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Tobacco and tobacco products — Non-destructive determination of strips density variation ratio in case — Ionizing radiation method

Tabac et produits du tabac — Détermination non destructive de la variation de densité des strips en caisse — Méthode par radiations ionisantes

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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 12030 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, Subcommittee SC 2, *Leaf tobacco*.

Introduction

The density variation ratio (DVR) is one of the significant factors that influence tobacco aging and primary processing (see Reference [1]). For example, the phenomena of oil loss, caking or even carbonization is easy to take place in an aging process because of the larger DVR. The block of strips, which has a larger DVR, is difficult to loosen in a conditioning cylinder. The larger DVR can lead to deterioration of taste and quality of cigarette and hence it is important to detect DVR of strips still in its case (i.e. before introducing it into a primary processing stream).

The standard method for detecting DVR of strips in case, used at present, is based on “9-point Static Detection’’ (see Reference [2]). This method results in damage to strips, being applicable for only off-line measurements and needing longer testing time. A non-destructive method, on the other hand, would be quicker, amenable to both off-line and on-line measurements and would result in no damage to strips and hence has been a subject of analytical research and development.

X-rays can easily penetrate the strips' case and their intensity correlates to the density of the material they pass through, which would be a strips column in the case. The ionizing method has been widely used in many other areas (see IEC 60692[3]) and IEC has developed relevant standards for safe X-ray apparatus (see IEC 60405[4]). The X-ray apparatus used in the proposed method complies with these International Standards and can therefore be considered to be a completely safe radiation source.

Thus the ionizing radiation (X-ray attenuation) method retains the advantages of a non-destructive method for measurement of density of strips in the case, is amenable for on-line as well off-line measurements, is quick, safe and relatively inexpensive. The present proposal elaborates on its application for measurement of density of strips in the case.

There are more possibilities of measuring the density variation ratio in case. Any system with the same accuracy can be used.

Tobacco and tobacco products — Non-destructive determination of strips density variation ratio in case — Ionizing radiation method

1 Scope

This International Standard specifies a method for determining density variation ratio (DVR) of strips in the case.

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 6488, *Tobacco and tobacco products — Determination of water content — Karl Fischer method*

ISO 16632, *Tobacco and tobacco products — Determination of water content — Gas-chromatographic method*

3 Terms and definitions

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

3.1
non-destructive determination
determination without any damage to the objects under study

3.2
density variation ratio
DVR
relative standard deviation of the strips density in case

4 Principle

The X-rays penetrate the tobacco strips in the case and lose their intensity depending on the density of the strips. The measured intensity of X-rays can thus be related to strips density according to Equation (1).

$$\rho = -\frac{1}{U_m \times d} \ln \frac{I_i}{I_0 \times B} \quad (1)$$

where

ρ is the strips density;

u_m is the mass attenuation coefficient;

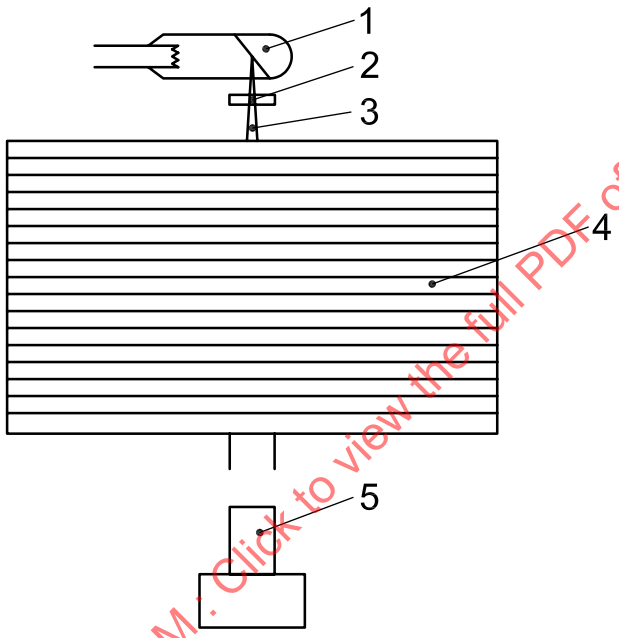
d is the thickness of strips (case height);

I_i is the intensity of X-rays after penetrating strips column;

I_0 is the initial intensity of X-rays;

B is the scattering factor.

The principle is illustrated in Figure 1.



Key

1 X-ray source
2 collimation hole
3 X-ray beam
4 strips case
5 NaI detector

Figure 1 — Principle of measurement

5 Apparatus

5.1 X-ray apparatus, complying with the following requirements. See Figure 2.

a) main components: one or three pairs of X-ray-producing tubes and X-ray detectors (see Annex B);

b) resolution: at least 1 kg/m^3 for strips density;

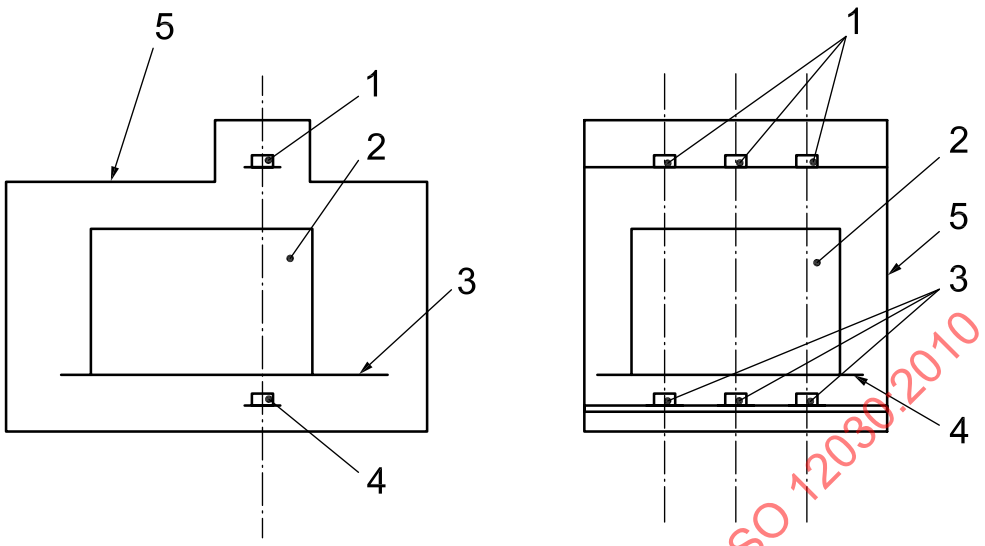
c) scope: 100 kg/m^3 to 600 kg/m^3 for strips density;

d) zero-drift: not more than 1 % over 24 h period;

e) output: density value of nine different detecting spots, ρ_1, $\rho_2 \ldots \rho_9$; DVR value;

f) safety requirement: maximum X-ray dose of 2,5 μSv/h at 1,0 m distance.

5.2 Analytical balance, with an accuracy of ± 0,1 g.



Key

1 X-ray-producing device
2 strips case
3 X-ray detector
4 conveyer belt
5 radiation shield

Figure 2 — Schematic diagram of X-ray apparatus

6 Procedure

6.1 Sampling

6.1.1 On-line determination

Every strips case shall be subjected to density determination.

6.1.2 Off-line determination

The recommended sampling rate for strips cases to be subjected for density determination is 2 %. If the total number of cases is less than 100, at least two cases representing the entire lot shall be subjected to density determination.

6.2 Experimental protocol

The main components of the instrument are the X-ray sources (5.1) with one or three pairs of X-ray-producing tubes and sensitive X-ray detectors. The X-ray-producing tubes emit X-rays constantly and the X-ray detector measures the intensity of the X-rays after they have passed through strips column in the strips case. The density determination device is fixed over a conveyor belt that carries the strips case. The number and location of detecting spots is given in 6.3.

The density value of nine different detection spots is determined as a first result, and then the DVR value is calculated.

6.3 Distribution of detection spots

The distribution of detection spots is shown in Figure 3.

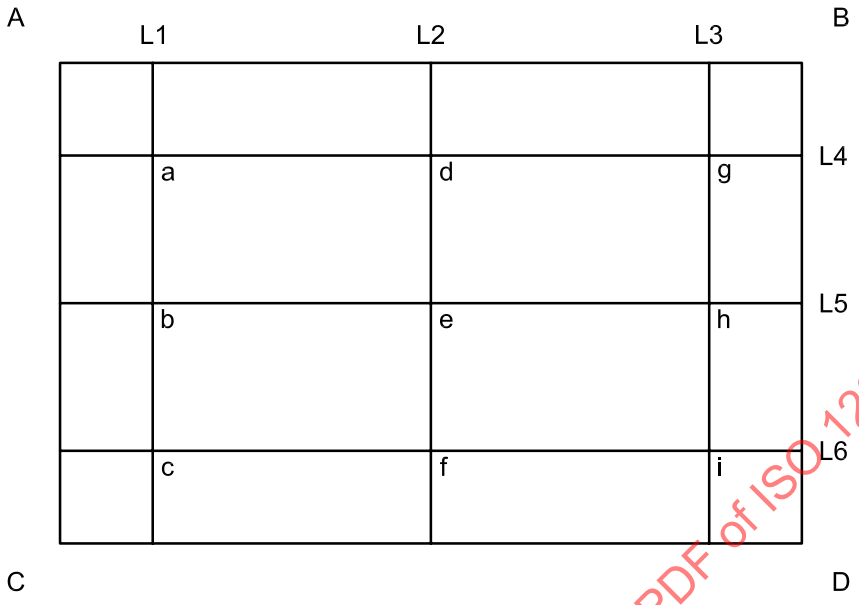


Figure 3 — Distribution of nine detection spots (top view of strips case)

The rectangle ABCD represents the top view of a typical strips case as placed under the X-ray device. Line 1 (L1), Line 2 (L2) and Line 3 (L3) are parallel with the edge AC; Line 4 (L4), Line 5 (L5) and Line 6 (L6) are parallel with edge AB. Detection spot locations are indicated by intersections of these lines *viz.*, spots a, b, c, d, e, f, g, h and i.

Spot 'e' is the centre of the top view of strips case. The distances between L1 and edge AC, L3 and edge BD, L4 and edge AB, L6 and edge CD are the same, being one eighth of the length of the strips case (edge AB).

6.4 Determination of water content

Water content is determined in accordance with ISO 6488 or ISO 16632.

Although the water content is not used in determining DVR, it may affect the results. The water content should, therefore, be determined and reported.

6.5 Determination of density

6.5.1 Calibration

The density determination instrument is calibrated with five standard pieces (plexiglass tubes containing strips with different densities) before actual usage as described in Annex A.

6.5.2 Determination of density

The intensity of X-rays (initial and after passing through strips column in strips case) is measured at nine spots and used to calculate strips density in nine different spots in the case.

CAUTION — Use the radiation shield as needed for safe operation.

7 Expression of results

The DVR value is calculated using the nine density values measured at nine different spots i.e. ρ_1, $\rho_2 \ldots \rho_9$ according to Equation (2).

$$\mathrm{DVR} = \frac{\rho_{\mathrm{sd}}}{\bar{\rho}} \times 100\ \% \tag{2}$$

where

DVR is the density variation ratio of strips in case, in percent;

ρ_{sd} is the standard deviation of nine different detecting spots, in kilograms per cubic metre;

$\bar{\rho}$ is the average density value of nine different detecting spots, in kilograms per cubic metre.

8 Test report

The test report shall include the following details:

a) all information necessary for the complete identification of the sample(s);

b) method and date of sampling;

c) date of testing;

d) water content, as a mass fraction in percent;

e) the analysis results and the units in which they are reported;

f) any special features observed during the analysis;

g) any working conditions that are not specified in this method or are considered as optional and that may have affected the results.

Annex A
(normative)

Determination of the density of standard pieces and calibration of density determination instrument

A.1 Standard pieces

Standard pieces are plexiglass cylinders with an outer diameter of 59 mm, a wall thickness of 5 mm with the tolerance being 0,1 mm and the height being equal to that of strips case. Two pieces of disc of the same material and thickness as the strips case are put in the two ends of standard pieces. The standard pieces are filled with the sample strips.

A.2 Determination of the density of standard pieces

The density of standard pieces is calculated by gravimetric method according to Equation (A.1).

$$\rho_{\text{actual}} = \frac{W_1}{V} \tag{A.1}$$

where

ρ_{actual} is the actual density value of a standard piece, in kilograms per cubic metre;

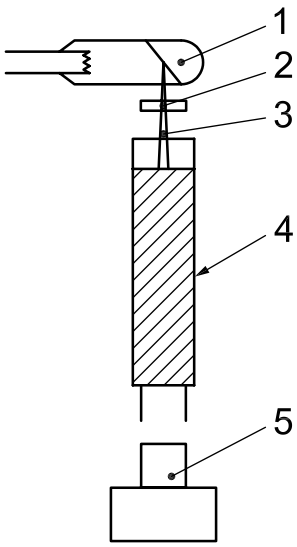
W_1 is the mass of strips within a standard piece, in kilograms;

V is the volume of a standard piece, in cubic metres.

A.3 Calibration

The density determination instrument needs to be calibrated before use. It is calibrated by five standard pieces with different densities between 200 kg/m^3 and 500 kg/m^3. It is recommended to use the strips with similar water content in the calibration tubes. The schematic diagram of the calibration apparatus is shown in Figure A.1.

Input the actual density value into the communication interface. The apparatus is adjusted automatically according to the actual density value and its corresponding I_i.



Key

1 X-ray source
2 collimation hole
3 X-ray beam
4 plexiglass cylinder
5 NaI detector

Figure A.1 — Calibration of density determination instrument with standard piece

A.4 Principle of calibration using standard pieces

The density value of a standard piece and the X-ray intensity after penetrating the material have the relationship shown by Equation (A.2).

$$\rho = K \ln(\frac{I_i}{I_0}) + C \tag{A.2}$$

where

K is a coefficient equal to $-\frac{1}{u_m \times d}$;

C is a coefficient equal to $\frac{1}{u_m \times d} \times \ln(B)$.

K and C can be calculated from the corresponding ρ_{actual} and I_i, and will be set as the defaults for particular samples.

Annex B
(informative)

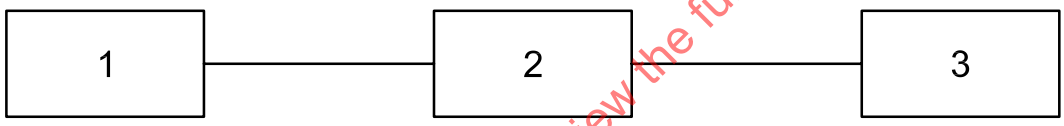
Main components of X-ray device

B.1 Principle of producing X-rays

A glass envelope under high vacuum, with a wire element at one end forms the cathode, and a heavy target at the other end forms the anode. Electrons from the cathode are accelerated on to a positively-charged anode by means of a high voltage. When electrons hit this material, some of the electrons will approach the nucleus of the metal atoms where they are deflected because of their opposite charges (electrons are negative and the nucleus is positive). This deflection causes the energy of the electrons to decrease, and this decrease in energy then results in forming an X-ray. X-rays are emitted from the anode in all directions. Shielding is used to create a narrow beam of X-ray.

B.2 X-ray producing device

X-ray producing device is composed of electrical source, X-ray tube and cooling system, seeing in Figure B.1.



Key

1 electrical source
2 X-ray tube
3 cooling system

Figure B.1 — X-ray-producing device

B.3 X-ray tube

An X-ray tube is composed of filament, cathode, anode, anode target and X-ray window. See Figure B.2.