

# INTERNATIONAL STANDARD

# ISO 15319

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## Recycled pulps — Estimation of visible contraries by instrumental means using reflected light

*Pâtes recyclées — Estimation des défauts visibles par des moyens  
automatiques en lumière réfléchie*



Reference number  
ISO 15319:1999(E)

## Foreword

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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.

International Standard ISO 15319 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulp*.

Annexes B and C form a normative part of this International Standard. Annex A is for information only.

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International Organization for Standardization  
Case postale 56 • CH-1211 Genève 20 • Switzerland  
Internet iso@iso.ch

Printed in Switzerland

## Introduction

This International Standard is complementary to ISO 5350-1, which concerns inspection of laboratory sheets by transmitted light, ISO 5350-2, which concerns inspection of mill sheets by transmitted light, and ISO 5350-3, which concerns inspection of mill sheets by reflected light. All these International Standards are intended for pulps in general and are primarily based on visual inspection, though procedures based on instrumental means are incorporated in the annexes of ISO 5350-1 and ISO 5350-2.

**NOTE** A high content of specks renders visual inspection very time-consuming to carry out.

In this International Standard, the inspection procedure and the instrument to be used are not described in detail, as hindrances should not be created for the future development of instruments for counting of contraries.

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# Recycled pulps — Estimation of visible contraries by instrumental means using reflected light

## 1 Scope

This International Standard specifies a method for the estimation of visible contraries in pulp by instrumental means using reflected light.

This International Standard cannot be used for inspection of pulps of low brightness, i.e. for evaluation of light contraries in dark pulp. Spots which look brighter than the surrounding area due to fluorescence will not be detected by the method described in this International Standard.

NOTE 1 The evaluation of contraries smaller than 0,04 mm<sup>2</sup> is not covered by this International Standard. See informative annex A.

NOTE 2 This International Standard is mainly intended for the assessment of product quality. Modifications may be needed if it is applied to process control.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method.*

ISO 7213, *Pulps — Sampling for testing.*

## 3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

### 3.1 contrary speck

any unwanted particle, embedded in the surface of the sheet, including dirt and shives, of specified minimum size and having a markedly contrasting light reflectance with respect to the surrounding area of the sheet, according to the comparison chart in annex B

## 4 Principle

Laboratory sheets are inspected in reflected light by instrumental means. The areas of all contraries larger than a specified value and having markedly contrasting light reflectance with respect to the surrounding sheet are recorded. The areas are added and the total area of the contraries is reported as square millimetres per square metre of laboratory sheets. If required, the areas or numbers of contraries in different size classes may also be reported.

## 5 Classification of contraries

Contraries having an area of at least 0,04 mm<sup>2</sup> and minimum contrast according to Table 1 shall be noted. See normative annex C.

It is usual to report only the total area, though when required, the area or number of contraries in different size classes may be reported. In this case, the classification given in Table 1 should be used.

**Table 1 — Classification of contraries according to area**

Class	Size mm <sup>2</sup>	Minimum contrast %
1	> 5,00	30
2	1,00 - 4,99	30
3	0,40 - 0,99	30
4	0,15 - 0,39	50
5	0,04 - 0,14	80

## 6 Equipment

**6.1 Disintegrator**, as specified in ISO 5263. The disintegrator is not needed if the sample to be inspected consists of pulp in slurry form.

**6.2 Sheet-making equipment**, for example as specified in ISO 5269, provided with a wire having a mesh aperture of 125 µm.

**6.3 Clean blotters**, of the size corresponding to the sheet-making apparatus.

**6.4 Drying plates**, of the same size as the laboratory sheets, made of corrosion-resistant metal or other suitable material, such as rigid plastics, glazed or polished on at least one side. It is essential that the wet sheet adheres firmly to the plate. The plate shall be flat and free from any perceptible bulges or distortions.

NOTE Drying plates need not be used for Rapid-Köthen sheet forming.

**6.5 Automatic device for counting of contraries**, using reflected light and so constructed that the contraries having areas and contrasts corresponding to Table 1 can be evaluated, counted and reported. 95 % of the light emitted by the device shall have a wavelength of 380 nm to 750 nm. The device shall not count, as contraries, particles which contrast because of their fluorescence. The repeatability of the device shall fulfil the requirement that the coefficient of variation does not exceed 15 % when the determination of the total area of the contraries in a specimen is repeated five times.

The instrument shall exhibit a maximum pixel size of 0,01 mm<sup>2</sup> and a minimum grey scale resolution of 0,5 % reflectance. The detection threshold changes step by step as indicated in Table 1. See also normative annex C.

**6.6 Comparison chart**, with a series of black and grey spots of different shapes, areas and contrasts. This shall be used for checking the instrument. The chart is included in normative annex B.

NOTE This chart is identical to the one used in ISO 15755.

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

Do not use the chart in ISO 5350-1 and 5350-2, as that chart is intended for examination by transmitted light.

## 7 Preparation of sample

### 7.1 Sampling

If the test is being made to evaluate a lot of dry pulp, the number of bales to be sampled and the method of taking the samples shall be in accordance with ISO 7213. When sampling unbaled wet laps, the edges of the laps and the top and bottom laps shall be avoided. At least ten samples shall be taken from a lot.

Inspect each sample separately. Take a set of test pieces from each sample, including at least four separate areas of the sample. Each test piece shall include the complete thickness of the pulp sheet. Keep the sets of test pieces separately and make sure that they are protected from contamination by dirt from the surroundings.

The total mass of a set of oven-dry test pieces shall be at least 60 g.

### 7.2 Pretreatment of the test pieces

#### 7.2.1 Sample in the form of sheets or laps

Determine the dry matter content of the sample as described in ISO 638.

Soak the set of test pieces, if dry, for at least 4 h. Use distilled or deionized water. Add water until the consistency is 50 g to 60 g of oven-dry pulp per 2 700 ml of water. Disintegrate the test pieces in the disintegrator (6.1). They should be disintegrated just until no fibre bundles remain. It is recommended that the set of test pieces is disintegrated for 1 min and then visually examined by diluting a sample portion to see that no fibre bundles are present, repeating the process until only individual fibres remain.

NOTE Care should be taken to avoid contamination of the pulp during the test. Ensure that the surface and the elements of the disintegrator and sheet-making equipment are clean and free from corrosion and deposits.

#### 7.2.2 Pulp in the form of a suspension (slurry)

Determine the dry matter content of the sample as described in ISO 4119.

To make the slurry homogeneous, add water and mix. Then proceed according to 7.3.

### 7.3 Sheet making

The number of sheets shall correspond to at least 50 g of oven-dry pulp, or allow for at least 300 contraries to be counted.

NOTE The above number of contraries are arrived at by accepting a relative error of about 10 % at 90 % confidence level ([1] in the bibliography).

Prepare a number of sheets with a grammage of 80 g/m<sup>2</sup> ± 3 g/m<sup>2</sup> in the sheet-making apparatus (6.2). Place each sheet, attached to a couch blotter (test sheet up) on top of a dry blotter (6.3). Place the drying plate (6.4), with its polished side down, on top of the test sheet followed by another dry blotter ready to receive the next couch blotter

and laboratory sheet. Press the pile of sheets, blotters and plates at about  $410 \text{ kPa} \pm 10 \text{ kPa}$  for  $5 \text{ min} \pm 1 \text{ min}$ . Separate the drying plates together with the attached laboratory sheets from the blotters. Dry the sheets in the laboratory atmosphere. The laboratory sheets should remain in contact with their drying plates during the entire drying period to prevent shrinkage.

If it is necessary to protect the sheets during drying, the blotter that was in contact with the laboratory sheet may be left there until the sheet is dry.

If the sheets are made using the Rapid-Köthen procedure, follow the instructions given in ISO 5269-2, where relevant. Mark the top side of the sheet in such a way that the result of the inspection will not be influenced by the mark.

## 8 Procedure

### 8.1 General

Apply the instructions provided by the manufacturer of the device for counting of contraries (6.5).

### 8.2 Checking and adjusting the instrument

Check the instrument regularly using the comparison chart (6.6). Place the chart on a white, flat tile or sheet of paper, of minimum brightness 85 %. All the contraries in the actual classes indicated on the comparison chart shall be noted by the instrument. If this is not the case, adjust the instrument, if possible, or contact the manufacturer.

The comparison chart shall be closely inspected to determine on which side the images are printed. If a device is used having a unidirectional light source with an incident angle off the normal, the printed side of the film should be placed against the tile or sheet of paper and held flat against the inspection surface using a modest weight. This procedure will minimize any shadows.

Adjust the instrument so that the total area it records for the contraries on the comparison chart is equal to the actual area of contraries, or the total area within each class is in agreement, if this measurement is required.

### 8.3 Inspection of the laboratory sheets

Subject the laboratory sheets to inspection by the instrument. For the number of sheets prepared, take half of the sheets and inspect them from the top or glazed side. Repeat the procedure with the other half of the sheets, inspecting them from the wire side. Read the result when the instrument has inspected at least 50 g of pulp or recorded at least 300 contraries. Determine and record the total area of sheets that was inspected.

## 9 Results

### 9.1 Calculation

Calculate the total area of contraries per area of laboratory sheets using the formula:

$$X = \frac{c}{b}$$

where

- $X$  is the total area of contraries per area of laboratory sheets, expressed in square millimetres per square metre;
- $c$  is the total area of contraries as indicated by the instrument, expressed in square millimetres;
- $b$  is the area of the inspected sheets, expressed in square metres.

Report the total area of contraries to two significant figures.

NOTE By special agreement, the area or number in each class may be reported.



## 9.2 Precision

Values for repeatability and reproducibility of this International Standard cannot be stated as there is only limited experience of instruments calibrated according to a standard. Some general information is found in the literature ([2] and [3] in the bibliography).

**NOTE** The variation within a lot can be estimated by calculating the standard deviation of the measured values of total area of the contraries from the set of samples taken from the lot. The coefficient of variation is obtained from the standard deviation.

## 10 Test report

The test report shall include the following particulars:

- a) all the information necessary for complete identification of the sample;
- b) reference to this International Standard;
- c) the results, expressed in square millimetres of contraries per square metre of laboratory sheets; by special agreement, the result can be given as the area or number of contraries in different size classes;
- d) number of revolutions used for disintegration;
- e) type or description of instrument used;
- f) any unusual features observed in the course of the test;
- g) any departure from this International Standard, or any other circumstances or influences regarded as optional, that could have affected the results.

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## **Annex A**

(informative)

### **Contraries smaller than 0,04 mm<sup>2</sup>**

In some cases, it is of interest to know the amount of sub-visible specks, below 0,04 mm<sup>2</sup>, when evaluating recycled pulps. The success of such an evaluation depends on the sensitivity of the counting device as regards size and contrast. It also depends on the brightness of the sample. Consequently, no general instructions can be given as regards contrast and size classes of specks below 0,04 mm<sup>2</sup>.

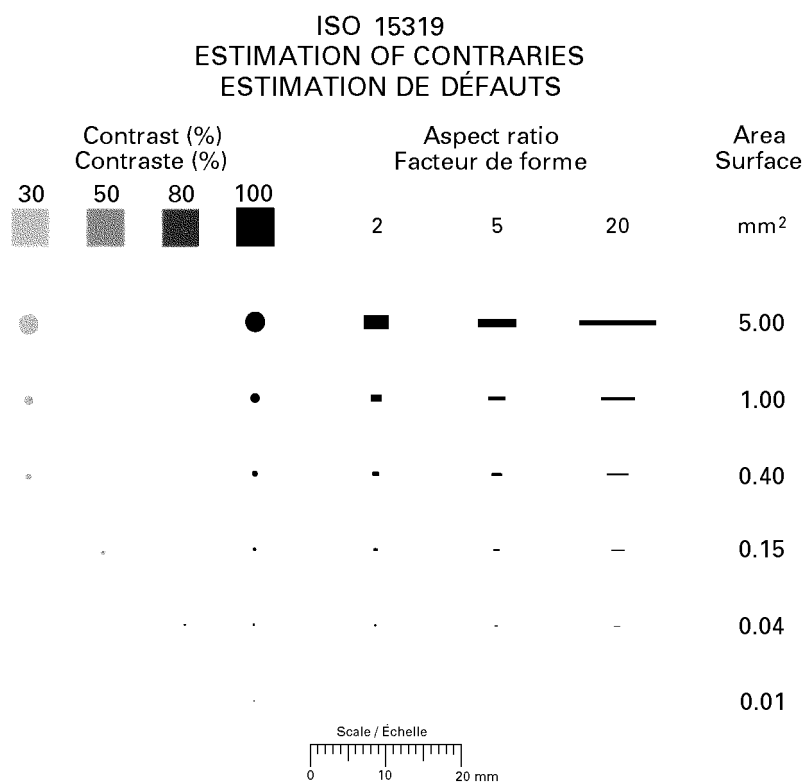
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## Annex B (normative)

### Comparison chart

This chart is identical to the chart included in ISO 15755.

Use the chart shown here.



**Figure B.1 — ISO 15319 Recycled pulp — Estimation of contraries, Comparison chart**

The spots having the minimum contrast to be considered in each size class are indicated, i.e. 30 %, 50 % and 80 % minimum contrast for contraries equal to or larger than respectively 0,4 mm<sup>2</sup>, 0,15 mm<sup>2</sup> and 0,04 mm<sup>2</sup>.

Copies of this International Standard printed by ISO include a comparison chart reproduced on transparent film. Users having only an electronic copy, or a printed copy reproduced from an electronic copy, may obtain an original transparent film comparison chart from the ISO Central Secretariat (price code: A).

## Annex C (normative)

### Contrast and density

The parameter used to measure the difference in light intensity between a foreign particle and its background is called "contrast". This is derived from the ratio of the physical intensity of the light reflected from the particle to the intensity of the light reflected from the surrounding sheet. The darker the contrary compared to its surrounds, the higher is the contrast. In this International Standard the local neighbourhood of the contrary is considered as the background.

$$C = 100 \times \left( 1 - \frac{R_p}{R_s} \right)$$

where

$C$  is the contrast, expressed as a percentage;

$R_p$  is the reflectance of the particle, expressed as a percentage;

$R_s$  is the reflectance of the surrounding area, expressed as a percentage.

The contrast needed to make a contrary visible depends on the size of the contrary; small spots are visible only when they have a sharp contrast to the surrounding pulp, whereas large spots are visible at low contrast ([4] in the bibliography).

For the purposes of this International Standard, the contrast values, which are derived from the optical intensity and density values, are formulated so that a value of zero is obtained when the intensities of the light reflected from the speck and the surrounding pulp are equal.

## Bibliography

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- [6] ISO 15755:1999, *Paper and board — Estimation of contraries*.
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- [8] ISO 5350-2:1998, *Pulp — Estimation of dirt and shives — Part 2: Inspection of mill sheeted pulp*.
- [9] ISO 5350-3:1998, *Pulp — Estimation of dirt and shives — Part 3: Inspection by reflected light*.

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