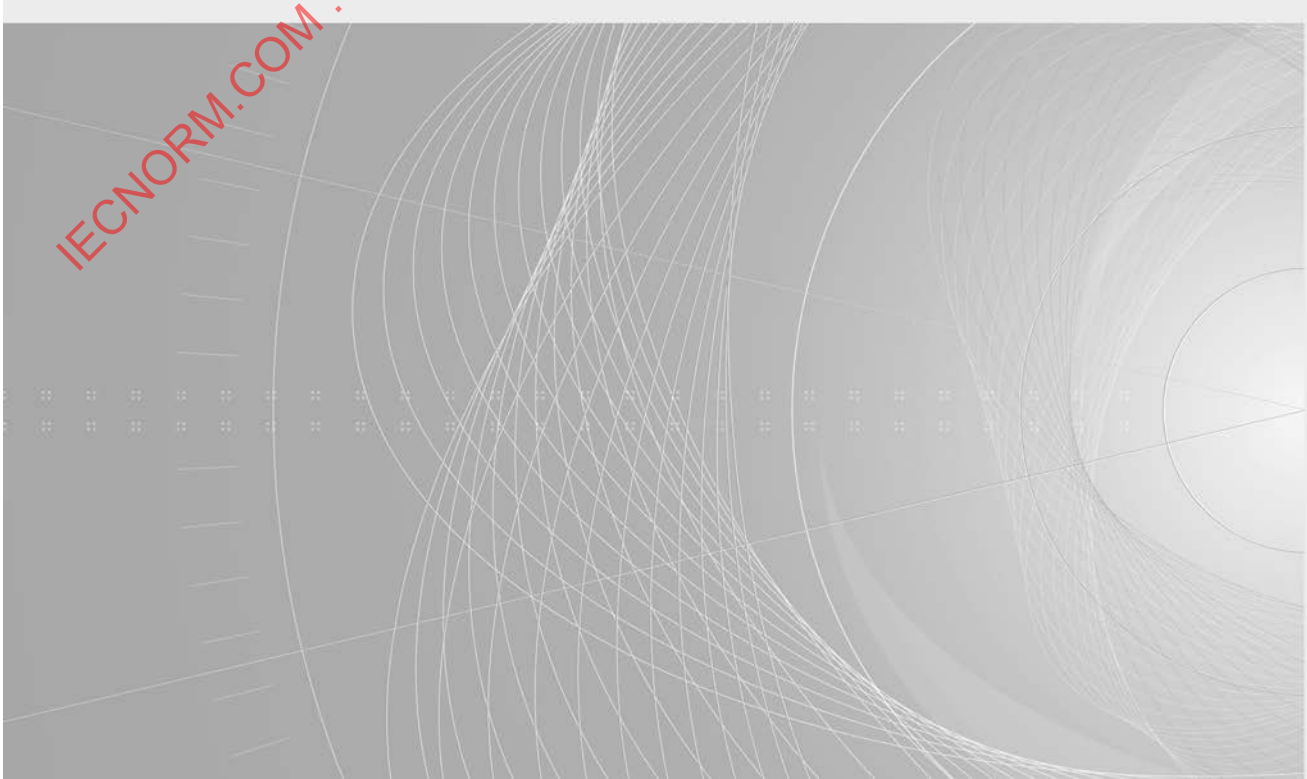
IEC 62899-201

Edition 1.0 2018-11

INTERNATIONAL STANDARD

AMENDMENT 1

Printed electronics –
Part 201: Materials – Substrates



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FOREWORD

This amendment has been prepared by IEC technical committee TC119: Printed Electronics.

The text of this amendment is based on the following documents:

CDV	Report on voting
119/189/CDV	119/206A/RVC

Full information on the voting for the approval of this amendment can be found in the report on voting indicated in the above table.

The committee has decided that the contents of this amendment and the base publication will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

2 Normative references

Add the following new normative references:

IEC 62321-3-1, *Determination of certain substances in electrotechnical products – Part 3-1: Screening – Lead, mercury, cadmium, total chromium and total bromine using X-ray fluorescence spectrometry*

IEC 62899-202-5, *Printed electronics – Part 202-5: Materials – Conductive ink – Mechanical bending test of a printed conductive layer on an insulating substrate*

ISO 187, *Paper, board and pulps – Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

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

ISO 217, *Paper – Untrimmed sizes – Designation and tolerances for primary and supplementary ranges, and indication of machine direction*

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

ISO 535, *Paper and board – Determination of water absorptiveness – Cobb method*

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

ISO 1924-2, *Paper and board – Determination of tensile properties – Part 2: Constant rate of elongation method (20 mm/min)*

ISO 1924-3, *Paper and board – Determination of tensile properties – Part 3: Constant rate of elongation method (100 mm/min)*

ISO 1974, *Paper – Determination of tearing resistance – Elmendorf method*

ISO 2471, *Paper and board – Determination of opacity (paper backing) – Diffuse reflectance method*

ISO 2493-1, *Paper and board – Determination of bending resistance – Part 1: Constant rate of deflection*

ISO 2493-2, *Paper and board – Determination of bending resistance – Part 2: Taber-type tester*

ISO 2758, *Paper – Determination of bursting strength*

ISO 2759, *Board – Determination of bursting strength*

ISO 3696, *Water for analytical laboratory use – Specification and test methods*

ISO 3781, *Paper and board – Determination of tensile strength after immersion in water*

ISO 3783, *Paper and board – Determination of resistance to picking – Accelerated speed method using the IGT-type tester (electric model)*

ISO 5626, *Paper – Determination of folding endurance*

ISO 5635, *Paper – Measurement of dimensional change after immersion in water*

ISO 6588-1, *Paper, board and pulps – Determination of pH of aqueous extracts – Part 1: Cold extraction*

ISO 6588-2, *Paper, board and pulps – Determination of pH of aqueous extracts – Part 2: Hot extraction*

ISO 8791-2, *Paper and board – Determination of roughness/smoothness (air leak methods) – Part 2: Bendtsen method*

ISO 8791-4, *Paper and board – Determination of roughness/smoothness (air leak methods) – Part 4: Print-surf method*

ISO 9220:1988, *Metallic coatings – Measurement of coating thickness – Scanning electron micro-scope method*

ISO 11556, *Paper and board – Determination of curl using a single vertically suspended test piece*

ISO 11798, *Information and documentation – Permanence and durability of writing, printing and copying on paper – Requirements and test methods*

ISO 12192, *Paper and board – Determination of compressive strength – Ring crush method*

ISO 15359, *Paper and board – Determination of the static and kinetic coefficients of friction – Horizontal plane method*

ISO 15754, *Paper and board – Determination of z-directional tensile strength*

3 Terms and definitions

Add, after 3.18, the following new terms and definitions:

3.19
paper
material without conductivity in the form of a coherent sheet or web, excluding sheets or laps of pulp as commonly understood for paper-making or paper-dissolving purposes and non-woven products, made by deposition of vegetable, mineral, animal or synthetic fibres, or their mixtures, from a fluid suspension onto a suitable forming device, with or without the addition of other substances

Note 1 to entry: There are also a number of synthetic products with paper-like qualities that are applicable as substrates for printed electronics. For the purposes of this document these can be treated as paper for testing as substrates.

[SOURCE: ISO 21067-1:2016, A.1.1, modified – "without conductivity" and NOTE have been added.]

3.20
board
paper (3.19) of a relatively high rigidity

Note 1 to entry: The term "paper" may be used for both paper and board. Materials of grammage less than 225 g/m^2 are generally considered to be paper, and materials of grammage of 225 g/m^2 or more to be board.

[SOURCE: ISO 5127:2017, 3.3.5.2.02]

3.21
pinhole
small hole in a printed feature that is a result of a surface inhomogeneity on the substrate

Note 1 to entry: This can be a consequence of a number of causes, and potential examples are listed below:

- a small hole in the surface of the substrate;
- a hole large enough to permit the transfer of an applied functional ink;
- a local variation in the wetting properties of the surface that results in uneven wetting of an applied functional ink.

4.2 Structures of substrates

Add, at the end of the subclause, the following new text:

For paper substrates, all paper and paper board substrates may be used. For example, uncoated or coated one-layer paper, as well as similarly uncoated or coated multilayer paperboards can be used, and laminated papers/boards, polymer coating papers/boards, and papers/boards coating with extrusion/dispersion can also be used.

4.3.3.3 Other materials

Replace the existing title and text with the following new title and text:

4.3.3.3 Paper substrate

When paper substrate is used as a sheet, the dimensions of the sheets shall be as specified in ISO 216. Designation and tolerances for primary and supplementary ranges, and indication of machine direction shall be as specified in ISO 217.

When paper substrate is supplied by a roll, the standard widths of the roll shall be according to ISO 217. Winding length on a roll shall be as agreed between user and supplier.

4.3.4.3 Other materials

Replace the existing title and text with the following new title and text:

4.3.4.3 Paper substrate

The thickness of substrates is not limited. It shall be as agreed between user and supplier. The method for measuring thickness shall be according to ISO 534.

Determination of grammage shall be according to ISO 536.

5.1 Sampling

Replace the existing text with the following new text:

In case paper substrates are used, the test methods in this document may be applied to the evaluation of a single sheet or to the set of sheets. When the test methods are applied to the set of sheets, the sheet shall be sampled randomly with no replacement. In cases where the results of the tests are reported for a set of sheets, the total number of sheets in the set to be tested and the number of sheets measured shall be reported.

5.3 Atmospheric conditions for evaluation test

Add, at the end of 5.3, the following new text:

The paper and paper board substrates shall be tested under standard atmospheric conditions, at a temperature of 23 °C ± 1 °C and a relative humidity of (50 ± 2) %.

NOTE These are the standard atmospheric conditions as specified by ISO 187.

5.4.3 Other materials

Replace the existing text with the following new text:

Paper and paper board substrates need conditioning. Unless otherwise specified, the test specimens to be evaluated shall be conditioned according to ISO 187.

8 Characteristics and evaluation method of other materials (ceramics, metal, paper, others)

Replace the existing title and text with the following new title and text:

8 Characteristics and evaluation method of paper substrates

8.1 General

The paper and paper board substrates used in printed electronics (PE) shall be tested according to the methods specified in Table 2. Unless there is a prior agreement between the user and supplier these test methods shall be applied without modification. In cases where the test has been modified, the changed condition shall be described in the report.

Table 2 – Test methods for paper and paper board substrates used in PE

Items		Standards that each test method is defined
Surface condition	Surface roughness	Subclause 8.2 in this document
	Flatness (curl)	ISO 11556
	Defects	Subclause 8.3 in this document
	Coefficient of friction	ISO 15359
	Contact angle	Subclause 8.4 in this document
	Surface pH	Subclause 8.5 in this document
	Composition of surface	Subclause 8.6 in this document
	Thickness of coating layer	Subclause 8.7 in this document
Mechanical properties	Bursting strength	ISO 2758 (for paper), ISO 2759 (for paper board)
	Tensile strength	Constant rate of elongation method: ISO 1924-2 (for 20 mm/min) ISO 1924-3 (for 100 mm/min) ISO 3781 (for after immersion in water) z-directional tensile strength: ISO 15754
	Young's modulus	ISO 1924-3
	Bending radius	(IEC 62899-202-5)
	Tearing resistance	ISO 1974 (Elmendorf method)
	Bending resistance (bending stiffness)	ISO 2493-1 (constant rate of deflection) ISO 2493-2 (taber-type) ISO 5626 (folding endurance) ISO 11798 (mechanical strength)
	Resistance to picking	ISO 3783
	Compressive strength	ISO 12192 (ring crush method)
Chemical properties	pH of aqueous extracts	ISO 6588-1 (cold extraction) ISO 6588-2 (hot extraction)
	Water vapour transmission rate	ISO 15106-2
	Oxygen gas transmission rate	ISO 15105-2
	Water absorptiveness	ISO 535 (Cobb method)
	Dimensional stability (humidity)	ISO 5635
Electrical properties	Volume resistance and surface resistance	IEC 62899-201
	Electrical strength	IEC 62899-201
Optical properties	Opacity	ISO 2471 (diffuse reflectance)

8.2 Surface roughness

8.2.1 General

The surface roughness of paper substrates is classified according to the following three classes:

class (a) optical roughness at < 1 µm;

class (b) micro roughness at 1 μm to 100 μm;

class (c) macro roughness at >100 μm

In traditional technology, class (a) was not recognized as an important area. However, the progress of the technology requires the region of class (a) to be considered, whereas the importance of class (c) might be reduced in comparison. The measurement methods for classes (a) and (b) are specified in this document.

8.2.2 Measurement method for class (a)

8.2.2.1 General

The gloss method or the white light interferometry (WLI) shall be used in the detection of optical roughness (< 1 μm). The method which is used in this measurement may be decided according to prior agreement between the user and supplier.

NOTE The WLI is used for high smoothness glossy paper such as a uniformly nanoparticle-coated product.

When the 60° gloss is over 65 or the average roughness (Ra) is less than 0,6 μm, the distinctness of image (DOI) measurement may be used.

8.2.2.2 Gloss method

The 60° gloss shall be used in this measurement. The appropriate gloss meter may be used, but the measurement method should be according to ISO 2813.

NOTE The equipment which is specified in ISO 8254-1 is also used widely in the paper industry. However, the method of ISO 8254-1 is limited to 75° gloss. When the equipment is able to measure the 60° gloss, it will be possible to use it in this measurement.

Since the value of specular reflection light intensity which is obtained during the measurement of gloss and the surface roughness have a relation in Formula (1), the surface roughness (R_q (RMS)) is calculated by Formula (2). The value of specular reflection light intensity can also be obtained by dividing the value of the glossiness by the specularly reflected light intensity of the glass surface with the refractive index n = 1,567.

$$\frac{I}{I_0} = F\exp\left[-\left(\frac{4\pi\sigma\cos\theta}{\lambda}\right)^2\right] \tag{1}$$

$$\sigma = \frac{\lambda}{4\pi\sigma\cos\theta}\sqrt{-\ln\left(\frac{I}{I_0 F}\right)} \tag{2}$$

where

I is the specular reflection light intensity;

I_0 is the incident light intensity;

F is the Fresnel coefficient;

σ is the surface roughness (R_q);

θ is the measurement angle (60°); and

λ is the wavelength.

8.2.2.3 WLI method

The WLI method shall be used with following requirements:

a) the inclination of the sample shall be corrected by tilt-stage before measurement;

b) the measurement equipment shall be placed on an anti-vibration table;

c) enough data shall be collected to achieve statistical significance over the roughness length scale of the substrate.

R_a and R_k shall be calculated according to the method described in 6.1.2.2.1.

8.2.2.4 DOI method

The wave scan DOI meter shall be used. The equipment shall use a laser light source to illuminate the substrate surface at a 60° angle. The image clarity value (C, %) is obtained as the result of the measurement, and R_a shall be calculated using the C value.

NOTE The DOI meter which is described in ASTM D5767 can be used in this measurement.

8.2.3 Measurement method for class b)

The method specified in ISO 8791-2 (Bendtsen method) or ISO 8791-4 (PPS (ParkerPrint-surf)) shall be used as the measurement of surface roughness of class (b).

8.2.4 Calculation

R_a and R_k shall be calculated as the parameters on the surface roughness according to the method described in 6.1.2.2.1.

8.2.5 Report of the results

The report shall include the following items:

a) measurement method and measurement equipment used;

b) measuring place and measurement size;

c) number of the sampling data;

d) result of R_a or R_q measurement;

e) result of R_k and number of protrusions (if detected).

8.3 Defects (pinholes) of coating layer

8.3.1 General

In many cases, paper substrates and paper board substrates are coated with polymeric layers. It may be possible to detect defects of the polymeric layer using the method described in 8.2. However, it would be necessary to examine the defects in a wider area on the surface of the paper substrate. In particular, pinholes are the defect which has a negative impact such as disconnection of the circuit. The method for detecting these defects is specified in 8.3. This method is applicable to all kinds of polymeric coated paper and paper board substrates.

It should be noted that there may be a case where it is not possible to detect all pinholes. It is necessary to pay particular attention in the case where the ink to be used has a large difference on the wettability from the ethanol solution.

8.3.2 Test specimens

At least five specimens shall be tested.

Except by prior agreement between the user and supplier, the size of the specimens shall be 12 cm x 12 cm.

In some cases the size of the specimens may be adjusted based on the size of the test equipment, in such cases a prior agreement between the user and supplier shall be made and the size of the test sample used shall be reported.

8.3.3 Colour solution

A solution of 0,5 % (weight per volume) of dyestuff ethanol shall be prepared. E131 Blue (CAS 3536-49-0) or Crossing Scarlet MOO (CAS 5413-75-2) shall be used as dyestuff. When insoluble matters are observed in the solution, the solution should be filtered before use.

8.3.4 Procedure

The specimen shall be placed on an appropriate plane. The colour solution shall be poured onto the surface of the specimen, covering it completely. After 5 min, the colour solution shall be poured off and wiped off the surface with some tissue paper or a sheet of unprinted kitchen roll. The number of coloured pinholes shall be counted and expressed by the number of pinholes in the sample per square metre. This procedure is repeated against five specimens. The five results shall be averaged.

8.3.5 Report of the results

The report shall include:

a) type of substrate, sample identification;

b) area of the test piece;

c) result of the test (averaged value).

8.4 Contact angle

8.4.1 General

When a droplet of liquid is dropped on the surface of a paper substrate, in many cases the droplet is absorbed into the substrate. In other words, the contact angle between the droplet and the substrate changes depending on the time. In the case of paper substrates where the surface is coated with a polymer and the contact angle does not change, the contact angle may be measured by the method specified in ISO 15989. However, for most paper substrates where the droplet of liquid is absorbed into the substrate, the contact angle shall be measured by the following method.

8.4.2 Outline of the measurement method

A drop with a specified volume of liquid is automatically applied to the surface of the paper substrate. Images of the contact angle between the droplet and the surface are captured by a video camera at specified time intervals. The contact angles at various time intervals are determined by image analysis techniques. The contact angles are determined at specified check points, and other related parameters such as the rate of change for the contact angles and the droplet volume can be determined by this method.

NOTE A meaningful property of the test liquid is that it has low to medium viscosity (e.g. below 100 mPa·s), and it has a boiling point above for example 60 °C.

8.4.3 Parameters

Parameters related to the contact angle shall be defined as in Figure 6.

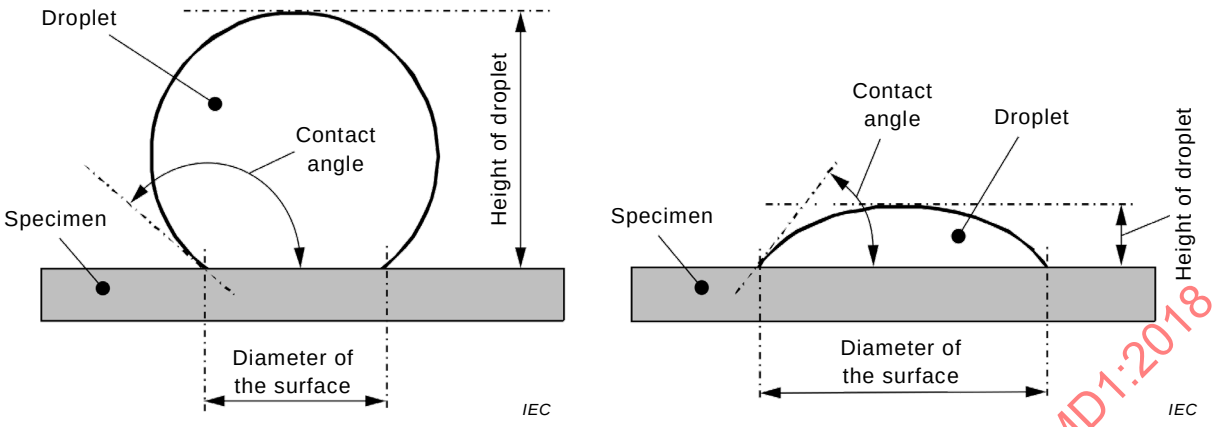

Figure 6 – Parameters related to the contact angle

8.4.4 Test apparatus

An automated contact angle tester shall comprise the following components:

- a light source,
- a video camera,
- a specimen stage,
- a liquid delivery system, and
- a computer with appropriate software.

The lamp housing shall be constructed so that the heat generated during lamp operation does not reach the test specimen or the droplet. The instrument shall have a contact angle precision of ±1° and a time precision of ±0,1 ms.

The specimen stage shall be designed so that the surface of the specimen becomes flat. The specimen is positioned horizontally with respect to the video camera, and shall be supported sufficiently to withstand the influence of capillary forces.

The drop volume should be in the range from 1 µl to 4 µl. It shall be possible to set the measurements start to the moment when the drop touches the substrate. The time resolution of the measurement should be 1 ms or better. The time stamp should be contained within the captured image with an accuracy of ±2 ms. A minimum of fifty video images shall be captured during the first second. After the first second, the images may be captured less frequently.

The inner wall of the dispensing system should be hydrophobic, for example by applying a PTFE coating.

The adjustment, calibration, and maintenance of the apparatus shall be performed following the instructions from the manufacturer.

The contact angle and the volume of the droplet shall be calculated as a function of time. The contact angle and the volume at any specified time (check points) should be calculated, and an average contact angle and volume should be calculated.

8.4.5 Test method

Except by prior agreement between trade partners, water with a purity of grade 3 or better as specified in ISO 3696 shall be used in this test.

Care shall be taken not to touch the surface of the specimen. The specimen shall not be cleaned unless otherwise specified. In some cases the wetting characteristics are different between the machine and cross directions. Care should be taken with the direction of the substrate. The size of the specimen shall be determined in accordance with the test apparatus. When the specimen curls it may be fixed by adhesive tape or other means in order to achieve a flat surface.

The test begins from the time when the droplet was dropped on the surface of the substrate by a trigger from the sync pulse from the video camera. Except by prior agreement between the trading partners, images shall be captured for at least 15 s.

At least three check points shall be set in the test. Except by prior agreement between the trading partners, the check points shall be either 0,1 s, 1,0 s and 2,0 s, or 0,1 s, 1,0 s and 10 s. The test shall be repeated ten times, changing the specimen position each time. The results (contact angles and droplet volumes) at each check point shall be averaged.

8.4.6 Report of the results

The report shall include the following items:

a) identification of the test specimen(s);
b) test liquid (if other than reagent water);
c) initial droplet volume (if other than standard volume);
d) centre-to-centre distance between two consecutive droplets applied on the same specimen surface;
e) stroke applied;
f) selected check points (seconds);
g) average contact angle and coefficient of variation at the selected check points;
h) average droplet volume and coefficient of variation at the selected check points.

8.5 Surface pH

8.5.1 General

For a pH measurement of paper, an aqueous extracts method is generally used. Since the surface pH of paper substrates affects printability directly this becomes one of most important factors in printing technology. In 8.5, the measurement of surface pH is specified.

8.5.2 Test specimens

The paper substrate to be measured shall be used as it is without any processing. It may be cut to the appropriate size for testing. Care should be taken not to touch the surface of the test specimens. The specimens shall not be cleaned unless otherwise specified by the manufacturer.

8.5.3 Measurement

The pH meter to be used for this measurement shall be equipped with a composite glass electrode with a flat head. The electrode shall be a single electrode formed by integrating the glass electrode and the reference electrode, and shall be soaked in a small amount of water. The pH meter shall be calibrated by standard buffers of pH 4,01 and pH 6,86. When a surface pH of greater than 8,0 is anticipated, the pH meter should be calibrated by a standard buffer of pH 9,18. The electrode shall be used after immersion into distilled water for at least 30 min.

The surface pH shall be measured as the pH of a wetting liquid where one or two drops have been placed onto the substrate. Distilled or deionised water, or 0,1 mol/L of KCl aqueous solution shall be used as the wetting liquid. The pH of the distilled or deionised water shall be in the range of 6,2 pH to 7,3 pH.

NOTE 1 If the variation of the measured pH becomes large, it is possible to use 0,1 mol/L of KCl aqueous solution to decrease the variation.

The pH value shall be measured after sufficient time has passed for the pH measured to reach equilibrium.

NOTE 2 In general, it often takes around 1 min or 2 min to reach equilibrium.

The electrode shall be washed with distilled water prior to each measurement.

The surface pH shall be measured for at least five sheets. The measured pH value shall be averaged to obtain the result value.

8.5.4 Report of the results

The report shall include:

a) identification of the test specimen(s);

b) number of measured sheets;

c) result of surface pH measurement (average of measured value).

8.6 Composition of surface (screening of metal composition)

The X-ray fluorescence spectrometry (XRF) shall be used in the screening of the metal composition on paper substrates and paper board substrates. The method (non-destructive approach) specified in IEC 62321-3-1 shall be used. In this measurement, at least Ca, Si, Al, and Ti shall be examined.

If quantification by the XRF method is necessary, the Ca and Ti content can be measured by the methods specified in ISO 10716 and ISO 5647, respectively.

The results of the screening shall be reported, including the following items:

a) type of substrate, sample identification;

b) detected metal composition, amount of metal(s);

c) Ca and/or Ti content measured by other methods (if necessary).

8.7 Thickness of coating layer

8.7.1 General

The thickness of any coating layer shall be evaluated by observation with a scanning electron microscope (SEM). It is necessary to prepare cross-sections for observation with a SEM. The requirements and procedure are specified in 8.7.

8.7.2 Instrumentation

The requirements of the apparatus (SEM), magnification, and calibration shall be according to ISO 9220.

8.7.3 Preparation of cross-sections

Various methods may be used for the preparations of cross-sections. Irrespective of how they are created, cross-sections shall meet the following requirements:

a) the cross-section shall be perpendicular to the plane of the coating layer;

b) the entire thickness of the coating image shall be simultaneously in focus at the magnification to be used for the measurement;

c) all material deformed by cutting or cross-sectioning shall be removed;