
**Recycled pulps — Estimation of Stickies
and Plastics —**

**Part 2:
Image analysis method**

*Pâtes recyclées — Estimation des matières collantes et des matières
plastiques —
Partie 2: Méthode par analyse d'image*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 15360 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15360-2 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*.

ISO 15360 consists of the following parts, under the general title *Recycled pulps — Estimation of Stickies and Plastics*:

- Part 1: *Visual method*
- Part 2: *Image analysis method*

Annexes A and B form a normative part of this part of ISO 15360.

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Introduction

This part of ISO 15360 is complementary to ISO 15360-1, which concerns estimation of Stickies and Plastics in recycled pulp by visual examination. ISO 15360-2 is particularly useful for pulps having a high content of Stickies or Plastics or both where visual inspection is very time-consuming to carry out.

Two alternatives are given for the estimation of Stickies, while there is only one procedure for the estimation of Plastics.

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Recycled pulps — Estimation of Stickies and Plastics —

Part 2: Image analysis method

1 Scope

This part of ISO 15360 specifies a method to estimate Stickies and Plastics in all grades of recycled pulp. It applies the principles for the separation of Stickies and Plastics described in ISO 15360-1. The visualization and counting techniques described in that standard are replaced here by image analysis.

This part of ISO 15360 permits the use of different laboratory screening devices as well as screens of different sizes when applying the specified method. It is impracticable to define more closely the equipment or screen sizes to be used, because of the wide range of Stickies and Plastics found in recycled pulps, and the various screening devices which are being successfully used for their measurement.

NOTE This method will only estimate those Stickies and Plastics which are tacky and which are retained on the screen of a given slit size under the temperature and pressure conditions of this test method. It should be noted that this will probably not be the total amount of Stickies and Plastics that are actually present in a given pulp sample.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 15360. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 15360 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 638, *Pulps — Determination of dry matter content*

ISO 4119, *Pulps — Determination of stock concentration*

ISO 5263, *Pulps — Laboratory wet disintegration*

ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method*

ISO 7213, *Pulps — Sampling for testing*

ISO 8486-1, *Bonded abrasives — Determination and designation of grain size distribution — Part 1: Macrogrits F4 to F220*

DIN 54516:1985, *Testing of paper and board — Determination of plybond resistance*

TAPPI T 541 om-89, *Internal bond strength of paperboard (z-direction tensile)*

3 Terms and definitions

For the purposes of this part of ISO 15360, the following terms and definitions apply.

3.1

Stickies

diverse group of materials that are retained on a laboratory screen of given slit aperture (100 µm or 150 µm) and which adhere to objects that they touch

NOTE 1 Stickies can adhere to objects at ambient conditions or they can adopt adhesive characteristics when subjected to elevated temperature, elevated pressure or change of pH.

NOTE 2 The following is a non-exhaustive list of Stickies: products derived from residues of materials such as inks, tars, hot melts, waxes, and multivalent metal ion soaps or different types of pressure-sensitive adhesives (tapes).

NOTE 3 A Stickey particle can be a composite of adhesive material together with non-adhesive plastic fragments and cellulosic fibres.

3.2

Plastics

non-adhesive polymers that are retained on a laboratory screen of a given slit aperture (100 µm or 150 µm), but excluding cellulosic materials

NOTE The following is a non-exhaustive list of Plastics: polymeric materials derived from such sources as polyethylene, polypropylene, polyester, UV-cured coatings and polystyrene.

4 Principle

A sample of pulp in disintegrated form is washed through a laboratory screen of a given slit aperture until the filtrate is clear. The material is removed and transferred to a filter paper. The Stickies are marked either with white aluminium oxide powder or by particles of coating that they pull out from a special coated paper. The Plastics are highlighted by dyeing the filter paper black and hiding the Stickies with black silicon carbide powder. The Stickies and Plastics are measured using an image analyser. The total numbers of Stickies and Plastics are determined and reported. Other report formats are possible, including the numbers of Stickies and Plastics of various areas, or a histogram showing the area distribution.

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5 Apparatus and equipment

5.1 Disintegrator as specified in ISO 5263.

5.2 Laboratory screening equipment, fitted with a screen complying with the specification as described in annex A.

NOTE For the purpose of this method, a screen is that part of the laboratory screening equipment which separates the Stickies and Plastics from the cellulose fibre. Various laboratory screening equipment may use different terminology to describe the separating medium (e.g. "slots" instead of "slits").

5.3 An image analyser system for illuminating, viewing, and detecting images. The image analyser system used shall be capable of scanning or viewing the total area of the filter paper upon which the Stickies or Plastics are collected (20 cm diameter or larger). The image analyser system incorporates the following components.

5.3.1 Specimen stage, generally consisting of a flat plate on one side of which the specimen illuminant and detector are located. The specimen stage shall be shielded to prevent influence from ambient light. The prepared specimen is a piece of filter paper upon one side of which the Stickies and Plastics have been collected. The side of the prepared specimen upon which the Stickies and Plastics have been collected faces the illuminant and image detector. The exact configuration of the specimen stage will depend upon the image detector used.

5.3.2 Image detector, either a scanner or camera based image detector, with at least 256 grey levels of sensitivity and a physical pixel resolution below 50 µm/pixel such that four contiguous pixels will have a combined area of 0,01 mm² or less. The image detector shall view the specimen normal to its surface.

5.3.3 Illuminant, un-polarized with wavelength concentrated in the visible portion of the spectrum, such that 95 % of the light reflected from a white surface will be between 380 nm and 750 nm. As a minimum, the illuminant shall

consist of two members providing illumination at an incident angle of $45^\circ \pm 5^\circ$. The illumination from the two members shall oppose each other at an angle of 180° . A preferred illuminant consists of four members each providing incident illumination at $45^\circ \pm 5^\circ$, placed such that the four members oppose one another at angles of 90° . The most preferred illuminant provides illumination that is diffuse or axially symmetric with an incident angle of $45^\circ \pm 5^\circ$. The uniformity of illumination over the area of the specimen stage before any software corrections shall be within $\pm 4\%$.

5.3.4 Image analysis software, capable of determining the average intensity ("grey level") of a detected image, for example, a Stickers or piece of Plastic, as well as the average background intensity surrounding the speck when examining the picture elements of a digitized Stickers or Plastics speck using a "centre-surround" filtering technique [1] to [4]. The normal size of the software filter is a $1,0 \text{ mm}^2$ area centred on the Stickers or Plastic. The software shall be capable of adjusting this area as required to completely surround the detected image of a Stickers or Plastic. The detection threshold is 10 % of the 100 % contrast scale on the comparison chart (annex B). The detection threshold is applied between the contrary and the average background intensity as defined by the centre surround filter.

5.4 Calibration chart as shown in annex B. The chart is a film with a series of black and grey spots of different shapes, areas and contrasts. The chart is used for checking the performance of the image analyser system.

5.5 Büchner funnel with a sintered glass filtering base of 20 cm diameter or larger. The funnel may be substituted by a Rapid-Köthen sheet former (ISO 5269-2).

5.6 White or black filter paper, comprised of 100 % cellulose fibres, qualitative, medium fast grade, to fit the funnel or sheet former (5.5).

5.6.1 White filter paper for marking the Stickers by metallic powder.

5.6.2 Black filter paper for marking the Stickers by coating particles pulled out from a coated paper.

5.7 Release paper, silicone coated.

5.8 Oven, capable of maintaining a temperature of $105 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$.

5.9 Heated press capable of applying a pressure of $95 \text{ kPa} \pm 5 \text{ kPa}$ at $94 \text{ }^\circ\text{C} \pm 4 \text{ }^\circ\text{C}$ for 10 min. A Rapid-Köthen sheet dryer operated at these conditions is suitable (ISO 5269-2).

5.10 Shallow glass dish, approximately $25 \text{ cm} \times 20 \text{ cm}$. The exact size of the glass dish is unimportant, provided the smaller dimension is greater than the diameter of the filter paper.

5.11 Metal plates, the top plate of which is circular with a diameter of $28 \text{ cm} \pm 1 \text{ cm}$ and a mass of $6,0 \text{ kg} \pm 0,1 \text{ kg}$ (5.6.1), and the bottom plate of which is the same size as the top plate or larger. The bottom plate may be of any convenient shape, for example circular with a minimum diameter of 28 cm, square with a minimum length of 28 cm, or other shape as desired.

5.12 Filter washing device (see 10.4.2) to wash the filter with a gentle stream of water at a pressure of about 0,1 MPa (1 bar), flow rate of about 10 ml/min and distance of delivery tip from filter of about 180 mm.

5.13 Water resistant black felt pen.

6 Reagents

6.1 For marking the Stickers by metallic powder adhering to the Stickers

6.1.1 White powdered aluminium oxide, purified Al_2O_3 with a grain size distribution of F220 according to ISO 8486-1

6.1.2 Aqueous solution of a black dye which is substantive to cellulose. Commercial black inks have been found suitable.

6.1.3 Black powdered silicon carbide, SiC, with a grain size distribution of F220 according to ISO 8486-1.

6.2 For marking the Stickies by coating particles pulled out from a coated paper

6.2.1 Coated paper as specified in the following: wood-free sized base paper, grammage about 70 g/m² to which is added a coating consisting of calcium carbonate and a synthetic binding material on one side of 50 g/m² to 55 g/m², brightness (ISO) 85 % ± 3 %, plybond resistance of coating 2,0 kN/m ± 0,5 kN/m tested according to DIN 54516 or 5,5 kPa ± 1,5 kPa tested according to TAPPI T 541 for a total sheet grammage of 120 g/m² to 125 g/m², size large enough to completely cover the black filter paper carrying the Stickies. When placed in contact with Stickies under the temperature and pressure specified in 5.9, the coating adheres to the Stickie particle, causing it to appear white in colour. To verify coating plybond resistance under the conditions of this test, the coated sheet may be processed according to 10.4.1 but without any Stickies on the filter paper, in which case no white particles shall be removed from the coated paper. Similarly, processing a trial sample of material containing Stickies shall cause the Stickies to be fully visualized as white areas on the black filter paper.

7 Sampling

If the estimation of the Stickies and Plastics is to represent the Stickies and Plastics in a lot of pulp, the number of samples to be tested and their selection shall be in compliance with ISO 7213. If the test is made on another type of sample, report the source of the sample and if possible the sampling procedure used.

From the sample received, select specimens so that they are representative of the whole sample.

8 Adjustment and calibration of the image analysis system

Activate the image analyser (5.3) according to the manufacturer's instructions, and allow it to warm up.

Calibrate the image analyser system software using the calibration chart (5.4) and the manufacturer's instructions for the software used. Use the chart to verify that the image analysis system correctly measures the size of the spots at 100 % contrast to within ± 5 %. If this criterion is not met, correct the calibration by consulting the calibration instructions accompanying the image analyser (5.3).

Set the image analysis software to separate the particles into classes based upon their measured area. The bottom limit for the smallest class will depend upon the size of the opening of the screen used. The number of particle size classes can be varied based on the information desired. The largest class size shall have no upper limit, so that all the particles present will be reported. The software will generally have several calculation capabilities such as counting the total number of Stickies in the various size classes chosen, determining the total physical area of the Stickies measured and plotting of histograms and frequency distributions.

9 Pretreatment of the sample

Determine the dry matter content according to ISO 638.

Soak air-dried pulp samples for at least 4 h in water (tap water may be used), but a wet lap pulp may be disintegrated immediately. Pulp stock with a concentration of 10 % or less need not be disintegrated.

Using the procedure described in ISO 5263, disintegrate a sample of pulp appropriate to the screening equipment used. The exact amount of pulp which must be processed may be varied based upon the level of Stickies and Plastics present. A total amount of 50 g oven-dried pulp shall be used for recycled pulps such as deinked pulp where Stickies and Plastics levels are moderately low. In pulps with higher levels of Stickies or Plastics the quantity may be reduced to 10 g oven-dried pulp.

When necessary, determine the concentration of the pulp stock, according to ISO 4119.

Run the analysis in triplicate. Thus for a complete examination of Stickies and Plastics, approximately 150 g of pulp are generally required.

10 Procedure

10.1 Processing the sample through the screening device

Following the relevant instructions from the manufacturer of the screening device used, process the prepared pulp stock (clause 9) until a clear filtrate is obtained. Note the processing time.

10.2 Distribution of the separated Stickies or Plastics on the filter paper

Some commercially available laboratory screening devices automatically transfer the separated Stickies and Plastics to a filter paper, in which case this step is omitted.

For laboratory screening devices where the separated Stickies and Plastics remain on the screen, proceed as follows.

Remove the screen from the screening device and stand it vertically in a suitable container. Wash the screen slits first from the underside and then from the topside of the screen with a fine jet of high pressure water. Be careful that all the material removed from the screen remains in the container. Use the minimum amount of water required to remove all of the screen rejects.

Retain the screen for subsequent visual examination.

Place a white filter paper (5.6.1) onto the filtering surface of a Büchner funnel (5.5). Filter the wash solution containing the Stickies and Plastics through the filter paper under vacuum until all free water is removed. Ensure a uniform distribution of Stickies and Plastics on the filter paper by using care in adding the suspension to be filtered. In those cases where the level of collected particles is so high that they touch or overlap on the filter paper, several filter papers may be required, or the analysis can be performed using a smaller mass of pulp. When the collected particles are divided among several filter papers, examine the total area of all of the filter papers in the image analyser system.

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NOTE A Rapid-Köthen sheet former may be substituted for the Büchner funnel.

When all the suspension has been filtered, examine the screen for remaining Stickies and Plastics. If any remain, transfer them to the filter paper.

10.3 Determination of Stickies by marking with metallic powder

10.3.1 Heat setting

Remove the filter paper from the collection device and cover the top side (the side where the Stickies and Plastics have been deposited) with the siliconized side of a silicone coated release paper (5.7). Place the filter paper and siliconized sheet with the filter paper on the bottom in a heated press (5.9) at a pressure of $95 \text{ kPa} \pm 5 \text{ kPa}$ and $94 \text{ °C} \pm 4 \text{ °C}$ for $10,0 \text{ min} \pm 0,5 \text{ min}$.

10.3.2 Dyeing

Remove the siliconized sheet retaining the Stickies and Plastics on the filter paper. Examine the collected particles visually. Remove any metal filings, or other non-cellulosic extraneous material obviously not a Stickey or Plastic as defined in clause 3. In removing these extraneous materials, make certain that Stickies or Plastics particles, including those which may be adhering to extraneous materials, are not removed. Shives and wood fibres need not be removed, as they will be dyed black and will not interfere.

Add a solution of an aqueous cellulose substantive black dye (6.1.2) to a shallow glass dish (5.10) to a depth of about 15 mm.