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SCANDINAVIAN PULP, PAPER AND BOARD
TESTING COMMITTEE

Filler and pigment (powder and slurry)

Preparation of tablets for the measurement of ISO brightness, Y-value and colour (C/2°)

0 Introduction

This SCAN-test Method replaces SCAN-P 43:95 and SCAN-P 49:95 from which it differs in that it describes only the procedures for the preparation of opaque tablets from fillers and pigments in powder or slurry form, the measurements being performed according to current versions of ISO 2470 and ISO 5631. At the same time SCAN-P 43 and SCAN-P 49 have been withdrawn.

The results obtained for slurries may differ from those obtained for the corresponding air-dry samples because of differences introduced in the dispersing process.

Although the Method is concerned with the preparation of tablets, it also includes a brief description of the apparatus required and the procedure for carrying out the optical measurements.

1 Scope

This SCAN-test Method specifies a procedure for the preparation of opaque tablets from nearly white fillers and pigments used in the production of paper or board, on which ISO-brightness, Y-value and colour can be determined using existing ISO standards.

It is primarily intended for quality control purposes. It can be applied to both air-dry samples and slurries, provided the appropriate preparation procedure is adopted.

The results should not be used for the calculation of the optical properties of intermediate or end products.

2 References

ISO 2469	Paper, board and pulps – Measurement of diffuse reflectance factor
ISO 2470	Paper, board and pulps – Measurement of diffuse blue reflectance factor (ISO brightness)
ISO 5631	Paper and board – Determination of colour (C/2º) – Diffuse reflectance factor
SCAN-G 5	Pulp, paper and board – Basic equations for optical properties

Note – SCAN-test has withdrawn all methods for determination of optical properties of pulp, paper and board and refers instead to the corresponding ISO and/or EN Standards.

3 Definitions

For the purpose of this SCAN-test Method, the following definitions apply for filler and pigment:

3.1 *Reflectance factor, R* – Ratio of the radiation reflected by a body to that reflected by the perfect reflecting diffuser under the same conditions of illumination and detection (ISO 2469, ISO 5631).

Note 1 – In the context of ISO 2469 and related standards, the illumination is diffuse and the direction of detection is perpendicular to the surface of the specimen.

Note 2 – A gloss trap ensures that there is no or considerably reduced illumination from directions close to the direction of detection.

3.2 *Intrinsic reflectance factor,* R_∞, – 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 leads to no change in the measured reflectance factor (ISO 2469).

Note 3 – *Reflectivity* is synonymous with intrinsic reflectance factor.

Note 4 – The reflectance factor of a non-opaque tablet is dependent on the background and is not a material property.

3.3 *Diffuse blue reflectance factor (ISO brightness),* R_{457} – Intrinsic radiance factor measured with a reflectometer having the characteristics described in ISO 2469, equipped with a filter or corresponding function having an effective wavelength of 457 nm and a width at half height of 44 nm (ISO 2470).

Note 5 – The symbol R_B may be used instead of R_{457}.

Note 6 – The full definition given in ISO 2470 includes the provision that the instrument shall be adjusted so that the UV-content of the illumination incident upon the test piece corresponds to that of the CIE illuminant C, but this requirement is not included here, since it is assumed that fillers and pigments are not fluorescent. The UV-setting can affect the result if titanium dioxide is measured due to photochromic effects; in this case an adjustment to the CIE illuminant C conditions is recommended.

3.4 *Y-value (C/2°)* – Tristimulus value Y in the CIEXYZ-system of a layer of material of such a thickness that there is no change in Y when the thickness is increased.

Note 7 – The illumination is here CIE illuminant C.

3.5 *CIELAB colour (C/2°),* (L^*, a^*, b^*), – L^*, a^* and b^* values of the sample according to the CIELAB 1976 system, evaluated according to the CIE 1931 Standard Observer and the CIE illuminant C.

4 Principle

From the air-dry pigment or filler sample or from the dried slurry sample, opaque tablets are prepared. The reflectance factors of the tablets are measured in accordance with ISO 2470 or in accordance with ISO 5631 from which the colour coordinate values are calculated.

5 Apparatus

5.1 *Reflectometer*, as specified in ISO 2469 and calibrated in accordance with the provisions of ISO 2469.

5.1.1 *For the determination of ISO brightness*
An instrument having the spectral characteristics specified in ISO 2470.

5.1.2 *For the determination of the Y-value*
An instrument having the spectral characteristics specified in ISO 5631.

5.1.3 *For the determination of the colour*
An instrument having the spectral characteristics specified in ISO 5631.

5.2 *Working standards*, as specified in ISO 2469, ISO 2470 and ISO 5631.

5.3 *Reference standards,* non-fluorescent reference standards as specified in ISO 2469, ISO 2470 and ISO 5631.

Note 1 – If fluorescent materials are involved, then the fluorescent reference standards specified in ISO 2470 will also be required.

5.4 *Black cavity*, as specified in ISO 2469.

5.5 *Mill*, a fixed-blade mill capable of developing a blade tip speed of about 50 m/s, or any mill which will produce the same degree of pulverization.

Note 2 – The mill, Type A 10, produced by Janke & Kunkel, PO Box 44, DE-7813 Staugen, Germany, fulfils these requirements.

5.6 *Powder press* for the preparation of circular tablets, 45 mm in diameter and with a thickness of at least 4 mm.

Note 3 – A suitable press is manufactured by Carl Zeiss, DE-7082 Oberkochen, Germany.

Note 4 – Although it is not stated explicitly, it is assumed that the tablet is thick enough to be opaque.

5.7 *Detergent*, a dilute solution containing no coloured or fluorescent constituents.

6 Preparation of tablets

6.1 Drying

6.1.1 *For material available in a slurry*
Mix the slurry thoroughly. Weigh an amount containing approx. 100 g of air-dry sample. Spread out the slurry in a thin layer on a tray or dish of inert material. Dry it at (105 ± 3) °C until the sample is dry.

> *Note 1* – During the drying process the sample normally develops a light brown colour on the surface. This is caused by the caustic soda and the dispersing agent in the slurry. This coloured layer shall not be scraped off or removed in any way. It shall be included in the sample to be milled.

6.1.2 *For material available as an air-dried powder*
Take a sample of at least 100 g. If it consists of lumps or clods, crush these with caution in a mortar until all particles are less than 5 mm in diameter. Spread the material in a thin layer on a tray or dish of inert material. Dry it for 1 h at (105 ± 3) °C.

6.2 Milling

Mill the sample in the mill (5.5). If a Type A 10 mill is used, a suitable portion size is 10 g. For other type of mills, follow the instructions provided by the manufacturer.

The time required for milling must be determined by preliminary experiments. The milling time should be so long that further milling for a similar period of time causes no further increase in the reflectance factor values.

> *Note 2* – Normally a milling time of 2 min is sufficient if the Janke & Kunkel mill is used, but for fine particles a time of between 4 min and 5 min may be required. The mill should be tapped sharply from time to time, to ensure that material does not stick inside the lid and thus remain untreated.

6.3 Pressing

Follow the instructions pertinent to the powder press (5.6) and prepare at least 5 tablets. Check that the top surface of each tablet is absolutely even and free from glossy areas by viewing it at an oblique angle. An uneven surface with small cavities indicates that the sample was too moist for pressing.

After each pressing, clean all parts of the press that have been in contact with the sample using a soft brush and a small amount of the next sample to be tested. When a series of tests has been completed, clean the press with a soft brush, detergent (5.7) and distilled water. Rinse with plenty of distilled water and dry with filter papers.

7 Procedure

7.1 Calibration

Calibrate the reflectometer (5.1) as described in ISO 2469.

7.2 Measurement

Operate the reflectometer as specified in ISO 2470 and ISO 5631 as relevant.

Place a tablet in the measurement position, and read and record the reflectance factors (ISO brightness and R_x, R_y, R_z) of the tablet to the nearest 0,1 percentage point and the tristimulus values (X, Y, Z) of the tablet to the nearest 0,1 unit. Repeat the procedure for at least 4 more tablets.

The reflectance factors for different tablets prepared from the same sample should fall within a range of 0,5 percentage point. If this cannot be achieved, check in the first place the procedure used for the preparation of the tablets.

8 Calculation

Calculate the mean and the standard deviation of the results obtained and report, as relevant, the ISO brightness to the nearest 0,1 percentage unit and the Y-value to the nearest 0,1 unit.

Calculate the tristimulus values X, Y and Z, and the L^*, a^* and b^* values as described in ISO 5631.

> *Note* – Further details regarding the applicability of the equations described in ISO 5631 are given in SCAN-G 5.

9 Report

The test report should include reference to this SCAN-test Method and the following particulars:

(a) date and place of testing;
(b) precise identification of the sample, stating whether it was available as a slurry or as an air-dried powder;
(c) the ISO-brightness and Y-value, and the colour expressed as L^* $a^*$and b^* coordinates;
(d) the type of instrument used for the measurement;
(e) any departure from the procedure described in this Method or any other circumstances that may have affected the results.

10 Precision

10.1 ISO-brightness and Y-value

Six laboratories prepared tablets of kaolin and calcium carbonate from material provided as a powder. Each laboratory prepared five tablets.

The brightness and Y-value of these tablets were determined, and two samples of paper were also measured. The results (within lab and between labs) were as follows (see Table 1 and Table 2):

Table 1. ISO brightness according to ISO 2470 (in percentage units)

Lab	Kaolin		Calcium carbonate		Paper A	Paper B
	mean	s, within lab	mean	s, within lab	mean	mean
1	80,4	0,13	85,8	0,23	90,4	81,0
2	79,1	0,11	85,3	0,14	90,6	81,0
3	78,8	0,15	85,8	0,44	90,6	81,0
4	80,7	0,13	86,7	0,08	90,4	81,0
5	80,4	0,13	86,1	0,24	90,4	81,0
6	78,4	0,11	85,7	0,21	90,3	80,8
mean	79,6	0,13	85,9	0,25	90,5	81,0
s, between labs	0,98		0,47		0,12	0,08

Table 2. Y-value according to ISO 5631 (dimensionless)

Lab	Kaolin		Calcium carbonate		Paper A	Paper B
	mean	s, within lab	mean	s, within lab	mean	mean
1	85,5	0,10	90,5	0,18	94,0	85,1
2	-	-	-	-	-	-
3	84,4	0,12	90,2	0,30	94,1	84,7
4	85,8	0,13	91,0	0,04	94,2	84,8
5	85,4	0,08	90,5	0,21	94,0	84,7
6	83,5	0,14	90,2	0,16	93,9	84,5
mean	84,9	0,12	90,5	0,18	94,0	84,8
s, between labs	0,95		0,33		0,11	0,22

The results clearly show that the greatest source of variation is between laboratories in the procedure for preparing the tablets. The measurements on the paper samples show that only a small part of the deviation is attributable to variation in the optical measurements.

10.2 Colour

Five laboratories prepared tablets of kaolin and calcium carbonate from material provided as a powder. Each laboratory prepared five tablets. The CIELAB coordinates were calculated from spectral data obtained in an abridged spectrophotometer. The results were as follows:

Table 3. Colour according to ISO 5631 (dimensionless)

Lab	Kaolin				Calcium carbonate			
	L^*	a^*	b^*	ΔE	L^*	a^*	b^*	ΔE
1	93,7	-0,79	4,42	0,14	95,9	-0,08	3,39	0,25
2	93,6	-0,82	4,64	0,39	96,1	-0,07	3,32	0,07
3	94,2	-0,83	4,13	0,46	96,4	-0,18	3,28	0,27
4	94,1	-0,80	4,08	0,39	96,2	-0,13	3,32	0,06
5	93,3	-0,67	4,21	0,50	96,1	-0,12	3,37	0,05
mean	93,8	-0,78	4,30	0,38	96,1	-0,12	3,34	0,14
s, between labs	0,37	0,06	0,23		0,18	0,04	0,04	

Note – In this analysis, the deviations, ΔE*, of each laboratory from the mean L^*, a^*, b^* values are calculated using the equation:

$$\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad [1]$$

11 Literature

11.1 Baetke F.: Die Praxis der Weissgradmessung von Zellstoffen. Das Papier *15* (1961):7, 287 - 295

11.2 ASTM E308-99 Standard method for computing the colors of objects by using the CIE System

11.3 CIE Publ. 15.2 Colorimetry

11.4 ISO/CIE 10527 (1991) CIE Standard colorimetric observers

SCAN-test Methods are issued and recommended by KCL, PFI and STFI-Packforsk for the pulp, paper and board industries in Finland, Norway and Sweden.
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