# **DE GRUYTER** OPEN

Beiträge zur Tabakforschung International/

Contributions to Tobacco Research

Volume 27 · No. 1 · January 2016

DOI: 10.1515/cttr-2016-0002

# Leak-Based Method for the Measurement of Air Permeability of Papers \*

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# **SUMMARY**

The air permeability of cigarette paper is currently assessed according to the international standard ISO 2965 by applying a constant pressure difference of 1 kPa between the two faces of a sample and by measuring the corresponding airflow

Lower Ignition Propensity regulations have led tobacco manufacturers to use specific cigarette papers with narrow bands of low air permeability and diffusion capacity to achieve regulatory compliance. The international standard ISO 2965 was revised in 2009 to take into account the specific geometry and characteristics of the bands and to include suitable narrow measuring heads. The consequence was a significant reduction of the measured airflow levels with banded papers and a need for equipment covering specifically low airflow ranges.

The well-known pressure-airflow relationship across cigarette paper enables the development of an alternative method to ISO 2965 which does not require direct airflow measurement, and therefore airflow meters which are costly parts of the current measuring devices. The alternative method is based on the measurement of the change of the pressure over time after an initial pressure difference was applied between the two faces of the paper. The consecu-

tive analysis of the pressure difference profile, impacted by the leak across the paper, enables the derivation of the air permeability.

The related theoretical aspects were developed for both viscous and inertial airflows, and experimental investigations were conducted with banded and conventional cigarette papers as well as a permeability calibration standard. Results obtained with the proposed method showed good consistency with ISO 2965 measurements and a lower repeatability, demonstrating that a leak-based method could be a simple and reliable alternative. [Beitr. Tabakforsch. Int. 27 (2016) 3–10]

# ZUSAMMENFASSUNG

Die Luftdurchlässigkeit von Zigarettenpapier wird derzeit nach der internationalen Norm ISO 2965 untersucht, indem eine konstante Druckdifferenz von 1 kPa zwischen den beiden Flächen eines Prüfmusters angelegt wird und der entsprechende Luftstrom gemessen wird.

Vorschriften für eine verminderte Zündneigung haben Tabakhersteller dazu veranlasst, spezielle Zigarettenpapiere mit schmalen Bändern geringer Luftdurchlässigkeit und Diffusionskapazität zu verwenden, um die gesetzlichen

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Bestimmungen zu erfüllen. Die internationale Norm ISO 2965 wurde 2009 überarbeitet, um die spezifische Geometrie und die besonderen Merkmale der Bänder zu berücksichtigen und geeignete schmale Messköpfe aufzunehmen. In der Folge wurden die gemessenen Luftstromraten mit gebänderten Papieren erheblich reduziert und spezielle für niedrige Luftstromraten geeignete Geräte wurden notwendig.

Die bekannte Beziehung zwischen Druck und Luftstrom durch Zigarettenpapier ermöglicht die Entwicklung einer alternativen Methode zur ISO 2965. Diese erfordert keine direkte Luftstrommessung und demzufolge auch keine Luftstrommessgeräte, welche teure Bestandteile der gegenwärtigen Messgeräte sind. Die alternative Methode basiert auf der Messung der Veränderung des Drucks über die Zeit, nachdem ein anfänglicher Druckunterschied zwischen den beiden Flächen des Papiers angelegt wird. Aus der anschließenden Analyse des Druckdifferenzprofils und seiner Beeinflussung durch den Durchstrom durch das Papier kann die Luftdurchlässigkeit abgeleitet werden.

Die diesbezüglichen theoretischen Aspekte wurden sowohl für viskose als auch träge Luftströme entwickelt, und es wurden experimentelle Untersuchungen mit gebänderten und konventionellen Zigarettenpapieren sowie einem Kalibrierstandard für die Durchlässigkeit durchgeführt.

Die mit der vorgeschlagenen Methode erzielten Ergebnisse zeigten eine gute Übereinstimmung mit den Messungen nach ISO 2965 und eine geringere Wiederholgenauigkeit. Dies zeigt, dass eine Methode, die auf der Durchstömung basiert, eine einfache, zuverlässige Alternative sein könnte. [Beitr. Tabakforsch. Int. 27 (2016) 3–10]

#### **RESUME**

La méthode actuelle de mesure de la perméabilité à l'air des papiers à cigarette, décrite dans la norme internationale ISO 2965, consiste à appliquer une différence de pression constante de 1 kPa entre les deux faces de l'échantillon de papier et à mesurer le débit d'air correspondant.

La règlementation sur la réduction du potentiel incendiaire des cigarettes a conduit les industriels du tabac à utiliser des papiers à cigarette spécifiques présentant des bandes étroites de perméabilité réduite. La norme internationale ISO 2965 a été révisée en 2009 afin de prendre en compte les spécificités géométriques et les caractéristiques de ces bandes ainsi que les nouvelles têtes de mesure adaptées à celles-ci.

L'utilisation des bandes à perméabilité réduite a pour conséquence une réduction drastique des niveaux de débits d'air mesurés et donc un besoin en équipement de mesure spécifique couvrant cette gamme de faibles débits.

Une méthode basée sur la relation connue entre pression et débit à travers le papier à cigarette a été développée comme alternative à la norme ISO 2965 pour répondre à ces nouvelles contraintes. Cette méthode ne requiert pas de mesure directe de débit. Le dispositif de mesure ne comporte donc pas de débitmètre, qui représente une partie coûteuse des instruments actuels.

Avec cette nouvelle méthode, l'évolution de la pression au cours du temps est mesurée après qu'une différence de pression est appliquée entre les deux faces du papier. Le

profil de pression créé par la "fuite" d'air à travers le papier est analysé pour en déduire la perméabilité à l'air.

Cet article présente une description théorique de cette méthode pour des débits laminaire et turbulent ainsi qu'une application expérimentale avec des papiers à bandes, des papiers à cigarette ordinaires ainsi qu'avec un étalon de perméabilité.

Les résultats obtenus avec cette nouvelle méthode sont en accord avec les résultats mesurés avec la méthode normalisée et montrent même une meilleure répétabilité. Cette nouvelle approche apparaît donc comme une alternative simple et fiable. [Beitr. Tabakforsch. Int. 27 (2016) 3–10]

### INTRODUCTION

Air permeability is a specified and characterized cigarette paper quality control parameter that used to be a key indicator of the ISO 12863 or ASTM E2187 test performance (1, 2). Even if diffusion capacity<sup>1</sup> tends nowadays to supplant air permeability as an ignition propensity predictor, both parameters remain of particular interest for characterization of banded papers. Air permeability is defined as the flow of air passing through 1 cm<sup>2</sup> surface of the test piece at a pressure difference of 1 kPa, and expressed in cubic centimeters per minute, according to the current standard ISO 2965 (3).

Other standards related to fabrics, paper and board, as well as polymeric materials and cellular flexibles have also been developed for "air permeability measurement" (4–11) with slight differences in the definition but with a same method principle, i.e., airflow measurement under a given pressure difference.

The principle of ISO 2965 consists in fixing a paper sample in an appropriate measuring head and applying a pressure difference of 1 kPa between the two faces of the sample. The resulting flow rate per square-centimetre corresponds to the air permeability expressed in cm<sup>3</sup> min<sup>-1</sup> cm<sup>-2</sup>. When the relationship of flow versus pressure difference is nonlinear, it is an ISO 2965 requirement to then conduct a second measurement under a pressure difference of 0.25 kPa to fully characterize the product.

In the course of the 2000's, the cigarette fire ignition propensity regulation has led the tobacco product manufacturers to use so-called banded papers or lower ignition propensity (LIP) papers. Those papers are characterized by narrow bands presenting lower air permeability and diffusion capacity. The consequence of using such papers was a drastic reduction of the measured airflow levels on bands and a need for equipment covering specifically low airflow ranges. The ISO 2965 standard was revised in 2009 to take into account the specific geometry and characteristics of the bands. The possibility to use a suitable narrower measuring head was included but the method was not specifically adapted for low flow rate measurements.

<sup>&</sup>lt;sup>1</sup> Diffusion capacity is defined as the capacity of a paper sample to allow carbon dioxide to diffuse through a defined area of the sample into nitrogen without any significant differential pressure across the sample.

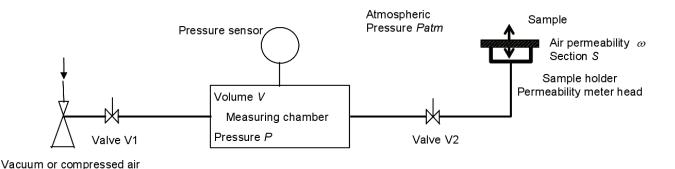


Figure 1. Measuring chamber and device illustration.

In most of the current air permeability measurement methods, accurate pressure regulators and flow meters are required. Therefore, the measuring devices are relatively complex and a flow calibration is necessary. Moreover, the measurement accuracy of air permeability of banded papers might be impaired with standard equipment due to particularly low levels of the measured airflow.

An alternative method to overcome the complex technical specification and experimental limitations i.e., a method with no pressure regulator, no flow meter and therefore no need of calibrated permeability standards is presented in this paper. This new method could be particularly suitable for low permeability levels, so useful for LIP papers. The principle is based on the measurement of the change of the pressure over time after an initial pressure difference was applied between the two faces of a sample. The theoretical aspects were developed and an experimental investigation was conducted with banded paper, a conventional cigarette paper and a permeability calibration standard.

# **METHOD**

The proposed method uses a measuring chamber of fixed volume of air V at a pressure P, in which air can enter or exit through an opening with a cross-sectional area S covered by a permeable material (Figure 1). The airflow Q through the permeable material depends of the section area S, the pressure difference  $\Delta P$  between the inside and the outside of the chamber and the air permeability  $\omega$  of the material that covers the opening. Only the case of a negative relative pressure in the chamber will be developed in the following sections considering that i) the relative pressure is negative in a cigarette when smoked (pressure below atmospheric pressure), and ii) the theoretical approach and the experimental measurements for a positive relative pressure are similar.

The airflow across cigarette papers is the addition of a viscous and an inertial component (12). Thus, the relationship between airflow and pressure difference is given by equation [1].

$$Q = \omega_v \times S \times \Delta P + \omega_i \times S \times \Delta P^{\alpha}$$
 [1]

where

Q is the airflow across the paper, expressed in cm<sup>3</sup> min<sup>-1</sup>  $\omega_{\nu}$  is the permeability of the paper related to the viscous flow, expressed in cm<sup>3</sup> (min<sup>-1</sup> cm<sup>-2</sup> (kPa<sup>-1</sup>))

- $\omega_i$  is the permeability of the paper related to the inertial flow, expressed in cm³ (min⁻¹ cm⁻² (kPa⁻¹/α)) under 1 kPa
- $\Delta P$  is the pressure difference between the two faces of the paper, expressed in kPa
- S is the area through which the airflow occurs, expressed in cm<sup>2</sup>
- α is a constant between 0.5 and 1.0 depending on the rate of inertial airflow.

The permeability of a cigarette paper is defined by convention as the airflow across a section of 1 cm<sup>2</sup> of paper when a pressure difference of 1 kPa is applied between the two faces. The total permeability  $\omega_T$  is then in that case  $\omega_V + \omega_D$ .

# Viscous airflow model

Low permeability levels are usually associated with small paper pore global volume as shown by EITZINGER *et al.* (13) for naturally porous cigarette papers. The airflow through such papers is then essentially viscous, and simply proportional to the pressure difference. Equation [1] can then be simplified and the airflow can be expressed by equation [2].

$$Q = \omega_{v} \times S \times (P_{atm} - P) = \omega_{v} \times S \times \Delta P$$
 [2]

where

 $P_{atm}$  is the atmospheric pressure outside the measuring chamber, expressed in kPa

*P* is the pressure in the measuring chamber with  $P < P_{atm}$ , expressed in kPa.

In the case considered here, the airflow given by equation [2] is the entering airflow because the pressure is lower inside the chamber than outside. If the inside pressure was higher, the exiting airflow would be considered and  $(P_{atm} - P)$  would be replaced by  $(P - P_{atm})$  in equation [2]. The volumetric airflow Q entering the measuring chamber can also be expressed as a function of the number of mole n entering the chamber per unit of time. And from the perfect gas law, the differential equation [3] can be derived.

$$Q = \frac{dn}{dt} \times (Molar\ Volume) = \frac{dn}{dt} \times \frac{RT}{P_{atm}}$$
 [3]

where

Q is expressed in  $m^3$  s<sup>-1</sup>

T is the gas temperature entering the chamber, expressed in K

R is the universal gas constant ((8.314 Pa)  $m^3$  mol<sup>-1</sup> K<sup>-1</sup>))

n is the number of mole entering the chamber, expressed in mole

t is the time expressed in seconds.

By combining equations [2] and [3], the air permeability can be linked to the molar flow and the pressure difference as given by equation [4].

$$\frac{dn}{dt} = \frac{1}{RT} \times \omega_{v} \times S \times P_{atm} \times \Delta P$$
 [4]

In the measuring chamber of fixed volume, the change of the number of mole n over time can also be linked to the change of pressure and temperature by the perfect gas law as given by equation [5].

$$\frac{dn}{dt} = \frac{V}{RT} \times \frac{dP}{dt} - \frac{PV}{RT^2} \times \frac{dT}{dt}$$
 [5]

By combining equations [4] and [5] through the equalization of the derivative of n in accordance with the mass conservation principle, the differential equation [6] is obtained.

$$\frac{d\Delta P}{dt} = \left[ \frac{1}{T} \times \frac{dT}{dt} - \frac{\omega_{\nu} \times S \times P_{atm}}{V} \right] \times \Delta P - \frac{P_{atm}}{T} \times \frac{dT}{dt} \quad [6]$$

Assuming that the temperature is equal and constant inside and outside the measuring chamber as this is usually the case in laboratory conditions, equation [6] can be simplified as expressed with equation [7].

$$\frac{d\Delta P}{dt} + \frac{\omega_{\nu} \times S \times P_{atm}}{V} \times \Delta P = 0$$
 [7]

The analytical solution of this differential equation is given by equation [8].

$$\Delta P(t) = \Delta P_{init} \times e^{-\frac{\omega_{v} \times S \times P_{atm}}{V} \times t}$$
[8]

where

 $\Delta P_{init}$  is the initial pressure difference before the change starts to be recorded.

The change of pressure over time is then linked to the initial pressure difference  $\Delta P_{init}$ , the atmospheric pressure  $P_{atm}$ , the section area S, the volume V and the permeability  $\omega_v$  of the paper.

Observing that the atmospheric pressure, the section through which the leak occurs and the chamber volume can be pre-determined, the air permeability can be derived from two different approaches. The first approach consists of the measurement of two different pressure differences at two different times,  $\Delta P_1$  at time  $t_1$ , and  $\Delta P_2$  at time  $t_2$ . Equation [8] gives the relationship between the pressure differences  $\Delta P_1$  and  $\Delta P_2$  from which the permeability can be extracted.

$$\Delta P_2 = \Delta P_1 \times e^{-\frac{\omega_v \times S \times P_{atm}}{V} \times \Delta t}$$
[9]

where

 $\Delta t = t_2 - t_1 \text{ with } t_2 > t_1.$ 

The permeability can then be easily derived from equation [10].

$$\omega_{v} = \frac{V}{S \times P_{atm} \times \Delta t} \times ln \left(\frac{\Delta P_{1}}{\Delta P_{2}}\right)$$
 [10]

This first approach uses only two data sets for deriving the permeability  $\omega_{\nu}$  which minimizes the number of pressure recordings but exposes the permeability determination to the variability of each single measured dataset. The estimation of  $\omega_{\nu}$  can be improved by a second approach considering the overall change of pressure over time and by including all measured values in the data treatment. From the measurement of  $\Delta P$  over time, a function f can be defined by equation [11].

$$f(t) = \frac{V}{S \times P_{atm}} \times ln\left(\frac{\Delta P_{init}}{\Delta P(t)}\right) = \omega_v \times t$$
 [11]

The estimation of the slope of the function f versus time, for example by linear regression, provides a global estimation of the air permeability  $\omega_v$ .

Viscous and inertial airflow model

The airflow across certain materials is not linear because it is a sum of viscous and inertial components. In this case, an equivalent but more convenient mathematical form of equation [1] is given by equation [12] (14).

$$Q = \omega_T \times S \times \Delta P^k$$
 [12]

where

 $\omega_T$  is the total air permeability, expressed in cm<sup>3</sup> (min<sup>-1</sup> cm<sup>-2</sup> (kPa<sup>-1/k</sup>)) under 1 kPa

k is a constant between 0.5 and 1.0 depending on the rate of inertial flow.

Again, only the case of a negative relative pressure in the chamber will be considered here observing that the approach for a positive relative pressure is similar.

Equation [3] is still valid for such an airflow, and by combining it with the previous equation, the following equation is obtained.

$$\frac{dn}{dt} = \frac{1}{RT} \times \omega_T \times S \times P_{atm} \times \Delta P^k$$
 [13]

By using equation [13] and assuming an equal and constant temperature inside and outside the chamber, equation [5] can be transformed into the differential equation [14].

$$\frac{d\Delta P}{dt} + \frac{\omega_T \times S \times P_{atm}}{V} \times \Delta P^k = 0$$
 [14]

By defining the function g such as

$$g(\Delta P) = -\frac{V}{S \times P_{atm}} \times \frac{d\Delta P}{dt} = \omega_T \times \Delta P^k,$$

it is possible to derive the total air permeability and the factor k from linear regression to  $ln\left(g\left(\Delta P\right)\right)$  as expressed with equation [15].

$$ln(g(\Delta P)) = ln(\omega_T) + k \times ln(\Delta P)$$
 [15]

#### EXPERIMENTAL DEVICE

Based on the principles presented in the previous section, the Company Sodim SAS (Fleury-Les-Aubrais, France), a manufacturer of metrology equipment specialized in tobacco product testing, has developed a prototype depicted in broad outline below.

As represented on Figure 1, a cylindrical cavity of known volume is linked on the left-hand side to a vacuum generator (or to compressed air) and on the right-hand side to a measuring head holding a sample. A vacuum generator was used for the experiment. A valve V1 is placed between the cavity and the vacuum (or the compressed air) generator and a valve V2 is placed between the cavity and the measuring head. A pressure sensor is connected to the cavity in order to record the pressure inside the cavity.

When the measurement process starts, valve V1 is opened and valve V2 is closed. The cavity is put under vacuum (or under pressure) until the targeted pressure is reached. V1 is then closed and the sample is inserted in the measurement head. V2 is opened and the pressure inside the cavity increases (or decreases if compressed air) progressively because of air entering (or exiting) through the permeable sample.

According to the equations given previously, the air permeability of the sample can be calculated from the recording of the pressure over time.

#### RESULTS AND DISCUSSION

The proposed method was first applied to bands of LIP papers and then to conventional cigarette papers with a measuring head of  $0.3~\rm cm^2$  (dimensions  $0.2~\rm cm \times 1.5~\rm cm$ ), a chamber volume of 377 mL and an initial pressure difference of nearly  $1~\rm kPa$ .

Air permeability was estimated by linear regression from

equation [11]. All results were compared to results measured according to ISO 2965 conditions with an air permeability meter Sodim (Model P3 - flow range from 0 to 3 L/min). This was considered as the reference method. The same samples were used with both measuring devices removing the potential variability that would have been observed with different samples of paper.

Additionally, measurements were also performed with a permeability calibration standard consisting of a metal sheet with a small hole of known diameter (a circular measuring head of 1 cm<sup>2</sup> was used). Here, the flow was not viscous and sufficiently low to record enough pressure changes over time. A too rapid pressure change would have made the measurement and the data interpretation particularly difficult due to limitation related to the sampling frequency (one measurement every 55 ms). In this case, equations [14] and [15] were used to derive the air flow rate.

# Experimental results with a banded paper

A banded LIP paper was placed in the measuring chamber and the change of pressure over time was recorded by the pressure sensor. Function f (equation [11]) was calculated.  $\Delta P$  and f were then plotted (Figure 2).

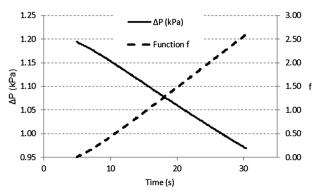


Figure 2. Example of  $\Delta P$  and function f over time for a LIP paper band measurement.

f changed linearly after a few seconds of stabilization, as was expected for a viscous flow. The slope was estimated subsequently by linear regression, and the permeability  $\omega_{\nu}$  was calculated according to equation [11].

Ten bands for each of five samples of LIP paper were first measured with the proposed alternative method and then with the ISO 2965 reference method. The permeability results for each band are represented on Figure 3.

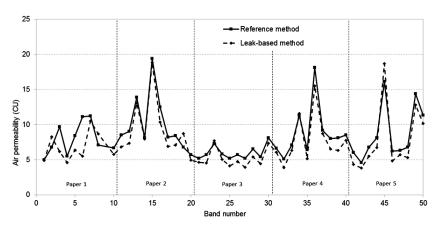


Figure 3. LIP paper measurements with the reference method and the leak-based method.

Table 1. Air permeability - Comparison of the averages of ten LIP bands for five samples.

Sample N°	Air permeability	Air permeability	Standard deviation	Standard deviation
	Reference method (CU)	Leak-based method (CU)	Reference method (CU)	Leak-based method (CU)
1	7.9	6.7	2.2	2.0
2	10.1	9.2	4.1	4.0
3	6.0	5.2	1.0	1.3
4	8.8	7.8	3.7	3.4
5	8.7	7.8	3.9	4.7

The average and standard deviation of each sample results obtained with both methods were then compared (Table 1). The air permeability level was low as expected for LIP bands, i.e., 8.3 CU on average. The difference of 0.9 CU between both methods was particularly narrow for such low level, and the standard deviations, which also depends on the bands variability, were in the same range.

The accuracy of the proposed alternative method was assessed by comparing the difference between the alternative method and the current ISO method against the published reproducibility (3). The difference of 0.9 CU obtained between both methods was significantly lower than the published method precision of 5.13 CU for a LIP band paper with air permeability of 5.52 CU (3).

Repeatability was also tested on a single band to assess the precision of the method by repeating the measurement ten times. The results are presented in Table 2.

Table 2. Air permeability - Comparison of coefficients of variation calculated from ten replicates measured on LIP band under repeatability conditions.

Method	CoV (%) (Repeatability conditions)		
Reference (Sodim P3)	2.9		
Leak-based	0.3		

The leak-based method was more repeatable with a coefficient of variation of 0.3% compared to 2.9% for the reference method; however, the flow rate level was in the lower range of the Sodim P3 equipment where the flow stabilization could be technically more difficult to achieve.

Experimental results with a non-banded (i.e., conventional) cigarette paper

After a first application of the proposed method to LIP papers, the same experiment was carried out with a conventional cigarette paper. The pressure difference  $\Delta P$  and function f were plotted over time (Figure 4).

After a few seconds of stabilization, f changed linearly with respect to time. The slope was estimated by linear regression, and the permeability  $\omega_v$  was calculated from equation [11].

The air permeability of ten positions of a cigarette paper were measured with both a Sodim permeameter P3 and with the leak-based method. Results are represented on Figure 5.

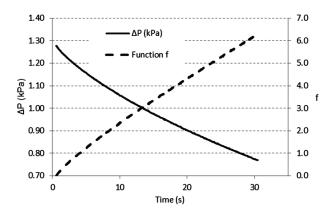


Figure 4. Example of  $\Delta P$  and function f over time for a conventional cigarette paper measurement.

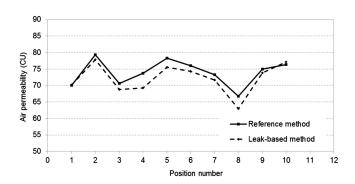


Figure 5. Conventional cigarette paper measurements with the reference method and the leak-based method.

The averages, the standard deviations and the coefficients of variation (CoV) were calculated and then compared for both methods (Table 3).

The average measured air permeability were 73.9 and 72.2 CU, respectively for the reference and the alternative methods. The standard deviations were in the same range i.e., 3.9 and 4.5 CU respectively, as well as the coefficients of variation of 5.3% and 6.3% respectively.

Table 3. Air permeability - Comparison of values measured on conventional cigarette paper (ten measurements at different positions).

Method	Air permeability- Average (CU)	Standard deviation (CU)	CoV (%)
Reference (Sodim P3)	73.9	3.9	5.3
Leak-based	72.2	4.5	6.3

The published reproducibility limits for air permeability measured on cigarette paper (3) were plotted against air permeability levels and compared with the difference between the two methods (Figure 6). The difference of 1.8 CU was far below the estimated reproducibility level of 10.9 CU.

In addition, ten measurements were made under repeatability conditions on a same paper and position with the two methods in order to compare the respective coefficients of

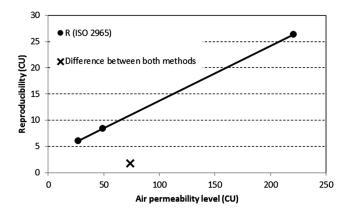


Figure 6. Reproducibility limit as a function of air permeability level (low range only).

variation. Results are given in Table 4 and show that the variability of the alternative method is particularly low. By comparing data obtained with LIP banded papers with those obtained with conventional cigarette paper, it can be observed that the coefficient of variation is lower with the reference method probably due to higher flow rate.

Table 4. Air permeability - Comparison of coefficients of variation calculated from ten replicates measured on conventional paper under repeatability conditions.

Method	CoV (%) (Repeatability conditions)
Reference (Sodim P3)	0.7
Leak-based	0.1

Experimental results with a permeability calibration standard

In a third and last experiment, the method was applied to a permeability calibration standard consisting of a metal sheet with a hole of 100 µm diameter. Such a standard combines non-linear air flow behavior with a low flow rate level. Indeed, the leak-based method is not suitable for high flow because a fast drop of pressure over time would reduce the number of records and would make the interpretation of the experimental data difficult.

The results obtained were compared to standard calibrations performed under ISO 2965 conditions in Sodim SAS laboratory. It is important to observe that, in the particular case of such metal sheet calibration standards, comparison of flow rate makes more sense than comparison of air permeability.

The change of pressure over time was recorded and the function *f* was calculated (Figure 7).

The non-linearity of function f confirmed that the airflow was at least partly inertial. In that case, the viscous and inertial model was used. The function g was calculated by power regression of the measurements and plotted against the calibration of the standard under ISO 2965 conditions noting that  $g(\Delta P) = Q$  (Figure 8).

Visually, the scatter plot obtained with the leak-based method suggested a good overall consistency with the standard calibration results, but shows that the method does

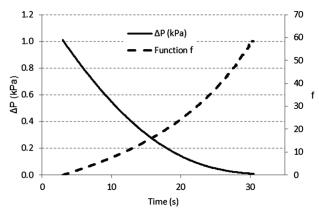


Figure 7. Example of  $\Delta P$  and function f change over time for an air permeability calibration standard.

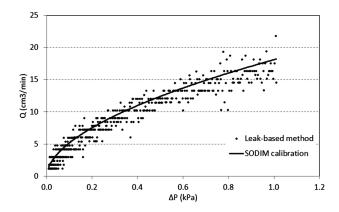


Figure 8. Leak-based method results against Sodim calibration standard

not seem suitable for an accurate determination of the airflow. The fast decrease of the pressure over time probably reaches the limit of the pressure sensor and sampling frequency. This has not been investigated further considering that the application of the leak-based method was mostly relevant for low airflows.

# **CONCLUSIONS**

The leak-based method has shown to be an alternative to the ISO 2965 method to determine the air permeability for LIP and conventional cigarette papers with lower repeatability. A mathematical model for the viscous and inertial flow through paper has been employed to determine the air permeability by measuring the pressure difference over time. The model has also been applied to a permeability calibration standard with consistent experimental results. This new method measuring a pressure difference over time is simple, reliable and does not require pressure regulator, flow meter and flow calibration which could reduce the frequency of calibration. This could be a simple way for quality control either at the supplier's site or at the manufacturer's site. The main limitation is that it could take a longer time to perform a measurement in order to have sufficient data over time to interpret.

#### REFERENCES

- International Organization for Standardization (ISO): International Standard ISO 12863:2010. Standard Test Method for Assessing the Ignition Propensity of Cigarettes; ISO, Geneva, Switzerland, 2010. Available at: http://www.iso.com (accessed January 2016).
- American Society for Testing and Materials (ASTM): ASTM Standard E2187–09. Standard Test Method for Measuring the Ignition Strength of Cigarettes; Available at: http://www.astm.org/Standards/ E2187.htm (accessed January 2016).
- 3. International Organization for Standardization (ISO): International Standard ISO 2965:2009. 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; ISO, Geneva, Switzerland, 2009. Available at: http://www.iso.com (accessed January 2016).
- American Society for Testing and Materials (ASTM): ASTM Standard D737-04 (Reapproved 2012). Standard Test Method for Air Permeability of Textile Fabrics; Available at: http://www.astm.org/Standards/ D737.htm (accessed January 2016).
- International Organization for Standardization (ISO): International Standard ISO 5636-1:1984. Paper and Board -- Determination of Air Permeance (Medium Range) -- Part 1: General Method; ISO, Geneva, Switzerland, 1984. Available at: http://www.iso.com (accessed January 2016).
- International Organization for Standardization (ISO): International Standard ISO 5636-2:1984. Paper and Board -- Determination of Air Permeance (Medium Range) -- Part 2: Schopper Method (Withdrawn); ISO, Geneva, Switzerland, 1984. Available at: http://www.iso.com (accessed January 2016).
- 7. International Organization for Standardization (ISO): International Standard 5636-3:1992. Paper and Board -- Determination of Air Permeance (Medium Range) -- Part 3: Bendtsen Method; ISO, Geneva, Switzerland, 1992. Available at: http://www.iso.com (accessed January 2016).
- International Organization for Standardization (ISO): International Standard ISO 5636-4:2005. Paper and Board -- Determination of Air Permeance (Medium Range) -- Part 4: Sheffield Method.; ISO, Geneva, Switzerland 2005. Available at: http://www.iso.com (accessed January 2016).

- International Organization for Standardization (ISO): International Standard ISO 5636-5:2003. Paper and Board -- Determination of Air Permeance and Air Resistance (Medium Range) -- Part 5: Gurley Method; ISO, Geneva, Switzerland, 2003. Available at: http:// www.iso.com (accessed January 2016).
- International Organization for Standardization (ISO): International Standard ISO 7231:2010. Polymeric Materials, Cellular, Flexible -- Determination of Air Flow Value at Constant Pressure-Drop; ISO, Geneva, Switzerland, 2010. Available at: http://www.iso.com (accessed January 2016).
- 11. International Organization for Standardization (ISO): International Standard ISO 9237:1995. Textiles -- Determination of the Permeability of Fabrics to Air; ISO, Geneva, Switzerland, 1995. Available at: http://www.iso.com (accessed January 2016).
- 12. Baker, R.R.: The Viscous and Inertial Flow of Air Through Perforated Papers; Beitr. Tabakforsch. Int. 14 (1989) 253–260.
- 13. Eitzinger, B.: The Pore Size Distribution of Naturally Porous Cigarette Paper and its Relation to Permeability and Diffusion Capacity; Beitr. Tabakforsch. Int. 26 (2015) 312–319.
- Selke, A.W. and J.H. Mathews: The Permeability of Cigarette Papers and Cigarette Ventilation; Beitr. Tabakforsch. Int. 9 (1978) 193–200.

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