to the processes used in the recycling of paper and board in the paper industry. These processes include mainly the pulping of paper and board for recycling, the separation of non-cellulose components, and the cleansing of the recovered pulp. Different types of equipment are used depending on the type of paper and board to be recycled and the end product requirements.

Recyclability: Ability to be treated in a recovered paper treatment plant according to recognised rules of engineering so as to ensure that the secondary fibre furnish allows the undisturbed and cost-effective manufacture of a recycled fibre-based new paper of acceptable quality.

5. PRINCIPLE

The recyclability of materials or products made predominately from paper and board is determined by means of laboratory procedures that emulates the most relevant industrial phases in a conventional paper and board recycling mill dedicated to the recycling of paper and board. In particular, this method defines the parameters of interest for the verification of recyclability as follows:

- Ease at which the fibres can be separated using equipment of a recycling mill with conventional process.
- The potential to form sheets out of the recovered fibres without significant disruption.
- The visual appearance when formed into sheets.
- The level of coarse and fine rejects.
- The level of fragmentation of disrupting materials (adhesives, metals, plastic film).
- The level of or colloidal substances below 12 microns resulting from non-paper components in the tested sample.

A flowchart showing the different phases of the method is shown in annex A.

6. TEST EQUIPMENT AND MATERIALS

6.1 Test equipment

1. Analytical balance with accuracy of ± 0.01 g
2. Barrels for collecting the accept from coarse and fine screening
3. Beakers
4. Büchner funnel (diameter 125 mm and 150 mm) equipped with suction flask and water jet pump
5. Couching roller for the sheet formation
6. Cutting mat for photo documentation (optional)
7 Cuvette heating block (temperature 150 °C ± 5 °C) for the COD determination (optional)
8. Cuvette rack for the COD determination (optional)
9. Drying oven (temperature 60 °C ± 1 °C, temperature 105 °C ± 2 °C and temperature 130 °C ± 2 °C)
10. Eppendorf variable pipette 1,000 – 5,000 µL for the COD determination (optional)
11. Glass Bottle to store the filtrate (optional)
12. Image analysis system comprising4:
13. Scanner (e.g. EPSON V-750 PRO) with minimal optical resolution of 2000 dpi
14. Software for analysing area and size distribution of adhesive particles (macrostickies), complaint with ISO 15360-2
15. LED light Panel for the photo documentation (optional)
16. Perforated plate (hole diameter 5 mm) for coarse screening in Somerville
17. Photometer measuring device for the COD determination (optional)
18. Metal plates (pressure 1.18 kPa or 3.7 kg, 20 cm diameter) for the sheet adhesion test
19. Metal plates (pressure 0.95 kPa or 6 kg, 28 cm diameter) for the macrostickies determination (optional)
20. Rapid-Koethen sheet former compliant with ISO 5269-2 (If another sheet former is used, it has to be proved that this makes no difference to the method)
21. Refrigerator to store the filtrate (optional)
22. Scissors / cutting machine / punch
23. Slotted plate (slot size 150 µm) for fine screening in Somerville
24. Somerville-fractionator compliant with TAPPI/ANSI T275
25. Standard disintegrator compliant with ISO 5263-1
26. Stopwatch / Timer Somerville-fractionator
27. Submersible pump (optional)
28. Thermometer digital
29. Vacuum desiccator

6.2 Materials

1. Aluminium trays for the determination of the evaporation residue
2. Black water-based ink, e.g. Pelikan No. 4001, compliant with ISO 15 360 (optional)
3. Carrier board and cover sheets
4. Corundum powder, compliant with ISO 15 360 for the macrostickies determination (optional)
5. Cuvette tests e.g. COD cuvette test 15-150 mg/LO_2 (optional)
6. Filter paper grade 388 diameter 125 mm (basis weight 84 g/m², filtration speed 10 s/10 ml, deposition range 12 - 15 µm)
7. Filter paper grade 388 diameter 150 mm (basis weight 84 g/m², filtration speed 10 s/10 ml, deposition range 12 - 15 µm)
8. Filter paper grade 1289 diameter 240 mm (basis weight 84 g/m², filtration speed 20 s/10 ml, deposition range 8 – 12 µm) (optional)
9. Silicon paper (60 g/m2) compliant with ISO 15 360 for the macrostickies determination (optional)

7. SAMPLE PREPARATION

The quantity of tested material or product must be sufficient to carry out all the measurements indicated by the method. An indicative quantity is 250 g air-dry weight.

Perform a double determination of the moisture content of the product or material in compliance with ISO 638-1.

Weigh one aliquot of air-dried material or product, with a precision of ± 0.01 g corresponding approximately to (50 ± 1) g dry weight.

If the tested product weigh less than 50 g, weigh an additional fraction of product in order to obtain a sample with a total aliquot of 50 g dry weight.

If the product or material weights more than 50 g, it is necessary to ensure that the sample contains the same proportion of elements different from the base product or material (e.g. labels, seals, hot-melt application, metallisation, ink application, etc) as the tested product or material.

Any relevant information allowing a correct and proportional sampling must be present in the technical data sheet provided with the sample. The technical data sheet must contain the minimum content of information indicated in the Annex G.

Cut the sample into pieces of 3 cm x 3 cm (± 0.5 cm) in size.

To avoid problems with the functioning of the disintegrator, easily-removable non-paper components, like metal clips and parts of rigid plastic material, can be removed and weighed separately from the rest of the sample, as dry removed components. The weight should not be included in the 50 g oven dry material for disintegration, but added to the sum of the coarse reject.

All sample quantities indicated hereinafter refer to the calculated dry weight of matter dried in an oven at (105 ± 2) °C.

If the material or product contains wet strength agents (WSA) and has been produced less than 30 days before the test, it has to be stored for the remaining time needed to reach 30 days from the date of production. Alternatively, it can be aged at 60°± 1 C for 72 hours. The accelerated aging is needed to mimic the natural aging of the material between production and recycling where for example a post curing of wet strength agents may still happen. In case of materials and products without WSA, make sure the sample is at least 15 days old from the date of production, therefore no aging is necessary.

For more details about the sample preparation, like examples for complex sample preparation, the sample preparation of packaging aids and the photo documentation, check Annex E Detailed work description Sample preparation, Version 1.

8. GENERAL PROCEDURE

The method comprises the following phases:

- Disintegration;
- Filtrate Analysis;
- Determination of the 5 mm hole residue (Coarse Reject);
- Determination of the consistency after the coarse screening (AC);
- Sheet adhesion test and visual appearance test of the accept of the coarse screening;
- Determination of the 150 µm slot residue (Fine Reject);
- Reject Characterisation
- Sheet adhesion test and visual appearance test of the accept of the fine screening
- Determination of the content of adhesive particles (macrostickies);

8.1 Disintegration

This section entails the sample disintegration and preparation of the total stock for the subsequent analyses.

Disintegrate (50 ± 1) g oven dry sample using the disintegrator compliant with ISO 5263-1, but diluting the sample with tap water at (40 ± 1) °C, the pH mildly alkaline (7 – 8). The total volume of sample and water must be approximately 2,000 g, so that a stock concentration of 2.5 % is achieved. No pre-wetting or soaking of the sample is allowed. The disintegration time is 10 min (30,000 revolutions).

The disintegration time can be prolonged to 20 minutes, in a second batch under the same conditions, when the Total Screening Reject (TSR) is 15 % (TSR= CR + 0.9*FR) or higher and a significant amount of fibres is present in the coarse reject (CR) and / or the fine reject (FR).

In this case, the results of the measurement taken after 10 min and 20 min disintegration must be included in the Test Report.

For more detailed information about the disintegration, check the detailed work description in Annex F Detailed work description Recyclability Laboratory Test Method - Part I.

8.2 Filtrate Analysis

This section describes the determination of the evaporation residue and, optionally, the chemical oxygen demand in the filtrate of the total stock.

Perform the filtration of the total stock immediately after the disintegration. Homogenise the total stock and filtrate a known amount of it (approximately 100 g) over a filter paper (diameter 150 mm) using the Büchner funnel, if possible, without moisturising the filter paper. Use the filtrate to rinse the suction flask and return the filtrate and the filter cake to the total stock.

Filter again 200 g of total stock, retrieve the filtrate from the suction flask and filter it again using the same paper filter. Save the filtrate for the further procedures and return the filter cake to the total stock.

For more detailed information about the filtration, like the photo documentation of the filtrate, check the detailed work description in Annex F Detailed work description Recyclability Laboratory Test Method - Part.

8.2.1 Evaporation residue

After the filtration, proceed with the determination of the evaporation residue. Pour a known amount (approximately 70 g) of filtrate on a previously weighed aluminium tray and dry it in the oven at (105 ± 2) °C until reaching a constant mass in accordance with DIN 38409-1. Perform a double determination of the evaporation residue.

If necessary, store the remaining filtrate in the refrigerator at 4 °C it for further analyses.

Determine the evaporation residue of the tap water used in the disintegration.

The evaporation residue (ER) in (g residue / kg packaging) is calculated as follows:

$$ER\ filtrate\ (g\ residue/g\ filtrate) = \frac{m_3\ (g) - m_1(g)}{m_2\ (g)}$$

$$ER\ sample\ (g\ residue/g\ filtrate) = ER\ filtrate\ (g/g) - ER\ tap\ water\ (g/g)$$

$$ER\ Packaging\ (\square) = \frac{\square}{\square} c\ (\ )$$

$Packaging\ mass\ (\square)\ (\square)$ m1 = mass of the empty aluminium tray

m_2 = mass of the filtrate that was taken in

m_3 = mass of tray after drying

c = stock concentration after disintegration, expressed in decimals

8.2.2 Chemical oxygen demand – COD (optional)

Perform a double determination of the chemical oxygen demand immediately after the filtration or until 24 h after it, if the filtrate is stored in the refrigerator.

Select the COD cuvette with the expected measuring range for the tests according to ISO 15705.

Following the cuvette test instructions, homogenise the cuvette solution and the filtrate, then pipette the volume of filtrate required for the test range into the cuvette.

If the cuvette solution immediately presents a green coloration, discard the sample and either use a higher measuring range or dilute the filtrate with deionised water.

Invert the cuvettes to stir the preparation and heat them in the heating block for 2 h at 148 °C or as describes acc. to the cuvette testing kit. Then, invert the cuvettes again and cool them to room temperature in the cuvette rack. Lastly, measure the COD using a photometer with absorbance of 600 nm. Repeat the same procedure using tap water from the same source that was used for the disintegration of the sample. If the COD of the tap water is higher than 15 mg O_2/L, it is recommended to measure this blank value, each time a sample is analysed.

The COD of the sample in (g O_2 / kg packaging) is calculated as follows:

$$COD\ sample\ (\frac{mg\ O_2}{L}) = COD\ filtrate\ (\frac{mg\ O_2}{L}) - COD\ tap\ water\ (\frac{mg\ O_2}{L})$$

$$COD\ (\frac{\square}{kg\ Packaging}) = \frac{\square\ sample\ (\frac{\square}{L})\ (\frac{\square}{\square})}{c\ (\ )\ (\frac{\square}{\square})}$$

8.2.3 Biological oxygen demand – BOD_5 (optional)

When the COD results is available, select the appropriate range of the BOD cuvette and perform the biological oxygen demand acc. to the ISO 5815-1:2019-07 as double determination.

Homogenise the filtrate and dilute it according to the manufacturer's instruction for the selected measurement range of the BOD cuvette test. If the measured COD value is so high that no BOD measurement range of the manufacturer can be used, a pre-dilution is necessary. In these cases, also note down the pre-dilution factor as well as the normal dilution factor of the selected measured value range. Carry out the cuvette test according to the manufacturer's instructions. The BOD is measured after 5 days using a photometer with absorbance of 600 nm.

The calculation of the BOD is similar to the calculation of the COD. Please note that the dilution of the filtrate for the cuvette range is already considered during the photometer measurement and only in cases of an additional pre-dilution, this pre-dilution factor has to be considered in the calculation.

The BOD of the sample in (g O_2 / kg packaging) is calculated as follows:

$$OD\ sample\ (\frac{\square}{L}) = BOD\ filtrate\ (\frac{\square}{L})\ x\ predilution\ factor - BOD\ tap\ water\ (\frac{\square}{L})$$

$$BOD\ (g\ O_2/kg\ Packaging\ ) = \frac{(\frac{\square}{L})}{c\ (\ )}$$

If the tap water did not have to be considered in the calculation of the COD due to a lower value than 5 mg O_2/L, then the tap water also does not have to be considered in the BOD calculation.

8.3 Determination of the 5 mm hole residue (Coarse Reject)

This section entails the determination of the coarse reject from the total stock according to the TAPPI/ANSI T 275.

Right after the filtration, as described in 8.2, proceed with the coarse screening using the Somerville fractionator equipped with a perforated plate containing holes with 5 mm of diameter and set with a water flow of (8.6 ± 0.2) l min. The required characteristics of the referred plate are set out in the Annex I Thickener Coarse screening plate.

Wait until the screening plate is covered by approximately 2.5 cm of water to pour in the total stock (including the filter cake mentioned in the filtrate analysis). Perform the coarse screening for 5 min, starting to counting the time when the sample starts to overflow the weir.

Collect the accept of the coarse screening in a specific container to be used for the subsequent procedures: determination of the concentration, sheet formation, fine screening and macrostickies determination (optional).

On completion of the test, first proceed with the reject characterisation described below (topic 8.8). Then, transfer all the reject remaining on the plate to a specific container and wash the plate with a sufficient amount of water to ensure it is completely clean, making sure that any fragments trapped in the holes of the screening plate are also recovered and added to the reject. Any particles and fragments that are trapped in the body of the Somerville fractionator have to be transferred as good as possible, to the coarse screening accept.

Filter the reject over a rapid paper filter with 125 mm of diameter previously calibrated in the oven at (105 ± 2) °C using a Büchner funnel. Then, dry the filter paper in the oven at (105 ± 2) °C, until reaching a constant mass according to ISO 638-1.

Calculate the dry weight of the coarse reject, net of the weight of the paper filter, and express the result as a percentage with respect to the dry weight of the initial sample. The results can be rounded to the first decimal place. Report any dry-removed components as part of the coarse reject.

For more details like the photo documentation of the coarse reject check the detailed work description in Annex F Detailed work description Recyclability Laboratory Test Method - Part.

8.4 Determination of the concentration after the coarse screening (AC)

This section entails the determination of the stock consistency according to EN ISO 4119 (stock consistency between 0.05 % and 0.3 %).

Homogenise the accepted fraction of the coarse screening and transfer a known amount of it (approximately 1000 ml) into the tared beaker.

Filter the 1000 ml of the accept over a rapid paper filter with 125 mm – 150 mm of diameter previously calibrated in the oven at (105 ± 2) °C using the Büchner funnel. Then, place the filter paper between two cover sheets and dry each side of it in the dryer of the sheet former for 7 min (93 ± 4) °C. Leave the filter paper to cool down in the desiccator before weighing it.

Determine the dry mass of the filter cake as follows:

$$c\,(\%) = \frac{m_{3\,(g)} - m_{2\,(g)}}{m_{1\,(g)}}\, x\ 100$$

m_1 = mass of sample before drying
m_2 = mass of filter paper without sample
m_3 = mass of sample with filter paper after drying

8.5 Sheet adhesion test and visual appearance test of the accept of the coarse screening

This section entails checking the adhesiveness and visual appearance of the sheets produced from the accepted fraction of the coarse screening.

After homogenising the accepted fraction, take a sufficient amount of pulp to form two hand sheets with (60 ± 2) g/m² (corresponding to approximately 1.8 g in dry weight for each sheet). If the target weight (60 ± 2) g/m² is not reached, adjust the amount of pulp required for sheet formation.

For performing the sheet adhesion test, place the lab sheet between a carrier board (bottom side) and a cover sheet (top side) and pre-dry them into the dryer of the sheet former for 7 min (93 ± 4) °C. Then, dry them, without removing the support and cover, in the oven at (130 ± 2) °C between two preheated brass or metal plates (pressure of 1.18 kPa or 3.7 kg) for 2 min. Finally, cool them down in the desiccator for approximately 10 min.

Then, perform the sheet adhesion test immediately after taking the lab sheet out of the desiccator by separating the carrier board first and then the cover sheet with a fluent motion from lab sheet itself and assess any damage or breakages using a bright oblique light, acc. to the following levels:

- Level 1 Tackiness absent: the handsheet can be separated completely from the carrier board and cover sheet without any damage or breakages. A few single fibre pickups can be present on the carrier board and cover sheet. Visible damage to the handsheets and fragments of paper on the carrier board and cover sheet are not permitted.

- Level 2 Tackiness partly present: the handsheet can be separated completely from the carrier board and cover sheet. Fibre tears and particles occur on the carrier board, the cover sheet and the handsheet itself.

- Level 3 Tackiness present: the handsheet cannot be separated from its carrier board and the cover sheet without a visible damage to the handsheet itself. A breaking of the handsheet or holes (> 1mm [in two dimensions]) occur.

It needs to be ensured that the rating is an average of all sheet adhesion tests carried out. The rating should comply to the overall impression of the sheet adhesion test. In case of a single hole, e.g. despite the absence of fibre tears. A single occurrence of a defect can be neglected.

Example pictures of the three categories as well as a video showing the performance of the sheet adhesion test can be accessed in the Annex C Sheet Adhesion Test Version January 2025.

Assess the visual impurities by inspecting the lab sheets used in the sheet adhesion test for the presence of the impurities outlined in the Annex B Visual impurities. This inspection should be conducted visually with and without a source of transmitted light. Based on the observations, assign the appropriate rating given by the decision tree.

For more details like the photo documentation of the sheet adhesion test and visual impurities check the detailed work description in Annex F Detailed work description Recyclability Laboratory Test Method – Part I.

8.6 Determination of the 150 µm slot residue (Fine Reject)

This section entails the determination of the fine reject present in the accept after the coarse screening.

Proceed with the fine screening using the Somerville fractionator equipped with a plate containing slots with 0.15 mm of width and set with a water flow of (8.6 ± 0.2) l/min.

Homogenise the accept from the coarse screening and take an aliquot of 20 g oven-dry for the fine screening.

Wait until the screening plate is covered by approximately 2.5 cm of water to pour in the 20 g oven-dry accept. The pouring time should not be longer than 4 min. Perform the fine screening for 20 min, starting to counting the time when the sample starts to overflow the weir. During the screening, collect at least the first 50 l of the sorted material to be used for assessing the second adhesive test and visual impurities.

If possible collect the total amount fine screening accept and work with a thickener to do so. Use the collected and thickened pulp to perform the sheet adhesion test and the assessment of visual impurities. Description of the Thickener in the Technical Annex I Thickener Coarse screening plate. The use of the entire fine screening accept material is preferred for reasons of representativeness. Make sure that, in case a thickener is used, no unforeseen washing of the suspension is performed.

On the completion of the test first proceed with the reject characterisation described below (topic 8.8). Then, transfer all the reject remaining on the plate to a specific container and wash the plate with a sufficient amount of water to ensure it is completely clean, making sure that any fragments trapped in the slots are also recovered and added to the reject.

Filter the reject over a rapid paper filter with 125 mm – 150 mm of diameter previously calibrated in the oven at (105 ± 2) °C, using a Büchner funnel. Then, place the filter paper between two cover sheets and dry the filter paper in the oven at (105 ± 2) °C, until reaching a constant mass according to ISO 638-1.

Calculate the dry weight of the fine reject, net of the weight of the paper filter, and express the result as a percentage with respect to dry weight of the accepted pulp aliquot used for the test and to the dry weight of the initial sample. The results can be rounded to the first decimal place.

For more details like the photo documentation of the coarse reject, check the detailed work description in Annex F Detailed work description Recyclability Laboratory Test Method - Part I.

8.7 Sheet adhesion test and visual appearance test of the accept of the fine screening

Please perform the sheet adhesion test and visual appearance inspection as described in Chapter 8.5.

8.8 Reject characterisation (RC)

This section describes the procedure for the reject characterisation according to the Annex D Reject characterisation. The reject characterisation should be carried out after the coarse and fine screening steps are complete, while the rejects are still laying on the screening plates.

In the absence of rejects on screening plates, tick the box “reject absent” and the characterisation process is concluded. On the other hand, if there are rejects left on the plate, observe them to check to which category they belong (cellulose fibres and flakes, polymer barrier coating, adhesives, metallised film, metal and aluminium, mix and others). Then, if applicable, also check their degree of disintegration (not disintegrated and partly disintegrated material) and fragmentation (unfragmented, partly fragmented and completely fragmented). Tick the boxes in the decision tree accordingly to what you have observed. You can tick as many boxes as you need. Further comments can be added in the field: additional comments. Photograph the reject, with special attention on the different reject categories found.

Observations: The category mix should only be selected if there are fibres and/or flakes combined with polymer barrier, metallised films, metal/aluminium, and/or others The field significant (S) refer to the reject amount and is optional, but still important, since the presence of significant amount of fibres is one of the conditions for the 20 min disintegration option.

For visual examples of the reject characterisation check the Annex D Reject characterisation.

8.9 Measurement of adhesive particles - macrostickies (optional)

This section entails evaluating the quantity of adhesive particles (so-called macrostickies) present in the accepted fraction of the coarse screening. Proceed with the measurement of macrostickies according to ISO 15360-2.

Perform the screening in the Somerville fractionator equipped with the 150 µm perforated plate for 10 min and set to work with a water flow of (8.6 ± 0.2) l/min.

Homogenise the accept from the coarse screening and take two aliquots of 5 g oven dry pulp for double macrostickies determination. If the accepted material is not enough to complete the test, the steps 8.1, 8.3 and 8.4 must be repeated to recover sufficient material.

Wait until the screening plate is covered by approximately 2.5 cm of water to pour in the first aliquot of 5 g equivalent oven-dry of the accept. Perform the fine screening for 10 min, starting to count when the sample starts to overflow the weir.

On completion of the test, transfer the reject on the plate in a suitable container and wash the plate with a sufficient amount of water to ensure it is completely clean, making sure that any fragments trapped in the slots are also recovered and added to the reject.

If the reject quantity is too high, so that stickies may overlap each other, it should be split on to several filter papers. A reduction of screening amount or an extension of screening time is not foreseen.

In case the reject should be split onto several filter papers, divide the residue into 2 or more equal parts on 2 or more filters; ideally do not divide into more than 5 equal parts, so as to have at least a residue corresponding to 1 g of accepted stock on each filter.

If after a deviation onto five filters, overlapping of particles is still overserved, further distribution of the Macrostickies has to be performed, as long as no overlapping is observed. In that case, the second Macrostickies screening of 5g can be skipped.

To carry out the division, once the residue has been collected in the container, bring it to a known volume and, under constant stirring, divide into 2 or more equal parts in different containers. With these, make a corresponding number of filter sheet samples.

Place a labelled filter sheet (grade 1289 diameter 240 mm) on the Rapid Koethen sheet former, moisten and smooth it. Then, pour the reject or one aliquot of reject in the Rapid Koethen sheet former to aspirate the water from it. Ensure that any particles adhered to the sheet former column are recovered and added to the reject.

Carefully remove the filter from the sheet former, place it between the carrier board (bottom side) and a silicon paper (siliconized side in contact with the stickies). Dry it in the dryer of the sheet former for 10 min (93 ± 4) °C.

Pour the black ink into a container onto a plate and dip the filter sheet into the ink. Place the filter sheet over a piece of blotting paper to absorb the ink excess. Place the dyed filter sheet between a carrier board (bottom side) and a silicon paper (top side). Dry it in the dryer of the sheet former for 10 min (93 ± 4) °C.

Pulverise the sticky side of the filter sheet with a closed layer of corundum powder using 100 g powder per filter. Place the filter sheet with the underside on one carrier board and cover it with another carrier board. Place the filter sheet between two preheated metal plates (pressure of 0.950 kPa or weighing 6 kg) for 10 min in a drying oven at (105 ± 2) °C.

If you make sure that the powder is kept clean, it can be reused up to 10 times.

Immediately after the filter is removed from the oven, gently remove the excess of corundum powder using a soft and gentle cosmetic brush and check if the stickies overlap each other or if there are other sources of contamination. If so, either remove the hydrophobic contamination (e.g. plastic pieces) by hand using tweezers or colour them black using a permanent marker. If the stickies overlap each other, repeat the determination splitting the reject into two or more filters as described above. Perform the same procedure with the second aliquot of 5 g oven dry pulp.

Use an image analysis software (e.g. PTS-DOMAS multispec) to measure the macrostickies area on each filter sheet.

In case the reject was divided into several filter sheets, measure the macrostickies area on all the filter sheets.

According to the image analysis software used, set the dimensional limits of the particle classes at a minimum of 0.15 mm and a maximum of 2 mm in equivalent diameter.

The area of macro stickies with diameter smaller than 2 mm must be calculated in mm^2 per kg of initial sample.

Report the average value of the macrostickies area, measured in all the filter sheets obtained in the different repetitions, rounding the result to the nearest ten.

9. TEST REPORT

The test report must include at least the following information:

a) Reference to this standard;
b) Description of the material or product specifying the following:
   > Reference of the tested sample (product name or number), reference to the technical data sheet, production date of sample
   > A summary of the main information included on the sample data sheet (grammage, materials and shares in case of multilayer materials, adhesives, sealing, printing, metallisation, accessory components or other specific features useful to identify the sample)
   > Specific sample preparation, if any (e.g. emptying, removal of manually separable accessories intended to be removed before disposal)
   > Finished product or intermediate (component/ constituent)
      i. Semi-finished - **sheets** of packaging material / substrate (paper, cardboard, solid board, corrugated board)
      ii. Semi-finished - **sheets** of packaging material / substrate with "upgrading" (polymer/metal coating, print, varnish)
      iii. Finished product - intermediate, not yet ready to be used
      iv. Finished product - ready to be used
      v. Finished product - used
   > Type of software used for image analysis
   > Photographic documentation of the material or product and its specimen during the testing, using transmitted and oblique light. See Annex F for more details.
   > Results of the test expressed in compliance with the criteria established in respectively paragraphs:
      • Coarse reject, expressed as a percentage, description of fibres/non-fibres and fragmentation
      • Dissolved and colloidal substances, expressed as in mg/g tested sample
      • Adhesiveness of the accept of the coarse screening, expressed as "absent", "partly present" or "present" and visual appearance, expressed with a level rating
      • Fine reject expressed as a percentage, description of fibres/non- fibres and fragmentation
      • Adhesiveness of the accept of the fine screening, expressed as "absent", "partly present" or "present" and visual appearance, expressed with a level rating
   > Date and place of the test
   > Any deviation from the specified test procedure
   > In the event that it has not been possible to perform all steps of the test method in accordance with this standard or it is not possible to determine one or more measurement parameters due to the nature and/or characteristics of the sample material or product, the circumstance must be reported by the laboratory in the test report

Examples:

   > Pulping resistance prevents the disintegrator from working or there is a risk of damage to the equipment
   > Presence of dense flakes or foams prevents the transfer of the accept to the next stage
   > Presence of metal particles or wet resistant resins distorts the assessment of macro stickies

Additionally, the test report may include the following information:

a) An indication of the adhesive particles (macrostickies) content expressed as a total area (including those greater than 2 mm equivalent diameter), expressed as mm^2 of the macrostickies' area per kg of the sample as-is, in compliance with the ISO 15360-2 standard
b) An indication of the ash content of the paper product or material determined in compliance with the ISO 1762 standard
c) Results of the test expressed in compliance with the criteria established respectively in paragraphs 8.5 and 8.7 (adhesiveness, expressed as "absent", "partly present" or "present" and optical inhomogeneity, expressed with a level rating of an additional adhesiveness and optical inhomogeneity) performed on the accept of coarse screen phase
d) Any specific comments, such as:
   > specific observations, e.g. changes in consistency after disintegration, long drainage time during sheet formation, foam formation
e) Further photographic documentation regarding the results, as detailed in point f)

Table of annexes
(see separate annex documents)

A) Annex part I method flowchart[4]

B) Annex visual impurities[5]

C) Annex Sheet adhesion[6]

D) Annex reject characterisation[7]

E) Annex detailed work description: sample preparation[8]

F) Annex detailed work description: recyclability laboratory test method – part I: recycling mill with conventional process[9]

G) Technical data sheet[10]

H) Description of the plate for evaluation of the coarse reject[11]

I) Description of possible Thickener[12]

J) Laboratory report template[13]

[4] 4evergreen, January 2025. Part I - Recycling mill with conventional process.
Available here: https://4evergreenforum.eu/wp-content/uploads/4evergreen-Annexes-PART-I-25.02.pdf
[5] Ibid
[6] Ibid
[7] Ibid
[8] Ibid
[9] Ibid
[10] Available here: https://4evergreenforum.eu/wp-content/uploads/Technical_Sample_Datasheet_4Evergreen_v25.01.xlsx
[11] Technical annexes to the Harmonised European laboratory test method to generate parameters enabling the assessment of the recyclability of paper and board products in recycling mills with conventional process (Part I).
Available here: https://www.cepi.org/wp-content/uploads/2025/02/REC-25-023_Technical-Annex_Thickener_Coarse-screening-plate.pdf
[12] Ibid
[13] Available here: https://4evergreenforum.eu/wp-content/uploads/Laboratory-report-template_method-Part-I_V25.2.xlsx

About Cepi

Cepi is the European association representing the paper industry. We offer a wide range of renewable and recyclable wood-based fibre solutions to EU citizens: from packaging to textile, hygiene and tissue products, printing and graphic papers as well as speciality papers, but also bio-chemicals for food and pharmaceuticals, bio-composites and bioenergy. We are a responsible industry: 85% of our raw materials are sourced from within the European Union, 92% of the water we use is returned in good condition to the environment. We are the world champion in recycling at the rate of 79.3% At the forefront of the decarbonisation and industrial transformation of our economy, we embrace digitalisation and bring 25 billion value addition to the European economy and €5 billion investments annually. Through its 19 national associations, Cepi gathers 490 companies operating 870 mills across Europe and directly employing more than 180,000 people.

Cepi RENEWABLE RECYCLED RESPONSIBLE EUROPEAN

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