



**Cooperation Centre for Scientific Research
Relative to Tobacco**

Physical Test Methods Sub-Group

**CORESTA Recommended Method
No. 77**

**DETERMINATION OF DIFFUSION
CAPACITY BY MEASUREMENT OF
CO₂ TRANSFER RATE THROUGH
MATERIALS USED AS CIGARETTE
PAPERS AND CIGARETTE PAPERS
HAVING AN ORIENTED ZONE OF
REDUCED DIFFUSION CAPACITY**

June 2019



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Status: Valid

Note:

Document history:

Date	Information
April 2014	Version 1
June 2019	Version 2 – Systematic review

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DETERMINATION OF DIFFUSION CAPACITY BY MEASUREMENT OF CO₂ TRANSFER RATE THROUGH MATERIALS USED AS CIGARETTE PAPERS AND CIGARETTE PAPERS HAVING AN ORIENTED ZONE OF REDUCED DIFFUSION CAPACITY

(June 2019)

0. INTRODUCTION

In 2010, ISO 12863 was published, which describes a standard test method for assessing the capability of a smouldering cigarette to extinguish when placed on a defined substrate [1]. It is known from several studies that the transfer of carbon monoxide (CO), carbon dioxide (CO₂) and oxygen (O₂) through the cigarette paper has a significant influence on the behaviour of cigarettes during smouldering and puffing and may therefore be related to the self-extinguishment of cigarettes in the test described by ISO 12863 [2, 3, 4]. Air permeability, the measurement of which is described by CRM N° 40 [5], has been shown to correlate with the transfer of CO or CO₂ through the cigarette paper and has previously been used to specify and characterise papers for low ignition propensity cigarettes. Further studies, however, have shown that the transfer rate of CO₂ as determined according to this CRM and the diffusion capacity calculated from the measured CO₂ concentration, correlate better with the results from the ISO 12863 test than air permeability measurements [6, 7, 8, 9, 10]. Consequently, the CORESTA Physical Test Methods Sub-Group has developed this CORESTA Recommended Method (CRM).

1. FIELD OF APPLICATION

This Recommended Method is applicable to the determination of the diffusion capacity of CO₂ into nitrogen (N₂) through paper materials used as cigarette papers, including materials having an oriented zone of reduced diffusion capacity. It includes paper materials from bobbins as well as from manufactured cigarettes.

2. NORMATIVE REFERENCES

ISO 187:1990, Paper, board and pulps - Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples.

3. DEFINITIONS

For the purpose of this CRM the following definitions apply.

3.1 Diffusion

Thermal motion of all molecules at temperatures above absolute zero. It explains the net flux of molecules from a region of higher concentration to one of lower concentration. The rate of this movement is a function of temperature, pressure, viscosity and the size and mass of the molecules.

3.2 Diffusion capacity

Capacity of a paper sample to allow CO₂ molecules to diffuse through a defined area of the sample into N₂ without any significant differential pressure across the sample.

3.3 Differential pressure

Difference in pressure between the two faces of the test piece during measurement.

3.4 Transfer rate

Amount per unit time of CO₂ molecules passing through a defined area of a paper sample into N₂ without any significant differential pressure across the sample.

4. PRINCIPLE

The sample to be tested is held in a measurement head, thereby dividing the head into two chambers of equal dimensions. Equivalent constant volume flows of different gases are introduced to flow in the same direction past the sample within each chamber. Gases are exchanged between the chambers by diffusing through the sample. In this CRM the diffusion process is monitored by the diffusion of CO₂ into N₂. The steady-state concentration of CO₂ in the outflow gas is measured and used to determine the diffusion capacity of the sample. The principle of measurement is illustrated in Figure 1.

The direction of CO₂ transfer through the sample shall be that which would occur when the sample is used on the finished product, where known, i.e. from the side facing the tobacco towards the side facing ambient air.

NOTE 1 The concentration of CO₂ in the outflow gas is influenced by the differential pressure across the sample. The differential pressure shall be recorded and kept to a minimum.

NOTE 2 The concentration of CO₂ in the outflow gas is influenced by ambient temperature, relative humidity and atmospheric pressure. Therefore temperature and relative humidity shall be monitored and should preferably comply with ISO 187. The ambient pressure shall be recorded.

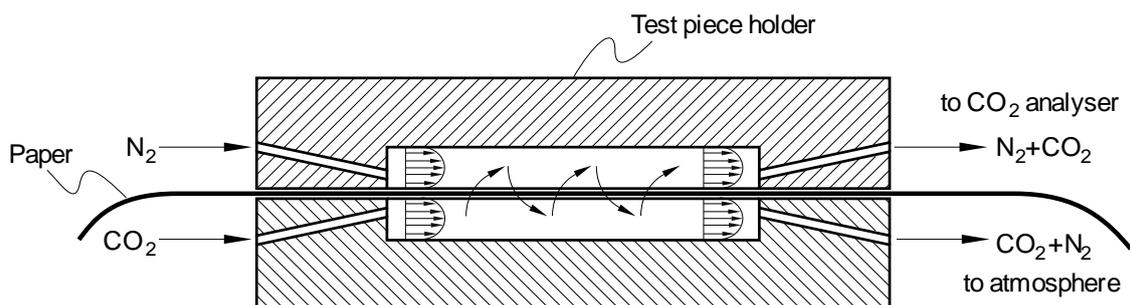


Figure 1 – Principle of measurement.

5. APPARATUS

NOTE 1 This section describes the basic requirements for the principle of the measurement. It is not a specification for instrument design

5.1 Test piece holder

Test piece holder for clamping the test piece, free from leaks with a rectangular surface area of 20 mm² - 80 mm². The corner radii shall not be greater than 0,5 mm. The long side shall have a length of 10,00 mm ± 0,05 mm to 20,00 mm ± 0,05 mm with a resulting short side of 2,00 mm ± 0,05 mm to 4,00 mm ± 0,05 mm. The depth of the chamber shall not exceed the chamber width. The gas flow shall be established along the long side of the chamber. To facilitate checking of the differential pressure between the two measurement chambers, both chambers shall have a suitable connection point.

The apparatus shall be designed in such a way that the difference between the pressure in the measurement chamber and atmospheric pressure does not exceed 1 kPa during measurement.

NOTE 1 The smaller the open area the higher will be the variation of individual measurements due to the inevitable inhomogeneity of the samples and due to a stronger influence of edges and sidewalls of the chamber on the flow and diffusion process. While areas as small as 20 mm² are allowed in this CRM, they should be limited to certain applications, and the open area should preferably be at least 30 mm².

5.2 Flow controller

Device to produce flows of different gases at given but adjustable constant flow rates at the two surfaces of the test piece.

5.3 Infrared analyser

A non-dispersive infrared analyser satisfying the following conditions:

Accuracy	< ± 0,1 % vol. CO ₂
Resolution	0,01 % vol. CO ₂
Linearity	< ± 0,1 % vol. CO ₂
Repeatability	0,1 % vol. CO ₂ under conditions of constant temperature and pressure

5.4 Pressure gauge

Pressure gauge suitable for measuring pressure differences within a range of at least ±25 Pa, with an absolute error of no more than 2 Pa in order to measure the differential pressure between the chambers.

5.5 Flow meter

Flow meter suitable for measuring the gas flow with an error not greater than 1 % of the measured value within the measuring range.

5.6 Conditioning enclosure

Conditioning enclosure capable of maintaining the conditions given in ISO 187. (See 7.2).

5.7 Thermometer

A thermometer for measuring temperature of the measuring chamber to the nearest 1 K.

5.8 Barometer

A barometer for measuring atmospheric pressure to the nearest 0,1 kPa.

5.9 CO₂ supply

CO₂ gas with a minimum purity of 99,995 % (grade 4.5).

NOTE 1 Some instruments may require mixed gases. In these cases refer to the manufacturer's recommendations and use gases with a composition known to an equivalent accuracy.

5.10 N₂ supply

N₂ gas with a minimum purity of 99,99 % (grade 4.0).

6. SAMPLING

Take a sample which is representative, on a statistical basis, of the population of test pieces to be characterized, for example according to ISO 186 for paper samples taken from bobbins [11], or according to ISO 8243 when sampling cigarettes [12].

Samples shall be free of visible defects and creases which may impair measurement performance. Touching the samples by hand in the area to be measured could influence the result and should be avoided. If this is not possible it is recommended to use cotton or powder-free nitrile gloves or tweezers.

7. PROCEDURE

7.1 Check of differential pressure

Connect a differential pressure gauge to both connection points. Reset or adjust the gauge to zero while the test head is opened. Separate both chambers from each other by using a sealing foil placed in the measurement area. Close the measurement head and perform a measurement. The determined value should be $0 \text{ Pa} \pm 5 \text{ Pa}$. A differential pressure check shall be performed periodically.

NOTE 1 The differential pressure during a typical measurement will differ from the determined value using the sealing foil. This is caused by the different weight and size of the CO₂ and N₂ molecules introducing a net mass flow through the sample. More N₂ molecules will diffuse through the paper than CO₂ molecules. The higher the transfer rate the larger the difference becomes. Values between 2 Pa and 10 Pa absolute are not unusual. No re-adjustment during measurement should be performed.

7.2 Preparation of the test pieces

Select at random from the sample taken in accordance with 6, the number of the test pieces required for the test plus an additional three test pieces to be used as spares. If necessary, the test pieces shall be made suitable for testing (cut to the required dimensions, eliminate folds, seams etc.). If the paper samples are taken from cigarettes about 5 to 10 cigarettes should be sufficient to provide the required number of paper samples.

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

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 stating the conditions used shall be included with the test report.

Sample bobbins, where it is not possible to expose all surfaces to the conditioning atmosphere, may require an extended period of conditioning. The time required should be determined by practice and experience and should, for example, not be less than 48 hours.

The period of time for conditioning shall be reported with the results.

NOTE 1 The standard conditions used in the paper and board industry (and adopted for this method) are given in ISO 187:1993 as:

Temperature (23 ± 1) °C
Relative Humidity (50 ± 2) % RH.

NOTE 2 In the tobacco industry, and particularly for manufactured cigarettes, the testing environment is given in ISO 3402:1999 [13] as:

Temperature (22 ± 2) °C
Relative Humidity (60 ± 5) % RH.

NOTE 3 For testing the ignition propensity of cigarettes, the conditioning environment is given in ISO 12863:2010 [1] as:

Temperature (23 ± 3) °C
Relative Humidity (55 ± 5) % RH.

7.3 Calibration

The instrument can be calibrated by the use of pure N₂ as zero gas and a CO₂ / N₂ gas mixture of a known concentration. It is recommended to use a certified test gas with an analytical accuracy of better than 1 % relative. The used concentration shall be in the same range as the expected concentration during the measurement.

The calibration gas should be stored under the same conditions as the gases for normal operation. The test gas shall pass through the measuring chamber and the gas analyser in the same direction as during a measurement.

The flow rate of the test gas should be the same as the flow rates of the gases during a measurement, if an analyser without internal compensation is used.

7.4 Insertion of a test piece

All test pieces shall be placed in the test piece holder so that the CO₂ will diffuse from the inner side face towards the outer side face of the sample as it is used in the manufacture of the finished product e.g. a cigarette, where this is known.

The positioning of the test piece in the test piece holder is illustrated in Figures 2 and 3. Touching the samples by hand in the area to be measured could influence the result and should be avoided. If this is not possible, it is recommended to use cotton gloves, powder-free nitrile gloves or tweezers.

7.4.1 Materials with uniformly distributed characteristics

Place the test piece so that the width of the opening in the measurement head is parallel to the width of the test piece and centred on the test piece (see Figure 2).

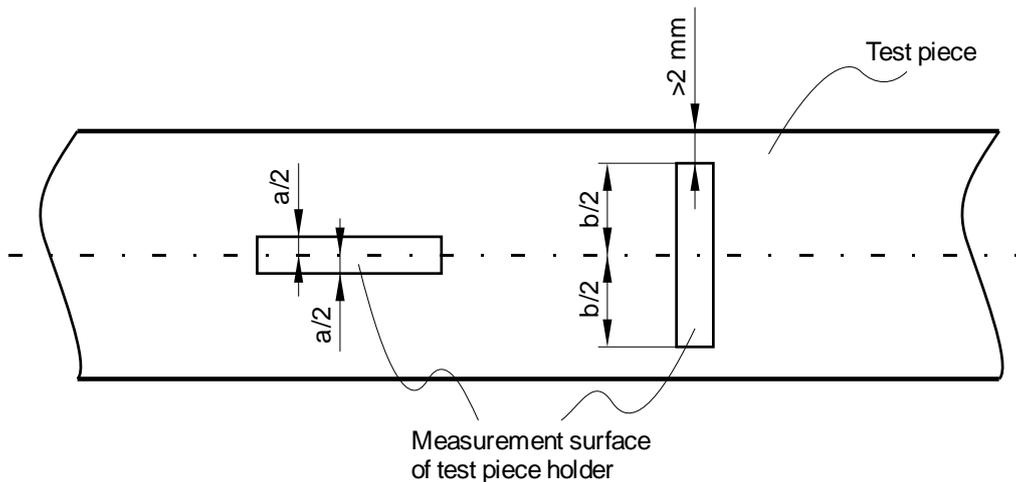


Figure 2 – Positioning of the test piece.

7.4.2 Materials with an oriented zone of reduced diffusion capacity (banded zone)

The banded zone shall be oriented along, and parallel to, the direction of the long side of the test surface (see Figure 3).

The edges of the banded zone shall not be less than 0,3 mm from the edges of the test surface. If for technical reasons this cannot be achieved this shall be noted in the test report.

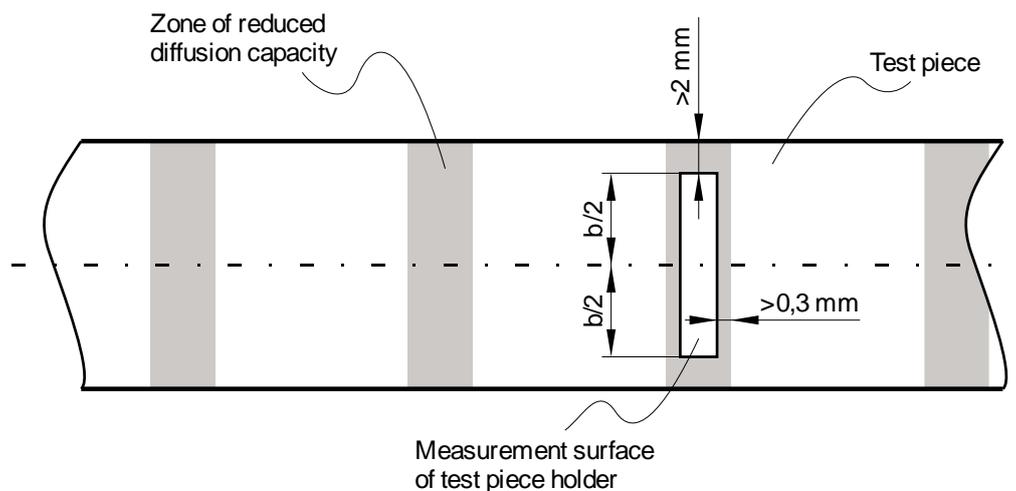


Figure 3 – Positioning of the test piece with oriented zones of reduced diffusion capacity.

7.5 Measurement

Insert a test piece in the test piece holder. Establish a suitable gas flow with a stability of better than 2 % along the two faces of the test piece until an acceptable stability of CO₂ concentration in N₂ has been reached. Measure the volumetric CO₂ concentration in the N₂ at the outlet of the measuring chamber.

The determined diffusion capacity can vary throughout the length of the test piece. For this CRM, it is advised to take 10 individual measurements at different positions to calculate the mean. In practice, laboratories often take a different number of measurements depending upon the application of the measurement, for example, during large-scale production of papers having an oriented zone of reduced diffusion capacity, five measurements may be used to calculate a

mean value. If results of higher precision are required 20 measurements can be used. Proceed in the same way with all the test pieces. Make sure that the measured areas do not overlap and do not measure the same position twice.

7.6 Calculation of diffusion capacity

The relationship between the diffusion capacity of the test piece and the measured CO₂ concentration in the outflow gas depends on the specific setup of the instrument, i.e. the chamber geometry, the volumetric flow rate of the gases and temperature and absolute pressure inside the measurement chamber. Instrument manufacturers use mathematical models to describe the flow and diffusion processes inside the measurement chamber to relate the CO₂ concentration to diffusion capacity. For the specific experimental setup shown in Figure A1 an algorithm to calculate diffusion capacity from the CO₂ concentration is described in Annex A.1. For head designs as shown in Figure A2, the calculation is given by Annex A.2.

The diffusion capacity shall be calculated for each measured CO₂ concentration. The mean value and standard deviation of the diffusion capacity shall be calculated and reported.

8. EXPRESSION OF RESULTS

The value of the sample mean diffusion capacity shall be the mean value of the individual determinations (See 7.6).

The diffusion capacity shall be expressed in cm·s⁻¹, rounded to the nearest 0,001 cm·s⁻¹ and reported together with the temperature and pressure for which the diffusion capacity is valid. Preferably it shall be reported for a temperature of 296,15 K and a pressure of 101,325 kPa

9. REPEATABILITY AND REPRODUCIBILITY

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 limit r on average not more than once in 20 cases in the normal operation of the method.

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

An international collaborative study, involving 12 laboratories using 15 different instruments or instrument configurations and five samples, conducted in 2013 according to ISO 5725-2:1994, showed that when materials used as cigarette papers and cigarette papers having an oriented zone of reduced diffusion capacity were measured in accordance with this Recommended Method, the following values for repeatability and reproducibility were obtained.

	Diffusion Capacity	Repeatability			Reproducibility		
Sample	Mean Value	Standard Deviation	Coefficient of Variation	r	Standard Deviation	Coefficient of Variation	R
	cm/s	cm/s	%	cm/s	cm/s	%	cm/s
Zones of reduced diffusion capacity on cigarette paper	0,0702	0,0029	4,18	0,0081	0,0098	14,03	0,0273
	0,1061	0,0058	5,53	0,0162	0,0400	37,79	0,1110
	0,0517	0,0032	6,25	0,0090	0,0169	32,72	0,0469
	0,0708	0,0043	6,10	0,0120	0,0102	14,45	0,0284
Tipping Base Paper	0,4713	0,0015	2,43	0,0317	0,0383	8,12	0,1061

Table 1 – Repeatability and reproducibility data for an average of 10 values

	Diffusion Capacity	Repeatability			Reproducibility		
Sample	Mean Value	Standard Deviation	Coefficient of Variation	r	Standard Deviation	Coefficient of Variation	R
	cm/s	cm/s	%	cm/s	cm/s	%	cm/s
Zones of reduced diffusion capacity on cigarette paper	0,0702	0,0057	8,11	0,0158	0,0112	16,00	0,0312
	0,1061	0,0117	11,04	0,0325	0,0415	39,17	0,1152
	0,0517	0,0069	13,42	0,0192	0,0181	35,08	0,0503
	0,0708	0,0072	10,16	0,0199	0,0123	17,40	0,0342
Tipping Base Paper	0,4713	0,0129	2,74	0,0358	0,0400	8,50	0,1110

Table 2 – Repeatability and reproducibility data for an individual measurement

In Table 1 repeatability and reproducibility data are given for a test result obtained as an average from 10 individual measurements as advised by this method, while in Table 2 one test result is defined as an individual value obtained from a single measurement. All values for repeatability and reproducibility may only be valid for the particular papers used. As it is required in this method that each measurement is done on a different position, the inhomogeneity in the test pieces contributes to the within laboratory variance.

For the interpretation of the reproducibility data it has to be noted that no reference method, reference materials or reference standards for diffusion capacity measurement were available at the time of the collaborative study. Therefore the instruments were not calibrated in the proper sense by adjusting the instrument reading to an accepted value of a reference material. This adds to the variation between laboratories and consequently, by averaging 10 values, reproducibility is improved less than repeatability. Furthermore, results of the collaborative study showed interactions between some paper samples and instruments so that certain instrument type/paper sample combinations delivered values substantially different from values obtained with the same paper sample on other instrument types. This mainly applies to the two paper samples showing a reproducibility coefficient of variation of more than 30 %.

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 CRM, or regarded as optional, as well as any circumstances that may have influenced the results.

Specifically the test report shall contain:

- a) date of measurement;
- b) name of operator;
- c) instrument identification, e.g. the serial number;
- d) head dimensions;
- e) date of sampling and sampling method;
- f) identification and full description of the material tested;
- g) the condition atmosphere and period of conditioning time;
- h) barometric pressure at the time of testing;
- i) measuring head temperature at time of testing;
- j) the diffusion capacity in $\text{cm}\cdot\text{s}^{-1}$ to three decimal places and the temperature and pressure for which the diffusion capacity is valid;
- k) elementary statistics relating to the results:
 - number of measurements;
 - mean value and standard deviation;
 - coefficient of variation of diffusion capacity to two decimal places

ANNEX A
(Informative)

A.1 Calculation of diffusion capacity for the test piece holder of Drake et al.

The results obtained by this CRM as described in section 7.5 can be used to determine the diffusion capacity of the test piece. The calculations are based on a mathematical model described by Drake et al. [14]. This model provides the volumetric concentration of CO₂ in the outflow gas for a given diffusion capacity of the test piece. The mathematical model is a system of partial differential equations, which allows an approximate analytic solution.

To compensate for temperature and pressure variation, the model was extended to cover the effects of non-standard temperature and pressure in the measurement chamber. The model can be understood as a function $f(\cdot)$ mapping the diffusion capacity D^*_0 at standard conditions and the values of actual temperature T and pressure p in the measurement chamber to the volumetric concentration of CO₂ in the outflow gas, $C_{CO_2,out} = f(D^*_0, T, p)$. As the function $f(\cdot)$ cannot be directly inverted to calculate the diffusion capacity, the diffusion capacity has to be determined by numerically solving this nonlinear equation. For given temperature and pressure, the concentration of CO₂ in the outflow gas is strictly increasing with increasing diffusion capacity so that the solution of this equation is unambiguous and convergence even of simple numerical methods is guaranteed.

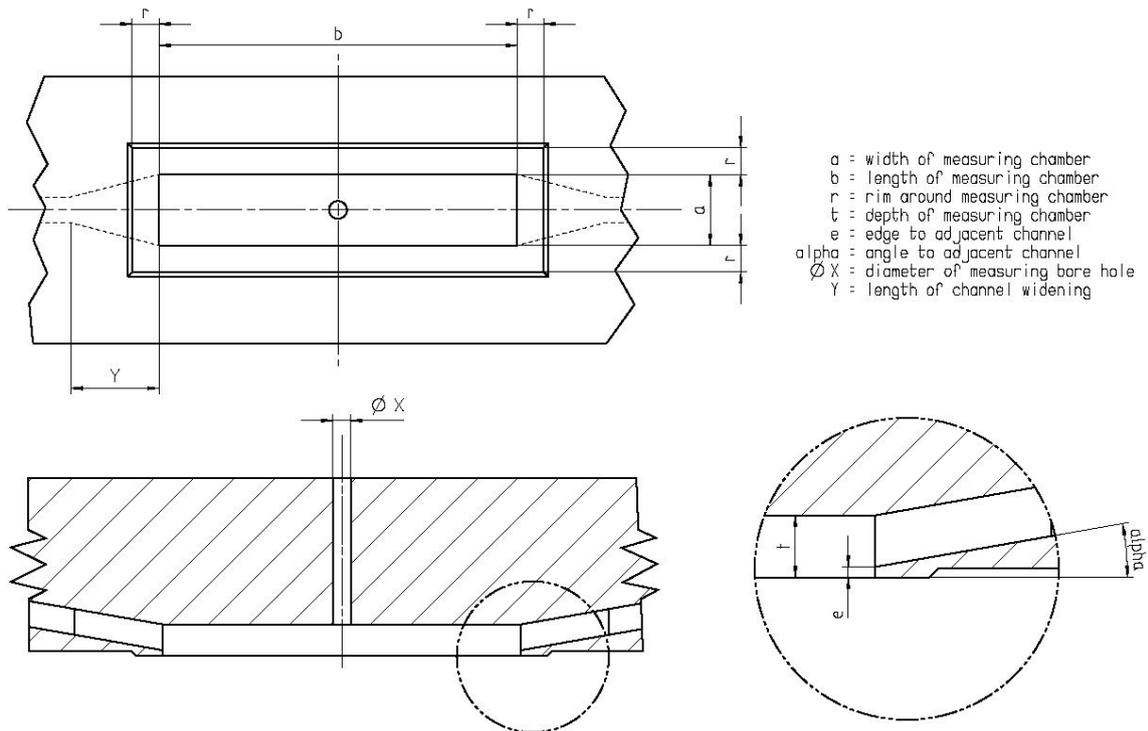


Figure A1 - Design and dimensions of test piece holder.

A.1.1 Evaluation of an extended version of the model by Drake et al.

The algorithm described below allows the evaluation of $C_{CO_2,out} = f(D^*_0, T, p)$, i.e. the calculation of the CO₂ concentration in the outflow gas for given diffusion capacity D^*_0 at standard conditions and temperature T and pressure p in the measurement chamber.

(A0) Input D^*_0, T, p

(A1) Calculate $D^*(T, p) = D^*_0 \left(\kappa \sqrt{\frac{T}{T_0}} + (1 - \kappa) \left(\frac{T}{T_0} \right)^n \frac{p_0}{p} \right)$

(A2) Calculate $D_g(T, p) = D_{g,0} \left(\frac{T}{T_0} \right)^n \frac{p_0}{p}$

(A3) Calculate $\beta = \frac{2t}{D_g(T, p)} D^*(T, p)$

(A4) For $i=1,2,\dots,10$ provide the 10 smallest solutions of $\beta = \alpha_i \cdot \tan(\alpha_i)$ for α_i with $\alpha_i > 0$ and an absolute residual error $|\beta - \alpha_i \cdot \tan(\alpha_i)| < 10^{-9}$. Any method for the numerical solution of nonlinear equations, such as a bisection method or Newton's method can be used.

(A5) Calculate $\gamma = \frac{ab D_g(T, p)}{\dot{V} 6t}$

(A6) Evaluate $\chi = 2\beta^2 \sum_{i=1}^{10} \frac{\exp(-6\gamma\alpha_i^2)}{\alpha_i^2(\alpha_i^2 + \beta^2 + \beta)}$

(A7) Calculate $C_{CO_2,out} = C_{CO_2,in} \frac{(\chi - 1)}{2}$

(A8) Output $C_{CO_2,out}$

with

T	Temperature in the measurement chamber [K]
p	Absolute pressure in the measurement chamber [kPa]
T ₀	Standard temperature as given by this CRM (296,15 K)
p ₀	Standard pressure as given by this CRM (101,325 kPa)
κ	Share of Knudsen diffusion in total diffusion, recommended value 0,034
n	Exponent for temperature dependence of diffusion, recommended value 1,88
a	Chamber width [cm]
b	Chamber length [cm]
t	Chamber depth [cm]
\dot{V}	Volumetric flow of CO ₂ and N ₂ through the chamber [cm ³ ·s ⁻¹]
C _{CO₂,in}	Volumetric concentration of CO ₂ in the inflow gas [%]
C _{CO₂,out}	Volumetric concentration of CO ₂ in the outflow gas [%]
D [*] (T,p)	Diffusion capacity of the test piece at conditions T and p [cm·s ⁻¹]
D [*] ₀	Diffusion capacity of the test piece at conditions T_0 and p_0 [cm·s ⁻¹]
D _g (T,p)	Diffusivity of CO ₂ in N ₂ in the free gas phase at T and p [cm ² ·s ⁻¹]
D _{g,0}	Diffusivity of CO ₂ in N ₂ in the free gas phase at T_0 and p_0 , recommended value 0,0163 cm ² ·s ⁻¹

NOTE The above units are just a proposal, any consistent set of physical dimensions works with the above algorithm. All variables denoted by Greek letters are dimensionless quantities.

A.1.2 Determination of diffusion capacity

While many numerical methods for the solution of non-linear equations may be appropriate to solve the equation $C_{CO_2,out}=f(D^*_0,T,p)$, a simple bisection method is described below, as more advanced methods frequently require the derivative $\partial f(\cdot)/\partial D^*_0$, which is not easily available. The method works as long as the diffusion capacity is greater than or equal to $0 \text{ cm}\cdot\text{s}^{-1}$ and less than or equal to $4 \text{ cm}\cdot\text{s}^{-1}$.

The input to the algorithm is the measured volumetric concentration of CO_2 in the outflow gas, $C_{CO_2,out}$, and the temperature T and pressure p in the measurement chamber.

- (B0) Input $C_{CO_2,out}$, T , p
- (B1) Set $D^*_{0,min} = 0 \text{ cm}\cdot\text{s}^{-1}$
- (B2) Set $D^*_{0,max} = 4 \text{ cm}\cdot\text{s}^{-1}$
- (B3) Set $D^*_{0,middle} = \frac{1}{2}(D^*_{0,min} + D^*_{0,max})$
- (B4) Evaluate $C_{CO_2,middle} = f(D^*_{0,middle}, T, p)$ by algorithm (A0)-(A8)
- (B5) If $C_{CO_2,out} \geq C_{CO_2,middle}$ then $D^*_{0,min} = D^*_{0,middle}$
- (B6) If $C_{CO_2,out} < C_{CO_2,middle}$ then $D^*_{0,max} = D^*_{0,middle}$
- (B7) If $|D^*_{0,min} - D^*_{0,max}| > \varepsilon$ go to step (B3)
- (B8) Set $D^*_0 = D^*_{0,middle}$
- (B9) Output D^*_0

The value for ε is the error tolerance of the resulting diffusion capacity; a recommended value is $10^{-6} \text{ cm}\cdot\text{s}^{-1}$. Note that with a starting interval width of $4 \text{ cm}\cdot\text{s}^{-1}$, the diffusion capacity can be determined to a precision of better than $10^{-6} \text{ cm}\cdot\text{s}^{-1}$ by repeating steps (B3)-(B7) 22 times.

A.1.3 Example evaluation of the modified model of Drake et al.

An implementation of the model by Drake et al. was evaluated at standard conditions for a set of diffusion capacity values with the following numerical values and a head design according to Figure A1.

$$\begin{array}{ll}
 a & = 0,3 \text{ cm} & \dot{V} & = 7,5 \text{ cm}^3 \cdot \text{s}^{-1} \\
 b & = 2,0 \text{ cm} & C_{CO_2,in} & = 100 \% \\
 t & = 0,175 \text{ cm} & &
 \end{array}$$

For variables not mentioned here, the recommended value was chosen. Table A1 shows the diffusion capacity and the volumetric concentration of CO₂, as obtained by algorithm (A0)-(A8) with a temperature of 296,15 K and a pressure of 101,325 kPa.

$C_{CO_2,out}$	D_0^*
[%]	[cm.s ⁻¹]
0,000000	0,000
0,119211	0,015
0,158600	0,020
0,236862	0,030
0,314443	0,040
0,391350	0,050
0,467592	0,060
0,543176	0,070
0,618109	0,080
0,692399	0,090
0,766053	0,100
1,469170	0,200
2,116063	0,300
3,263902	0,500
4,248339	0,700
5,099422	0,900
5,840678	1,100
6,490707	1,300
7,064352	1,500
7,573560	1,700
8,028028	1,900
8,435684	2,100
8,803064	2,300
9,135586	2,500
9,437778	2,700
9,713443	2,900

Table A1 - Correspondence between CO₂ concentration in the outflow gas and diffusion capacity at standard conditions.

A.2 Calculation of diffusion capacity for an alternative test piece holder

This part of the Annex presents an approach to the design of the measurement head and the calculation of diffusion capacity that has built on the work of Drake et al. [14]. The same measurement principle remains, in that different gases are passed over the two sides of the paper and the rate of exchange of gases through the paper is determined and expressed as the flow velocity of the diffusing species through the paper, in $\text{cm}\cdot\text{s}^{-1}$. This is the diffusion capacity D^* . The analysed gas that is transferred through the paper is CO_2 and the gas into which it diffuses is pure N_2 . The CO_2 can be supplied either in pure form or diluted in N_2 .

A.2.1 Design of the test piece holder

A schematic illustration of a measurement head designed to deliver a flow of gas along the surface of the paper under measurement is presented in Figure A2.

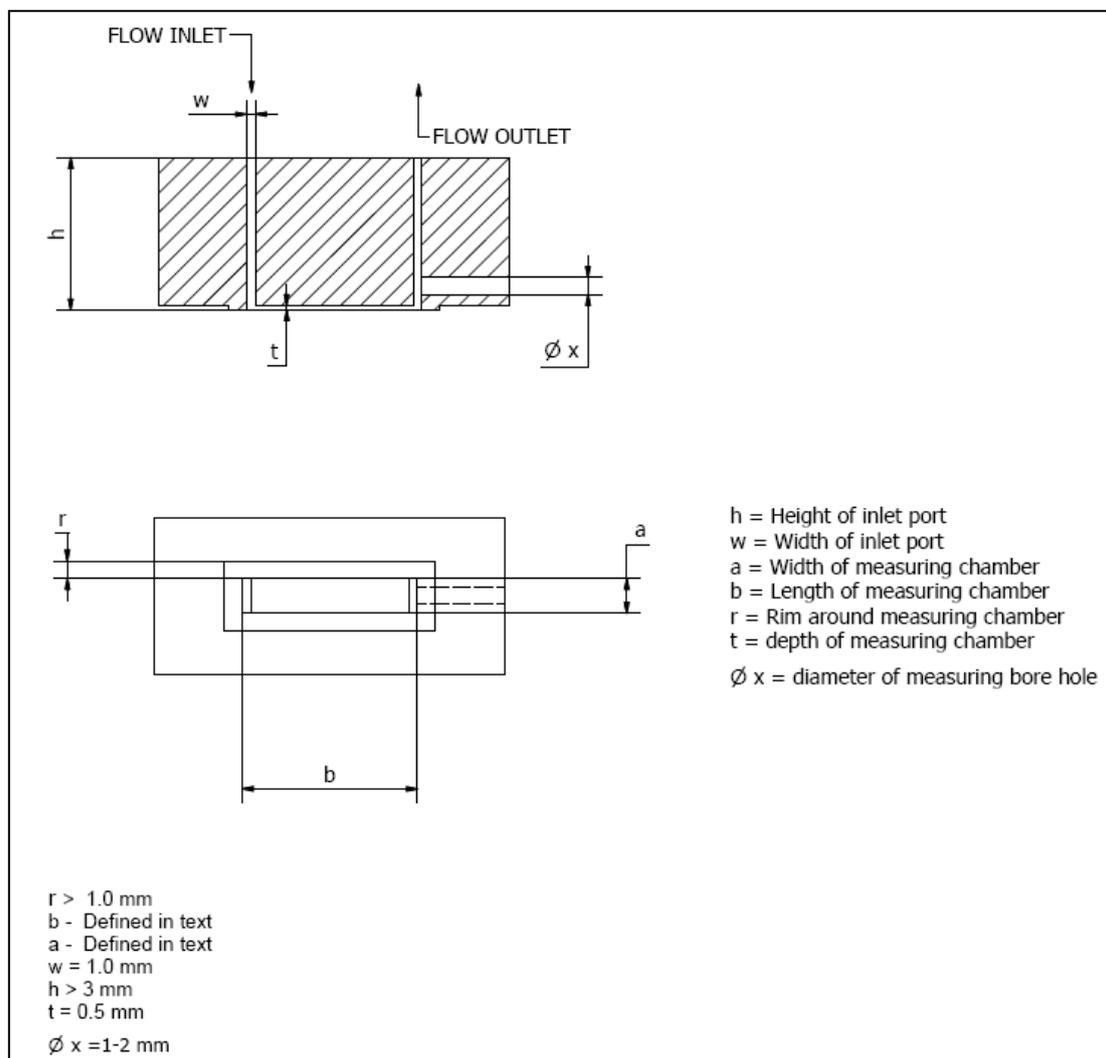


Figure A2 - Design and dimensions of the test piece holder

Laminar flow is developed in the slot delivering the gas to the chamber and the measurement cell is sufficiently shallow (typically 0,5 mm) such that non-turbulent flow is maintained around the sharp 90° corner and then parallel to the paper and into the exhaust path. This has been verified using a Computational Fluid Dynamics (CFD) model of the design.

A.2.2 Calculation of diffusion capacity

A.2.2.1 Gas flow rate

The volumetric gas flow rates over the opposite surfaces of the paper are set at the same level, a volumetric flow rate of 460 standard cm³ per minute at 0 °C and 101,325 kPa (7,66 cm³·s⁻¹) for a measurement area A of 20 mm × 4 mm. The standard volumetric flow rate \dot{V}_{Std} is converted into the volumetric flow rate \dot{V}_{amb} at ambient conditions of temperature and pressure as follows:

$$\dot{V}_{amb} = \dot{V}_{Std} \frac{T_{amb}}{273,15} \frac{101,325}{p_{amb}}$$

with

- \dot{V}_{Std} Volumetric flow at 0 °C and 101,325 kPa in [cm³·s⁻¹]
- \dot{V}_{amb} Volumetric flow rate of CO₂ and N₂ in the chamber at T_{amb} and p_{amb} in [cm³·s⁻¹]
- T_{amb} Ambient temperature in [K]
- p_{amb} Ambient pressure in [kPa]

A.2.2.2 Calculation of D^*

Calculation of diffusion capacity requires two steps:

1. Determination of D_{raw}^*

This is defined in this system as the calculation of net diffusion of CO₂ through the paper without any compensation for the change in gas compositions on the opposite faces of the paper due to the gases that diffuse through the paper.

Thus

$$D_{raw}^* = \dot{V}_{amb} \frac{C_{CO_2,out}}{C_{CO_2,in}} \frac{1}{A}$$

with

- \dot{V}_{amb} Volumetric flow rate at ambient conditions T_{amb} and p_{amb} in [cm³·s⁻¹]
- D_{raw}^* Uncompensated diffusion capacity in [cm·s⁻¹]
- $C_{CO_2,out}$ Volumetric concentration of CO₂ in the outflow gas in [%]
- $C_{CO_2,in}$ Volumetric concentration of CO₂ in the inflow gas in [%]
- A Measurement head area in [cm²]

2. Determination of the corrected value D^*

This procedure corrects for the change in gas compositions on the opposite faces of the paper due to the diffusing gases. It calculates the diffusion capacity D^* of the paper in cm·s⁻¹, which is always greater than D_{raw}^* . This is because gas that diffuses across the paper is initially of the inlet composition that is used in the calculation of D_{raw}^* . However, as the gases pass over the paper, the CO₂ concentration on the CO₂ side is diluted by N₂ diffusing through from the N₂ side. Also, some CO₂ that has passed into the N₂ side diffuses back into the CO₂ side.

These effects combine to reduce the net transfer of CO₂ through the paper compared to having pure gas on each side and hence must be compensated to yield the corrected D^* value. The compensation parameters were calculated using the same CFD model as was used to verify the flow through the head.

The following expression to calculate D^* from D_{raw}^* is valid for $t = 0,5$ mm, $a = 20$ mm and $b = 4$ mm, see Figure A2.

$$D^* = 1,0347 \cdot D_{raw}^* + 0,11079 \cdot (D_{raw}^*)^2$$

with

D_{raw}^* Uncompensated diffusion capacity in [$\text{cm} \cdot \text{s}^{-1}$]

D^* Diffusion capacity in [$\text{cm} \cdot \text{s}^{-1}$]

A.2.3 Example of calculation

Input values

A	Measurement area	20 mm × 4 mm
\dot{V}_{Std}	Volumetric flow rate of CO ₂ and N ₂	460 standard cm ³ ·min ⁻¹
$C_{CO_2,in}$	CO ₂ source gas	18,00 % CO ₂ in N ₂
T_{amb}	Ambient temperature	22,5 °C
P_{amb}	Ambient pressure	100,5 kPa
$C_{CO_2,out}$	Analysed CO ₂ in N ₂ outlet	2,530 %

Calculated values

\dot{V}_{amb}	Volumetric flow rate	8,366 cm ³ ·s ⁻¹
D_{raw}^*	Uncompensated diffusion capacity	1,470 cm·s ⁻¹
D^*	Diffusion capacity	1,760 cm·s ⁻¹

This is a typical diffusion capacity of cigarette base paper.

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