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T 567

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WORKING GROUP CHAIRMAN N/A

SUBJECT CATEGORY Optical Properties

RELATED METHODS See "Additional Information"

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Determination of effective residual ink concentration (ERIC) by infrared reflectance measurement

(Five-year review of Official Method T 567 om-15)

(no changes from Draft 1; editorial corrections incorporated)

1. Scope

This method provides a means for determining the Effective Residual Ink Concentration (ERIC) in deinked pulp and paper made from recycled feedstock. The presence of ink influences the brightness and color of recycled paper. Trace amounts of residual ink can leave recycled paper darker and grayer than paper made from virgin pulp, however, deliberate bleaching or incidental bleaching by deinking chemicals can recover some brightness loss if most of the ink has been removed. Counteracting the tinting power of residual ink can be easier if one can monitor the effective concentration of the ink. Brightness is not only affected by the presence of ink but also by other absorbers of visible wavelengths of light such as lignin and dye. For this reason brightness has been found to be an ineffective way to monitor the deinking process. The ERIC method employs reflectance measurements in the

infrared region of the spectrum where the absorption coefficient for the ink is several orders of magnitude greater than the absorption coefficient for the fiber and other components, resulting in a sensitive means for determining the concentration of ink *(1).* The ERIC measurement is dependent on the distribution of ink particle sizes and is most effective for submicron particles *(2).*

2. Summary

The function defined by Kubelka and Munk as *absorption coefficient* may be determined from reflectance measurements taken on ink alone and on recycled paper which contains ink. If the ink in a sheet of paper is of a type that has an absorption coefficient of 10,000 m^2/kg *(4),* and if the residual ink in the sheet has increased the sheet's absorption coefficient by 1 m^2/ kg, then the "effective concentration" of the residual ink can be estimated at 1/10,000 or 100 ppm (parts per million). The term *effective concentration* is used because the ERIC value is relative rather than absolute. The absorption coefficient, as determined by this method, depends strongly upon the kind of ink, the particle size of the ink and the dispersion or agglomeration of the ink.

NOTE 1: Highly dispersed (or deagglomerated) ink provides more surface area for light absorption than an equal concentration of agglomerated ink. Visual assessments of recycled paper brightness are also affected more by a large number of deagglomerated ink specks than by a few agglomerated specks. For this reason, the ERIC method provides better correlation with visual assessments than other methods, e.g. image analysis of visible specs as described in TAPPI T 563 "Equivalent Black Area (EBA) and Count of Visible Dirt in Pulp, Paper and Paperboard by Image Analysis."

3. Significance

The ERIC method converts near infrared reflectance (NIR) measurements to light absorption which is related to ink concentration. The infrared area of the spectrum is chosen for these measurements because in this area of the spectrum, ink (not dye, lignin or other colorants) is the predominant absorber of infrared light *(1).* Once the absorption coefficient of the residual ink is known, an effective residual ink concentration value may be determined. This value can be used as a control parameter, to assist in the deinking/decolorization process, or as an end product specification. The estimation of residual ink by optical measurements in the infrared area is more accurate and much less time consuming for submicron ink particles than determination by image analysis methods *(4)*. Also image analyzers typically respond to ink particle sizes of 5 microns and above whereas the ERIC method is sensitive to the smallest particles below 10 microns. This follows from the exponentially increasing number of particles occurring at diminishing sizes.

4. Definitions

4.1 *Diffuse reflectance factor.* The ratio of the radiance of the specimen to that of a perfectly reflecting diffuser, each being irradiated hemispherically and viewed identically.

4.2 *Absorption coefficient.* The fraction of light incident upon an infinitesimally thin layer of material that is absorbed (converted to heat or other form of energy) divided by the (infinitesimal) grammage of the layer. The absorption coefficient (*k*) is expressed in reciprocal grammage units. A multiplier (1,000) is used to convert m^2/g to the standard units, m^2/kg.

4.3 *Scattering coefficient.* The fraction of light incident upon an infinitesimally thin layer of material that is scattered backwards by that layer, divided by the (infinitesimal) grammage of the layer. Scattering coefficient (*s*) is expressed in reciprocal grammage units. A multiplier (1,000) is used to convert m^2/g to the standard units, m^2/kg.

4.4 *Effective residual ink concentration.* Ratio of the absorption coefficient of pulp or paper, which contains ink, to the absorption coefficient of the ink itself as determined at 950 nm wavelength. ERIC units are expressed as parts per million (ppm).

5. Apparatus

5.1 *Reflectometer.* An instrument designed for the measurement of diffuse reflectance which employs the geometric and photometric characteristics specified in T 525, and the following spectral characteristics:

5.1.1 *Spectral characteristics.* The effective wavelength of the reflectometer, must be 950 ± 5.0 nm, the bandpass at 50% peak height must not exceed 150 nm, and the bandpass at 10% peak height must not exceed 250 nm. This spectral response is arrived at with a combination of lamps, integrating sphere, glass optics, filters or other spectrally selective device and photoelectric cells.

NOTE 1: The instrument used for ERIC analysis should contain a means of qualifying the sheet such that its opacity will not exceed 97.0 at 950 nm.

5.2 The following *ancillary items* must be available:

5.2.1 *Reference standard.* A white standard material with R_{950} value provided by an ISO Level III Authorized Laboratory (*5*).

5.2.2 *Instrument standard.* Opal glass, ceramic or spectralon standard which can be measured after calibration to the reference standard and maintained for future reference.

NOTE 2: Some instruments are equipped with an integral instrument standard.

5.2.3 *Black cavity.* A cavity coated with black felt, black paint or other coating material, having a known cavity reflectance of less than 1% at 950 nm.

6. Test specimens

6.1 Measurement of the ERIC value of a sample is highly dependent upon the sample being analyzed. In order for the ERIC analysis to be at its most effective level, the sample must be:

a. Uniform in terms of ink distribution, formation, and grammage in all three dimensions.
b. The paper backing opacity must not exceed 97.0 at 950 nm.

NOTE 3: It is usually possible to meet these criteria when dealing with machine made paper, however, it is sometimes difficult to produce a handsheet from pulp (containing ink) that completely qualifies. If it is not possible to meet the opacity requirement, it will not be possible to determine the actual ERIC content. If the sample has an opacity near 100%, the pad reflectance (R_∞) and the single sheet reflectance over black (R_0) are nearly equal, which leads to inaccurate calculations of scattering and absorption coefficients. However, it is possible to analyze relative ERIC values among a collection of samples, provided the scattering coefficients of the various samples under investigation does not vary substantially.

6.1.1 *Machine made paper and pulp handsheet test specimen preparation.* Cut sample sheets to at least 63.5 × 63.5 mm (2.5 × 2.5 inch) test pieces. Provide a stack of test pieces sufficient to be opaque (i.e. where no change in reflectance results from doubling the number of sheets in the stack).

6.1.2 *Pulp handsheet preparation.* Making handsheets from pulp (containing ink), that are suitable for ERIC analysis, requires care and attention. The commonly selected Büchner funnel method of preparing a specimen, TAPPI T 218 “Forming Handsheets for Reflectance Testing of Pulp (Büchner Funnel Procedure),” is not effective for ERIC analysis for the following reasons:

a. The resulting specimen is typically too opaque, its formation is poor and its grammage is not uniform. Büchner funnel specimens exhibit many times the variation in ERIC values obtained for handsheets made from the sheet machine.
b. The filter paper on the backside of the specimen and the high consistency act as drainage barriers, causing a high concentration of ink on the backside of the specimen. This results in the specimen being very “sided” (one side of the sheet being darker than the other side).
c. The high consistency and slow drainage rate is inconsistent with machine made paper.
d. Intimate contact is not achieved.

The preferred method for making handsheets for ERIC analysis is via the sheet machine procedure, TAPPI T 272 “Forming Handsheets for Reflectance Testing of Pulp (Sheet Machine Procedure),” but using a handsheet weight of 1.2 grams. These lightweight handsheets exhibit similar (but, due to the higher degree of washing, not the same) optical properties as those obtained on machine made papers. It is important to visually inspect the handsheet after preparation to make certain that its formation is uniform. Poor formation will result in misleading ERIC results.

6.1.3 It is important when making handsheets from pulp (containing ink) that the pH of the pulp must not be adjusted as this will affect the ERIC results. Sheets should be made in the sheet machine using type II reagent water *(6)* as described in ASTM D 1193 or better and the slurry allowed to find its own pH level. The pH shall then be measured just before the deckle is drained and included in the report.

NOTE 4: The instrument used for ERIC analysis should contain a means of qualifying the sheet such that its opacity will not exceed 97.0 at 950 nm.

7. Calibration

7.1 Select the appropriate filter or spectral equivalent for reflectance measurement at 950 nm. Calibrate the instrument relative to the reference standard using the instrument manufacturer's instructions. The black cavity must be employed in the calibration routine to assure proper calibration at the low end of the reflectance scale. After calibration read and record the value of an instrument standard for future reference (not mandatory).

NOTE 5: The instrument standard may be used from time to time to check the stability of calibration.

8. Procedure

8.1 Select the 950 nm filter position or modified spectral equivalent.

8.2 Place the stack of test pieces on the instrument and read and record the reflectance of the top sheet while backed with the other sheets in the stack (R_∞).

8.3 Place the top sheet on the instrument and back it with the black cavity. Read and record its reflectance (R_0).

8.4 A minimum of five specimens must be measured.

8.5 Repeat the above measurements for the opposite side of the sheet.

NOTE 6: If sidedness is present in the sample to the extent that the ERIC value on one side differs from that on the other side by 25 ppm or more, then the ERIC values for each side and an average of both sides should be reported.

NOTE 7: Handle the specimens by the corners or edges to avoid contamination in the area of measurement.

9. Calculations

The *scattering coefficient (s)* of the sample at 950 nm may be calculated by:

$$s = [1000/ (w (1/R_\infty - R_\infty))] \ln [(1- R_0R_\infty) / (1 - R_0/ R_\infty)]$$

where w = grammage (g/m^2) and where R_0 and R_∞ are expressed as decimals.

The *absorption coefficient* (k) of the sample at 950 nm may be calculated by:

$$k = s [(1 - R_\infty)^2 / 2 R_\infty]$$

where R_∞ is expressed as a decimal.

$$\text{ERIC} = (k_{\text{sheet}}/k_{\text{ink}})10^6$$

Black inks have been shown to have an absorption coefficient of about 10,000 m^2/kg *(3)*. This may be considered to be a default value for this method (i.e. k_{ink} = 10,000). If an *insitu* means is available for determining the absorption coefficient for the ink, the value used should be reported *(3)*.

10. Report

10.1 Report a precise identification of the sample.

10.2 Refer to this TAPPI method.

10.3 Report the average ERIC value of the sample as well as the standard deviation, minimum, maximum and number of readings. The ERIC value must be reported unitless or in parts per million (ppm). Optionally report the scattering and absorption coefficients obtained for the sample to the nearest 0.1 m^2/kg.

10.4 Indicate whether the sample is machine made paper or a handsheet.

11. Precision

11.1 The within laboratory repeatability and reproducibility was computed from the averages from the reported standard deviations in accordance with TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility.”

11.2 The following estimates of repeatability and reproducibility are based on an interlaboratory test conducted using 7 laboratories and three samples of recycled printing paper. The precision data is based on 10 determinations per test and one result per lab, per material.

11.3 Repeatability and reproducibility are estimates of the maximum difference (at 95%) which should be expected when comparing test results for materials similar to those described above under similar test conditions. These estimates may not be valid for different materials or testing conditions.

	Property Measurements			
Material	*Grand mean*	*Repeatability r and %r*	*Reproducibility R and %R*	*Labs included*
A	71.4	0.9 1.1%	8.9 12.4%	7
B	134.7	4.0 3.0%	8.5 6.4%	7
C	258.5	6.5 2.2%	13.1 5.1%	7
	Mean	3.8 2.1%	10.2 8.0%	

12. Keywords

Ink, Absorption, Scattering, Reflectance, Opacity, Handsheets, Paper, Kubelka-Munk equation

13. Additional information

13.1 Effective date of issue: To be assigned.

13.2 Related methods: TAPPI T 525, T 218, T 272, ISO 2469, ASTM D 1193, ISO 22754, PAPTAC E.8.

13.3 In the 2009 revision, language in 6.1.2 was changed to clarify the handsheet weight referred to from TAPPI T 272.

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