
**Bidis — Determination of total and
nicotine-free dry particulate matter
using a linear routine analytical
smoking machine**

*Bidis — Détermination de la matière particulaire totale et de la
matière particulaire anhydre et exempte de nicotine au moyen d'une
machine à fumer analytique de routine*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

Introduction

A bidi (also spelt as beedi) is a hand-made smoking article made by rolling the tobacco filler consisting of air-cured, sun-cured and/or other suitable tobacco variety in Tendu (*Diospyrus* spp.) or Ashriti (*Bauhinia* spp.) leaf. The wrapper leaf is a forest product. Bidis are either conical or cylindrical in shape available in various lengths from 55 mm to 90 mm. Nearly 50 % of the weight of a bidi is contributed by the non-tobacco leaf wrapper.

A conical bidi is completely closed at the broad end (lighting end) by folding and tucking in the wrapper while the flattened narrow end (mouth end) is partly closed and is secured with a thread or any other means to prevent unfolding of the wrapper.

A filter bidi will have a filter plug on one end while the lighting end will be completely closed by folding and tucking in the wrapper leaf.

The components of bidis (tobacco filler and non-tobacco wrapping leaf) are natural products which cause intrinsic variability in bidis. The variability is further increased by the way these components are put together to produce a bidi.

Bidis form an important segment of smoking articles on a global level, are consumed in a large number of countries and hence an International Standard on the analysis of bidi smoke is justified.

The method allows bidis to be smoked on a routine analytical linear smoking machine using a strictly controlled set of parameters and collection of total particulate matter from smoke for further analysis. Thus, it enables the determination of nicotine-free dry particulate matter (NFDPM) and nicotine in bidis, when smoked by this procedure, to be compared and ranked on the basis of machine yield.

Experience and knowledge gained from the use of analytical cigarette-smoking machines have highlighted a need to specify certain requirements, which are addressed in this document.

No machine smoking regime can represent all human smoking behaviour:

- machine smoking testing is useful to characterize bidi emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;
- smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is misuse of testing using ISO standards.

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Bidis — Determination of total and nicotine-free dry particulate matter using a linear routine analytical smoking machine

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies methods for the determination of total particulate matter and for the subsequent determination of nicotine-free dry particulate matter present in the smoke from bidis generated and collected using a routine analytical smoking machine.

The smoking method described is suitable for routine analytical smoking machines which are equipped with a suitable bidi holder complying with the requirements of 5.8. The method is equally suitable for rotary machines after due validation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3308:2012, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

ISO 4387:2000, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*

ISO 10315, *Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method*

ISO 10362-1, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3308, ISO 3402 and ISO 4387 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

butt length

length of unburnt bidi remaining at the moment when the smoking is stopped

[SOURCE: ISO 3308:2012, 3.2, modified — “cigarette” has been replaced by “bidi”.]

3.2

puff volume

volume leaving the butt end of a bidi and passing through the *smoke trap* (3.7)

[SOURCE: ISO 3308:2012, 3.8, modified — “cigarette” has been replaced by “bidi”.]

3.3

puff number

number of puffs necessary to smoke a bidi to a specified *butt length* (3.1)

[SOURCE: ISO 3308:2012, 3.9, modified — “cigarette” has been replaced by “bidi”.]

3.4

puff frequency

number of puffs in a given time

[SOURCE: ISO 3308:2012, 3.10]

3.5

puff termination

termination of the connection of the port to the suction mechanism

[SOURCE: ISO 3308:2012, 3.11]

3.6

bidi holder

device for holding the mouth end of a bidi during smoking

[SOURCE: ISO 3308:2012, 3.14, modified — “cigarette” has been replaced by “bidi”.]

3.7

smoke trap

device for collecting such part of the smoke from a sample of bidis as is necessary for the determination of specified smoke components

[SOURCE: ISO 3308:2012, 3.15, modified — “cigarette” has been replaced by “bidi”.]

3.8

channel

element of a smoking machine consisting of one or more *bidi holders* (3.6), one trap and a means of drawing a puff through the trap

[SOURCE: ISO 3308:2012, 3.17, modified — “cigarette” has been replaced by “bidi”.]

3.9

mainstream smoke

all smoke which leaves the butt end of a bidi during the *smoking process* (3.13)

[SOURCE: ISO 3308:2012, 3.20, modified — “cigarette” has been replaced by “bidi”.]

3.10

total particulate matter
crude smoke condensate
TPM

that portion of the *mainstream smoke* (3.9) which is trapped in the *smoke trap* (3.7), expressed as milligrams per bidi

[SOURCE: ISO 4387:2000, 3.1, modified — “cigarette” has been replaced by “bidi”.]

3.11**dry particulate matter
dry smoke condensate
DPM**

total particulate matter (3.10) after deduction of its water content, expressed as milligrams per bidi

[SOURCE: ISO 4387:2000, 3.2, modified — “cigarette” has been replaced by “bidi”.]

3.12**nicotine-free dry particulate matter
nicotine-free dry smoke condensate
NFDPM**

dry particulate matter (3.11) after deduction of its nicotine content, expressed as milligrams per bidi

[SOURCE: ISO 4387:2000, 3.3, modified — “cigarette” has been replaced by “bidi”.]

3.13**smoking process**

use of a smoking machine to smoke bidis from lighting to final puff

[SOURCE: ISO 4387:2000, 3.4, modified — “cigarette” has been replaced by “bidi”.]

3.14**smoking run**

specific *smoking process* (3.13) to produce such smoke from a sample of bidis as is necessary for the determination of the smoke components

[SOURCE: ISO 4387:2000, 3.5, modified — “cigarettes” has been replaced by “bidis”.]

3.15**bidi laboratory sample**

bidi sample received in the laboratory for inspection or testing

[SOURCE: ISO 4387:2000, 3.6, modified — the term “laboratory sample” has been replaced by “bidi laboratory sample” and the definition has been revised.]

3.16**bidi conditioning sample**

bidis taken randomly from the laboratory sample for conditioning prior to selection and tests

[SOURCE: ISO 4387:2000, 3.8, modified — the term “conditioning sample” has been replaced by “bidi conditioning sample” and the definition has been revised.]

3.17**bidi test sample**

bidis for test selected by mass and length from the conditioned *bidi laboratory sample* (3.15)

[SOURCE: ISO 4387:2000, 3.7, modified — the term “test sample” has been replaced by “bidi test sample” and the definition has been revised.]

3.18**bidi test portion**

group of selected and marked bidis prepared for a single determination and which is a random sample from the test sample

[SOURCE: ISO 4387:2000, 3.9, modified — the term “test portion” has been replaced by “bidi test portion” and the definition has been revised.]

4 Principle

Bidis are conditioned and then selected by mass and length. The selected bidis are smoked on a routine analytical smoking machine with simultaneous collection of total particulate matter on a glass fibre filter pad fitted in a smoke trap. If used, the consistency of the laboratory smoking process and subsequent analytical procedures are controlled by using monitor test pieces specified in ISO 16055. The smoking regime/protocol is maintained as specified in ISO 4387 except for the puff frequency which is changed to two puffs per minute and only two bidis are smoked per smoke trap to avoid overload of the filter pad. This puff frequency is necessary as bidis extinguish if not puffed frequently. The mass of total particulate matter so collected on each trap is determined gravimetrically. The total particulate matter is extracted from the trap for the determination of the water and nicotine contents by gas chromatography.

In laboratories that are not in a position to use gas chromatographic methods, reference should be made to ISO 3400 for the determination of total nicotine alkaloids in smoke condensates by spectrometric method, and the determination of water in smoke condensate should be performed by the method described in ISO 10362-2. In such cases, values obtained for nicotine and water in smoke condensate may be used with the addition of a note in the expression of the result.

NOTE 1 ISO 3308 and ISO 4387 deal with the routine analytical smoking machine and smoking cigarettes on a smoking machine, respectively. Most of the requirements given in ISO 3308 and ISO 4387 are equally applicable to bidi smoking. Any laboratory familiar with cigarette smoke analysis will be able to adopt these standards for bidi smoke analysis by judiciously substituting cigarettes with bidis in these standards as appropriate.

NOTE 2 ISO 3308 and ISO 4387 employ a cigarette holder for smoking cigarettes on a routine analytical smoking machine. However, in case of smoking bidis on a smoking machine, a bidi holder (5.8) is required in place of the cigarette holder due to the bidi's unusual shape and the size of its mouth end.

5 Apparatus

The usual laboratory apparatus and, in particular, the following items.

5.1 Routine analytical cigarette-smoking machine.

5.2 Soap bubble flow meter, graduated at 35 ml to an accuracy of $\pm 0,2$ ml and with a resolution of 0,1 ml.

5.3 Apparatus for the determination of puff duration and frequency.

5.4 Analytical balance, suitable for measuring to the nearest 0,1 mg.

The weighing of filter pad holders may be affected by static electricity, necessitating the use of an antistatic device.

5.5 Length measuring device, suitable for measuring to the nearest 0,5 mm.

5.6 Smoke trap sealing device, end caps made from a non-hygroscopic and chemically inert material.

5.7 Gloves, made of cotton, or the non-talc surgical type.

5.8 Bidi holder, a device for holding the mouth end of a bidi during smoking.

The outer surface of the bidi is uneven and irregular and the mouth end is narrow and elliptical, hence a special holder is required. The bidi holder shall ensure a leak-proof hold of the mouth end of a bidi for smoking on the smoking machine. The bidi holder shall cover 9,0 mm, with a range of 8,0 mm to 9,5 mm, from the butt end of a bidi and shall be impermeable to smoke components and to air.

The bidi holder shall also be provided with devices for attaching it to the standard filter holder and in turn to the smoking port of the smoking machine so that the bidi holder is held rigidly and that no air enters the smoke trap and no smoke is lost from the smoke trap during smoking. The axis of the holder shall be within 0° to $+5^\circ$ of the horizontal and the holder shall ensure that the bidi is held within $\pm 5^\circ$ of the holder axis.

Bidi holders complying with these requirements are available commercially for smoking machines. The user instructions provided by the suppliers shall be followed for preparing the bidi holder, assembling the smoke trap and inserting the bidi into the holder.

An example of a bidi holder suitable for linear smoking machines is given in [Annex A](#) for information.

5.9 Glass fibre filter pads. It is recommended to choose the size of the glass fibre filter pads in relation to the mass of collected smoke condensate in accordance with ISO 3308.

The pads shall be conditioned in the test atmosphere specified in ISO 3402 for at least 12 h before use.

6 Preparation of bidi test portion for smoking

6.1 Conditioning of bidi laboratory sample

A sufficient number of bidis randomly drawn from the laboratory sample is conditioned as specified in [6.4.1](#).

6.2 Selection of conditioned bidis

6.2.1 General

The test sample bidis shall be selected from the conditioned sample ([6.1](#)) based on both mean mass and mean length.

Due to the inherent high variability in the bidi length and mass, it is necessary to select bidis on length and mass to achieve reproducible results in the total particulate matter collected.

6.2.2 Selection by mass

6.2.2.1 Weigh individually 100 bidis taken randomly from the conditioned laboratory sample ([6.1](#)) to the nearest 1 mg and calculate the mean mass and SD.

6.2.2.2 Bidis are then selected from the conditioned sample ([6.1](#)) on the basis of mean mass ± 30 mg.

6.2.3 Selection by length of mass selected bidis ([6.2.1](#))

6.2.2.1 Measure individually the length of 100 bidis taken randomly from the conditioned laboratory sample ([6.1](#)) to an accuracy of 0,5 mm and calculate the mean length and SD.

6.2.2.2 Bidis, already selected on a mass basis ([6.2.1.2](#)), are further selected on the basis of mean length ± 2 mm.

Bidis selected on mass and length basis will constitute the test sample for smoking and will be stored in the conditioning atmosphere ([6.4.1](#)).

6.3 Marking the butt length and insertion depth

6.3.1 Standard butt length and insertion depth

The standard butt length to which the bidis shall be marked shall be 23 mm. The standard insertion depth of the bidi in the bidi holder shall be 9 mm from the mouth end.

NOTE Butt length is the unburnt length of bidi remaining at the moment when smoking is stopped.

6.3.2 Marking of butt length and insertion depth

6.3.2.1 Draw a line, using a soft tipped marker, at 23 mm to an accuracy of 0,5 mm, from the mouth end.

6.3.2.2 Draw another line, using the soft tipped marker, at 9 mm to an accuracy of 0,5 mm from the mouth end. The marked bidis will constitute the test portion and will be stored in the conditioning atmosphere (6.4.1) till ready for smoking.

6.4 Conditioning and test atmosphere

6.4.1 Conditioning of bidis

All bidi samples shall be conditioned and treated as specified in ISO 3402.

Transfer the conditioned sample/test portions to the selection/smoking location in airtight containers (just large enough to contain the portions) unless the selection/smoking location and the conditioning location are adjoining and have identical atmospheres.

6.4.2 Testing atmosphere

The testing atmosphere in the laboratory where the smoking is to be carried out shall be in accordance with ISO 3402.

Measure the temperature and relative humidity of the air surrounding the smoking machine and note the atmospheric pressure.

6.5 Smoking and collection of particulate matter

6.5.1 Smoking plan

Choose a smoking plan; examples of plans are given in ISO 4387:2000, Annex A.

A minimum of 10 bidis shall be smoked in each smoking run. Smoke bidis in batches of two per channel when using a 44 mm filter pad or five when using a 92 mm filter pad.

The plan should include the use of a test portion of monitor test pieces. The test pieces are included in the plan as if they were another set of samples prepared and smoked in accordance with the procedure for smoking and collection of total particulate matter and its determination and treatment for further analysis.

Given the lack of availability of validation data for monitor test pieces under this regime, it is recommended to use the monitor test pieces under the conditions given in ISO 16055 to check the suitability of the machine and process.

6.5.2 Preparation of smoke traps

6.5.2.1 General

For all operations, the operator shall prevent contamination from the fingers by wearing gloves of suitable materials (5.7).

6.5.2.2 Insert conditioned filter pads (5.9) into their holders, and assemble, placing the rough side of the filter pad so that it will face the oncoming smoke. After assembly, examine the filter holders to ensure that pads have been properly fitted. Cover the front and back apertures of the trap with the sealing devices (5.6). Weigh the smoke traps to the nearest 0,1 mg.

If the smoke trap is designed to contain a perforated disc (washer), insert it and fit the sealing devices (5.6). Weigh the assembled smoke traps to the nearest 0,1 mg.

Because of absorption of water by smoke traps and solvent, it is necessary to determine a value for the sample blank. Prepare sample blanks by treating additional smoke traps (at least 2 per 40 bidis) in the same manner as that used for smoke collection.

6.5.2.3 Remove and preserve the sealing devices from the smoke trap. Attach the bidi holder.

These two steps should be carried out only after the machine is warmed up and ready for puff volume check (6.5.4), if required.

6.5.3 Puffing parameters

6.5.3.1 General

The smoking machine shall comply with the following puffing parameters. These checks should be performed in accordance with ISO 4387 by substituting cigarettes with bidis, as appropriate.

6.5.3.2 Puff duration

The standard puff duration shall be $(2 \pm 0,02)$ s.

6.5.3.3 Puff frequency

The puff frequency shall be 2 puffs per minute with 1 puff every $(30 \pm 0,5)$ s measured over 10 consecutive puffs.

6.5.3.4 Puff volume

The standard puff volume shall be $(35 \pm 0,3)$ ml.

6.5.4 Setting up the smoking machine

Switch on and allow the machine to warm up with automatic cycling for at least 20 min. After warm up, check that the puff duration and puff frequency on each channel are in accordance with the puffing parameters specified in 6.5.3. Fit the smoke trap and bidi holder assembly (6.5.2.3) onto the smoking machine.

The puff volume should be checked if it is suspected that the smoking machine is subject to a large change in temperature during use.

6.5.5 Procedure for smoke run

Insert the marked bidis (6.3) into the bidi holders; avoid any leaks or deformations.

Any bidis found to have obvious defects, or which have been damaged during insertion, shall be discarded and replaced with spare marked bidis.

Ensure that the bidis are positioned correctly so that the axes of the bidis coincide with the axes of the ports. Adjust the position of each bidi so that when the burning coal reaches the butt mark, the puff termination device is activated.

Zero the puff counters and light each bidi at the beginning of its first puff. Should it be necessary to relight a bidi, a hand-held electrical lighter may be used at the second puff. When each butt mark has been reached, remove the burning coal from the bidi and note the final reading of the puff counters.

After the smoking process is complete, leave the bidi butt in place for at least 30 s to enable deposition of any residual smoke in the trap.

In case a bidi extinguishes during a smoke run, the smoking on that channel shall be stopped and the filter pad discarded.

Avoid disturbance of the smoking by artificial removal of ash. Allow the ash to fall naturally into the ashtray.

If required, a new bidi shall be inserted immediately and the smoking process repeated to its completion. Immediately begin the determination of total particulate matter as described in [6.6](#).

6.6 Determination of total particulate matter

Remove the smoke traps from the smoking machine (gloves should be worn) and remove the bidi holder from the smoke trap. Cover the front and back apertures of the trap with the sealing devices ([5.6](#)). Weigh the smoke traps to the nearest 0,1 mg.

6.7 Calculation of total particulate matter

The difference in the mass of the smoke trap on each channel before and after smoking gives the amount of total particulate matter (TPM) per trap for the number of bidis smoked on that channel.

The TPM content, m_{TPM} , for each channel, expressed in milligrams per bidi, is given by [Formula \(1\)](#):

$$m_{\text{TPM}} = \frac{m_1 - m_0}{q} \quad (1)$$

where

m_0 is the mass of the smoke trap before smoking, in milligrams;

m_1 is the mass of the smoke trap after smoking, in milligrams;

q is the number of bidis smoked into the trap.

6.8 Treatment of total particulate matter

6.8.1 Extraction procedure

Remove the sealing devices from the smoke trap (gloves shall be worn). Open it and remove the filter pad with forceps. Fold it twice, total particulate matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded pad in an appropriately shaped dry flask (maximum 150 ml for 44 mm pads and maximum 250 ml for 92 mm pads). Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned filter pad and add these to the flask. Pipette solvent (propan-2-ol containing the internal standards for both nicotine and water determinations) into the flask (20 ml for 44 mm pads and 50 ml for 92 mm pads) (see ISO 10315 and ISO 10362-1).

Stopper the flask immediately and shake gently on an electric shaker for at least 20 min, ensuring that the pad does not disintegrate. The shaking time should be adjusted to ensure full extraction of the nicotine and water in the particulate matter.

Follow the same procedures with each of the blank smoke traps used for the determination of water.

6.8.2 Determination of water

Carry out the determination of water in the solution in each flask in accordance with ISO 10362-1.

The DPM content, m_{DPM} , for each trap, expressed in milligrams per bidi, is given by [Formula \(2\)](#):

$$m_{\text{DPM}} = m_{\text{TPM}} - m_{\text{W}} \quad (2)$$

where

m_{TPM} is the TPM content, in milligrams per bidi;

m_{W} is the water content in the TPM, in milligrams per bidi.

6.8.3 Determination of nicotine

Carry out the determination of nicotine in the solution in each flask in accordance with ISO 10315.

6.8.4 Calculation of NFDPM

The NFDPM content, m_{NFDPM} , for each trap, expressed in milligrams per bidi, is given by [Formula \(3\)](#):

$$m_{\text{NFDPM}} = m_{\text{DPM}} - m_{\text{N}} \quad (3)$$

where

m_{DPM} is the DPM content, in milligrams per bidi;

m_{N} is the nicotine content in the TPM, in milligrams per bidi.

7 Test report

The test report shall show the sample details, the method used and the results obtained accompanied with the following text clarifying that the results are valid only for the selected test sample:

“The test results reported here are valid only for this set of test sample which has been selected on the basis of mean mass (± 30 mg) and mean length (± 2 mm) for smoking.”

It shall also mention any operating conditions not specified in this document, or regarded as optional, as well as any circumstances that may have influenced the results.

The test report shall include all details required for complete identification of the sample. If appropriate, the information given in a) to d) shall be recorded.

a) Characteristic data about the bidis

All details necessary for the identification of the bidis smoked shall be given. In case of commercial bidis, this should include:

— name and address of the manufacturer;

- product name.
- b) Data about the laboratory sample:
 - type of sampling procedure;
 - date of sampling;
 - place of sampling;
 - sampling point and address;
 - number of bidis in the laboratory sample;
 - length of the bidis in millimetres (mean, range and SD);
 - mass of the bidis in milligrams (mean, range and SD).
- c) Description of the test:
 - reference to this document, i.e. ISO 17175;
 - date of the test;
 - type of smoking machine used;
 - type of bidi holder used;
 - type of smoke trap used;
 - total number of bidis smoked;
 - number of bidis smoked on each trap;
 - butt length;
 - room temperature (in degrees Celsius) during smoking operation;
 - relative humidity (in per cent) during smoking operation;
 - atmospheric pressure (in kilopascals) during smoking operation.
- d) Test results

The expression of the laboratory data depends on the purpose for which the data are required and the level of laboratory precision. Confidence limits shall be calculated and expressed on the basis of the laboratory data before any rounding has taken place.

Details should include the following:

- average length of the bidis to the nearest 0,5 mm;
- average mass of the bidis to the nearest 1 mg;
- butt length to which the bidis were smoked;
- average number of puffs per bidi for each channel to the nearest 0,1 puff;
- TPM content (in milligrams per bidi) for each channel to the nearest 0,1 mg and the average per bidi to the nearest 1 mg;
- DPM content (in milligrams per bidi) for each channel to the nearest 0,1 mg and the average per bidi to the nearest 1 mg;

- nicotine content (in milligrams per bidi) for each channel to the nearest 0,01 mg and the average per bidi to the nearest 0,1 mg;
- NFDPM content (in milligrams per bidi) for each channel to the nearest 0,1 mg and the average per bidi to the nearest 1 mg.

8 Repeatability and reproducibility

A collaborative study was conducted in 2015 involving five laboratories and one bidi sample to validate the method. The inter-laboratory test data and data analysis are given in a collaborative study report¹⁾. In spite of paucity of data points, r and R values are given in [Annex B](#) for information validating the method.

1) The full study report is available upon request from the secretariat of ISO/TC 126.