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T 568

BALLOT NO. 2 SARG

DRAFT NO. 01

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WORKING GROUP
CHAIR To be Determined

SUBJECT
CATEGORY Pulp Properties

RELATED
METHODS See "Additional Information"

Physical area of sub-visible contraries in pulp, paper, and paperboard by image analysis

(Five-year review of Official Method T 568 om-18)

1. Introduction

The level of sub-visible contraries, such as microscopic ink particles, present in pulp, paper or paperboard can impact its usefulness in a specific end-use application. For someone controlling or monitoring the de-inking process, the absolute physical area of ink coverage, or the number of ink specks present in an inspection area may be of greatest importance.

2. Scope

2.1 This method uses image analysis to determine the level of sub-visible contraries in pulp, recycled pulp, paper, and paperboard in terms of Equivalent Physical Diameter (EPD) of contraries within the EPD range of 8 micrometers to 160 micrometers, reported in parts per hundred as well as the number of specks per square centimeter of sample. Extension to other speck sizes (for example those greater than 160 micrometers), may require changes in equipment, calculation procedures, or both, and is not covered in this test method.

2.2 Paper and board surfaces are far from uniform below 100 micrometers (*1*). To obtain the truest physical size distribution of the sub-visible contraries requires the specific illumination and optics described in this method.

2.3 The specimen to be evaluated must have a brightness, as determined by TAPPI T 452 "Brightness of Pulp, Paper, and Paperboard (Directional Reflectance at 457 nm)," of approximately 20% or greater. It may be necessary to reform some pulp sheets into handsheets if the surface is too rough or textured to obtain a sharp image over the entire viewing area. Reforming the sheet may result in the loss of some ink particles.

2.4 Specks larger than 160 micrometers, and of sufficient contrast, are considered visible and should be measured according to TAPPI T 563 "Equivalent Black Area (EBA) and count of visible dirt in pulp, paper and paperboard by image analysis".

3. Significance

3.1 Cleanliness and shade are two appearance attributes influencing the marketability of recycled paper. Any particles of residual ink too small to be seen with the naked eye could still influence the shade of the sheet. In principle, one might want to control the deinking process completely from these two attributes; however, greater insight can be obtained from the direct measurement of changes in the particle-size histogram (*2, 3*).

3.2 The measurement of sub-visible residual ink specks differs in several ways from the measurement of cleanliness with a standard dirt counter as described in T 563. The standard dirt count incorporates the visual impact of different colored specks with the calculation of equivalent black area, EBA. The physical size of residual (sub-visible) ink specks, rather than their psychophysical impact, is of interest. Secondly, visible dirt counting is performed at such low magnification that the reflectance variation associated with formation requires a special center surround filter described in T 563. This surround filter is not required at sub-visible dimensions when employing the optics described in the apparatus section of this method.

4. Related documents

TAPPI T 452 Brightness of pulp, paper and paperboard (Directional reflectance at 457 nm)

TAPPI T 563 Equivalent Black Area (EBA) and count of visible dirt in pulp, paper and paperboard by image analysis

5. Definitions

5.1 *Sub-Visible Contraries* - any foreign matter embedded in or on the sheet, which, when examined by reflected light has a darker contrast relative to the sheet surface and has an equivalent physical diameter of 8 micrometers or greater as determined by this test method.

5.2 *The Equivalent Physical Diameter (EPD)* - the shape of ink specks, and other sub-visible contraries, is irregular. The EPD is the diameter of a circle equivalent in area to the area of the detected speck. The EPD of a speck is calculated from the total area of all adjacent and contiguous pixel areas below the detection threshold. The EPD is calculated according to:

$$\text{EPD} = 2\ (\text{speck area}/\pi)^{1/2}$$

5.3 *Gray Level* - Image analyzer detectors digitize the signal of each picture element (pixel) into discrete values called gray levels. The upper and lower limits of the gray level scale can be adjusted to arbitrary settings to achieve calibration to established reflectance scales. The gray level scales are frequently calibrated against an absolute scale such as the percent reflectance at specific wavelengths.

5.4 *Detector Integration Time* - the amount of time, in milliseconds, for the image detected by a CCD sensor to reach a mean gray level (GL) of 180.

5.5 *Percent Coverage* - is the summed area of all the detected specks, expressed in mm^2, taken as a ratio over the examined area normalized to 1 cm^2 and multiplied by 100%. Percent coverage can also be expressed as parts per hundred (PPH).

6. Apparatus

6.1 *Description of the apparatus:*

6.1.1 *Detector:* The detector consists of a CCD sensor matrix, optics, illuminant and specimen stage. The sensor matrix is densitometric with at least 256 gray levels (G.L.) of sensitivity with the physical pixel resolution having an effective diameter of 5 µm or less (see Note 1). The detector must view the specimen perpendicular to its surface. The illumination is diffuse, un-polarized and is concentrated in the portion of the spectrum such that 95% of the spectrum will be between 650 nm and 710 nm. The illumination must be uniform such that the illumination at the specimen stage, before any software corrections, must be within ± 3% coefficient of variation (COV). The specimen stage must be shielded to prevent influence from ambient light, be matte, and have a reflectance exceeding 90% between 380 nm and 750 nm.

NOTE 1: Imaging precision improves with resolution. Therefore it is advisable to have as high a pixel resolution as is practical for the smallest dirt specks. However, the pixel resolution must not be less than 1.5 µm (i.e. twice the peak wavelength of the illuminant). The practical limit of resolution will be limited by the quality of the near IR optics used. Frequently, this is several times greater than the wavelength of illumination.

6.1.2 *Analyzer:* An analyser incorporating the EPD calculations and using a specified thresholding technique (3, Appendix A.1).

6.1.3 *The calibration plates* must be matte, blemish free, nonfluorescent at the wavelengths of measurement, and have at least one solid white area and one solid black area. Additionally the calibration plates must meet the specifications in Appendix A.2 and the white plate must have a reflectance greater than 80% at 660 nm.

6.2 *Calibration:* Turn on the apparatus and light source. Allow the equipment to warm up and adjust the hardware settings according to the manufacturer's instructions or recommendations.

6.2.1 *Contrast calibration:*

6.2.1.1 *Zero check:* In the absence of light the sensor must take more than 200 ms to reach a GL of 150 (i.e. the dark current drift of the CCD sensor). Typically a GL of 50 is reached after 200 ms of integration. If after 200 ms, the check value exceeds 150 GL, then the sensor must be serviced or replaced.

6.2.1.2 *Span check:* Place the calibration plate in the field of view. The check value of the plate is determined according to the formula in the Appendix A.1.3.1.

6.2.1.3 *Calibration:* Place the calibration plate with the greatest contrast flat on the stage. If a device is used to hold a sample flat on the stage, then this same device must be used to hold the calibration plate flat.

6.3 *Maintenance:* Follow the manufacturer's instructions regarding the equipment maintenance. It is important that the specimen stage remain above 90% reflectance at 660 nm.

6.3.1 Store the calibration plates in the dark or in a black plastic envelope to prevent discoloration and loss of contrast. The calibration plates are cleaned DRY to remove dust or lint. Avoid scratching the surface as this may cause the plates to be out of specification. Periodically measure the %-reflectance, at 660 nm, of the white square. If the calibration plates fall outside the specifications given in Appendix A.2, then they must be replaced.

6.3.2 Unless the ambient light changes, or the hardware settings have changed, it is not necessary to recalibrate the hardware for a particular analysis. It should be noted that, with time, light sources age and hence, there may be a loss of intensity. Thus, full calibration checks and hardware adjustments must be made as frequently as specified by the manufacturer.

7. Sampling and test specimens

7.1 Sample the paper or paperboard in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Fibreboard, or Related Product."

7.2 *Paper and Paperboard:* From each test unit, select five or more sheets having a total surface area (both sides) of at least 1 m^2. A minimum of 5 cm^2 will be examined by the image analysis apparatus of this method.

NOTE 2: There may be instances where less than 5 cm^2 is examined. This may be acceptable when the quantity of ink specks in the paper or paperboard exceeds the minimum required to reach a chosen level of counting precision (see section 8.3) and the sample is representative of the manufacturing process.

7.3 *Pulp*

7.3.1 *Pulp Sheets:* From each test unit, select ten or more sheets having a total exposed area (both sides) of at least 10 m^2 (see Note 2). Keep the specimen sheets clean between two outer extra sheets.

7.3.1.1 Some pulp sheets contain deep corrugations which may cast shadows or prevent consistent detection of the dirt specks at some locations in the sheet. Reform such pulp sheets into handsheets for examination.

7.3.2 *Slush or flash dried pulp:* Take a sample and form into specimen sheets in a carefully cleaned stainless steel sheet machine according to TAPPI T 272 "Forming Handsheets for Reflectance Testing of Pulp (Sheet Machine Procedure)." Make a sufficient number of sheets so that they have a total exposed area (both sides) of at least 4000

cm^2. Restrain dry the sheets taking care to avoid contamination and wrinkling.

8. Procedure

8.1 Carefully brush away any loose surface dirt specks. Avoid measuring specimens which contain smudge marks and/or wrinkles as these could dramatically affect the results.

8.2 A minimum of 5 cm^2 are be examined by the image analysis apparatus of this method. Typically, the field of view is 0.1 cm^2. Consequently, 50 fields should be measured to reach a total of 5 cm^2. Take these 50 fields uniformly over the total surface area of 1 m^2. Follow the measurement procedure outlined in the instruction manual provided by the instrument manufacturer. Take care to ensure that the specimen is held flat enough so that all dirt specks in the field of view are in focus (see 7.3.1.1).

8.3 Choose a consistent target of counting precision (% uncertainty) and measure enough paper surface to reach that precision. By the nature of random sampling of contraries, if one sheet of paper is found to contain N specks, then replicate sheets of paper from the same lot should be found to contain $N \pm \sqrt{N}$ specks two times out of three. For example, if a certain area of paper is found to contain 100 specks, another region with the same area should have 100 ± 10 specks, for a counting precision of 10%. If another specimen only contains 25 specks, then replicate areas should contain 25 ± 5 specks for a counting precision of 20%. Therefore, to reach a consistent target of 10% in the count, measure as much paper surface as needed to reach a minimum count of 100 specks.

8.4 The precision of percent coverage (PPH) is more complicated to estimate than the counting precision of the specks count, because the total count is inflated by many small specks that contribute relatively little to the total PPH. To reach a target precision on PPH, count at least twice as many specks as would be needed to reach the same precision in the particle count. For example, one would count 200 dirt specks to reach a 10% precision on PPH.

9. Report

9.1 Report the total percent coverage as parts per hundred (Note 3).

9.2 Report the total speck count per square centimeter.

9.3 Report the % uncertainty, *P*, given by:

$$P = \frac{100\%}{\sqrt{N}}$$

Where: *N* is the total accumulated dirt count.

NOTE 3: The presence on many large specks may distort the percent coverage result if an insufficient number of fields are measured.

9.4 If the samples are pulp, report if the test was made on brightness pads or handsheets.

9.5 Report any deviations from this method.

9.6 If a minimum size other than 8 micrometers has been used it must be reported. Different upper or lower size limits will give different results for 9.1, 9.2 and 9.3.

10. Precision

10.1 See Tables 1 and 2.

10.2 The estimates of repeatability and reproducibility are based on data obtained from an inter-laboratory trial using TAPPI T 1200 and include test results from three instruments on two different types of samples. Laboratories were asked to calibrate the instrument prior to performing the tests. A summary of the results is displayed in Tables 1 and 2. The repeatability and reproducibility reported in the tables are estimates to the maximum differences, which should be expected 95% of the time or in 19 out of 20 instances when comparing two test results for materials similar to those described in this study under similar test conditions.

Table 1: Precision statement results for deink pulp (DIP) handsheets made from pulp after the primary cleaner. The average %-Uncertainty for these measurements was P = 1.2 %

Deinked Pulp (DIP) Handsheets			
	Grand mean	*Repeatability (%r)*	*Reproducibility (%R)*

PPH	1.41	12.5	21.2
#/cm^2	5518	7.8	31.4

Table 2: Precision statement results for Newsprint (with 10% DIP). The average %-Uncertainty for these measurements was P = 2.7 %

Newsprint (10% DIP)			
	Grand mean	*Repeatability (%r)*	*Reproducibility (%R)*
PPH	0.10	10.7	48.3
#/cm^2	377	11.0	21.2

11. Keywords

Brightness, Dirt, Dirt count, Image analysis, Paper, Paperboard, Pulp

12. Additional information

12.1 Effective date of issue: To be Assigned

12.2 In the 2007 revision, the reproducibility was included in the precision statement. The 2012 revision included only editorial changes.

Literature cited

1. Jordan, B.D., Nguyen, N.G., Trepanier, R.J., "Measuring the Particle-Size Distribution of Residual Ink in Recycled Paper," TAPPI J., 76(10), 110-116 (1993)
2. Trepanier, R.J., "Novel High Resolution Image Analysis for Measuring Residual Ink in Pulp, Paper and Board," Pulp & Paper Canada, 95(12): T547-T549(1994)
3. Trepanier, R.J., Jordan, B.D., Nguyen, N.G., Patschka, H., "High Magnification Image Analysis with Novel Background Reflectance Technique for Measuring Residual Ink in Sheets," J. Pulp & Paper Sci., 23(3): J129(1997)

References

Leighton, W.G., Miranda, J.F., "Residual Ink Size Distribution via Image Analysis," Proceedings of the TAPPI 1992 Workshop on Image Analysis, Cincinnati, OH, April 1992.

McNabb, C.A., "Monitoring Recycled Deinking Process Performance Through Image Analysis," Proceedings of TAPPI P&PQ Conference, Atlanta, GA, November 1993

Appendix A.1

A.1.1 *Detector Integration Time.* The measurement sensitivity is automatically adjusted for maximum signal to noise ratio between the ink specks and the background (i.e. paper). A maximum signal to noise ratio is obtained by integrating (exposing) the CCD detector to the field of view until a mean image gray level of 180 is obtained. At this point there is a distribution of gray levels, which includes both the ink specks and the background paper, with a distribution mean of 180 GL.

A brighter sample will take less time to reach 180 GL relative to less bright samples. In both instances comparable, and high signal to noise ratios, are achieved. Integration times are dependent on the charging rate of the CCD detector. These rates vary between detectors. Integration times of about 25 ms for xerographic paper (approx. 85% brightness) and about 80 ms for linerboard (approx. 40% brightness) are typical.

A.1.2 *The contrast threshold.* The apparatus detects specks above an effective diameter of 8 µm. The pixels, corresponding to ink specks, are detected if their gray levels are lower (darker) than the detection threshold. The threshold is set to be more than 2-sigma below the distribution mean of 180 GL, as obtained in A.1.1. For example: If the area of paper which has been imaged has pixel gray level distribution of 180 ± 25 GL, then adjacent pixels below

130 GL would be detected as dirt specks.

A.1.3 *The calibration check span process.*

A.1.3.1 The instrument must be calibrated such that plates of known reflectance, %*R* (660 nm), can be used to verify the performance of the instrument. The relationship between the %*R* (660 nm) and the detector integration time to reach a GL of 180 must be a straight line.

$$\%R\ (660\ \text{nm}) = m\ (1/T) + d$$

where:

d = instrumental offset (i.e. sensor dark level current, etc...) GL at $(1/T) = 0$
m = instrumental exposure rate (%R-ms)
%*R* (660 nm) = measured gray level (GL)
T = exposure time (ms) to reach an average GL of 180 for specific %*R*

The slope, *m*, and zero intercept, *d*, is unique to the sensor and optics of the individual instrument (3) and is determined during manufacturing.

A.1.3.2 Black and white plates of known %*R* (660 nm) are measured on the instrument. If the instrument is functioning well, then the integration time required to reach 180 GL must be within ± 2.5% of the values calculated from the calibration equation in A.1.3.1.

If the plates are outside the ± 2.5% tolerance, but within ± 10%, it should be possible to recalibrate the instrument and determine new *m* and *d* values. If the plates are outside ± 10%, then contact the manufacturer since the replacement of the illuminant, optics and/or the detector may be necessary.

Appendix A.2

A.2.1 *The calibration plates.* The reflectance, at 660 nm, is 5 ± 0.5% for the black plate and 85 ± 1% for the white, a difference of about 80%. These plates should have a low fluorescence. However, fluorescence is not a major concern with the illumination used in this method. The calibration plates used for the present method may each have their own assigned value of reflectance at 660 nm.

A.2.2 *The calibration specifications:* The black and white plates calibration standards of T563 should be capable of serving as calibration standards for this method. Measuring %-reflectance, at 660 nm, as described in TAPPI T 442 “Spectral reflectance factor, transmittance, and color of paper and pulp (polychromatic illumination)” is required.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.

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