Annexes to the Harmonised European laboratory test method to generate parameters enabling the assessment of the recyclability of paper and board products in standard paper and board recycling mills

Table of Annexes:

A) Flowchart: Harmonised European Laboratory Test Method to produce parameters enabling the assessment of the recyclability of paper and board products in standard paper and board recycling mills.

Cepi RENEWABLE RECYCLED RESPONSIBLE EUROPEAN PAPER

HARMONISED EUROPEAN TEST METHOD FOR PAPER AND BOARD RECYCLABILITY ASSESSMENTS

VERSION 2 (2022)

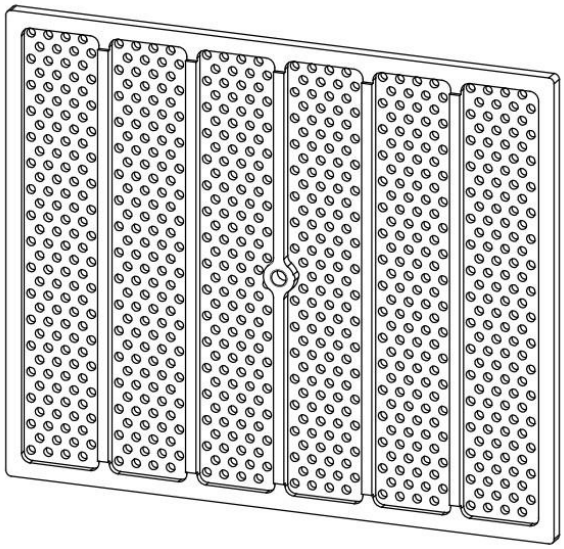
Test procedure			Measurement	Parameter for recyclability assessment
paper & board material and product	**amount**	250 g (at least one piece)		
	ageing	Sample without wet strength agents (WSA) should be older then 15 days, then no artificial ageing needed. If WSA present and sample not older then 30 days, artificial ageing needed acc. to Ingede Method 11 (3d, 60 °C)		
representive sample				
preparation	**size**	3 cm x 3 cm (+/- 0.5 cm)		
	amount	50 g aliquot of dry weight		
	method	cutting		
disintegration (acc. ISO 5263-1)	**device**	Standard disintegrator	start again with 20 min	
	consistency	2.5 %		
	time	10 min		
	temperature	40°C +/- 1°C		
filtrate analysis	**amount**	100 + 200 g	**amount of dissolved and colloidal substances**	**quantity dissolved and colloidal substances (DCS)**
	device	Buchner funnel ø 150 mm		
	porosity of the filter paper	12 - 15 µm		
	procedure	Double filtration using the same filter		
filtrate / return filter cake	**evaporation residue** (ISO 638)	Amount of the filtrate: approx. 70 g		
		Devices: Aluminium tray & Oven		
		Drying conditions: 105 ± 2 °C		
		Double determination		
filtrate	**chemical oxygen demand - COD** (ISO 6060; DIN 38409-H41-H44) **optional**	Amount: as described in the cuvette test		
		Devices: Cuvettes, Photometer		
		Temperature & Time: 148°C; 2h		
		Double determination		
coarse screening	**device**	Somerville Fractionator	**coarse reject determination** (if coarse reject contains significant amount of fibrous material and the coarse reject amount is above the Score Card Threshold, then 20 min disintegration)	**quantity coarse reject (CR)**
	size	hole ø 5 mm		
	time	5 min		
	water flow	8.6 l/min		
	amount	total		
sheet forming (accept coarse screening)	**device**	Rapid-Koethen sheet former	**sheet adhesion test**	**removability of impurities potential of tacky and visual impurities**
	grammage	60 g/m²	**visual appearance test**	
	number	2		
fine screening (acc. TAPPI T275 sp18)	**device**	Somerville Fractionator	**fine reject determination**	**quantity fine reject (FR)**
	size	slot // 150 µm		
	time	20 min		
	water flow	8.6 l/min		
	amount	20 g aliquot of dry weight		
If possible: work with a drainage				
sheet forming (accept coarse screening)	**device**	Rapid-Koethen sheet former	**sheet adhesion test**	**recyclate quality tacky impurities (SA) visual impurities (VI)**
	grammage	60 g/m²	**visual appearance test**	
	number	2		
macrostickies analysis (acc. ISO 15360) **optional**	**device**	Somerville Fractionator	**macrosticky assessment**	**recyclate quality tacky impurities (macrostickies)**
	size	slot // 150 µm		
	time	10 min		
	water flow	8.6 l/min		
	amount	2 x 5 g aliquot of dry weight		

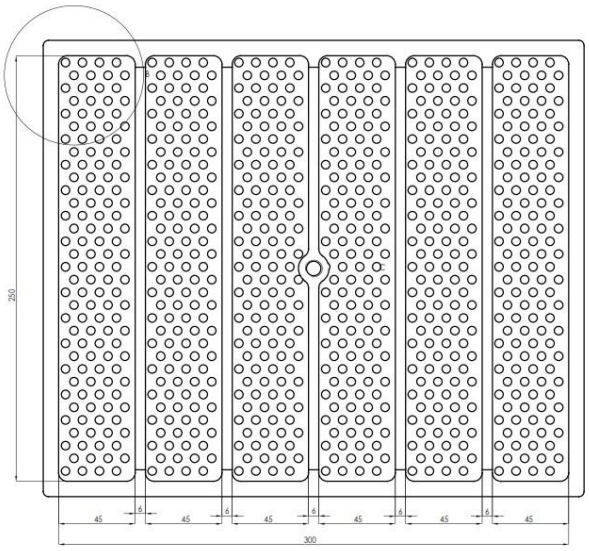
B) Description of the plate for evaluation of the coarse reject

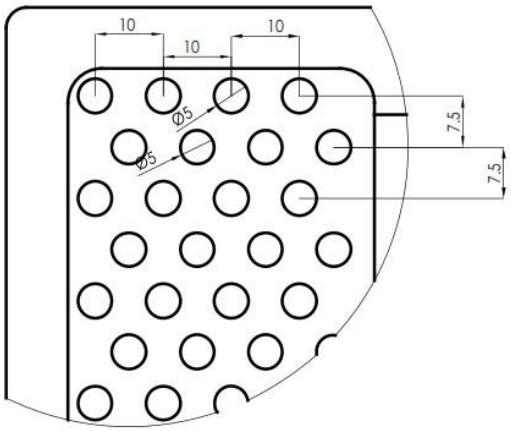
The plate for the evaluation of the course reject must have the following characteristics:

- Holes with a diameter of 5 mm;
- The overall area containing the holes must be 300 mm x 250 mm
- The holes are arranged in 6 columns, with each column occupying an area of 45 mm x 250 mm and with 6 mm of space between each column;
- Each column must comprise 33 lines of holes;
- Each line must contain 4 holes of a 5 mm diameter and the space between the lines must be 7.5 mm (the centre distance of the holes);
- The space between the holes along each line must be 10 mm (centre distance of the holes).
- An example of such a plate is shown in Figure B.1.

Figure B.1. Schematic representation of plate for separation of the coarse reject from the accept







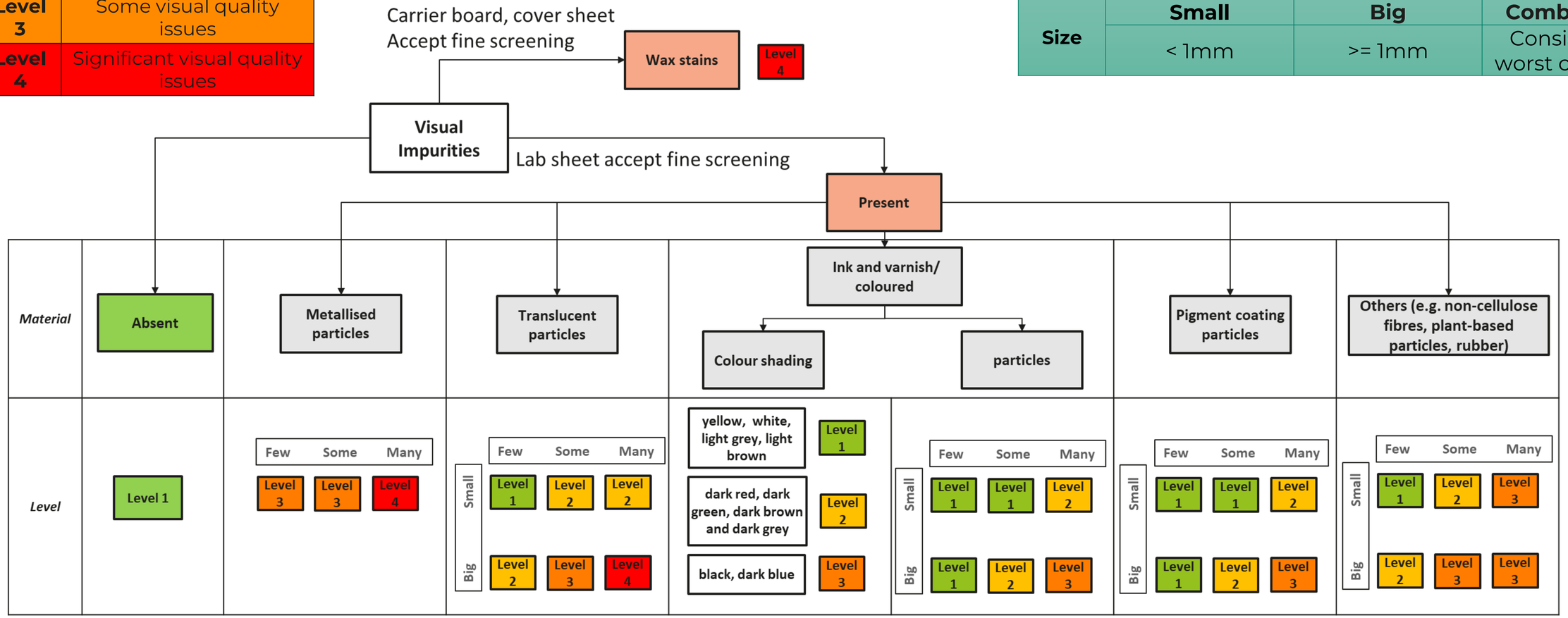
Source: Aticelca

C)Decision Tree for the evaluation of the visual appearance

VI: Decision tree – table version (28-09-2022)

Level 1	No visual quality issues
Level 2	Minor visual quality issues
Level 3	Some visual quality issues
Level 4	Significant visual quality issues

Visual impurities			
Amount	**Few**	**Some**	**Many**
	< 10	10 – 100	> 100
Size	**Small**	**Big**	**Combination**
	< 1mm	>= 1mm	Consider the worst case level



NOTE: All applicable columns should be evaluated. Worst case level allocation applies.

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D)Description of possible Thickener

Tank equipped with a 200 mesh wire on its bottom allowing the pulp to be thickened by natural gravity. The diameter of the thickener should be of enough dimension (approximately 50-75 cm) to avoid plugging the mesh during utilisation. It is possible to unclog the mesh manually during the thickening step in order to prevent overflow.
An example of such an equipment is shown in Figure C.1.

Figure 1.C.

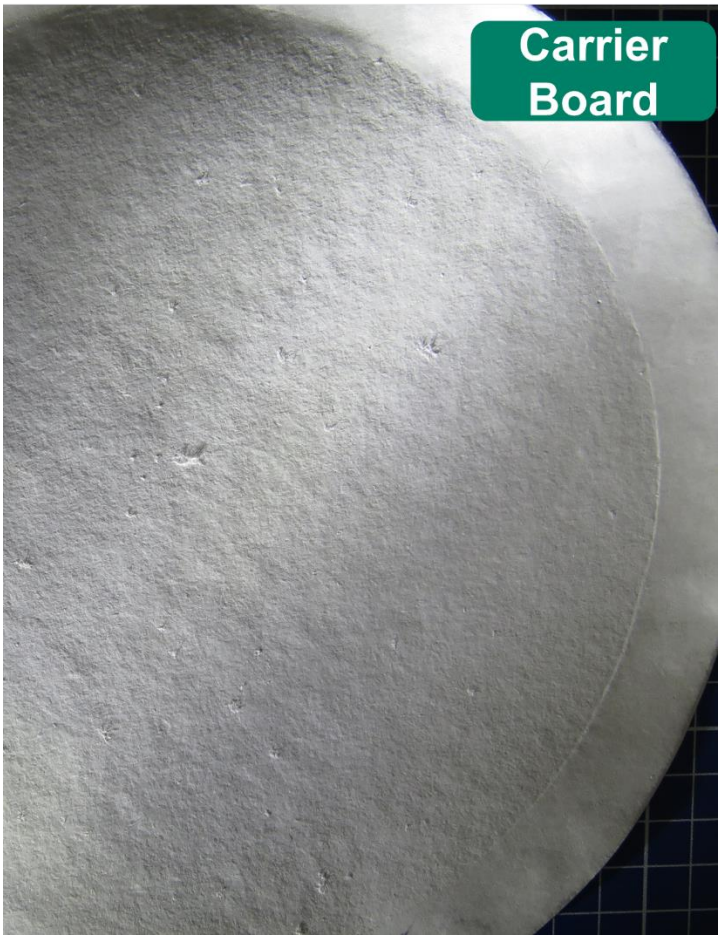
Source: Centre Technique du Papier

Figure 2.C. Top view of thickener as used in CTP

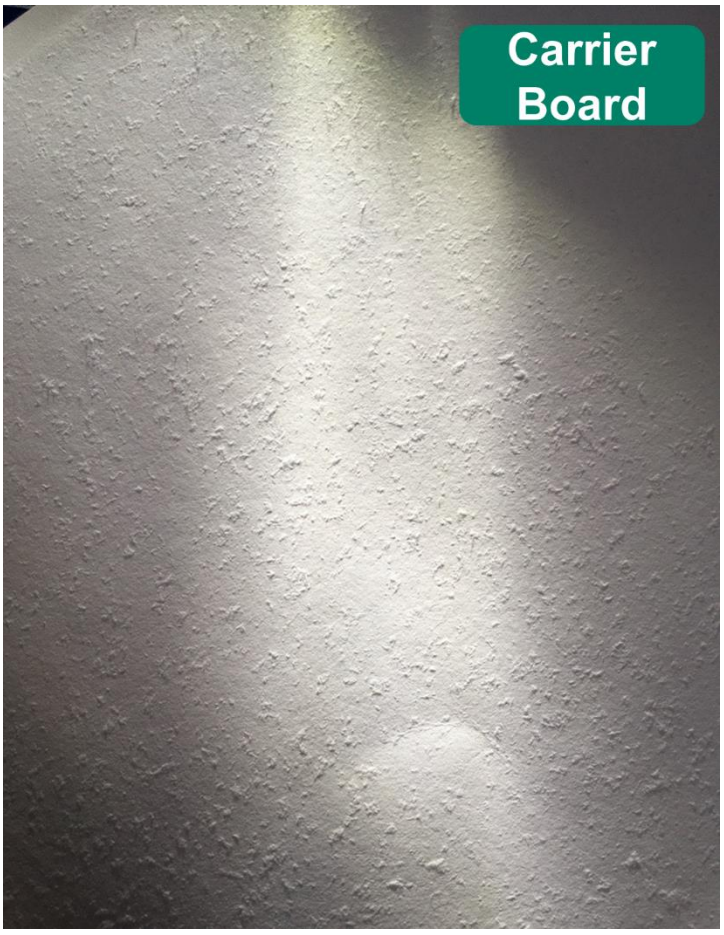
Source: Centre Technique du Papier

E) Sheet Adhesion test reference pictures of the carrier board after sheet adhesion test

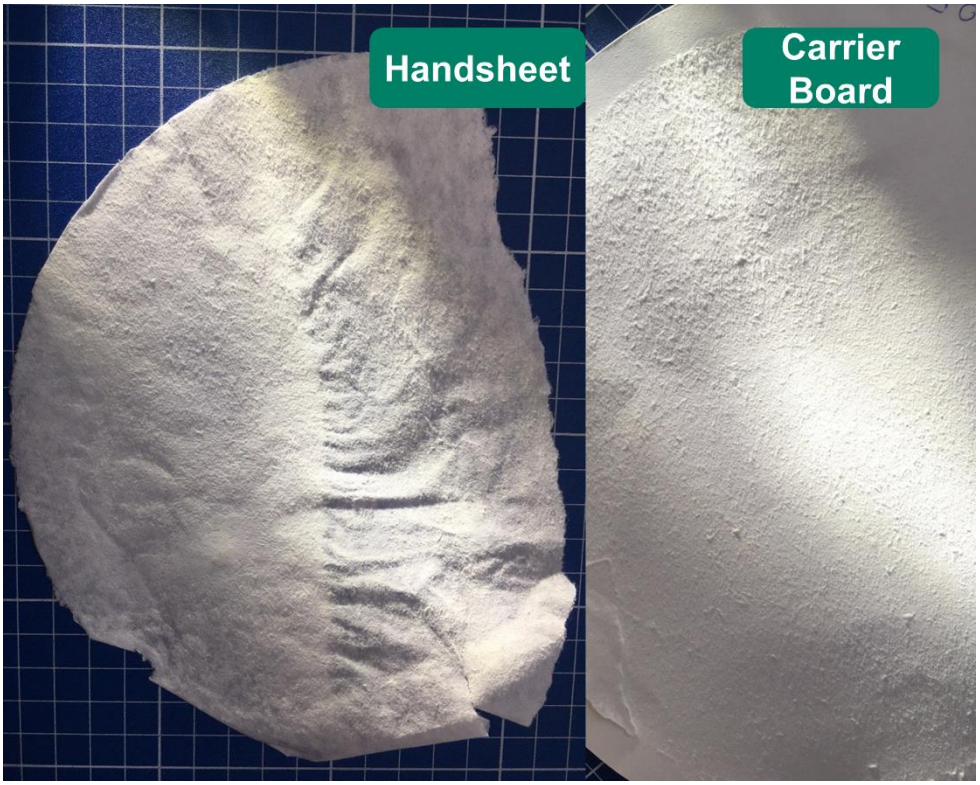
Adhesives absent:

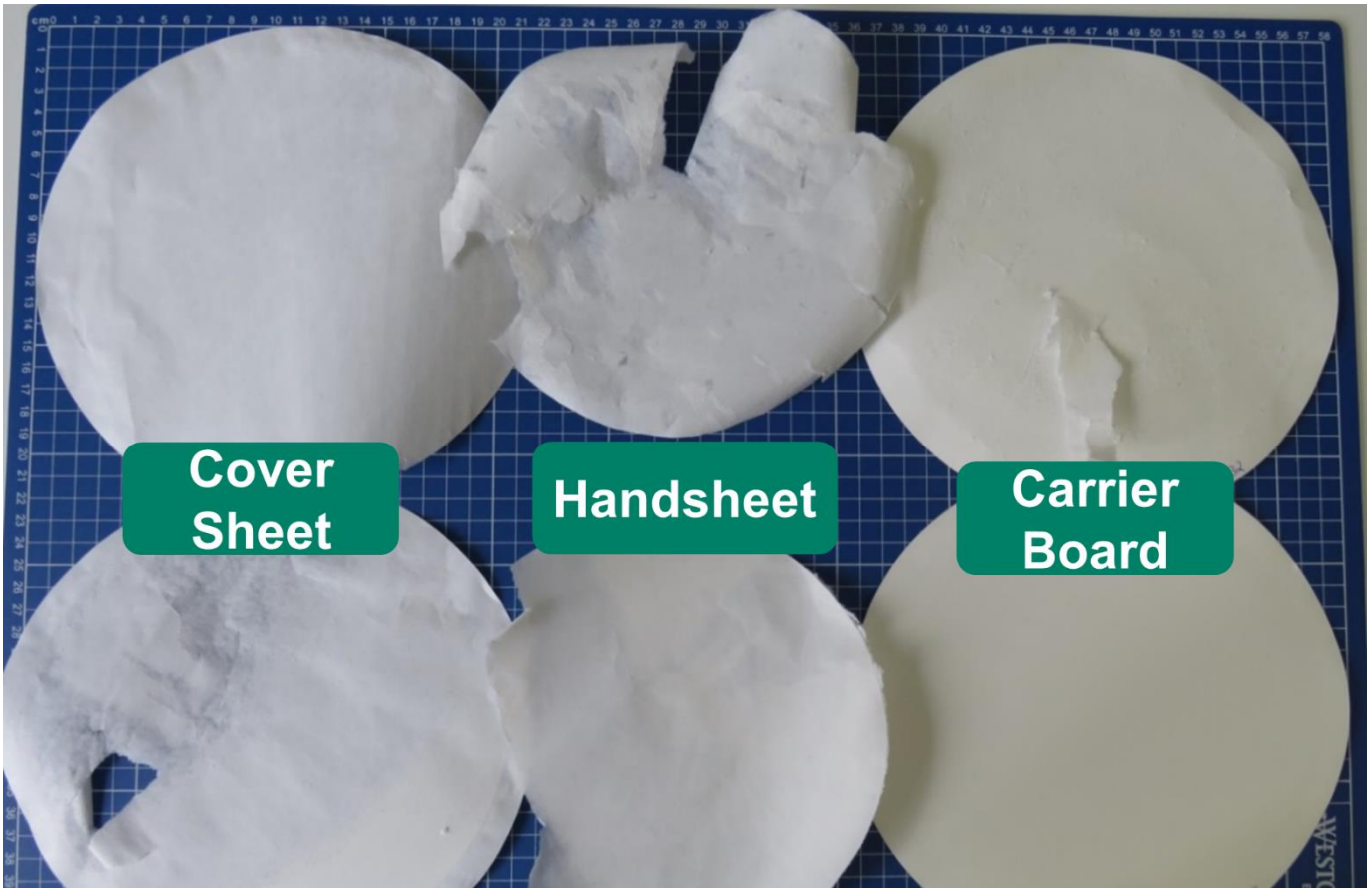


Adhesives partly present:



Adhesives present:





F) Technical data sheet

MINIMUM REQUIREMENT FOR TECHNICAL DATA SHEET TO BE PROVIDED TO THE LABORATORY

	SECTION A - GENERAL DATA			**UNIT**
A1	Company name			text
A2	Product name			text
A3	Description of the material/product and its function			text
A4	Is it a base paper material or is it a finished product?			material/product
	SECTION B - PRODUCT DETAILS	**Fill section B only if it is a product**		
B1	Dimensions of the product	Width and tolerance		mm
B2	Dimensions of the product	Lenght and tolerance		mm
B3	Dimensions of the product	Height and tolerance		mm
B4	Weight of the product (emptied if it is a packaging) and tolerance			g
B5	Is it a used product (e.g. is it a packaging already filled and then emptied)?			yes/no
	SECTION C - PAPER BASED MATERIAL DATA	**Fill section C if it is a material or if it is a product. Duplicate this section if the product is composed of more than one paper based material**		
C1	Composition and characteristics of the paper based material	Paper and board	Grammage and tolerance	g/m2
C2	Composition and characteristics of the paper based material	Paper and board	Thickness and tolerance	µm
C3	Composition and characteristics of the paper based material	Paper and board	Presence of a coating	yes/no
C4	Composition and characteristics of the paper based material	Paper and board	Presence of fillers	yes/no
C5	Composition and characteristics of the paper based material	Paper and board	Presence of wet strenght polymers	yes/no
C6	Composition and characteristics of the paper based material	Paper and board	Presence of artificial fibres	yes/no
C7	Composition and characteristics of the paper based material	Paper and board	Is it printed, varnished, lacquered, etc.?	yes/no
C8	Composition and characteristics of the paper based material	Non-paper layer, if any (e.g. plastic, aluminium, etc.)	Description of the material (e.g. PE, PLA, etc.)	text
C9	Composition and characteristics of the paper based material	Non-paper layer, if any (e.g. plastic, aluminium, etc.)	Grammage and tolerance	g/m2
C10	Composition and characteristics of the paper based material	Non-paper layer, if any (e.g. plastic, aluminium, etc.)	Thickness and tolerance	µm
C11	Composition and characteristics of the paper based material	Other non-paper layer, if any (e.g. plastic, aluminium, etc.)	Description of the material	text
C12	Composition and characteristics of the paper based material	Other non-paper layer, if any (e.g. plastic, aluminium, etc.)	Grammage and tolerance	g/m2
C13	Composition and characteristics of the paper based material	Other non-paper layer, if any (e.g. plastic, aluminium, etc.)	Thickness and tolerance	µm
C14	Composition and characteristics of the paper based material	Overall paper based material, including other material layers	Grammage	g/m2
C15	Composition and characteristics of the paper based material	Overall paper based material, including other material layers	Thickness	µm
	SECTION D - GLUE	**Fill section D only if it is a material or a product. Duplicate this section if the product includes more than one glue**		
D1	Presence of glue, if any	Describe the type of glue		text
D2	Presence of glue, if any	Describe the use of the glue		text
D3	Presence of glue, if any	Weight and tolerance of the glue		g
	SECTION E - NON-PAPER BASED COMPONENTS	**Fill section E only if it is a product. Duplicate this section if the product includes more than one non-paper based material**		
E1	Other non-paper components (e.g. staples, labels, handles, etc.)	Describe the component		text
E2	Other non-paper components (e.g. staples, labels, handles, etc.)	Weight and tolerance of the component		g
E3	Is the non-paper component easily removable from the product?			yes/no
	SECTION F - OTHER REMARKS			
F1	Other remarks			text
	SECTION G - DATE OF PRODUCTION			
G1	Date of production of the sample provided to the laboratory			dd/mm/yyyy

G)Laboratory report template

Please download the template **here**.

H) Detailed work description

Content

List of Figures

List of abbreviations and symbols

AC	Accept from coarse screening
AF	Accept from fine screening
COD	Chemical oxygen demand
OD	Oven dry
ER	Evaporation residue
RK	Rapid Koethen
WSA	Wet strength agents

1 Purpose and scope

This work instruction specifies the procedure for the:

> **determination of the recyclability according to "**Harmonised European laboratory test method to produce parameters enabling the assessment of the recyclability of paper and board products in standard paper and board recycling mills Version 2 (September 2022)"

This document is applicable for the test rounds that are organised within the scope of 4evergreen Workstream 1. The contents aim to address specific difficulties and/or unclarities that were brought forward during the lab alignment meetings.

2 Test equipment and materials

2.1 Test equipment

1. Analytical balance with accuracy of ± 0.01 g
2. Barrels for collecting the accept from coarse and fine screening
3. Metal plates (pressure 1.18 kPa or 3.7 kg, 20 cm diameter) for the sheet adhesion test
4. Metal plates (pressure 0.95 kPa or 6 kg, 28 cm diameter) for the macro stickies determination (optional)
5. Beakers
6. Büchner funnel (diameter 125 mm and 150 mm) compliant to ISO 12331-11 and equipped with suction flask and water jet pump.
7. Couching roller for the sheet formation
8. Cutting mat for photo documentation (optional)
9. Cuvette heating block (temperature 150 °C ± 5 °C) for the COD determination (optional)
10. Cuvette rack for the COD determination (optional)
11. Drying oven (temperature 105 °C ± 2 °C and temperature 130 °C ± 2 °C)
12. Eppendorf variable pipette 1,000 – 5,000 µL for the COD determination (optional)
13. Glass Bottle to store the filtrate (optional)

14. LED light Panel for the photo documentation (optional)
15. Perforated plate (hole diameter 5 mm) for coarse screening in Somerville
16. Photometer measuring device for the COD determination (optional)
17. 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.)
18. Refrigerator to store the filtrate (optional)
19. Scissors / cutting machine / punch
20. Slotted plate (slot size 150 µm) for fine screening in Somerville
21. Somerville-fractionator compliant with TAPPI/ANSI T275
22. Standard disintegrator compliant with ISO 5263-1
23. Stopwatch / Timer Somerville-fractionator
24. Submersible pump (optional)
25. Thermometer digital
26. Vacuum desiccator

2.2 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 macro stickies determination (optional)
5. Cuvette tests e.g. COD cuvette test 15-150 mg/L O_2 (optional)
6. Deionised water
7. Filter paper grade 388 diameter 125 mm (basis weight 84 g/m², filtration speed 10 s/10 ml, deposition range 12 - 15 µm)
8. Filter paper grade 388 diameter 150 mm (basis weight 84 g/m², filtration speed 10 s/10 ml, deposition range 12 - 15 µm)
9. Filter paper grade 1289 diameter 240 mm (basis weight 84 g/m², filtration speed 20 s/10 ml, deposition range 8 – 12 µm) (optional)
10. Silicon paper (60 g/m²) compliant with ISO 15 360 for the macro stickies determination (optional)

3 Sample preparation

3.1 Simple sample preparation

Step Nr.	Notes	Description
1	Material amount	• A minimum amount of 250 g air-dried material is needed to carry out all following measurements.
2	Dry content determination	• Place the sample in the oven at (105 ± 2) °C until it reaches a constant mass to determine the moisture content according to ISO 638. The sample must be cooled for approx. 30 min in desiccator before every weighting. • If the packaging sample is too large to fit in the oven, cut it beforehand and place it in the oven and then in the vacuum desiccator. • If the sample contains dry-removed parts (e.g. metal clips), do not count their weight within the 50 g oven dry (OD) material for disintegration. Instead, include it in the reject amount calculation.
3	Cutting the specimen	• If the specimen has the form of a roll, cut it to A4 size. • Put the A4 format sample into a cutting machine or punch and cut / punch it to (3 x 3 ± 0.5) cm. • At least 65 g air dry sample has to be cut / punched.

4	Aging	• If the sample contains wet strength agents (WSA) and was produced for less than 30 days, store it for the time needed to complete this time and after that proceed with the test. • Another option is to perform an accelerated aging by placing the sample in the oven at (60 ± 1) ° C for 72 hours. • In case of samples without WSA, make sure the sample is at least 15 days old from the date of production, and therefore no aging is necessary.
5	Documentation in Excel	• Enter the dry content of the sample in the Excel spreadsheet. • Calculate the sample weight equivalent to 50 g OD. • Enter the experiment date and processor abbreviation in the Excel spreadsheet.

3.2 Complex sample preparation

3.2.1 Example 1: paper cup

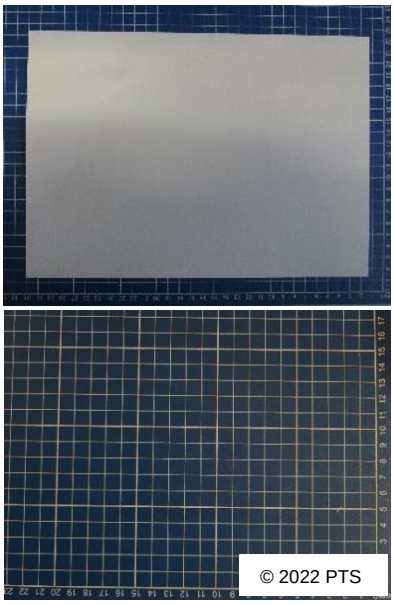
Step Nr.	Notes / Pictures	Description
1	Dry content determination	• Place the sample, or at least each different part of the sample (sealing, gluing, paper, etc) in the oven at (105 ± 2) °C to determine the moisture content according to ISO 638-1.

		• If the packaging sample is too large to fit in the oven, cut it beforehand and place it in the oven and then in the vacuum desiccator. • If the sample contains dry-removed parts (e.g. metal clips), do not count their weight within the 50 g oven dry (OD) material for disintegration. Instead, include it in the reject amount calculation. • The use of an electronic equipment instead of the oven mentioned in the standard needs to be reported. Some rejects can behave differently when electronic devices are used in the dry content determination.
2	Cutting the specimen  *Figure 1: Example of cutting preparation of a cup sample.*	• Cut the cup along the side gluing up to the bottom. • Remove the bottom by hand. • Cut off the side gluing seam. • Cut off the rolled edge. • Weigh all parts individually and enter their weights in the Excel spreadsheet. • Weigh out sample portions corresponding to 50 g OD and cut it to (3 x 3 ± 0.5) cm pieces.
3	Documentation in Excel	• Enter the dry content in Excel spreadsheet. • Enter the weight of each sample components in Excel spreadsheet. • Enter date and abbreviation of the processor in the Excel spreadsheet.

3.2.2 Example 2: Shopping bag

Step Nr.	Notes / Pictures	Description
1	Dry content determination	• Perform it as in topic 3.2.1
2	Cutting the specimen  *Figure 2: Example of cutting preparation of a shopping bag sample.*	• Cut the bag along the side gluing up to the bottom. • Cut off the bottom. • Cut off the side gluing seam. • Cut off the handle including adhesives. • Metal parts that are not shredded are marked as dry removed. • Weigh all parts individually and enter their weights in the Excel spreadsheet. • Weigh out sample portions corresponding to 50 g OD and cut it into (3 x 3 ± 0.5) cm pieces.
4	Documentation in Excel	• Perform it as in *topic 3.2.1*

3.3 Photo documentation

Step Nr.	Picture	Description
1	 *Figure 3: Paper sample positioned on the cutting mat. In detail the cutting map lines.*	• Photograph the front and back side of the specimen on the cutting mat (if available) so that the scale of the mat can be seen. • If possible, place the specimen in a way it lies against a mat line. A ruler can also be placed near to the sample. • The photo should be made as straight as possible (use cutting mat as an aid). • Photographs of specific sample portions are recommended for complex specimens.

3.4 Filter paper preparation

Step Nr.	Picture / Notes	Description
1	Drying  *Figure 4: Filter papers with respective weight written on the bottom edge.*	• Dry filter papers of grade 388 (indicate filter pore) for at least 30 min in the drying-oven at (105 ± 2) °C. • Cool them down in a desiccator. • Weigh them on the analytical balance and note the obtained weight at their bottom edge.

2	Labelling	• The previously weighed filter papers must be labelled according to the following information: ○ 3 filter papers for stock consistency. ○ 1 filter paper for the 5 mm hole residue. ○ 1 filter paper for the 150 µm slot residue.

4 Disintegration

Step Nr.	Notes	Description
Standard Disintegrator following the ISO 5263-1		
1	Dilution  *Figure 5: Paper sample diluted with tap water.*	• Dilute 50 g OD pulp using tap water at (40 ± 1) °C and with a mildly alkaline pH (7-8) until reaching 2,000 g, so that a stock consistency of 2.5 % is achieved. • Note the real sample amount you have weighted in. Considering you have 3 x 3 cm pieces, you may have to use a little less or a little more than the sample amount equivalent to 50 g oven dry.
2	Disintegration	• Transfer the diluted sample to the standard disintegrator container. • Set the revolution counter to 30,000 revolutions, which is equivalent to 10 min of disintegration time.

	 *Figure 6: Disintegrated sample in the standard disintegrator.*	• After completion of the disintegration process, flush the remaining stock from the cover plate and the rotor blade with a spray bottle. • Transfer the sample without losses to a beaker. A spray bottle with tap water can be used to recover the sample attached to the disintegrator.
3	Disintegrator check	• Check the disintegrator regularly to ensure the following conditions: o The rotor shaft must run smoothly and be always positioned centrally in the vessel. o The rotor must run at a specified speed. o The rotor blades must be correctly adjusted (this can be checked with the help of a gauge). o The rotor blades must have specified dimensions (see Annex Standard 5263-1) and cannot be damaged. o If the device is used properly, the other dimensions of the impact device should not change. However, they must be checked at regular intervals.

5 Filtrate Analysis

Step Nr.	Notes/ Pictures	Description
1	Sampling  *Figure 7: Total stock in a beaker.*	• Perform the filtration immediately after the disintegration step. • Homogenise the total stock in the beaker using a spoon or ladle. • Weigh out approx. 300 g of the total stock using the analytical balance.
2	Filtration  *Figure 8: Büchner funnel set for the pulp filtration.*	• Use the suction flask and the Buchner funnel exclusively for water analysis tests. • Wash the suction flask with tap water before using it. • Place a filter paper (150 mm) onto the Büchner funnel (150 mm). • Attach the vacuum hose and turn the vacuum pump on. • Filter 100 g of the total stock through the Buchner funnel, if possible, without moisturising the filter paper. To prevent the sample from reaching the flask without passing though the filter, first pour a few drops of the sample to set the filter on the funnel and then proceed with the rest.

- Use the filtrate to rinse the suction flask and return it to the total stock into the beaker.
- Separate the filter cake from the filter paper and return it to the total stock into the beaker.
- Place another filter paper (150 mm) onto the Büchner funnel (150 mm).
- Filter more 200 g of the total stock via a Büchner funnel as described above.
- Retrieve the filtrate from the suction flask and filter it via the Büchner funnel again using the same paper filter.
- Fill filtrate into a clean glass bottle and label it.
- Return the filter cake to the pulp stock as described above.
- Photograph the filtrate, especially if it presents some colouration.
- Proceed immediately to the determination of the evaporation residue.
- After use, wash the funnel and the flask with deionised water.

5.1 Evaporation residue

Step Nr.	Notes/ Pictures	Description
1	Evaporation residue  *Figure 9: Evaporation residue (white stain) left on the aluminium tray.*	• Pour a known amount (approx. 70 g) of filtrate on a previously weighed aluminium tray. • The size of aluminium tray must be compatible with the amount of liquid poured on it and the size of the oven. • Note down the mass of the empty aluminium tray “m1” and the mass of the filtrate that was taken in “m2” (approx.70 g). • Repeat the procedure to have a double determination of the evaporation residue. • Place the tray with the filtrate in the drying oven at (105 ± 2) °C to remove the solvent. • Determine the residue in line with ISO 638. • When there is apparently no more solvent in the tray, take it out of the oven for the first time. • In order to control the residue mass, cool the tray in the desiccator for approx. 30 min before weighing it. • Repeat the process until the residue reaches a constant mass value “m3”, and note it down. • Enter the all values in the Excel spreadsheet.

		• As the packaging sample is disintegrated with tap water, it is necessary to consider its influence on the evaporation residue. • Measure the evaporation residue of the tap water performing the procedures described in the topic 5.1. The water does not need to be filtrated as described in the topic 5. • Do not use the first jet of water from the tap (e.g. discard the first 2 L of water) to measure the evaporation residue because it can contain residues of contamination from the pipes.
2	Calculation	• The evaporation residue (ER) of the sample is calculated as follows: $ER\ filtrate\ (g\ residue/g\ filtrate) = \frac{m3\ (g) - m1(g)}{m2\ (g)}$ $ER\ sample\ (g\ residue/g\ filtrate) = ER\ filtrate\ (g/g) - ER\ tap\ water\ (g/g)$ m_1 = mass of the empty aluminium tray m_2 = mass of the filtrate that was taken in m_3 = mass of tray after drying The evaporation residue can be calculated in relation to the used stock suspension. As approx. 50 g of sample were disintegrated in 2 L of water, the packaging mass is 25 g fibres /L. Thus, the evaporation residue (g residue / kg packaging) is calculated as follows:

$$\frac{ER\ sample\ (g/g)}{Packaging\ mass\ (g/L)} = ER\ sample\ (L/g\ )$$

$$ER\ sample\ (L/g\ ) = ER\ sample\ (kg/g)$$

$$ER\ sample\ (kg/g)\ x\ 1{,}000{,}000 = ER\ sample\ (g\ residue/kg\ packaging)$$

The evaporation residue in percentage is calculated as follows:

$$ER\ sample\ (g\ residue/kg\ packaging) \div 1{,}000 = ER\ sample\ (g\ residue/g\ packaging)$$

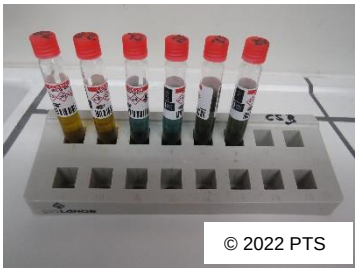
$$ER\ sample\ (g\ residue/g\ packaging) \times 100 = ER\ sample\ (\%)$$

The results must be given with two decimal places.

5.2 Chemical Oxygen Demand (COD) (optional)

Step Nr.	Notes/ Pictures	Description
1	Cuvette test  *Figure 10: Cuvette used in the COD measurement.*	• Conduct the COD determination preferentially directly after the filtration of the total stock. • In case a direct measurement is not possible, store the filtrate in glass bottle in the refrigerator at approximately 4°C for upmost 24 hours. • Record in the Excel spreadsheet if the filtrate was stored in the refrigerator and the duration of it.

- Select the COD cuvette with the expected measuring range for the tests according to ISO 6060-8.
- Perform a double determination (2 cuvettes).
- Write the sample number on the cuvette lid (the barcode must remain legible and the glass must be clean for measurement).
- Invert the cuvette to homogenise the solution.
- Shake the glass bottle containing the filtrate.
- Measure 2 ml filtrate using a pipette. Depending on the cuvette test range, a different volume of the filtrate is required (for details, see the instructions on the packaging).
- Add the filtrate to the cuvette carefully (run along the edge).
- Close the cuvette securely.
- If the cuvette solution immediately presents a green coloration as in Figure 10, discard the sample and either use a higher measuring range or use dilute the filtrate with deionised water.
- Invert the cuvette containing the preparation.
- Repeat the same procedure using the filtrate stored in the second glass bottle.

2	Boiling  *Figure 11: Cuvette in the heating block*  *Figure 12: Cuvette cooling on the cuvette rack.*	• Place the cuvettes in the heating block. • Set the heating block to work for 2 h and at 148 °C. • Remove the hot cuvette from the heating block. • Carefully invert the cuvette. • Cool the cuvette to room temperature on the cuvette rack.
3	Photometry measurement  *Figure 13: Cuvette on the photometer.*	• Start the photometer. • Do not to stir up sediment in the cuvette anymore. • The outside of the cuvette must be clean, wipe it clean if necessary. • Place the cuvette in photometer and perform the measurement (absorbance at 600 nm). • Enter the results in the Excel spreadsheet. • Repeat the same procedure using tap water instead of filtrate.

		• If the COD of the tap water is higher than 15 mgO_2/L, it is recommended to measure it every time a sample is going to be tested. • If the COD of the tap water is lower than 15 mgO_2/L, it is recommended to measure it every 3 months.
4	Calculation	• The COD of the sample is calculated as follows: $COD\ sample\left(\frac{mg\ O2}{L}\right) = COD\ filtrate\left(\frac{mg\ O2}{L}\right) - COD\ tap\ water\left(\frac{mg\ O2}{L}\right)$ • The COD value must be calculated in relation to the used stock suspension. As approx. 50 g of sample were disintegrated in 2 L of water, the packaging mass is 25 g /L. Thus, the COD is calculated as follows: $COD\ (g\ O2/kg\ Packaging\ ) = \frac{COD\ \left(\frac{mg\ O2}{L}\right)}{Packaging\ mass\ \left(\frac{g}{l}\right)}$ The results must be given with two decimal places.

6 Screening

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

Step Nr.	Notes	Description

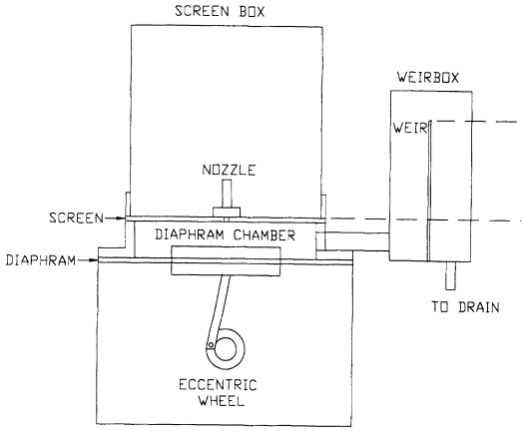
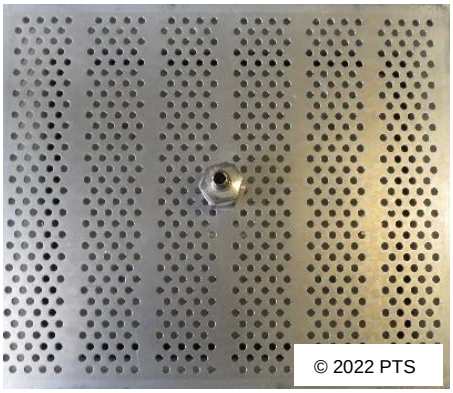
1	Sampling	• Take the total amount of the sample suspension for the coarse screening (including the filter cake recovered from the filtration as mentioned in *topic 5*).
2	Sampling Procedure  *Figure 14. Sketch of the Somerville set up (TAPPI, 2018).*  *Figure 15: Perforated plate (5 mm hole) UNI 11743*	• Place the 5 mm perforated plate into the Somerville fractionator. • Attach the instrument screen box tightly. • Attach the instrument hose onto the plate nozzle. • Place the glass protection cover over the screen box. • Assure that the weir is positioned in the bottom of the weir box. • Start the water flow and pour the total amount of the sample on the screening plate, when it is covered with approximately 2.5 cm of water. • When the sample starts to overflow through the weir, start the screening time using a timer. • During the washing process, the sorted suspension (AC) is collected in a barrel. • After 5 min, turn off the motor and the water supply. • Pull the weir out the weir box, so the water can be drained. • Open the unit when the water has completely drained off.

Figure 16: Coarse screening in the Somerville fractionator.

Figure 17: Coarse reject after the water draining.

- Carefully open the screening box.
- Transfer the fibres stuck on the screening box of Somerville into the barrel, where the accept material is collected.

3

Photo documentation

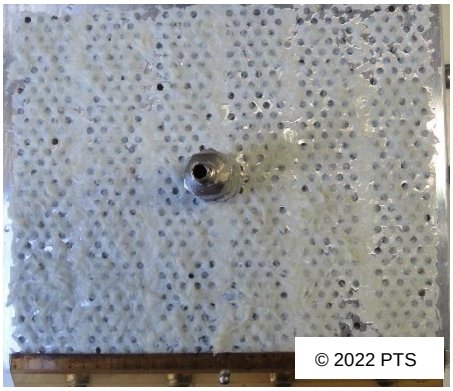


Figure 18: Coarse reject after the coarse screening

.

- Photograph the residue on the perforated plate using a ruler as a sense of scale as in Figure 18
- Photograph the perforate plate again with zoom on the reject.
- If applicable, separate the different components in the coarse reject and photograph them on a dark background using a sense of scale (Figure 19)
- Stretch the reject components to show their size clearly in the picture.

Figure 19: Detailed picture of reject components.

This is important to evaluate the reject fragmentation.

- If applicable, show in the picture fibres attached to non-paper components.

Measurement of the coarse reject

4 *Figure 20: Büchner funnel used for the residue filtration.*

Figure 21: Filter papers cooling in desiccator.

- Record in the Excel spreadsheet the approximate number of visible specks, sticky components and non-paper components.
- Carefully transfer the residue to a vessel using a dough scraper or squeeze bottle. Be sure that all fragments trapped on plate are also recovered.
- Place a weighed filter paper (125 mm grade 388, basis weight 84 g/m², filtration rate 10s/10ml. filtration range 12 - 15 µm) on the Büchner filter and pour the residue over it.
- Place the filter paper between two cover sheets and dry it in the dryer of the sheet former for 7 min (93 ± 4) °C.
- Turn around the filter paper and dry it again in the dryer of the sheet former for 7 min (93 ± 4) °C.

Figure 22: Coarse reject dried in the aluminium tray.

- Place the filter paper in the oven at (105 ± 2) °C, until the mass becomes constant as in *topic 3.1*.
- Allow the filter paper to cool in desiccator.
- Weigh the paper filter on the analytical balance.
- If the reject amount is too high to fit in the filter paper (Figure 22), place it in a weighed aluminium tray and dry it directly in the oven at (105 ± 2) °C until reaching a constant mass.
- Transfer the results to the Excel spreadsheet, and calculate the coarse reject (dry-weight) in respect to the starting sample.
- Round the coarse reject to the first decimal place.

6.2 Determination of the consistency after the coarse screening

Step Nr.	Notes	Description
1	Determination of the consistency	The determination of the stock consistency is carried out according to ISO 4119 as follows (stock consistency between 0.3 % and 1 %): • Carefully homogenise the screened stock collected in the AC barrel. • Tare the sampling vessel on the analytical balance.

- Transfer approx. 500 g suspension into the tared vessel.
- Weigh the mass of the sample taken ("m1").
- Place a filter paper (*weighted in topic 3.4* "m2") on the Büchner funnel (diameter 125 mm) and moisten it with water using a spray bottle.
- Make sure that all pores in the Büchner funnel are covered by the filter paper.
- Apply negative pressure by means of a vacuum pump.
- Place the sample over the paper filter into the Büchner funnel
- Rinse the sample vessel with water and pour the liquid to the funnel to not lose any sample residue.
- Wait until there is no more water in suspension in the Büchner funnel.
- Remove the filter paper containing the pulp cake from the funnel.
- Return any material remaining on the funnel wand to the paper filter.
- Place the filter paper between two cover sheets and dry it in the dryer of the sheet former for 7 min at (93 ± 4) °C.
- Turn around the filter paper and dry it again in the dryer of the sheet former for 7 min at (93 ± 4) °C.
- Transfer the filter paper to a desiccator to cool it down.
- Determine the dry mass of the filter cake on the analytical balance and document as "m3".

2	Calculation of the consistency	$$c\ (\%) = \frac{m_{3\ (g)} - m_{2\ (g)}}{m_{1\ (g)}} \times 100$$ 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 The results must be given with two decimal places.

6.3 Sheet formation after coarse screening, incl. quality assessment

Step Nr.	Notes	Description

1	Sheet formation (AC)
	 
	
	Figure 23: Sheet former and the couching roller.
		• Homogenise the total amount of the sample carefully before sampling.
		• Take approximately 2,000 g amount of pulp from the barrel, in which the accept material was collected during the screening.
		• Form a lab sheet in the Rapid Koethen sheet former according to the ISO 5269-2.
		• The target weight is (60 ± 2) g/m² (approximately 1.8 g) if it is not reached, adjust the amount of pulp required for sheet formation.
		• To form two lab sheets follow the steps:
		  ○ Pour the sample into the sheet former column.
		  ○ Bubble the sample for 8 s.
		  ○ Let the sample settle for 8 s.
		  ○ Drop the sample to 2 L.
		  ○ Drain: when the water has completely passed through the sieve, aspirate the water for 10 – 15 s, or until there is no more water in the column.
		  ○ Open sheet former column.
		  ○ Photograph the sheet.
		  ○ Place a carrier board over the formed sheet and roll a couching roller over it (one time

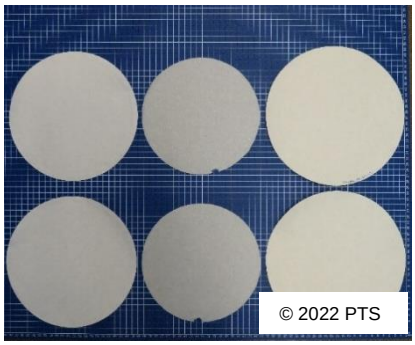
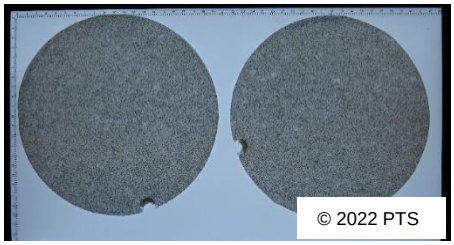
		lengthwise and one time cross-wise). o Remove the screen frame from the sheet former and knock the laboratory sheet out of it.
2	Photo documentation  *Figure 24: Lab sheet AC1.*	• Photograph each lab sheet as in Figure 24
3	Sheet adhesion test  *Figure 25: Drying oven used for the sheet adhesion test.*	• The two sheets (AC1, AC2) are used to perform the sheet adhesion test and to evaluate the optical inhomo-geneities. • Place the lab sheet between a car-rier board (bottom side) and a cover sheet (top side) and dry it in the dryer of the sheet former for 7 min at (93 ± 4) °C. • Place the lab sheet with carrier board and cover sheet flat between two preheated metal plates (pres-sure of 1.18 kPa or 3.7 kg) into the drying oven and dry it at (130 ± 2) °C for 2 min.

Figure 26: Lab sheet with carrier board and cover sheet placed between metal plates into the oven.

- Place the lab sheet with carrier board and cover sheet in the desiccator for 10 min to cool it down (time is measured using a stopwatch).
- After taking it out of the desiccator, perform both sheet adhesions tests immediately by separating the carrier board and the cover sheet from the lab sheet. Please note which of the cover sheet or carrier board was removed first.
- Weigh the lab sheet on the analytical balance.
- Record the observations in the laboratory excel spreadsheet.
- Observe the fibre tearing in oblique light.

4 Labelling

- Label all sheets with their number (AC1, AC2) and sample number (e.g. sample 1).
- The cover sheet and the carrier board must be labelled in the same way as the lab sheets.
- Regarding the carrier board, the side that was in touch with the lab sheet must be labelled.
- Regarding the cover sheet, the side that was not in touch with the lab sheet must be labelled

5	Photo documentation  *Figure 27: Hand sheets analysed in the sheet adhesion test (AC1, AC2) with incident light*  *Figure 28: Lab sheets (AC1, AC2) on the LED panel with transmitted light.*	• Photograph the sheets on the cutting map, using the auxiliary lines to align the photo. • Place the carrier board at the right-hand side and at the lowest line. • Place the lab sheet in the middle and two lines above the carrier board. • Place the cover sheet in the left-hand side and one line above the carrier board. • Photograph the lab sheets on the LED panel, showing the measurement scale.

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

Step Nr.	Notes/ Picture	Description
	Somerville fractionator according to TAPPI T275 sp18	
1	Sampling	• Homogenise the total amount of the sample carefully before sampling. • Take 20 g OD from the AC barrel for the fine screening.
2	Screening Procedure	• Place the 150 µm slotted plate into the Somerville fractionator.

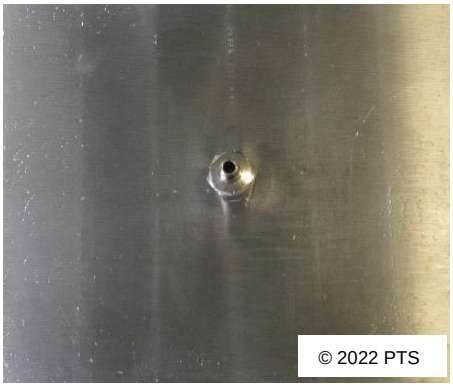


Figure 29. Slotted plate (150 µm slots).

Figure 30: Fine screening in the Somerville fractionator

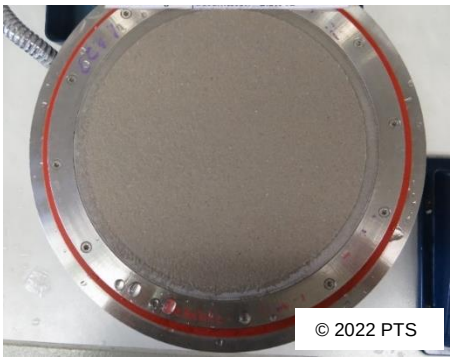
Figure 31: Fine reject after water draining.

- Set the Somerville as described in *topic 6.1*.
- Start the water flow and wait until the screening plate is covered with 2.5 cm of water.
- Start pouring the sample material on the plate.
- When the overflow through the weir starts, start the 20 min screening time using a stopwatch.
- The pouring time should be as short as possible and not be longer than 4 min.
- Record the time needed to pour the sample.
- If possible, collect all the accept material (AF) in a barrel and use with a thickener to reduce the water volume and perform the sheet adhesion test and the assessment of visual impurities.
- In case, no thickener is available, collect at least the first 50 L of AF in a Barrel and proceed the sheet adhesion test and the assessment of visual impurities.
- After 20 min, turn off the motor and then the water supply.
- Pull the weir out of the weir box, so the water can be drained.

		• Open the unit when the water has completely drained off. • Carefully open the screening box and perform the photo documentation. • Transfer the fibres stuck on the screening box of Somerville into the barrel, where the sorted material is collected.
3	Photo documentation  *Figure 32: Fine reject on the 150 µm plate.*  *Figure 33: Fine reject in detail.*	• Photograph the fine screening residue on the slotted plate using a sense of scale Figure 32. • Make a detail picture of the residues, as in Figure 33, if necessary.

4	Measurement of fine reject	• Record in the Excel spreadsheet the approximate number of visible specks, sticking components and non-paper components. • Carefully transfer the residue into a vessel using a dough scraper or squeeze bottle. Be sure that all fragments trapped on plate are also recovered. • Place a weighed filter paper (125 mm grade 388, basis weight 84 g/m², filtration rate 10s/10ml. filtration range 12 - 15 µm) on the Büchner filter and pour the residue over it. • Place the filter paper between two cover sheets and dry it in the dryer of the sheet former for 7 min at (93 ± 4) °C. • Turn around the filter paper and dry it again in the dryer of the sheet former for 7 min at (93 ± 4) °C. • Place the paper filter in the oven at (105 ± 2) °C, until the mass becomes constant as in *topic 3.1*. • Allow the paper filter to cool in desiccator. • Weigh the paper filter on the analytical balance. • Transfer the results to the Excel spreadsheet.

6.5 Sheet formation after fine screening, incl. quality assessment

Step Nr.	Notes	Description
1	Sheet formation	• Homogenise the total amount of the sample carefully before sampling. • Take approximately 9,000 g amount of pulp from the AF Barrel. • Form a sample sheet using the sheet former as in *topic 6.3*. • The target weight is (60 ± 2) g/m², if it is not reached, adjust the amount of pulp required for sheet formation.
2	Photo Documentation  *Figure 34: Lab sheet AF1.*	• Photograph each lab sheet as in Figure 34
3	Sheet adhesion test	• Perform it as in *topic 6.3*
4	Labelling	• Perform it as in *topic 6.3*
5	Photo documentation	• Photograph the sheets on the cutting map, using the auxiliary lines to align the photo as in *topic 6.3*.

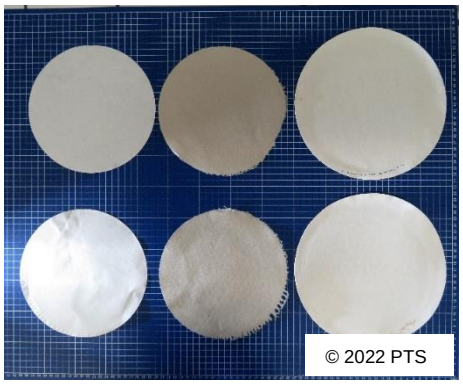


Figure 35. Hand sheets analysed in the sheet adhesion test (AF1, AF2) with incident light

Figure 36: Individual pictures of the cover sheet, lab sheet and carrier board with incident light.

- Photograph each sheet individually with an oblique light.
- Photograph the lab sheets on the LED panel, showing the measurement scale.

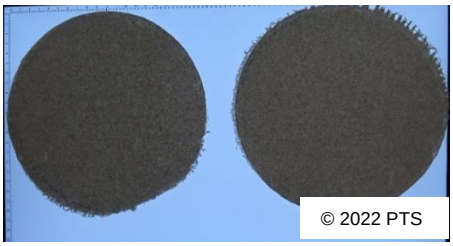
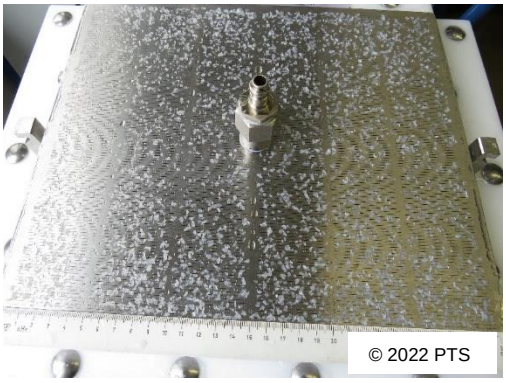
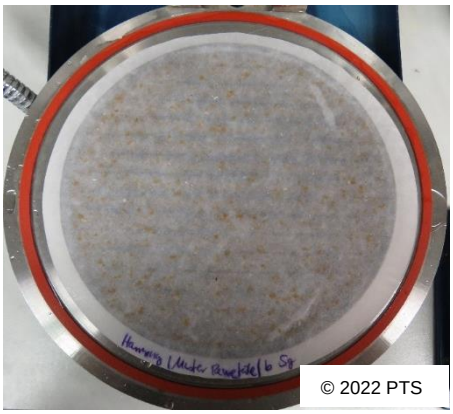


Figure 37: Lab sheets (AF1, AF2) on the LED panel with transmitted light.

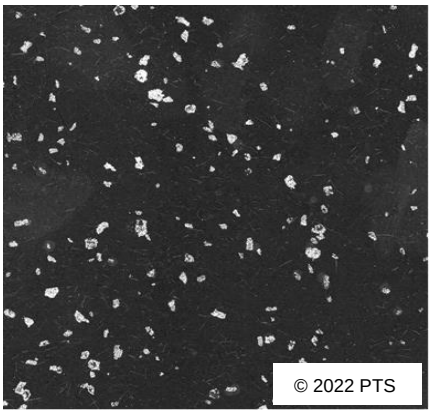
6.6 Macro stickies determination (optional)

Step Nr.	Notes	Description
Somerville fractionator according to ISO 15360		
1	Sampling	• Homogenise the accept from the coarse screening carefully before sampling. • Take 5 g OD from the AC barrel for the macro stickies determination. • Perform at least a double determination.
2	Macro stickies screening	• Place the 150 µm slotted plate into the Somerville fractionator • Set the Somerville as described in *topic 6.1*. • When the overflow through the weir starts, start the 10 min screening time using a stopwatch.

		• Turn off the motor and the water supply after the screening time. • Open the unit carefully when the water has completely drained off.
3	Photo documentation  *Figure 38: Macro stickies screening reject on the 150 µm plate.*	• Photograph the residue on the slotted plate using a sense of scale. • Take a detail picture if necessary.
4	Aspiration of the reject  *Figure 39: Aspirated reject on the filter paper.*	• Carefully transfer the residue into a beaker using a dough scraper or squeeze bottle. • Label the filter sheet (grade 1289 diameter 240 mm) using a pencil. Write down the sample name, the oven-dry quantity and the slot width. • Place the labelled filter sheet on Rapid Koethen (RK) sheet former, moisten it with a squeeze bottle, smooth it with the hands and close the sheet former. • Aspirate the water from the reject in the RK sheet former:

- Start the sheet former on the manual mode.
- Fill it with water to approx. 2 L.
- Add the reject rinsing out sample beaker.
- Bubble it for approx. 8 s.
- Switch to aspiration when the water has completely passed through the sieve and aspirate it dry for approx. 10 to 15 s, or until there is no more water in the column
- Rinse off adhering particles in the sheet former column using a squeeze bottle.
- If the stickies quantity on the filter paper is too high, so that stickies overlap each other, perform the screening step again with a lower sample amount. For example, reduce the sample quantity from 5 g OD to 2.5 g OD, and if necessary, reduce it further.
- If there are a lot of specks on the filter, the sorting time can also be increased without reducing the sample quantity of 5 g.
- Each deviation (reduction of the sample quantity/change of the sorting time) must be documented.

5	Specimen preparation	• Carefully remove the filter from the sheet former, place it with the underside on a carrier board and cover it with silicon paper (siliconized side in contact with the stickies). • Dry it in the dryer of the sheet former for 10 min at (93 ± 4) °C. • Pour the black ink (e.g. Pelikan No. 4001) onto a plate. • Dip the filter sheet completely into the ink. The entire filter surface must be covered in ink. • Place the filter sheet over a piece of kitchen paper to absorb the ink excess. • Place the dyed filter sheet with the underside on one carrier board and cover it with silicon paper. • Dry the sample in the dryer of the sheet former for 10 min at (93 ± 4) °C. • Pulverise the sticky side of the filter sheet with corundum powder evenly in excess (thin layer). • Place the filter sheet with the underside on one used carrier board and cover it with silicone paper and another carrier board. • Dry the sample between two preheated metal plates (6 kg, 28 cm diameter) for 10 min in a drying oven

		at (105 ± 2) °C. (Store the metal metal plates permanently in the oven at (105 ± 2) °C). • Remove the excess of corundum powder using a brush.
6	Visual Inspection  *Figure 40: Example of a prepared macro stickies specimen.*	• Visually check the filter sheets to avoid the stickies overlapping. • Remove any hydrophobic contamination (e.g. plastic pieces) by hand or using tweezers or colour them black using a permanent marker. • The surface of filter sheets should not contain bends or waves.
7	Image analysis using image analysis system m e.g. PTS-DOMAS Multispec (see separate description)	

7 Evaluation

Step Nr.	Notes	Description
1	The evaluation scheme for this method is published separately.	

8 Documentation

Excel Spreadsheet

List of changes

Rev.	Valid from	Author	Change(s)
01/2021-09-01	November 2021	Marie Geißler	First version of the detailed description
02/2021-12-01	December 2021	Marie Geißler	(Version used in the 3x3 validation.) - Determination of the dry matter content only in the oven acc. ISO 287 - Drying of the rejects also only in the oven - Defining the exact procedure for the Somerville Screening (2.5 cm water level before filling in the sample which is exact 20 g OD) - Cancelling of the macro stickies assessment, because it was not relevant for the 3x3 tests
03/2022-03-14	March 2022	Vanessa Wortmann	- Detailed definition for the procedure of the DCS (chapter 5) - Description of the procedure for the sheet adhesion tests - Including macro stickies as a parameter again for the for the completeness of the document (excluding procedure for image analysis)
04/2022-03-14	March 2022	Vanessa Wortmann	- Update on the procedure for drying the coarse reject (topic 6.1), and fine reject (topic 6.4) first in dryer of the sheet former and then in the oven. - Update on the procedure for drying the consistency filter paper (topic 6.2) two times in the dryer of the sheet former.
05/2022-03-23	March 2022	Vanessa Wortmann	- Filter paper with 125 mm was added to the materials list - The standard DIN 12331:1988-10 was replaced for the most updated version DIN EN ISO 3819:2016 – 05.

06/2022-05-23	May 2022	Vanessa Wortmann	- Aging in the sample preparation - Aluminium tray as an alternative to the filter paper in the coarse reject drying. - Photo documentation of the coarse reject on a dark background.
07/2022-06-21	June 2022	Vanessa Wortmann Marie Geißler	Update of the detailed description according to the CEPI revision 2022. - Artificial ageing of samples containing WSA: 60°C for 3 days - Natural ageing of sample without WSA: 15 days - pH of tap water used in the disintegration: mildly alkaline (7-8) - Pouring time in the fine screening: max. 4 min - Collection of the whole accept fraction from the fine screening (AF) and use of a thickener in ideal conditions. - Collection of at least 50 l of AF if a thickener is not available - Filtration proceeded only 2 times, one for rinsing the suction flask and another to determine the dissolved in colloidal substances.
08/2022-09-27	September 2022	Vanessa Wortmann	- Amount of material needed to complete all measurements was included. - Suppliers of lab equipment erased - Self-protection equipment erased - List of test equipment and materials more detailed - Use of two carrier boards in the analysis of macro stickies erased. - PTS logo updated. - Changes from the feedback of the Consultative Group Summer 2022