INTERNATIONAL STANDARD

ISO 2965

Third edition
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Materials used as cigarette papers, filter plug wrap and filter joining paper, including materials having a discrete or oriented permeable zone and materials with bands of differing permeability — Determination of air permeability

Matériaux utilisés comme papier à cigarettes, pour le gainage des filtres et comme papier manchette, y compris les matériaux possédant une zone perméable discrète ou orientée et les matériaux à bandes de perméabilité diverses — Détermination de la perméabilité à l'air

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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 2965 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, Subcommittee SC 1, *Physical and dimensional tests*.

This third edition cancels and replaces the second edition (ISO 2965:1997) which has been technically revised.

Introduction

Measurements of air permeability of materials used as cigarette papers have been made for many years. The methods have required development and change because of the changing nature of the paper products and changes in the magnitude of their air permeability.

This method has been developed with the technical resources of CORESTA (Cooperative Centre for Research Relative to Tobacco).

Materials used as cigarette papers, filter plug wrap and filter joining paper, including materials having a discrete or oriented permeable zone and materials with bands of differing permeability — Determination of air permeability

1 Scope

This International Standard specifies a method for the determination of air permeability (AP).

It is applicable to materials used as cigarette papers, filter plug wrap and filter joining paper, including materials having an oriented permeable zone or discrete permeable zones where the measured permeability is in excess of 10 $cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa. In addition, it is applicable to banded cigarette papers, with bands of width ⩾ 4 mm.

NOTE For an estimate of the air permeability of materials outside the scope of this International Standard, see Note 2 in 5.1.2 and Note 3 in 7.6.1.

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

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

3 Terms and definitions

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

3.1
air permeability
AP
flow of air, measured in cubic centimetres per minute, passing through 1 cm^2 surface of the test piece at a measuring pressure of 1,00 kPa

NOTE The air permeability units are $cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa.

3.2
measuring pressure
difference in pressure across the two faces of the test piece during the measurement

3.3
leakage
air flow unintentionally aspirated from the surrounding atmosphere or escaping into it through the sealing surface of the test piece holder and elsewhere

3.4
paper with uniformly distributed permeability
standard paper
paper with natural air permeability only

3.5
paper with oriented permeable zone
paper that has a continuous zone of higher air permeability obtained through perforation

3.6
paper with discrete permeable zone
paper that has higher air permeability obtained through perforation in discrete areas

3.7
banded paper
paper with bands of different air permeability

NOTE Papers of this type normally have bands with permeability significantly lower than that of the base paper.

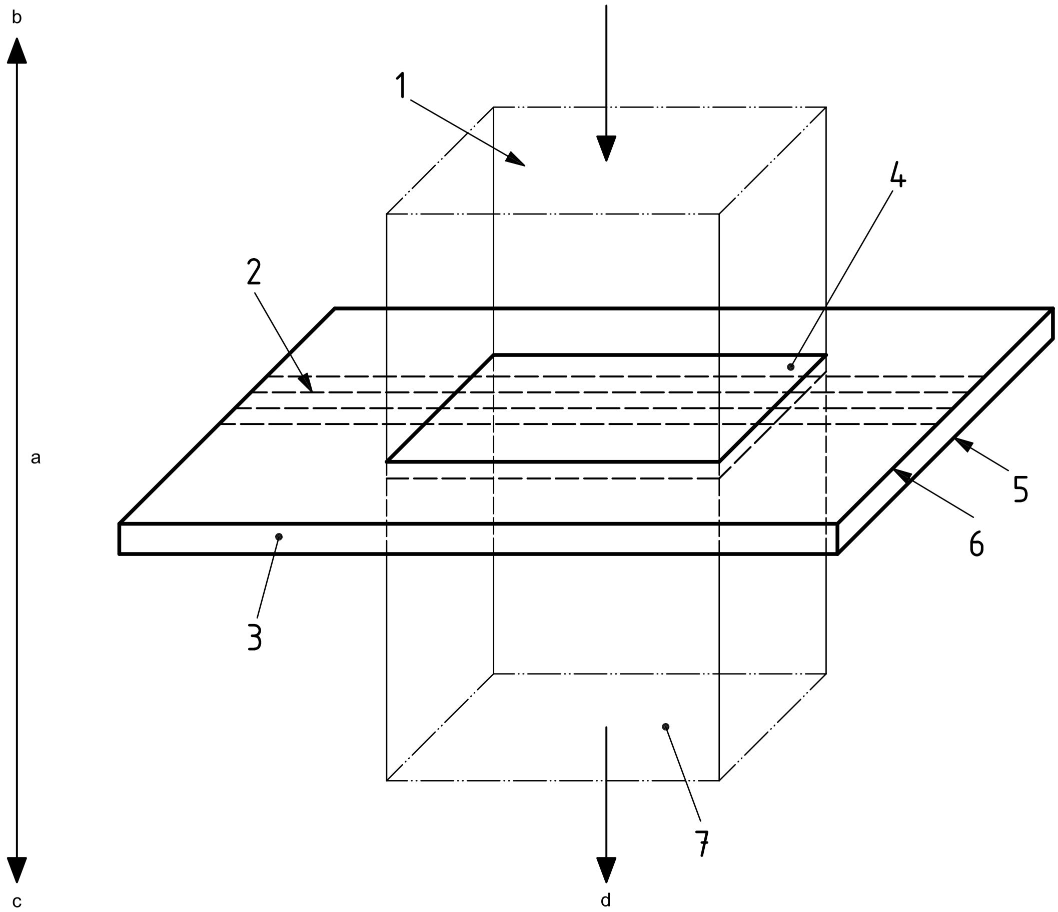
3.8
special paper
paper with modified air permeability

NOTE Papers of this type include those defined in paragraphs 3.5, 3.6 and 3.7.

4 Principle

A test piece is held in a suitable device. A pressure difference is applied across the test piece. The resultant flow of air through the test piece is measured.

The principle of measurement is illustrated in Figure 1.



Key

1 air stream
2 perforated zone (if present)
3 test piece
4 test surface of area (2 cm^2)
5 inner surface
6 outer surface
7 air stream

[a] Pressure difference.
[b] High pressure.
[c] Low pressure.
[d] Direction of air flow.

Figure 1 — Principle of measurement

The air flow through the test piece may be produced by applying a positive or negative pressure to one side of the test piece. The direction of air flow through the test piece shall be that which would occur when the sample is used in the finished product, where known, i.e. from the outside face towards the inside face.

If the air flow is produced by a positive pressure, the apparatus used should incorporate a filter which protects the test sample from contamination by oil, water and particles.

NOTE 1 With certain materials, the flow through the test piece can exhibit a non-linear relationship with the applied pressure drop. Thus the air flow through the test piece is determined at two pressure differences to establish whether the flow/pressure relationship across the paper is linear or non-linear. If it is non-linear, a second measurement of air flow is recorded at 0,25 kPa to fully characterize the material.

NOTE 2 Depending upon whether the volumetric air flow rate is measured up-stream or down-stream of the test piece, a difference of approximately 1 % of the flow rate can exist either side of the theoretical value at the centre of the test piece.

5 Apparatus

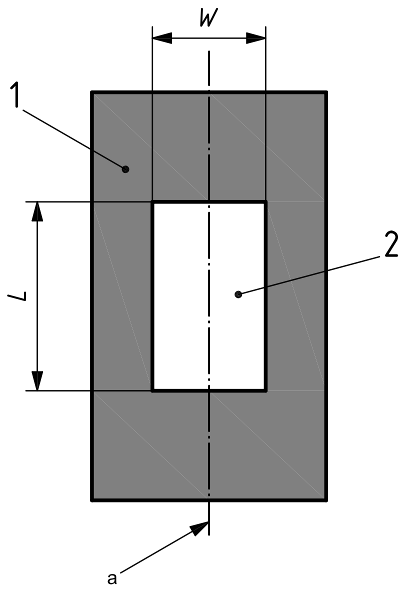
5.1 Test piece holder, for clamping the test piece, free from leaks.

5.1.1 For papers with uniformly distributed permeability and those with oriented or discrete permeable zones: the test piece holder has a rectangular surface area of 2,00 $cm^2 \pm 0,02\ cm^2$ with corner radii no greater than 0,1 cm. The long side, L, shall have a length of 2,000 cm ± 0,005 cm (see Figure 2).

5.1.2 For bands of differing permeability: the test piece holder has a rectangular surface area of 0,30 $cm^2 \pm 0,01\ cm^2$. The short side shall have a length of 2,00 mm ± 0,05 mm [see 7.5.6 and Figure 3 d)].

NOTE 1 The positioning of the test piece holder on the test piece differs for different types of papers (see 7.5 and Figures 2 and 3).

NOTE 2 An estimate of the air permeability of other speciality papers, outside the scope of this International Standard, may be required. In this case, specialized test piece holders with different surface areas might be necessary.



Key

1 test piece

2 measurement surface area of test piece holder

L is the long side of the test surface (see 5.1.1)

W is the width of the test surface

a Centreline of test piece.

Figure 2 — Positioning of test pieces for materials with uniformly distributed permeability

5.2 Pneumatic controller, to produce an air flow at a given but adjustable pressure difference between the two mating faces of the test piece holder.

5.3 Pressure gauge, suitable for measuring pressure differences to at least 0,001 kPa, having a relative error of no more than 2 % of the measured value within the measuring range.

5.4 Flow meter, suitable for measuring the air flow with a relative error no greater than 5 % of the measured value within the measuring range.

5.5 Conditioning enclosure, capable of maintaining the conditions given in ISO 187 (but see 7.3).

6 Sampling

Take a sample that is representative, on a statistical basis, of the population to be characterized.

Samples shall be free of visible defects and creases that may impair measurement performance.

7 Procedure

7.1 General

Since the pressure versus flow relationship of many papers is not linear, this procedure should be followed closely to allow proper comparison of results. If it is necessary to deviate from this procedure in any way (for example, to use a non-standard size of test piece holder or to modify the positioning of the test piece holder, due to sample dimensions) then this shall be noted in the Test Report [see 7.5 and 10 d)].

7.2 Leak check of the test piece holder

Follow the procedure given in Annex A. Perform a leak check daily, prior to use.

Air leaks between the mating faces of the test piece holder shall not be greater than 2,0 $cm^3 \cdot min^{-1}$.

Some users require determination of the effect of surface leakage through particular papers that contribute to the measured flow. In this case, if a value for leakage with the test piece in place is required, the procedure given in Annex C may be used. This should be determined and referred to in the test report.

7.3 Preparation of test pieces

Select at random from the sample, taken in accordance with Clause 6, the number of test pieces required for the test plus an additional three test pieces to be used as described in 7.6.1, Note 2.

If necessary make the test pieces suitable for testing (cut to the required dimensions, eliminate folds, seams, etc.).

Condition the test pieces, prior to measurement, in a conditioning enclosure set in accordance with ISO 187. Samples shall be held such that the conditioning air has free access to all their surfaces.

IMPORTANT — In laboratories unable to use the conditions given in ISO 187, the conditions given in ISO 3402 may be used. In these cases, a note shall be included with the test report.

NOTE Complete sample bobbins, where it is not possible to expose all surfaces to the conditioning atmosphere, might require an extended period of conditioning. The time required should be determined by practice and experience.

The period of time for conditioning is not given in this International Standard but the period of time retained should be reported with the results.

7.4 Calibration

Calibrate the instrument using the calibration standards and procedure referred to in Annex B.

7.5 Insertion of a test piece

7.5.1 General

All papers shall be placed in the test piece holder so that the measuring air will travel from the outside face towards the inside face of the paper as it is applied in the construction of the finished product, where this is known.

The positioning of the test pieces in the test piece holder is illustrated in Figures 2 and 3 (see also 5.1).

7.5.2 Materials with uniformly distributed permeability

Place in such a way that, if possible, the centre of the smaller dimension, W, of the test surface is at the centre of the width of the test piece (see Figure 2).

7.5.3 Materials with a narrow and oriented permeable zone

The permeable zone shall be oriented along, and parallel to, the direction of the 2 cm length of the test surface [see Figure 3 a)].

The edges of the permeable zone shall not be less than 1 mm from the edges of the test surface. Ideally, the test piece should extend over each edge of the test surface by at least 3 mm. If, for technical reasons, this cannot be achieved (i.e. the specimen under study is less than 16 mm total width or the permeable zone is less than 4 mm from one edge of the sample), this shall be referred to in the test report.

7.5.4 Materials with an extended and oriented permeable zone

The test piece holder shall be placed so that it covers the maximum possible width of the permeable zone and exposes the maximum possible permeable zone within the measurement surface area [see Figure 3 b)].

Ideally, the dimension L of the test surface shall extend at least 1 mm outside the edges of the permeable zone and the sample should extend over each edge of the test surface by at least 3 mm. Where this cannot be achieved (e.g. due to sample dimensions), this shall be referred to in the test report.

7.5.5 Materials with discrete permeable zones

The test piece shall be oriented so as to expose the greatest possible number of the permeable zones within the measurement surface area of the test piece holder [see Figure 3 c)].

Ideally, the 2 cm dimension of the test surface shall extend at least 1 mm outside the edges of the permeable zones and the sample should extend over each edge of the test surface by at least 3 mm. Where this cannot be achieved (e.g. due to sample dimensions), this shall be referred to in the test report.

7.5.6 Materials with bands of different air permeability

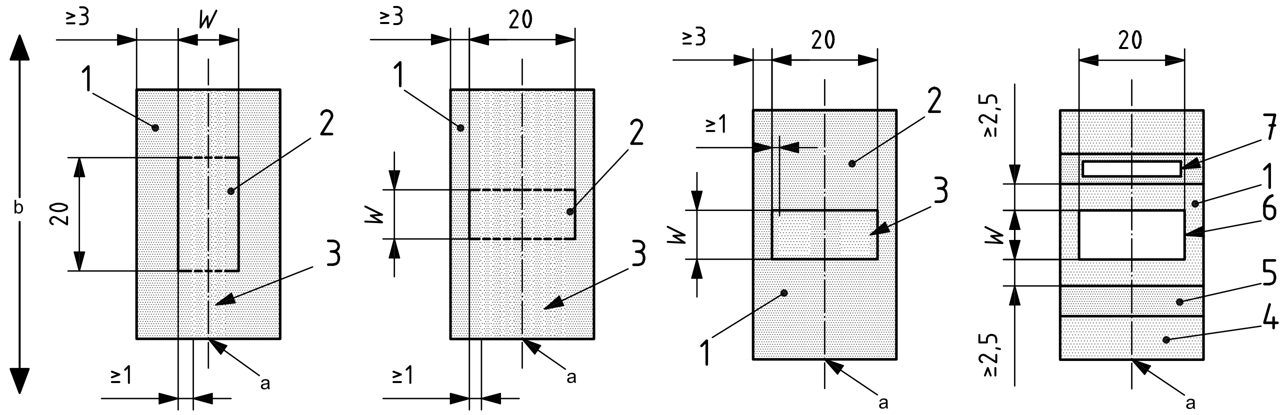
For measurement of the permeability of the bands, a 0,30 cm^2 test piece holder shall be used.

The test piece holder shall be oriented so that the long side of the test surface is parallel to the band and shall be positioned, as far as is practical, to centre the test surface within the band [see Figure 3 d)].

For measurement of the permeability of the base paper, a 2,00 cm^2 test piece holder shall preferably be used.

The test piece holder should be positioned between the bands so that the clamping face of the holder is at least 2,5 mm from the bands. It should be oriented so that the 2 cm dimension is parallel to the bands [see Figure 3 d)]. Where this is not possible, the size and orientation of the test piece holder used shall be noted in the test report.

Dimensions in millimetres



a) Narrow, oriented permeable zone (see 7.5.3)

b) Oriented permeable zone (see 7.5.4)

c) Discrete permeable zone (see 7.5.5)

d) Bands of different permeability (see 7.5.6)

Key

1 test piece

2 measurement surface area of test piece holder

3 permeable zone

4 base material with higher permeability (non-banded area)

5 band of modified permeability

6 position of test piece holder for determining *AP* of base material (using preferably 2,00 cm^2 test piece holder)

7 position of test piece holder for determining *AP* of band (using 0,3 cm^2 test piece holder)

[a] Centreline of test piece.

[b] Running direction of papers.

Figure 3 — Positioning of test pieces for materials with an oriented or discrete permeable zone and for papers with bands of different permeability

7.6 Measurement

7.6.1 General

Insert a test piece into the test piece holder. Establish an approximate pressure difference within the range 1,00 kPa ± 0,05 kPa across the two faces of the test piece. Accurately record this pressure and the corresponding flow rate.

NOTE 1 The permeability of test pieces can vary throughout their length. For this International Standard, the mean value of ten individual measurements is used to determine the value of air permeability of a test piece. In practice, laboratories often take a different number of measurements depending upon the application of the measurement.

Proceed in the same way with all the test pieces. The results are normalized as given in Clause 8.

NOTE 2 If further characterization of the material is required because it is believed that the flow-pressure relationship is non-linear, perform the following test for air flow rate-pressure relationships on three additional test pieces.

Set up, in turn, pressure differences of 0,25 kPa and 1,00 kPa across the test material, without moving the test material. Record the corresponding air flow rates Q_1 and Q_2 (in cubic centimetres per minute) across the test material. Calculate the ratio Y using Equation (1):

$$Y = Q_1/Q_2 \times 1{,}0/0{,}25 \quad (1)$$

Repeat the above procedure on two other test pieces and calculate the mean of the three values obtained for the value Y. If the mean value of Y does not deviate by more than 2 % from the value 1,00 (in practice if it is no greater than 1,02), the air flow rate-pressure relationship is **linear**. Otherwise, the relationship is referred to as **non-linear**.

If the test material has been shown to have non-linear air flow rate-pressure characteristics, the measurement of air flow rate at a single pressure difference is considered inadequate to characterize the material. The flow rate may be determined using the second pressure difference of 0,25 kPa.

Further information is given in Annex D.

NOTE 3 Materials that exhibit a linear characteristic and have a permeability of less than 10 $cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa may be re-measured in order to obtain an estimate of permeability using:

— a test piece holder with a single larger test surface area,

— a test piece holder containing multiple areas that perform simultaneous measurements of the standard single 2,00 cm^2 rectangular test surface area, each with dimensions described in 5.1, and

— a pressure drop of 2,0 kPa.

In this case, the method gives only the estimation of permeability.

7.6.2 Measurement of strips

Make ten consecutive measurements with a minimum distance of 20 mm between measurements.

7.6.3 Measurement of spills (papers recovered from manufactured products)

Make ten measurements comprising single measurements on each of ten spills. Ensure that the overlap seam is not included in the test surface.

8 Expression of results

The determination of the value of air permeability shall be the mean value from the individual measurements (see 7.6.2 and 7.6.3).

NOTE If a measuring head with multiple test surfaces is used as described in 7.6.1, Note 3, it should be understood that the measurement obtained is already an average of the number of test surfaces used in the measuring head. In addition, care has to be taken in the interpretation of r and R (see 9.1 and 9.2) when using these measuring heads.

The air permeability, AP, is expressed in cubic centimetres per minute per square centimetre measured at 1 kPa. Using a test surface of area 2 cm^2, it is given by Equation (2):

$$AP = Q/2 \qquad (2)$$

where Q is the numerical value of the air flow, in cubic centimetres per minute, passing through the test piece.

In practice, Q is not measured at precisely 1 kPa and a normalization procedure to correct to 1 kPa is required. In addition, other measuring heads, with areas other than 2 cm^2 may have been used (see 7.6.1, Note 3) and a correction for this is then also required.

The general equation is as given in Equation (3):

$$AP = \frac{Q}{A} \times \frac{p}{\Delta p} \qquad (3)$$

where

p is the numerical value of the pressure (1 kPa);

A is the numerical value of the surface area, in square centimetres, of the test piece subjected to testing;

Δp is the actual measurement pressure difference, in kilopascals, across the two surfaces of the test piece.

9 Precision

9.1 Repeatability

The difference between two single results found on matched test samples by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value, r, on average not more than once in 20 cases in the normal and correct operation of the method.

9.2 Reproducibility

Single results on matched test samples reported by two laboratories will differ by more than the reproducibility value, R, on average not more than once in 20 cases in the normal and correct operation of the method.

NOTE In practice, better values for reproducibility may be obtained when identical experimental conditions are used between client and supplier (in particular when common standards are used).

9.3 Results of an international collaborative study (Study 1)

A major international collaborative study was conducted in 1994, involving 24 laboratories and six samples of cigarette papers, filter plug wrap and filter joining paper. This study also included samples having an oriented permeable zone. The study showed that when these papers are measured in accordance with this method, the following values for repeatability, r, and reproducibility, R, were obtained.

Table 1 — Repeatability and reproducibility limits for Study 1

Mean air permeability [$cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa]	**Repeatability limit** r	**Reproducibility limit** R
26,9	2,37	6,01
49,2	4,15	8,37
221	17,4	26,3
1 334	96,6	133
2 376	281[a]	326
21 449	1 182	2 077

[a] See 9.4.

For the purpose of calculating r and R, one test result is defined as the mean value obtained from ten measurements from a single paper strip or the mean value obtained from ten separate paper spills removed from manufactured products.

The values for r and R given in Table 1 may only be valid for the particular papers used. It is not practical, in the context of collaborative studies, to conduct repeat tests on the same test piece. Therefore, the inhomogeneity in the test pieces contributes to the within-laboratory variance. This situation was referred to in ISO 5725:1986[1)] as quoted below.

"When tests have to be performed on solid materials that cannot be homogenized (such as metals, rubber or textile fabrics) and when the tests cannot be repeated on the same test piece, inhomogeneity in the test material will form an essential component of the precision of the measurement and the idea of identical material no longer holds good. Precision experiments can still be carried out, but the values of r and R may only be valid for the particular material used and should be quoted as such. A more universal use of r and R will be acceptable only if it can be demonstrated that the values do not differ significantly between material produced at different times or by different producers. This would require a more elaborate experiment than has been considered in this International Standard."

From the data obtained in the collaborative experiment, it is possible to estimate the within-laboratory component of variance with the day-to-day and strip-to-strip components of variance removed. This within-laboratory component of variance can then be used to derive alternative values for repeatability. These and the corresponding values for reproducibility are shown in Table 2

Table 2 — Alternative repeatability and reproducibility limits for Study 1

Mean air permeability [$cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa]	**Repeatability limit** r	**Reproducibility limit** R
26,9	1,57	5,72
49,2	3,12	7,89
221	11,7	22,9
1 334	45,2	95,1
2 376	249[a]	297
21 449	519	1 773

[a] See 9.4.

These values have been adjusted to be equivalent to values that might have been obtained from a similar analysis of the mean of ten readings replicated within a single strip.

9.4 Statistical discussion of r and R results for Study 1

From the results of the analysis shown in Tables 1 and 2 it can be seen that, in general, both r and R, when compared as a percentage of their mean value, are highest for the lower permeability papers, with a tendency for r and R, when expressed as a percentage of the mean, to decrease as the mean permeability increases.

However, the paper with footnote a in Tables 1 and 2 exhibits results that do not conform to this trend. Examination of Table 1 shows that the high value of R % for this paper (when compared to the other papers) is entirely due to the high value of the within-laboratory variability for this paper. There is no evidence to suggest that the between-laboratory variability is any higher for this paper (in terms of the percentage of the mean) than for any of the other papers tested in this study.

This was confirmed in the analysis by the within- and between-laboratory standard deviations. The values of the within-laboratory percentage standard deviation of the mean display the same pattern as the values of r %

1) International Standard withdrawn.

(as expected) but the values for between-laboratory percentage standard deviation of the mean, do not indicate an unexpectedly high value for this paper.

The results for this paper demonstrate that the values for r and R obtained from this study may only be applicable to the papers tested in this study.

9.5 Results of an international collaborative study (Study 2)

A second international inter-laboratory collaborative study was conducted in 2005 to estimate the r and R values for special cigarette and tipping papers, having artificially induced permeability by means of oriented, extended or discrete zones of perforation and banded papers. This study also included standard (naturally permeable) cigarette and plug wrap papers, allowing comparison with the results previously obtained. For each paper type, one measurement is the average of ten individual readings made at different positions on one strip of the paper. Five replicate measurements were made on different days using a new sample selected from each paper type.

Table 3 — Repeatability and reproducibility limits for Study 2 — Papers with low permeability

Sample description	Mean AP [$cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa]	Repeatability limit r	Reproducibility limit R	r	R
				as a percentage of mean AP	
Banded cigarette paper	5,52	3,97	5,13	71,92	92,93
Naturally permeable cigarette paper	31,75	3,30	3,70	10,45	11,72
Cigarette paper with extended permeable zone	99,00	8,78	17,66	8,87	17,84
	202,00	9,02	13,78	4,46	6,82
Cigarette paper with discrete permeable zones	341,79	34,46	40,18	10,08	11,76
	744,30	48,61	67,56	6,53	9,08
Filter joining paper	1 013,90	44,42	73,02	4,38	7,20
	3 709,80	141,00	533,08	3,80	14,37
Filter plug wrap	11 171,14	1 423,69	1 782,06	12,74	15,95

The values for r and R given in Table 3 may only be valid for the particular papers used. It is not practical, in the context of collaborative studies, to conduct repeat tests on the same test piece. Therefore, the inhomogeneity in the test pieces contributes to the within-laboratory and between-laboratory variances.

In an attempt to minimize the impact of the sample variability, a second inter-laboratory test was conducted. For each paper type, one measurement is the average of ten individual readings made at different positions on one strip of the paper. In this case, each laboratory measured a selected sample of each paper type on five different days. The sample was selected and marked so that each replicate measurement could be made at an identical position to the first measurement. Hence, within each laboratory, the samples used for each replicate were nominally identical and hence the r values obtained for each paper type were much lower than those obtained in the initial study.

NOTE 1 On a routine basis, a sample should not be measured more than once at the same position because of the risk of sample damage. However, for this test, special instructions and care were applied to avoid such damage, thus allowing the sample variation between replicates to be minimized.

The results of this second inter-laboratory study are given in Table 4.

NOTE 2 Banded cigarette paper was excluded from this type of trial because of the difficulties experienced when attempting to measure this paper several times at one position on the paper.

Table 4 — Repeatability and reproducibility limits for Study 2 — All papers

Sample description	Mean AP [cm^3·(min^{-1}·cm^{-2}) at 1 kPa]	Repeatability limit r	Reproducibility limit R	r as a percentage of mean AP	R as a percentage of mean AP
Naturally permeable cigarette paper	30,99	0,49	1,47	1,58	4,74
Cigarette paper with extended permeable zone	100,37	1,04	18,18	1,04	18,11
	208,69	2,92	45,96	1,40	22,02
Cigarette paper with discrete permeable zones	347,89	6,49	17,50	1,87	5,03
	754,35	13,46	42,23	1,78	5,60
Filter joining paper	1 006,50	9,28	26,85	0,92	2,67
	3 718,39	38,16	475,68	1,03	12,79
Filter plug wrap	10 710,06	122,91	833,22	1,15	7,78

9.6 Statistical discussion of r and R results for Study 2

From the results of the analysis shown in Table 3 it can be seen that, in general, both r and R, when compared as a percentage of their mean value, are highest for the lower permeability papers, with a tendency for r and R, when expressed as a percentage of the mean, to decrease as the mean permeability increases. Filter plug wrap and low permeability, discrete zone cigarette paper are exceptions to this trend.

From the analysis of the results of the second study, shown in Table 4, the r values, expressed as a percentage of the mean permeability value, are seen to be approximately 1 % for all papers. This value is closer to the actual repeatability of the method (but still includes some component of sample variability due to differences in AP along a sample strip).

The R values show considerable differences between paper types. These results indicate that a major proportion of the within-laboratory variation seen in Table 3 is due to the permeability variability between samples of the same type of paper. Further, the between-laboratory values support this view and show that some papers (particularly cigarette papers with extended permeable zones and high permeability filter joining paper) show a high variability between the samples measured at different laboratories.

NOTE For these series of inter-laboratory studies, all samples of one paper type were taken from one bobbin and were randomized before being distributed for the trial. Hence, all laboratories would be expected to receive nominally identical samples. High R values indicate larger sample-to-sample variability between the laboratories and hence a larger variability within the sample bobbin.

Sample-to-sample and within-sample variability can thus be seen to have a significant influence on the within- and between-laboratory variabilities for the measured AP and, as such, reinforces that the results quoted in Table 3, and the R results in Table 4, may only be directly applicable to the actual samples measured.

10 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, 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. Specifically, the test report shall contain the following:

a) date of sampling and sampling method;

b) identification and full description of the material tested, stating the properties (e.g. nature, width) of samples having oriented zone permeability;

c) date of testing;

d) precise and full measuring conditions (particularly if blowing or sucking has been used) and those deviating from this International Standard or any events that are likely to have affected the results;

e) the conditioning atmosphere and period of time for conditioning;

f) barometric pressure at the time of testing;

g) results in air permeability (AP) units [$cm^3 \cdot (min^{-1} \cdot cm^{-2}$) at 1 kPa];

h) elementary statistics relating to the results:

— number of measurements;

— mean and standard deviation values.

Annex A
(normative)

Leak testing of test piece holder

A.1 General

The performance testing of instruments for measuring air permeability of materials used as cigarette papers, filter plug wrap and filter joining paper (including materials having an oriented permeable zone) shall be conducted in accordance with manufacturers' instructions.

However, this annex describes a general test for assessing the value of any leakage (see 3.3) of air between the mating faces of the measuring head assembly.

A.2 Procedure

Seal the air flow path to the atmosphere from the measuring head assembly.

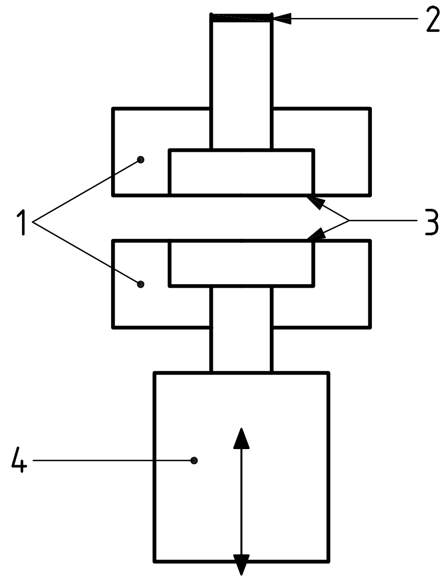
Operate the instrument in the normal manner to make an air permeability measurement ensuring that no sample has been placed between the mating faces of the test piece holder clamp assembly.

Record the leakage rate as indicated by the instrument. The mating faces of the measuring head clamp assembly shall create a seal, such that a flow measurement of no greater than 2 $cm^3 \cdot min^{-1}$ is recorded.

Repeat this procedure five times. If any value is greater than 2 $cm^3 \cdot min^{-1}$, the clamp assembly is deemed to be defective.

The readings shall be noted and reported with results of each test.

The principle of measuring the test piece holder clamp assembly leakage is illustrated in Figure A.1.



Key

1 test piece holder

2 sealed air flow path to atmosphere

3 sealing faces

4 air flow measuring device

Figure A.1 — Leak testing of test piece holder

Annex B
(normative)

Calibration of air permeability standards and air permeability measuring instruments

B.1 Essential properties of calibration standards

Air permeability calibration standards are used to calibrate measuring instruments for the determination of air permeability of materials used as cigarette papers, filter plug wrap and filter joining paper (including materials having discrete or oriented permeable zones and materials with bands of differing permeability).

The calibration standard shall have a known and repeatable value of volumetric air flow as measured at the exit of the standard, when subjected to a specified (1 kPa) static pressure. The flow-pressure characteristic of the standard shall remain constant and shall be largely unaffected by changing atmospheric conditions.

The calibration standard shall be inscribed with the value of volumetric air flow, at 1 kPa, corrected and, where necessary, compensated to the standard conditions of 22 °C and 1 013 hPa, quoted to a minimum accuracy of 0,5 %.

NOTE 1 Compensation for the effects of atmospheric pressure will be necessary if the calibration standard has a non-linear pressure-flow characteristic.

NOTE 2 The linearity of a calibration standard may be confirmed by measuring the flow at a differential pressure of approximately 0,75 kPa and 1,25 kPa. A standard may be considered to be linear when factor k (see Equation D.6) is greater than 0,95.

NOTE 3 See Annex E for guidance on the compensation of non-linear standards.

The precise construction of these calibration standards depends upon the design of the air permeability meter in which they are to be used.

The calibration standards shall be supplied with unique serial numbers and a traceable certificate of calibration (see B.6)

B.2 Procedure for calibration of standards

B.2.1 General

The laboratory testing atmosphere shall be controlled in accordance with ISO 187. In laboratories unable to use the conditions given in ISO 187, the conditions 22 °C ± 1 °C and (60 ± 2) % RH given in this International Standard and ISO 3402 may be used. The conditions used shall be stated in the certificate of calibration supplied with the standard.

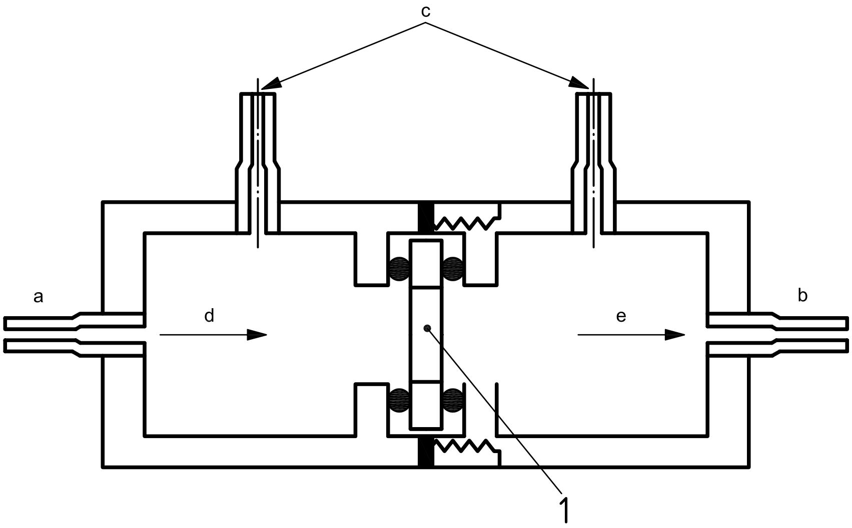
The standard shall be held in a calibration test holder, the mechanical arrangement of which shall not alter the characteristics of the standard.

The air flow through the calibration standard may be produced by applying a positive or negative pressure, for blowing or sucking instruments respectively, to one side of the calibration holder. The direction of air flow through the calibration standard shall be that which would occur when it is used to calibrate an air permeability measuring instrument.

The ambient atmospheric conditions and the volumetric air flow, temperature and pressure shall be measured at the exit of the calibration holder containing the standard. Depending upon the type and operation of gas calibrator employed and the intrinsic properties of the calibration standard, appropriate mathematical corrections shall be applied to correct the volumetric air flow to the standard conditions of 22 °C and 1 013 hPa.

NOTE Correction and compensation of the volumetric air flow is discussed in Annex E.

A schematic diagram of a typical calibration holder is shown in Figure B.1.



Key

1 calibration standard

a High pressure.

b Low pressure.

c Measuring pressure difference.

d Air in.

e Air out.

Figure B.1 — Apparatus for calibration of standards (schematic)

B.2.2 Method 1

The flow shall be adjusted such that a constant pressure of 1,000 kPa ± 0,005 kPa is applied across the calibration standard. Using a gas calibrator that does not generate a systematic influence on the flow measurement, measure the volumetric flow at the exit of the standard and the temperature and pressure of the air in the calibrator.

Repeat this procedure five times with each calibration standard to be calibrated. The value to be ascribed to the calibration standard shall be the mean of the five volumetric flow rates at standard conditions.

B.2.3 Method 2

Adjust the flow such that a constant pressure is maintained, in turn, at 5 % to 10 % above 1 kPa and at 5 % to 10 % below 1 kPa. At each point, the corresponding pressure drop across the calibration standard shall be recorded to the nearest 0,005 kPa. Using a gas calibrator that does not generate a systematic influence on the flow measurement, measure the volumetric flow at the exit of the standard and the temperature and pressure of the air in the calibrator.

A minimum of two measurements shall be made at each flow setting. The value to be ascribed to the calibration standard is the interpolated value of volumetric air flow at standard conditions when the pressure drop across the standard is at 1,000 kPa.

B.3 Calibration of instruments

The calibration and performance testing of instruments for measuring the air permeability of materials used as cigarette papers, filter plug wrap and filter joining paper (including materials having an oriented permeable zone) should be conducted in accordance with manufacturers' instructions.

B.4 Principle

To obtain the best accuracy, the instrument shall be calibrated over its specified range of measurement. Calibration shall be undertaken at measurement values corresponding to individual transducing elements used to achieve the instrument's measurement range.

B.5 Procedure

Follow the procedure given in the instrument manufacturer's instructions. A typical procedure is as follows.

— Install the calibration standard and allow it to equilibrate to the temperature of the measuring air.

— Connect a reference manometer into the measuring circuit to monitor the pressure drop applied across the calibration standard. The maximum relative error of the reference manometer shall be less than 0,5 % for any measured value.

— Establish an approximate pressure difference within the range of 1,0 kPa ± 0,1 kPa across the calibration standard.

— Adjust the instrument's measurement system to display the exact value indicated on the reference manometer.

— Disconnect the reference manometer and seal the connection point.

— Adjust the pressure drop across the calibration standard to 1,000 kPa ± 0,005 kPa and adjust the instrument's measurement system to display the value inscribed on the calibration standard.

— Repeat the above step for each of the calibration standards.

— Return the instrument to its measurement mode and make an air permeability measurement on each of the calibration standards to check that the measurement is within the tolerances of calibration of the calibration standards and the measurement specification of the instrument.

B.6 Calibration certificate

Each air permeability calibration standard shall be supplied with a calibration certificate that gives the unique reference number and assigned value for the standard. Additionally, the calibration certificate shall include information concerning the conditions of calibration and any calculations made in correcting or compensating the measured air flow value to the assigned value (under standard atmospheric conditions).

The certificate shall include all information necessary for the user to identify and apply the calibration standard, including (but not limited to) the following:

- ambient temperature, relative humidity and atmospheric pressure at the time of calibration;
- the volumetric air flow, gas temperature and pressure at the exit to the standard;
- the differential pressure across the standard at the time of calibration;
- the assigned (corrected and/or compensated) value of volumetric air flow;
- the differential pressure at which the assigned value applies;
- the standard atmospheric conditions to which the assigned air flow has been corrected;
- the correction and compensation formulae used with full explanation of any symbols used;
- the date of calibration;
- calibration technician's identification/name.

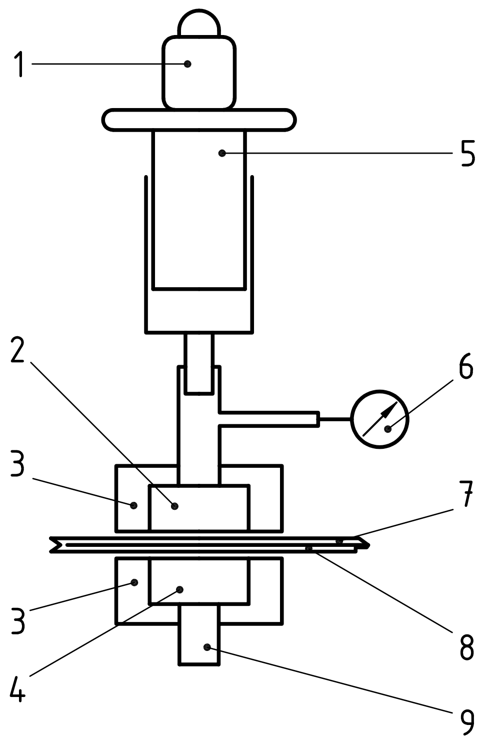
Annex C
(informative)

Determination of relevant surface leakage of test piece in the test piece holder

C.1 General

Surface leakage is aspirated from the surrounding atmosphere or escapes into it, unintentionally, past the sealing surface of the test piece holder.

The principle of measurement of relevant surface leakage is illustrated in Figure C.1.



Key

1	applied weight	6	pressure-measuring device
2	inlet cavity	7	test material
3	test piece holder	8	non-permeable membrane
4	outlet cavity	9	outlet orifice
5	syringe		

Figure C.1 — Principle of measurement of surface leakage

C.2 Procedure

Determination of the relevant surface leakage can be carried out as follows.

— Connect a calibrated syringe to the inlet side of the test piece holder.

— Connect a manometer to the junction of the syringe and the inlet side of the test piece holder, ensuring that all connections are air-tight.

— Insert a sample of the test material to be measured and a non-permeable membrane covering the whole test area, including the sealing surfaces, in the test piece holder. Ensure that the test material faces the inlet cavity of the test piece holder. The non-permeable membrane ensures that only that part of the total leak relevant to the determination of air permeability is considered.

— Close the test piece holder and weight the syringe to apply a pressure of approximately 1 kPa to the upper cavity of the test piece holder.

— Measure the leakage flow by timing the change in position of the piston in the syringe. A suitable length of time should be chosen such that an accurate assessment of surface leakage is possible.

— The pressure at the inlet side of the test piece holder should be monitored throughout this time and should remain close to 1 kPa.

— Any change in pressure may indicate unacceptable resistance in the syringe, in which case the test should be repeated.

NOTE This test can also be performed omitting the non-permeable membrane and sealing the outlet of the test piece holder.

Annex D
(informative)

Flow of air through porous materials

D.1 Theoretical considerations

The flow of air through a porous material is dependent on both viscous and inertial forces in the flowing air. The total flow of air through the material may be expressed as given in Equation (D.1):

$$Q = ZA\Delta p + Z'A\Delta p^n \quad \text{(D.1)}$$

where

Q is the total air flow in cubic centimetres per minute;

A is the area of the material, in square centimetres, exposed to the flowing air;

Δp is the pressure difference across the material in kilopascals;

Z is the component of air permeability of a porous material due to viscous forces, in cubic centimetres per minute, per square centimetre at one kilopascal;

Z' is the component of air permeability of a porous material due to inertial forces, in cubic centimetres per minute, per square centimetre at one kilopascal by n;

n is a constant whose value lies between 0,5 and 1,0, and is dependent on the size distribution of the spaces/holes in the material through which the air flows.

The general form of Equation (D.1), as written, has a non-linear relationship between air flow, Q, and pressure difference, Δp. Since the air permeability of material is defined as the flow of air through 1 cm^2 of the material when the pressure difference across it is 1,00 kPa, then from Equation (D.1) the “total air permeability” of the material is equal to ($Z+ Z'$).

Two extreme forms of Equation (D.1) can be considered.

a) With porous cigarette paper, for example, spaces in the material are small (typically 1 µm wide) compared to paper thickness (20 µm to 40 µm). Inertial forces in the air flow are negligible, $Z' = 0$, and Equation (D.1) reduces to Equation (D.2):

$$Q = ZA\Delta p \quad \text{(D.2)}$$

In this case, the relationship between air flow, Q, and pressure difference, Δp, is linear.

b) With perforated tipping paper, for example, the diameter of the perforation holes may be large (e.g. above 100 µm) compared to the paper thickness (e.g. 40 µm). In this case n = 0,5 and Equation (D.1) becomes quadratic, as given in Equation (D.3):

$$Q = ZA\Delta p + Z'A\sqrt{\Delta p} \quad \text{(D.3)}$$

If there are no spaces in the tipping paper, other than the perforation holes, then Z = 0 and Equation (D.3) reduces to Equation (D.4):

$$Q = Z'A\sqrt{\Delta p} \quad \text{(D.4)}$$

D.2 Characterization of materials having a non-linear air flow/pressure relationship

If the test material has been shown to have non-linear air flow rate/pressure characteristics, the values of Z, Z' and n can be calculated using the above equations from a regression of values of Q determined at a series of values of Δp.

As a minimum, the material should be characterized by two values of air flow rate, determined for pressure differences of 0,25 kPa and 1,00 kPa.

A more general form of Equation (D.1) is given in Equation (D.5):

$$Q = Z_T A \, \Delta p^k \qquad \text{(D.5)}$$

where

Z_T is the total air permeability of the paper;

k is a constant whose value lies between 0,5 and 1,0, depending on the size distribution of the spaces/holes in the material through which the air flows.

Q, A, Δp have the same meanings as in Equation (D.1).

The constant k can be determined using Equation (D.6) if the resulting air flows are measured for two different measuring pressures:

$$k = \frac{\lg \frac{Q_1}{Q_2}}{\lg \frac{p_1}{p_2}} \qquad \text{(D.6)}$$

where

Q_1 is the air flow, in cubic centimetres per minute, measured at pressure p_1;

Q_2 is the air flow, in cubic centimetres per minute, measured at pressure p_2.

For small differences between actual and nominal measuring pressure, the air flow may be calculated by means of the following equation without giving rise to appreciable error:

$$Q_1 = Q_2 (p_1/p_2)^k \qquad \text{(D.7)}$$

Annex E
(informative)

Compensation of calibration standards

E.1 Linear and non-linear standards

E.1.1 General

Air permeability calibration standards, as described in Annex B, require marking with the volumetric air flow obtained under standard atmospheric conditions and at a differential pressure of 1 kPa. When measured under non-standard conditions, some mathematical correction must be applied to the measured flow value to achieve this. The type of correction required depends on the nature of pressure-flow characteristic for the standard.

E.1.2 Standards with linear pressure-flow characteristic

These are dominated by viscous effects and have pressure-flow characteristics given by Equation (E.1):

$$\Delta p = \alpha \times Q \qquad \text{(E.1)}$$

where

Δp is the differential pressure across the standard;

Q is the volumetric flow through the standard;

α is the constant.

E.1.3 Standards with non-linear pressure flow characteristics

These may have various pressure-flow characteristics that may be generally represented by Equation (E.2):

$$\Delta p = (\alpha \times Q) + (\beta \times Q^n) \qquad \text{(E.2)}$$

where

Δp is the differential pressure across the standard;

Q is the volumetric flow through the standard;

α depends upon the air viscosity and the exact construction of the standard;

β depends upon the air density and the exact construction of the standard;

n is a value depending upon the construction of the standard.

Hence, the volumetric flow is dependent on both viscous and inertial forces in the flowing air.

E.2 Correction and compensation of measured volumetric air flows

E.2.1 Linear standards

These may be simply corrected by applying corrections for the differences in atmospheric temperature and pressure according to the perfect gas law, as given in Equation (E.3):

$$Q_s = Q_m \times \left(\frac{P_s}{P_{atm}} \times \frac{T_{gas}}{T_s} \right) \quad \text{(E.3)}$$

where

Q_s is the volumetric flow corrected to standard conditions;

Q_m is the volumetric flow under measurement conditions;

P_s is the standard atmospheric pressure (as defined by ISO 187 or ISO 3402);

P_{atm} is the atmospheric pressure at time of measurement;

T_{gas} is the gas temperature at time of measurement;

T_s is the standard temperature (as defined by ISO 187 or ISO 3402).

E.2.2 Non-linear standards

The correction and compensation of volumetric air flow of non-linear standards depends upon the exact nature of the standard. For the purposes of illustration, compensation for standards made from porous materials (for example sintered glass or metal beads) will be considered here.

Porous media exhibit a specific form of Equation (E.2):

$$\Delta p = (\alpha \times \eta \times Q) + (\beta \times \rho \times Q^2) \quad \text{(E.4)}$$

where

α and β are constants, dependent on the actual nature of the porous material;

η is the viscosity of the air flowing through the calibration standard;

ρ is the density of the air flowing through the calibration standard.

This equation may be rearranged into a linear relationship, as given in Equation (E.5):

$$\frac{\Delta p}{\eta Q} = \alpha + \beta \left(\frac{\rho Q}{\eta} \right) \quad \text{(E.5)}$$

The flow through the calibration standard should be measured at several different differential pressures and a plot made of $\frac{\Delta p}{\eta Q}$ against $\frac{\rho Q}{\eta}$.

The constants α (intercept) and β (slope) may be determined from the regression of this plot.

NOTE Values of air viscosity, η, and density, ρ, can be calculated from equations given in Reference [5].

The determined values of α and β characterize the flow behaviour of the calibration standard and can be used to compensate the measured flow under any particular conditions of temperature, pressure and relative humidity.

To achieve this, Equation (E.4) may be rearranged to give Equation (E.6) and solved for flow:

$$\beta \times \rho \times Q^2 + \alpha \times \eta \times Q - \Delta p = 0$$

Hence:

$$Q_s = \frac{-\alpha \times \eta_s + \sqrt{(\alpha \times \eta_s)^2 + (4 \times \rho_s \times \beta \times \Delta P)}}{2 \times \beta \times \rho_s} \qquad \text{(E.6)}$$

where

Q_s is the volumetric air flow compensated to standard atmospheric conditions;

η_s is the air viscosity under standard atmospheric conditions;

ρ_s is the air density under standard atmospheric conditions;

α and β are the previously determined characteristic constants for the calibration standard.

Bibliography

[1] ISO 5725:1986 [2], *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*

[2] CORESTA, Recommended Method No. 3:1976, *Determination of the air permeability of cigarette paper* [3]

[3] CORESTA, Recommended Method No. 18:1991, *Materials used as cigarette tipping papers having a perforated zone — Determination of unit air-flow and permeability*

[4] CORESTA, Recommended Method No. 40:1994, *Determination of air permeability of materials used as cigarette papers, filter plug wrap and filter joining paper including materials having an oriented permeable zone*

[5] RASMUSSEN, K., *Calculation methods for the physical properties of air used in the calibration of microphones*; Technical University of Denmark; Report PL-11b, 1997

2) International Standard withdrawn.

3) Method replaced by CORESTA Recommended Method No. 40.

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