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Pulp and paper — Determination of the effective residual ink concentration (ERIC number) by infrared reflectance measurement

Pâte et papier — Détermination de la concentration d'encre résiduelle relative (nombre ERIC) par mesurage de la réflectance infrarouge

Reference number
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Contents

Page

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22754 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

Introduction

This International Standard provides a means of assessing the effective residual ink concentration (ERIC) in paper made from recycled fibres. The presence of residual ink influences the brightness and colour of pulp and of paper made from recycled fibre. The effect of the residual ink can be counteracted more easily if the effective concentration of the ink can be monitored. Brightness is not, however, an effective parameter for monitoring the deinking process, since brightness is affected by the presence not only of ink but also of other light-absorbing materials in the blue region of the spectrum such as lignin and dyestuffs. The ERIC method employs reflectance measurements in the infrared region of the spectrum where the light absorption coefficient of the ink is several orders of magnitude greater than the absorption coefficients of the fibre and other components, and this provides a sensitive means of estimating the concentration of ink [1]. This International Standard is based on the TAPPI method T 567 pm-97.

Pulp and paper — Determination of the effective residual ink concentration (ERIC number) by infrared reflectance measurement

1 Scope

This International Standard specifies a method for the determination of the effective residual ink concentration (ERIC number) by infrared reflectance measurement.

This International Standard is applicable to all types of deinked, recycled pulp and to sheets of machine-made paper made from recycled pulp, where the residual ink is black. The method is applicable to materials available in sheet form only if the opacity at 950 nm is less than 97 %. The ERIC number obtained is dependent on the distribution of ink particle sizes, and the method is most effective for submicron particles [2]. The value obtained is reliable only if the test material is uniform in ink distribution, formation, and grammage such that presenting different parts of the sheet to the measuring aperture of the reflectometer produces very similar readings.

2 Normative references

The following referenced documents are indispensable for the application 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 186, *Paper and board — Sampling to determine average quality*

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

ISO 2469, *Paper, board and pulps — Measurement of diffuse radiance factor*

ISO 3688, *Pulps — Preparation of laboratory sheets for the measurement of diffuse blue reflectance factor (ISO brightness)*

ISO 14487, *Pulps — Standard water for physical testing*

3 Terms and definitions

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

3.1
diffuse reflectance factor
R
ratio of the radiation reflected by a body to that reflected by the perfect reflecting diffuser under the same conditions of diffuse illumination and normal detection

NOTE 1 The ratio is often expressed as a percentage.

NOTE 2 This International Standard prescribes diffuse irradiation and normal detection in an instrument calibrated in accordance with the provisions of this International Standard.

3.2
intrinsic reflectance factor
R_∞
diffuse reflectance factor of a layer or pad of material thick enough to be opaque, i.e. such that increasing the thickness of the pad by doubling the number of sheets results in no change in the measured reflectance factor

NOTE The reflectance factor of a non-opaque sheet is dependent on the background and is not a material property.

3.3
single-sheet reflectance factor
R_0
reflectance factor of a single sheet of paper with a black cavity as backing

3.4
light scattering coefficient at 950 nm by reflectance factor measurements
s_{950}
⟨Kubelka-Munk method⟩ coefficient calculated by application of the Kubelka-Munk equations to reflectance factor data obtained at a wavelength of 950 nm in an instrument having a geometry and having been calibrated as specified in 5.1, and taking into consideration the grammage

NOTE 1 Units: m^2/kg.

NOTE 2 The relevant equations are given in Clause 9.

3.5
light absorption coefficient at 950 nm by reflectance factor measurements
k_{950}
⟨Kubelka-Munk method⟩ coefficient calculated by application of the Kubelka-Munk equations to reflectance factor data obtained at a wavelength of 950 nm in an instrument having a geometry and having been calibrated as specified in 5.1, and taking into consideration the grammage

NOTE 1 Units: m^2/kg.

NOTE 2 The relevant equations are given in Clause 9.

3.6
effective residual ink concentration
ERIC number
ratio of the light absorption coefficient of pulp or paper containing ink to the light absorption coefficient of the ink itself, both being determined at a wavelength of 950 nm

NOTE The ERIC number is dimensionless.

4 Principle

The intrinsic reflectance factor and the single-sheet reflectance factor of the test material are determined at a wavelength of 950 nm. The light absorption coefficient of the test material is calculated from these data by application of the Kubelka-Munk equations. The ERIC number may then be calculated as the ratio of this value to the light absorption coefficient of the ink.

5 Apparatus

5.1 Reflectometer, having the geometric, spectral and photometric characteristics described in ISO 2469, equipped with a filter or other device enabling the reflectance factor to be measured at a wavelength of 950 nm, and calibrated in accordance with the provisions of ISO 2469.

For such measurements, the effective wavelength of the reflectometer shall be (950,0 ± 5,0) nm. The spectral characteristics shall be such that the bandpass width at half peak height shall not exceed 150 nm, and the bandpass width at 10 % peak height shall not exceed 250 nm.

5.2 Reference standard, issued by an ISO/TC 6 authorized laboratory in accordance with the provisions of ISO 2469 for calibration of the instrument and the working standards, and having an assigned reflectance value corresponding to a wavelength of 950 nm.

Use new reference standards sufficiently frequently to ensure that the reflectometer is maintained in agreement with the reference instrument.

5.3 Working standards, two plates of flat opal glass, ceramic or other suitable material, cleaned and calibrated as described in ISO 2469.

Calibrate the working standards sufficiently frequently to ensure that satisfactory calibration is maintained.

NOTE In some instruments, the function of the primary working standard may be taken over by a built-in internal standard.

5.4 Black cavity, having a known reflectance factor of less than 1 % at 950 nm. The black cavity should be stored upside down in a dust-free environment or with a protective cover.

NOTE The condition of the black cavity can be checked by reference to the instrument maker.

6 Sampling and preparation of test material

6.1 Sampling

The sampling procedure is not included in this International Standard. If the tests are being made to evaluate a lot, the sample should be selected in accordance with ISO 186. If the tests are made on another type of sample, make sure that the test material taken is representative of the sample received.

6.2 Preparation of the test pieces

6.2.1 General

The method of preparation of test pieces depends upon whether the material is available as a machine-made paper or as a pulp.

6.2.2 Machine-made paper

For material available as a machine-made paper, cut test pieces with dimensions of at least (63,5 × 63,5) mm. Prepare a pad of test pieces sufficient to be opaque as defined in 3.2. Mark the top side of the pad for identification.

6.2.3 Pulp

Make a set of laboratory sheets from the pulp containing ink according to the procedure described in ISO 3688, each sheet having a grammage of 60 g/m^2. Do not use a retention aid.

NOTE ISO 3688 specifies two procedures for the preparation of laboratory sheets. The procedure using a Büchner funnel or similar is not suitable for making laboratory sheets in this case.

Do not adjust the pH of the pulp. Prepare sheets using reagent water as described in ISO 14487 or better and allow the slurry to find its own pH level. Measure and record the pH just before the deckle is drained.

These lightweight laboratory sheets exhibit similar (but, due to the higher degree of washing, not the same) optical properties as those obtained on machine-made papers. Visually inspect each laboratory sheet after preparation to make certain that its formation is satisfactory. Discard any laboratory sheet that exhibits a poor formation. Prepare at least four laboratory sheets for each pulp sample.

Dry the laboratory sheets as specified in ISO 3688. Cut test pieces with dimensions of at least (63,5 × 63,5) mm. Prepare a pad of test pieces sufficient to be opaque as defined in 3.2. Mark the top side of the pad for identification.

Check the opacity of the sheet at a wavelength of 950 nm. If the opacity exceeds 97,0 %, defined as the ratio of R_0 to R_∞ measured as described in 8.2 and 8.3, prepare new laboratory sheets with a lower grammage.

Assemble at least ten test pieces in a pad with their top sides uppermost; the number should be such that doubling the number of test pieces does not alter the reflectance factor. Protect the pad by placing an additional sheet of paper or board on both the top and bottom of the pad. Avoid contamination and unnecessary exposure to light or heat.

Mark the top test piece in one corner to identify the sample and its top side, or to distinguish between the two sides.

6.3 Grammage determination

Determine the grammage of the sheets in accordance with ISO 536.

7 Calibration

Select the appropriate filter or spectral equivalent for reflectance measurement at 950 nm. Calibrate the instrument using the reference standard (5.2) or working standard (5.3) and following the instrument manufacturer's instructions.

8 Procedure

8.1 Select the 950 nm filter position or spectral equivalent.

8.2 Place the pad of test pieces in the measuring aperture of the instrument. Read and record the intrinsic reflectance factor of the pad (R_∞). Handle the test pieces by the corners or edges to avoid contamination in the area of measurement.

8.3 Remove the top test piece from the pad and place it in the measuring aperture of the instrument over the black cavity. Read and record the single-sheet reflectance factor (R_0).

NOTE Subclauses 8.2 and 8.3 describe the two independent measurements which are necessary for the determination of the ERIC number. This text is not intended to imply that the two measurements necessarily be made in this order.

8.4 Move the top test piece to the bottom of the pad, and repeat 8.2 and 8.3 until four pairs of readings have been obtained. If the sample material was provided in the form of a pulp, make sure that the four pairs of readings are made on test pieces from four different laboratory sheets.

This subclause implies that measurements of R_∞ and R_0 are to be made alternately, but this is not an essential requirement of this International Standard. The four measurements of R_0 may be made before or after the four measurements of R_∞ if such a procedure is preferred.

8.5 Invert the pad and repeat 8.2 to 8.4 until four pairs of readings have been obtained on the reverse side of the sheets.

9 Calculations

9.1 Calculate the values for the two sides separately, as follows in 9.2 to 9.4.

NOTE The calculation can begin with a calculation of the mean R_∞ and R_0 values for each side but, if this is done, it will not be possible to calculate the standard deviation.

9.2 Calculate the light scattering coefficient at 950 nm (s_{950}) of each test piece as given in Equation (1):

$$s_{950} = \frac{1000}{g} \times \frac{R_\infty}{\left(1 - R_\infty^2\right)} \times \ln \frac{R_\infty \left(1 - R_0 R_\infty\right)}{R_\infty - R_0} \tag{1}$$

where g is the grammage, expressed in grams per square metre (g/m^2) and where R_0 and R_∞ are expressed as decimal fractions.

9.3 Calculate the light absorption coefficient at 950 nm (k_{950}) for each test piece as given in Equation (2):

$$k_{950} = \frac{s_{950} \left(1 - R_\infty\right)^2}{2 R_\infty} \tag{2}$$

where R_∞ is expressed as a decimal fraction.

9.4 Calculate the ERIC number for each test piece as given in Equation (3):

$$\text{ERIC} = 10^6 \left(k_{\text{sheet}} / k_{\text{ink}}\right) \tag{3}$$

where

k_{sheet} is the light absorption coefficient of pulp or paper containing ink, determined at a wavelength of 950 nm;

k_{ink} is the light absorption coefficient of the ink itself, determined at a wavelength of 950 nm.

If the k_{ink} value is not known, a light absorption coefficient of k_{ink} equal to 10 000 m^2/kg shall be used as a default value [3]. If the default value is not used, report the value of k_{ink} that is used.

Calculate the ERIC number to the first decimal place (0,1) as a dimensionless number.

If the ERIC number on one side of the test material differs from that on the other side by more than 25, report the ERIC numbers for the two sides separately, together with the average number for the two sides.

10 Precision

The estimates of repeatability and reproducibility shown in Table 1 are based on an interlaboratory test conducted in 2003 at 7 laboratories using 3 samples of recycled printing paper. The precision data are based on 10 determinations per test and 1 result per laboratory per material.

Table 1 — Repeatability and reproducibility

Material	**Mean**	**Repeatability**		**Reproducibility**	
		r	%	R	%
A	71,4	0,9	1,1	8,9	12,4
B	134,7	4,0	3,0	8,5	6,4
C	258,5	6,5	2,2	13,1	5,1
Mean			**2,1**		**8,0**

Repeatability and reproducibility are estimates of the maximum difference (at 95 %) which is to be expected when test results for materials similar to those described above are compared under similar test conditions. These estimates may not be valid for other materials.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) date and place of testing;

c) precise identification of the sample;

d) for pulps, the pH at which the laboratory sheets were made (see 6.2.3);

e) the value of k_{ink} which has been used (see 9.4);

f) the mean ERIC number and the standard deviation; if required (see 9.4), also report separately the values for the two sides;

g) any departure from this International Standard or any circumstances or influences that may have affected the results.

Annex A
(informative)

Background to the ERIC measurement

The function defined by Kubelka and Munk as a light 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 typically has a light absorption coefficient of the order of 10 000 m^2/kg [3], and if the residual ink in the sheet has increased the sheet's light absorption coefficient by 1 m^2/kg, then the “effective concentration” of the residual ink can be estimated to be 1/10 000 or 100 ppm (parts per million). The term effective concentration is used because the ERIC number is relative rather than absolute. The light 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.

Highly dispersed ink provides a greater surface area for light absorption than the same amount of agglomerated ink. Visual assessments of recycled paper brightness are also affected more by a large number of dispersed ink specks than by a few agglomerated specks. For this reason, the ERIC method provides a better correlation with visual assessment than other methods, e.g. image analysis of visible specks.

The estimation of residual ink by optical measurements in the infrared area is more accurate and much less time-consuming in the case of submicron ink particles than determination by image analysis methods [4]. Image analysers typically respond to ink particle sizes of 5 µm and above whereas the ERIC method is sensitive to the smallest particles below 10 µm. This follows from the exponential increase in the number of particles occurring with diminishing size.

Bibliography

Literature cited

[1] JORDAN, B.D. and POPSON, S.J. "Measuring the Concentration of Residual Ink in Recycled Newsprint," *Journal of Pulp and Paper Science*, **20** (6): 161 (June 1994)

[2] TREPANIER, R.J., JORDAN, B., NGUYEN, N. and PATSCHKA, H.J. "High-Magnification Image Analysis with Novel Background Reflectance Technique for Measuring Residual Ink in Sheets," *Journal of Pulp and Paper Science,* **23** (3): J129 (1997)

[3] JORDAN, B. and O'NEILL, M. "The Kubelka-Munk Absorption Coefficients of Several Carbon Blacks and Water-Based Printing Inks," *Journal of Pulp and Paper Science,* **20** (12): 371 (December 1994)

[4] CARRE, B., GALLAND, G. and SAINT AMAND, F.J. "Control of Detachment and Removal of Ink by Image Analysis," Centre Technique de l'Industrie des Papiers (CTP), Grenoble, France, doc. # 1670, (9 March 1994)

[5] TAPPI T 567 pm-97, *Determination of effective residual ink concentration by infrared reflectance measurement*

Additional literature

[6] BEN, Y. and DORRIS, G.M. "Irreversible Ink Redeposition During Repulping. Part II: ONP/OMG Furnishes", *Journal of Pulp and Paper Science*, **26** (8): 289 (August 2000)

[7] CHAPMAN, JOHN B. and WALMSLEY, MICHAEL R.W. "Light Absorbing Characteristics of Laser Inks in Bleached Eucalypt Sheets," Australian Pulp and Paper Institute, University of Waikato, Hamilton, New Zealand

[8] CARRE, B., GALLAND, G. and SAINT AMAND, F.J. "Estimation of Ink Detachment and Removal," *Progress in Paper Recycling*, **80** (November 1994)

[9] CARRE, B., GALLAND, G., VERNAC, Y. and SUTY, H. "The Effect of Hydrogen Peroxide Bleaching on Ink Detachment during Pulping and Kneading," TAPPI Recycling Symposium, February 1995, New Orleans, LA, USA

[10] GALLAND, G., VERNAC, Y. and CARRE, B. "The Advantages of Combining Neutral and Alkaline Deinking, Part I: Comparison of Deinking of Offset and Flexo Printed Paper", *Pulp and Paper Canada*, 98.6 (1997)

[11] GALLAND, G., VERNAC, Y. and CARRE, B. "The Advantages of Combining Neutral and Alkaline Deinking, Part II: Comparison of Various Processes for Deinking Mixtures Containing Waterbased Printed Paper in the CTP Pilot Plant", *Pulp and Paper Canada*, 98.7 (1997)

[12] HAYNES, R.D. "Evaluation of Deinking Chemicals Based on Ink Removal and Water Quality Using Lock Cycle Testing", TAPPI Recycling Symposium, April 1997, Chicago, IL, USA

[13] HAYNES, R.D. "Pulper Chemistry: The Key to Improved Deinking in North America", TAPPI Recycling Symposium, March 1998, New Orleans, LA, USA

[14] HAYNES, R.D. "The Impact of Pulper Chemistry on Contaminant Removal and Water Quality", TAPPI Recycling Symposium, March 1999, Atlanta, GA, USA

[15] HAYNES, R.D. "The Impact of the Summer Effect on Ink Detachment and Removal", TAPPI Recycling Symposium, March 1999, Atlanta, GA, USA

[16] HAYNES, R.D. “Measuring Ink Content: From Pulper to Deinked Pulp”, TAPPI Recycling Symposium, March 2000, Washington D.C., USA

[17] POPSON, S.J., MALTHOUSE, D.D., CRAWFORD, T.C., POPSON, M.T. and CRAWFORD, P.M. “Measurement and Control of the Optical Properties of Paper,” Ninth Edition, Technidyne Corporation, New Albany, IN, USA

[18] SAFADI, T. and FABRIS, I. “Innovative Testing Club ERIC 950 Evaluation,” PIRA Reference M456, April 1995, PIRA International, Leatherhead, Surrey, UK

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