INTERNATIONAL STANDARD

ISO 15360-1

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Recycled pulps — Estimation of Stickies and Plastics —

Part 1: Visual method

Pâtes recyclées — Estimation des matières collantes et des matières plastiques —

Partie 1: Méthode visuelle

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

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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 15360-1 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulp*.

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.

Introduction

The production of pulp from de-inked recovered fibres and from brown or mixed waste grades is increasing in many parts of the world. Many of the recovered papers used in the production of recycled pulps contain adhesives, latex and other materials, which are either intrinsically "tacky" or can become so under appropriate conditions of temperature, pH and pressure. Residual particles of such materials can cause problems when the pulp is subsequently used in paper manufacture. In addition, recycled pulp feedstock is sometimes derived from material that has been plastics coated and the presence of plastics in the finished pulp can also cause problems, especially in the manufacture of coated papers. Moreover, plastics found in recovered paper may also come from a collection which has not been properly sorted.

International Standards exist for the determination of visible dirt and shives in pulp and these could be applied to de-inked pulp. However Stickies and Plastics are often similar to the pulp in colour and, even when large, are difficult to detect by visual inspection. Different techniques have thus to be employed.

This part of ISO 15360 is based on a visual identification and count of the Stickies and Plastics. Instrumental techniques are also available for estimating Stickies and Plastics. However, these are less widely used but may be the basis of a future International Standard for Stickies and Plastics in recycled pulps.

NOTE Different types of laboratory screening equipment, complying with this part of ISO 15360 may be used to isolate the Stickies and Plastics from the cellulose stock. It should be noted that different types of laboratory screening equipment may give different results. Furthermore, screening equipment of the same style fitted with different screens complying with this part of ISO 15360 may also give different results because of the difference in the distribution of slit sizes within the screen.

Recycled pulps — Estimation of Stickies and Plastics —

Part 1:
Visual method

1 Scope

This part of ISO 15360 specifies a method to estimate Stickies and Plastics in a wide variety of pulps including all recycled grades. It is not intended for the estimation of visible dirt and shives which is covered by the ISO 5350 series, or for visible contraries in recycled pulps, which is covered by ISO 15319 [1].

This method will only capture those Stickies and Plastics which are retained on the screen of a given slit size. 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 5269-1, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method.*

ISO 5350-1, *Pulps — Estimation of dirt and shives — Part 1: Inspection of laboratory sheets.*

ISO 5350-2, *Pulps — Estimation of dirt and shives — Part 2: Inspection of mill sheeted pulp.*

ISO 7213, *Pulps — Sampling for testing.*

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 (5.2) of given slit aperture (100 µm or 150 µm), and which adhere to objects which they touch; Stickies may adhere objects at ambient temperature or they may adopt adhesive characteristics when subjected to elevated temperature, elevated pressure or change of pH

NOTE 1 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.

NOTE 2 A Stickie particle can be a composite of adhesive material, together with non-adhesive plastics fragments and cellulose fibres.

3.2
Plastics
non-adhesive polymers that are retained on a laboratory screen (5.2) of a given slit aperture, 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.

3.3
screen
that part of the laboratory screening equipment which separates the Stickies and Plastics from the cellulose fibre

4 Principle

A sample of pulp in disintegrated form is passed through a laboratory screen of given slit aperture until the filtrate is clear. The material retained on the screen is removed and transferred to a filter paper. The Stickies and Plastics are identified and their total numbers and areas are estimated separately.

5 Apparatus and equipment

5.1 Disintegrator as specified in ISO 5263.

5.2 Laboratory screening equipment, fitted with a screen (3.3) complying with the specification described in annex A.

NOTE Various types of laboratory screening equipment may use different terminology to describe the separating medium (e.g. “slots” instead of “slits”).

5.3 Filter paper; a qualitative, medium/fast grade is recommended.

5.4 Tweezers.

5.5 Illumination device: a suitable light source for the investigation of filter papers containing Stickies and Plastics particles in reflected light. The light shall be strong enough to ensure that all particles having the minimum area agreed upon are visible.

5.6 Light table, as specified in ISO 5350-1 and ISO 5350-2. This is needed only when Stickies are estimated at elevated temperature and pressure, see 7.5.3.

5.7 Desk-top magnifying glass, magnification ×2 to ×4, or **microscope**, stereo type, magnification ×10 to ×40 (optional only).

5.8 Dissecting needles.

5.9 Heated press, capable of applying a load of 690 kPa ± 20 kPa at a constant temperature of 150 °C ± 10 °C.

5.10 Comparison chart, as shown in annex B.

5.11 Blotters, as used for the preparation of laboratory sheets in ISO 5269-1.

5.12 Oven, capable of maintaining an air temperature of 105 °C ± 2 °C.

6 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 accordance with ISO 7213. If the test is made using another type of sampling, make sure that the test pieces are representative of the sample received.

7 Procedure

7.1 Temperature

All operations described in this clause, with the exception of 7.5.3 (Stickies at elevated temperature), shall be carried out at room temperature (20 °C to 25 °C).

7.2 Pretreatment of sample

Determine the dry matter content as described in ISO 638.

Soak air-dried pulp samples for at least 4 h in water (tap water may be used). Wet-lap pulp may be disintegrated immediately (see 5.1). Pulp stock with a consistency of 10 % or less need not be disintegrated.

Using the procedure described in ISO 5263, disintegrate a sample of pulp of mass and concentration appropriate to the screening equipment used. (See the second paragraph of 7.2.) The conditions of disintegration shall be reported in clause 10.

NOTE The procedure for visible contraries in recycled pulp (ISO 15319) may be used. This procedure disintegrates 50 g to 60 g oven-dried pulp in 2 700 ml of water, until the stock is just clear of fibre bundles.

When necessary, determine the concentration of the stock as described in ISO 4119.

7.3 Screening of the disintegrated stock

Following the manufacturer's instructions for the relevant laboratory screening equipment (5.2), add a portion of the prepared stock to the screen (3.3). Process the stock until the filtrate is clear.

It is recommended that initially a total mass of 100 g oven-dried pulp should be processed. It may be necessary to process more or less than 100 g of pulp depending on the content of Stickies and Plastics.

7.4 Removal of material retained on the screen

Stand the screen vertically in a suitable container and wash the slits first from the underside and then from the top side of the slits, with a fine jet of high pressure cold water. Use an amount of washing water that is just sufficient, as far as possible, to remove all the material retained on the screen. Retain the screen for a subsequent visual re-examination.

Filter the suspension through the filter paper (5.3) ensuring a uniform distribution of Stickies and Plastics on the filter paper. The number of filter papers used is determined by the amount of material retained on the screen.

NOTE Some commercial laboratory screening equipment transfers the material from the screen to the filter paper automatically.

When all the suspension has been filtered, re-examine the screen. Any remaining particles which might be Stickies or Plastics should be removed from the screen with the aid of a pair of tweezers (5.4) and placed on one of the filter papers.

Place all the filter papers on separate blotters (5.11) and dry at 105 °C for 1 h in an oven (5.12).

7.5 Examination of the material retained on the screen

Examine the filter paper(s) in reflected light by eye to identify the types of particles present. Record the number of particles of Stickies and Plastics and estimate the area of each particle. Calculate the total area of each particle type.

A magnifying glass or stereo-microscope (5.7) may be used as an aid to identify the Stickies and Plastics, but must not be used to count the number of these particles present or to estimate their areas.

7.5.1 Identification and size estimates of the Stickies at ambient temperature

Stickies tend to be opaque and rounded in shape and may bind with toner, ink or dye particles, giving the aggregate a coloured appearance. Starting with the largest particle, probe with a dissecting needle (5.8). If the particle is adhering or embedded in the filter paper then estimate the particle size using the comparison chart (5.10). Circle each Stickie identified; repeat the estimate of size for each particle present. Record the total number of particles present and calculate the total area, in square millimetres, of the Stickies.

7.5.2 Identification and size estimates of the Plastics

Plastics are usually present in flakes or strands, often associated with fibre bundles. They may be clear or coloured, but rarely black. Starting with the largest particle not identified as a Stickie, examine each non-Stickie particle under a magnifying glass or microscope (5.7) and with tweezers. If the particle stretches and it fulfils the appearance criteria, estimate the particle size using the comparison chart (5.10). Circle and estimate the area for each Plastics particle identified. Record the total number of particles and calculate the total area, in square millimetres, of the Plastics.

7.5.3 Identification and size estimates of the Stickies at elevated temperature and pressures

Some Stickies, such as those derived from hot-melt adhesives, only become evident when subjected to elevated temperatures and pressures and become non-Stickies again when the temperature returns to ambient. Since there is a range of softening temperatures and pressures over which it occurs, estimation of these Stickies is optional. This part of the procedure may be used if it is suspected that the recycled pulp feedstock contains any bookbinding adhesives.

Place the filter paper(s) in the heated press (5.9) for 10 min ± 2 min at 690 kPa ± 20 kPa and 150 °C ± 10 °C.

These Stickies will be revealed as translucent spots in the filter paper. Place the filter paper on the light table (5.6). Circle and estimate the translucent areas using the comparison chart. Record the total number of translucent spots and calculate the total area, in square millimetres, of these Stickies. Report these values separately to the values for Stickies at ambient temperature. Record in clause 10 the temperature and pressure used to activate these Stickies.

8 Calculations

8.1 Number of Stickies and Plastics

Record separately the number of Stickies and, if tested, Plastics at ambient temperature, and Stickies at elevated temperature and pressure.

Calculate separately the total number of Stickies and Plastics per kilogram of pulp using the formula:

$$Y = \frac{a}{m} \quad (1)$$

where

Y is the total number of Stickies or Plastics, as relevant, expressed in number per kilogram of oven-dried pulp;

a is the observed total number of Stickies or Plastics, as relevant;

m is the oven-dried mass of the pulp selected for the test, in kilograms.

8.2 Area of Stickies and Plastics

Calculate separately the total area of Stickies and Plastics per unit mass of pulp using the formula:

$$X = \frac{A}{m} \quad (2)$$

where

X is the total area of the Stickies or the Plastics per unit mass of pulp, expressed in square millimetres per kilogram;

A is the total area of the Stickies or the Plastics, in square millimetres;

m is as defined in 8.1.

9 Precision

Five laboratories using either a Somerville Fractionator[1)] or a Pulmac Master Screen[1)] evaluated one batch of de-inked pulp for Stickies (including hot melts) and Plastics. Based on a count of the particles present, the results were as shown in Table 1.

1) The Somerville Fractionator and the Pulmac Master Screen are examples of suitable models of screening equipment available commercially. This information is given for the convenience of users of this part of ISO 15360 and does not constitute an endorsement by ISO of these models.

Table 1

Parameter	Number per kilogram of oven-dried pulp	
	Stickies	Plastics
Range of counts	30 to 70	30 to 100
Coefficient of variation, %	35 to 68	15 to 45

The variation includes inhomogeneity in the inspected batch and differences in the screen in addition to the variation in the procedure.

10 Test report

The test report shall include the following particulars:

a) reference to this part of ISO 15360;

b) all the information necessary for complete identification of the sample;

c) the number of Stickies at ambient and elevated temperature (if determined) and Plastics per kilogram of oven-dried pulp, and their total areas, expressed as square millimetres per kilogram of oven-dried pulp;

d) the mass of pulp inspected;

e) full details of conditions used in disintegration of the pulp (stock concentration and disintegrator revolutions);

f) full details of the type of commercial screening equipment used (make, model, together with number of slits, slit aperture and configuration of the screen);

g) the screening time;

h) the temperature and pressure conditions used to activate Stickies (hot melts) at elevated temperature, if tested;

i) any particular points observed in the course of the test;

j) any departure from this part of ISO 15360, or any other circumstances or influences regarded as optional, that could have affected the results.

Annex A
(normative)

Laboratory screening equipment

The laboratory screening equipment shall be fitted with a screen with slit widths of either 100 µm or 150 µm. The average slit width shall be ± 5 µm of the specified slit width. No individual slit width shall be more than 15 µm from the average of all slit widths.

In certain applications, other slit widths such as 80 µm or 200 µm may be used; this must be reported with the results.

References for two well-known models of screening equipment, the Somerville Fractionator[1)] and the Pulmac Master Screen[1)] are given in the bibliography ([4] and [5]). These references contain the details of the design and permitted tolerances of the screens used in this equipment. Other screens may also be used, provided that it can be demonstrated they meet the necessary tolerance requirements.

The responsibility of ensuring that the screen slits are within the prescribed tolerances is usually that of the manufacturer of the screening equipment, who shall provide evidence that this has been carried out. The screen shall be returned to the manufacturer at agreed intervals to check that the slits remain within the required tolerances.

The width of the screen slits is paramount in this test method. The screen shall be kept clean and the slits be examined at regular intervals during use with the aid of a magnifying glass to ensure that there is no deterioration to the edges of the slits, or any damage causing larger apertures to be evident.

It should be noted that different types of laboratory screening equipment may give different results. Furthermore, screening equipment of the same style fitted with different screens complying with annex A may also give different results because of the difference in the distribution of slit sizes within the screen.

Annex B
(normative)

Comparison chart

This chart is identical with the charts included in ISO 15319 [1] and ISO 15755 [2].

Use the chart shown here.

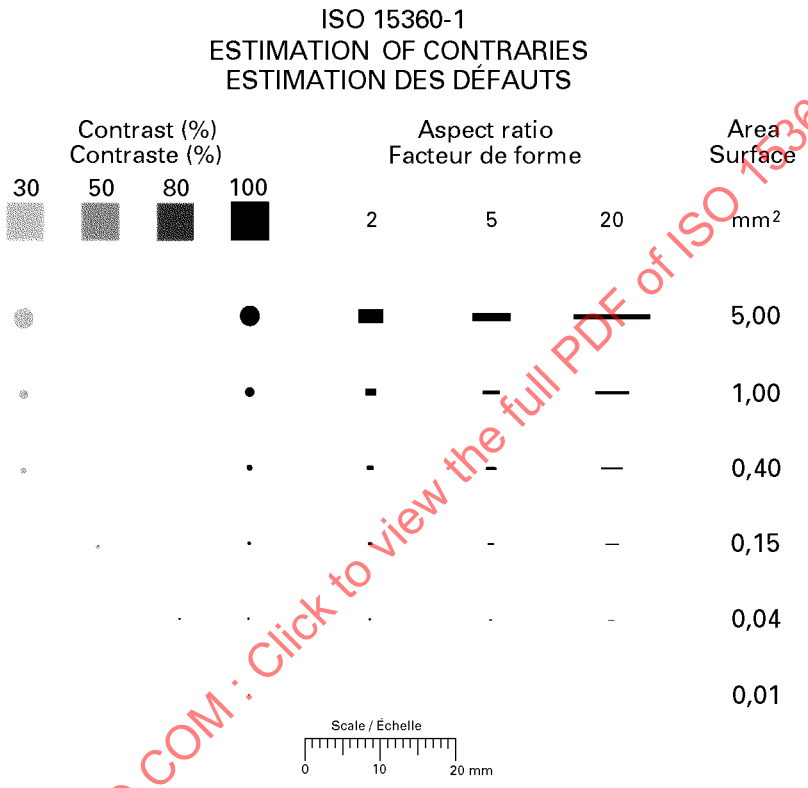

Figure B.1 — Estimation of particle size — Comparison chart

Do not use a photocopy of the chart in any inspection, because reproduction may change the size and contrasts of the spots.

The left part of the chart is intended for checking instrumental devices and is not used for this part of ISO 15360. The spots on the right-hand side indicate contraries of different aspect ratios. The contrast ratio is 100 %. This part of the chart is to be used as an aid for size classification.