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Recycled pulps — Estimation of stickies and plastics —

Part 3:
Determination by applying near-infrared measurement

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6 Paper, board and pulps.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Tacky particles, commonly named stickies, are a major issue in paper recycling since they can affect the processes as well as the quality of the recycled products. Therefore, a variety of test methods have been developed over decades. Existing laboratory test procedures for sticky contaminants in pulps made from paper for recycling or in recycled papers require an elaborate sample preparation to increase the concentration of these sticky contaminants by screening the pulp through a slotted plate and analysing the rejects. With the test procedure and equipment based on this document, the content of sticky contaminants can be determined at a laboratory handsheet or at the paper by means of near-infrared (NIR) measurement but also on filter paper where stickies have been concentrated and deposited with existing test procedures.

The approach of the measurement specified in this document is different to the established test methods since the amount and the chemical composition of polymeric substances are determined by applying NIR measurements. The content of these substances, which are typical constituents of adhesives are assigned as stickies. This is a further major difference to the existing methods, which typically analyse the sticky behaviour, but not the chemical composition. The results determined applying the method specified in this document correlate very well with established sticky measurement techniques, e.g. ISO 15360-2[4] or INGEDE Method 4.[5] The measurement procedure according to this document also enables to simultaneously analyse polymeric substances without tacky behaviour, typically named “plastics” in other methods.

Related publications originating from the years 2012 to 2016 are referred in the bibliography. [7] and [8] correspond to “3DStick”: Screening sequence is performed as INGEDE Method 4[5] and the retained particles are deposited onto a filter with “natural drying”. The particles are then analysed by

(i) Laser triangulation to assess the morphology of each particles detected (3 dimension information)

(ii) NIR analysis on each particles to determine their chemical nature including stickies (Hot-melt, PSA, PSA hotmelt, hot-melt, PVAc and VAE based adhesives) and plastic particles (polyethylene, polystyrene, PVC, Polypropylene, PET)

The information given are then

— 3 dimensions of each particles (length, width, thickness) with their chemical nature

— Number, surface and volume per kg for each families present on the tested sample

[9] and [10] correspond to patented solution very similar to the proposed standard

Recycled pulps — Estimation of stickies and plastics —

Part 3:
Determination by applying near-infrared measurement

1 Scope

This document specifies a method for the determination of stickies and non-tacky polymeric contaminants present in pulp or paper sheets near-infrared measurement. This document is applicable to recycled pulps and papers.

Sampling of pulp and paper as well as the preparation of handsheets are not part of this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 216, *Writing paper and certain classes of printed matter — Trimmed sizes — A and B series, and indication of machine direction*

ISO 534, *Paper and board — Determination of thickness, density and specific volume*

ISO 536, *Paper and board — Determination of grammage*

ISO 4119, *Pulps — Determination of stock concentration*

ISO 5263-2, *Pulps — Laboratory wet disintegration — Part 2: Disintegration of mechanical pulps at 20 degrees C*

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 5270, *Pulps — Laboratory sheets — Determination of physical properties*

ISO 15360-1, *Recycled pulps — Estimation of Stickies and Plastics — Part 1: Visual method*

ISO 15360-2:2015, *Recycled pulps — Estimation of Stickies and Plastics — Part 2: Image analysis method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 which they touch

Note 1 to entry: 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 to entry: 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 to entry: A stickie particle can be a composite of adhesive material together with non-adhesive plastic fragments and cellulosic fibres.

Note 4 to entry: Stickies according to this document are polymeric substances, which are usually found in adhesives.

[SOURCE: ISO 15360-2:2015, term 3.1, modified — Note 4 to entry added]

3.2
non-tacky polymeric contaminant
polymeric substance which is usually not found in adhesives, excluding cellulosic materials and inorganic substances

Note 1 to entry: ISO 15360-2 defines these substances as “plastics”.

4 Principle

This document specifies the determination of the chemical composition of an air dry paper sample applying a near-infrared (NIR) analysis system. The sample to be analysed can have various origins – pulp, paper machine, pilot plant, laboratory etc. – but shall be made using recycled fibres. The surface areas of particles, which are neither natural fibres nor inorganic substances are determined. The measurement differentiates between stickies or non-tacky polymeric contaminants according to their chemical nature.

5 Apparatus (near-infrared analysis system)

5.1 Hardware

The measuring device is a hyperspectral NIR imaging system. The illumination device, optics and sensor shall cover at least the spectral range from 1 400 nm to 1 800 nm. The optical system has a lateral resolution of at least 127 DPI (i. e. object pixel size ≤ 200 µm). The optical system may be of the wide-field type, or of the point-, line- or wavelength-scanning type.

Annex A details the characteristics of such compatible hyperspectral NIR systems.

It is recommended that the system allows analysing paper sheets of ISO A4 according to ISO 216 format or larger.

5.2 Software

The required software is a NIR evaluation system for qualitative and quantitative assessment of polymeric substances by size and number. The software shall consist of an NIR imaging algorithm, including chemometrics and classification.

For examples, see Annex B.

5.3 Penetration test reference piece

A film or plate with an approximate size of ISO A4 according to ISO 216 and a maximum thickness of 300 µm consisting of at least one polymeric substance, which is assigned to the sticky or non-tacky polymeric contaminants group (see 8.1).

5.4 Calibration of the NIR sensor

Calibration shall be done according to the manual of the respective device. The calibration shall be checked according Annex A.

6 Preparation of specimen

6.1 General

In the case of determination of plastics and stickies deposited on filter paper, the measurement can be performed directly, without the need of the penetration test defined in clause 7.4.

In the case of determination on paper sample, the NIR signal can be submitted through a limited specimen thickness only. For pulp or paper sheets, this specimen thickness is depending on the fibre type, the degree of densification, the filler content and the like. Therefore, a penetration test shall be performed to determine whether the thickness of the specimen is below the maximum allowable specimen thickness δ_{max} (see 7.4).

6.2 Pulp samples

6.2.1 Measurement of stickies and non-tacky contaminants deposited on filter

The stickies and plastics shall be isolated in accordance to ISO 15360-1 or ISO 15360-2 and deposited on filter paper before air drying. The oven dry mass of pulp used to concentrate the contaminants shall be determined.

6.2.2 Measurement of stickies and non-tacky contaminants directly on paper or handsheets

Determine the oven-dry mass of the stock used for preparing the handsheets according to ISO 4119.

Prepare handsheets having a thickness of maximum δ_{max} according to ISO 534 by using a sheet former according to ISO 5269-1 or ISO 5269-2. The cumulative area of multiple handsheets shall be a minimum of 0,1 m^2 and a maximum of 0,5 m^2. Within these limits, prepare enough handsheets to be able to measure 50 stickies respective non-tacky polymeric contaminants or more, whatever the interesting group of substances is.

EXAMPLE If using a Rapid-Köthen sheet former according to ISO 5269-2, usually four handsheets are prepared.

The polymeric substances, which have a detectable size for the measurement, but are not retained in the handsheet are negligible.

If the penetration test cannot be performed in advance (see 7.4), and therefore δ_{max} is unknown it is recommended to prepare handsheet samples having a thickness of max. 100 µm for recycled pulps.

When preparing several handsheets from the same pulp sample, the weight of the suspension used for the handsheet forming should be the same for each handsheet.

Determine the oven-dry grammage of the handsheets according to ISO 5270.

When material balances for stickies, or the sticky separation efficiency of stock preparation equipment respectively a sub-system are determined, the grammage of individual handsheets made from stock

samples taken at different positions within the process should not vary more than ±10 % from their average grammage.

6.3 Paper samples

Determine the oven-dry grammage of the paper samples according to ISO 536. The cumulative area of multiple paper samples shall be a minimum of 0,1 m^2 and a maximum of 0,5 m^2. Within these limits, prepare enough paper sheets to be able to measure 50 stickies respective non-tacky polymeric contaminants or more, whatever the interesting group of substances is. The paper samples can be cut to paper sheets, suitable for the measurements.

EXAMPLE If the format of the paper sheets was chosen as ISO A4 according to ISO 216 format, two ISO A4 sheets need to be cut from the paper sample.

To determine the sticky value in mm^2/kg when analyzing paper samples, the following cases shall be distinguished:

— If the paper sheet thickness is $\leq \delta_{max}$, place the paper sheets under the NIR sensor and perform the NIR analysis;

— If the paper sheet thickness is > δ_{max}, pulp the paper samples on laboratory scale according to ISO 5263-2, and prepare handsheets as specified in 6.2. The procedure to be followed is the same as specified for pulp samples.

NOTE The pulping can cause size reduction of the particles [stickies (3.1), non-tacky polymeric contaminants (3.2)] thus effecting the detectable amount and the size distribution.

7 Procedure

7.1 Parameters for the analysis

Define the size classes for the particles and select the available substances.

Size classes in circle equivalent diameter according to INGEDE Method 4[5] see Table 1.

Table 1 — Size classes in circle equivalent diameter

Dimensions in µm

Size classes	Lower limit	Upper limit
K1	100	200
K2	201	300
K3	301	400
K4	401	500
K5	501	600
K6	601	1 000
K7	1 001	1 500
K8	1 501	2 000
K9	2 001	3 000
K10	3 001	5 000
K11	5 001	10 000
K12	10 001	20 000
K13	20 001	50 000
K14	50 001	200 000

7.2 Placement of specimen

Place the specimen under the NIR sensor and fix it. When analysing laboratory handsheets, place them with the wire side facing up.

If analysing paper sheets $\leq \delta_{max}$, it is recommended that the side of the paper sample having the first contact with a heated drying cylinder is facing up. Use the same side facing up for all paper sheets of one paper sample. When several paper samples from the same paper machine are measured, ensure that always the same side of the paper samples are measured applying the NIR analysis.

If the paper sheet thickness is > δ_{max} the NIR measurements can be performed for only one side facing up, preferably the paper side having the first contact with a heated drying cylinder, and the specific sticky value can be determined related on the total surface area of the sample.

7.3 Measurement

After placing the specimen under the NIR sensor start the measurement. The specimen shall be scanned in individual stripes and the individual scans are assembled if using a line scan sensor. If using a matrix sensor, the entire sheet may either be analysed by one shot or by individual rectangular scans which are assembled. If using a single spot sensor, the entire sheet is analysed by 2D point scanning.

7.4 Penetration test

If necessary (according to clause 6.1), perform a penetration test by placing the penetration test reference piece under a specimen and carry out the NIR measurement. If the polymeric substances covered by the specimen can be detected by the NIR measurement, the specimen's thickness is $\leq \delta_{max}$. This is fulfilled if the covered area of polymeric substances detected is 90 % or more of the specimen's area. If the polymeric substances covered by the specimen cannot be detected by the NIR measurement, the specimen's thickness is > δ_{max}. In that case, prepare samples with lower thickness and repeat the penetration test.

8 Analysis

8.1 Determination of stickies and non-tacky polymeric contaminants

The planar geometrical properties of particles, which are neither natural fibres nor inorganic substances are determined by measuring their NIR spectral response and by comparing these responses with those of known chemical substances, which are summarized in a data base.

The assignment to the stickies and non-tacky contaminant groups can be done as follows (non-exhaustive):

a) stickies can be classified based on their origin:

   — Hot-melt glue (EVA based);

   — Pressure Sensitive Hot melt adhesive (PSA-Hotmelt);

   — Pressure Sensitive Adhesive (PSA);

   — Polyvinyl acetate glue (PVAc);

   — Copolymer Vinyl Acetate Ethylene based glue (PVAE).

b) non-tacky polymeric contaminants:

   — Polyethylene (PE);

   — Polystyrene (PS);

— Polyvinyl chloride (PVC);

— Polypropylene (PP);

— Polyethylene terephthalate (PET)

c) unknown and other polymeric contaminants.

The geometrical properties feature the following information:

1) surface area of the sticky and non-tacky contaminant particles;

2) size of particles as circle equivalent diameter;

3) length and width for particles having a circle equivalent diameter > 1 000 µm.

For every polymeric particle, which is detected, the information summarized above is stored individually, allowing comprehensive evaluation and clustering of results including the introduction of size classes.

NOTE A polymeric substance, which does not give any reflection in the NIR wavelength range, cannot be detected. For example, if a non-tacky particle is deeply dyed in black.

The software classifies the measured stickies and non-tacky polymeric contaminants as absolute values according to the following parameters:

a) number of stickies and non-tacky polymeric contaminants per size class and/or clustered for the chemical properties;

b) total area of stickies and non-tacky polymeric contaminants per size class and/or clustered for the chemical properties;

c) logarithmic area density per size class (calculation according to[6]) and/or clustered for the chemical properties.

The software classifies the measured stickies and non-tacky polymeric contaminants as specific values. For this purpose, the following steps shall be performed:

a) The total surface area of all specimens analysed by the NIR for one sample needs to be determined.

NOTE For Rapid-Köthen handsheets according to ISO 5269-2, the surface area of one individual handsheet is 314 cm^2 ± 2 cm^2.

b) Determining specific values for pulp samples, the absolute values of stickies and non-tacky polymeric contaminants are divided by the total oven-dry mass of the stock used for preparing the handsheets.

c) Determining specific values for paper samples, the absolute values of stickies and non-tacky polymeric contaminants are divided by the product of the surface area analyzed by the NIR measurements and the bone-dry grammage of the paper samples.

8.2 Calculation

8.2.1 Pulp samples

Applying this procedure, the following specific values for stickies and non-tacky polymeric contaminants are determined:

a) specific number of stickies and non-tacky polymeric contaminants per kg of pulp per size class:

$$Y_{i,\text{pulp}} = \frac{a_i}{m_{\text{pulp}}} \quad (1)$$

where

i is an indicator for the size classes K1 to K14;

$Y_{i,\text{pulp}}$ is the number of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry pulp of each size class;

a_i is the observed number of stickies or non-tacky polymeric contaminants, as relevant, of each size class;

m_{pulp} is the oven-dry mass of the pulp, used for handsheet preparation, in kg.

b) total specific number of stickies and non-tacky polymeric contaminants per kg of pulp:

$$Y_{\text{total,pulp}} = \sum_{i=\text{K1}}^{\text{K14}} Y_{i,\text{pulp}} \quad (2)$$

where

i is an indicator for the size classes K1 to K14;

$Y_{i,\text{pulp}}$ is the number of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry pulp of each size class;

$Y_{\text{total,pulp}}$ is the total number of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry pulp.

c) specific area of stickies and non-tacky polymeric contaminants per kg of pulp per size class:

$$X_{i,\text{pulp}} = \frac{A_i}{m_{\text{pulp}}} \quad (3)$$

where

i is an indicator for the size classes K1 to K14;

$X_{i,\text{pulp}}$ is the area of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry pulp of each size class;

A_i is the area of stickies or non-tacky polymeric contaminants, as relevant, of each size class;

m_{pulp} is the oven-dry mass of the pulp, used for handsheet preparation, in kg.

d) total specific area of stickies and non-tacky polymeric contaminants, per kg of pulp:

$$X_{\text{total,pulp}} = \sum_{i=\text{K1}}^{\text{K14}} X_{i,\text{pulp}} \quad (4)$$

where

i is an indicator for the size classes K1 to K14;

$X_{i,\text{pulp}}$ is the area of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry pulp of each size class;

$X_{\text{total, pulp}}$ is the total area of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry pulp.

8.2.2 Paper samples

Applying this procedure, the following specific values for stickies and non-tacky polymeric contaminants are determined:

a) specific number of stickies and non-tacky polymeric contaminants per kg of paper per size class:

$$m_{\text{paper}} = \frac{M \times g_{\text{paper}}}{1\,000} \tag{5}$$

where

m_{paper} is the oven-dry mass of the paper samples, in kg;

M is total measured surface area of the paper samples, in m^2;

g_{paper} the oven-dry grammage of the paper samples, in g per m^2.

$$Y_{i\text{, paper}} = \frac{a_i}{m_{\text{paper}}} \tag{6}$$

where

i is an indicator for the size classes K1 to K14;

$Y_{i\text{, paper}}$ is the number of stickies or non-tacky polymeric contaminants, as relevant, per kg of the paper samples of each size class;

a_i is the observed number of stickies or non-tacky polymeric contaminants, as relevant, of each size class.

b) total specific number of stickies and non-tacky polymeric contaminants per kg of paper:

$$Y_{\text{total, paper}} = \sum_{i=\text{K1}}^{\text{K14}} Y_{i\text{, paper}} \tag{7}$$

where

i is an indicator for the size classes K1 to K14;

$Y_{i\text{, paper}}$ is the number of stickies or non-tacky polymeric contaminants, as relevant, per kg of the paper samples of each size class;

$Y_{\text{total, paper}}$ is the total number of stickies or non-tacky polymeric contaminants, as relevant, per kg of oven-dry paper.

c) specific area of stickies and non-tacky polymeric contaminants per kg of paper per size class:

$$X_{i\text{, paper}} = \frac{A_i}{m_{\text{paper}}} \tag{8}$$

where

i is an indicator for the size classes K1 to K14;

$X_{i,\,\mathrm{paper}}$ is the area of stickies or non-tacky polymeric contaminants, as relevant, per kg of paper of each size class;

A_i is the area of stickies or non-tacky polymeric contaminants, as relevant, of each size class;

m_{paper} is the oven-dry mass of the paper samples, in kg [see Formula (5)].

d) total specific area of stickies and non-tacky polymeric contaminants, per kg of paper:

$$X_{\mathrm{total,\,paper}} = \sum_{i=\mathrm{K1}}^{\mathrm{K14}} X_{i,\,\mathrm{paper}} \tag{9}$$

where

i is an indicator for the size classes K1 to K14;

$X_{i,\,\mathrm{paper}}$ is the area of stickies or non-tacky polymeric contaminants, as relevant, per kg of paper of each size class;

$X_{\mathrm{total,\,paper}}$ is the total area of stickies or non-tacky polymeric contaminants, as relevant, per kg of paper.

9 Test report

The test report shall include at least the following information:

a) reference to this document;

b) identification of the sample;

c) oven-dry mass of stock used for preparing the handsheets (for pulp samples);

d) oven-dry mass of paper which is measured (for paper samples);

e) grammage of the test specimen, see 6.2 and 6.3;

f) thickness of the test specimen (optional), see 6.2 and 6.3;

g) result of the penetration test (if required), see 7.4;

h) specific number of stickies and non-tacky polymeric contaminants per kg of pulp respective paper (per size class and as total), see 8.2.1, Formula (1), Formula (2) and see 8.2.2, Formula (6), Formula (7);

i) specific area of stickies and non-tacky polymeric contaminants per kg of pulp respective paper (per size class and as total), see 8.2.1, Formula (3), Formula (4) and see 8.2.2, Formula (8), Formula (9);

j) any deviations from the procedure;

k) any unusual observations that may have affected the test results;

l) the date of the test.

Annex A
(normative)

Calibration check

A.1 General

The calibration shall be checked using reference sheets (A.2). They contain pieces of materials with which the identification of polymers as well as the accuracy of the measured areas can be checked.

A.2 Reference Sheets

The sheets consist of uncoated, wood free paper of 160 g/m^2 to 250 g/m^2. Two sheets are necessary: reference material of plastics and stickies (qualitative assessment (see A.2.1)) and reference material in different sizes and shapes (quantitative assessment (see A.2.2)).

A.2.1 Qualitative Assessment

Macroscopic pieces of the stickies and non-tacky polymeric contaminants (see 8.1) are attached to the paper sheet. The size shall be of 0,5 cm to 1 cm in diameter. The pieces shall be non-transparent and of flat surface. Reference method shall be IR spectroscopy.

A.2.2 Quantitative Assessment

Polymeric objects of different sizes and shapes are printed on the paper sheet. The printer shall use wax colours. The objects are at least: quadratic, rectangular, round and elliptic with a ratio of longer to shorter width of 1:1, 2:1 and 3:1. All objects shall have a representative in the middle of each size class (see 7.1). Reference method shall be visual assessment with a resolution of at least 1 200 dpi.

Annex B
(informative)

Example for measuring devices

B.1 PTS DOMASmultispec[1)]

NIR line or matrix camera with a wavelength range from 1 320 nm to 1 900 nm, lateral resolution of 200 µm × 200 µm or a higher resolution, illumination from one side using three times 230 W Halogen lamps with colour temperature 2 900 K, rated luminous flux 5 000 lm. Tolerances of these values within the usual manufacturing tolerances are permitted.

B.2 Techpap 3DStick[2)]

It is a fibre optic NIR hyperspectral system. The measurement point size is 200 µm in diameter and the image is obtained by a 2D point scan of the measurement head. The sensor is a NIR grating spectrometer covering the wavelength range 1 200 to 2 200 nm, with a linear array of 256 channels and a spectral resolution of 4 nm. Illumination is provided by filament emitters with a continuous black-body spectrum. Parabolic reflectors ensure uniformity of illumination. In addition, the system is equipped with a laser triangulation device, in order to measure the topography of the sample simultaneously with the NIR image and estimate the 3D volume of the contaminants (stickies, plastics …).

1) DOMASmultispec is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

2) 3DStick is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

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