68th TSRC

Leak-based method for measurement of low air permeability of cigarette papers

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Introduction

What is air permeability (*AP*)?

Definition from ISO 2965:**1998** standard- *Determination of air permeability - suitable for the materials used as cigarette papers, filter plug wrap and filter joining paper, including material having a discrete or oriented permeable zone*

⇒ *Flow of air, measured in cubic centimeters per minute, passing through 1 cm^2 surface of the test piece at a measuring pressure of 1.00 kPa*

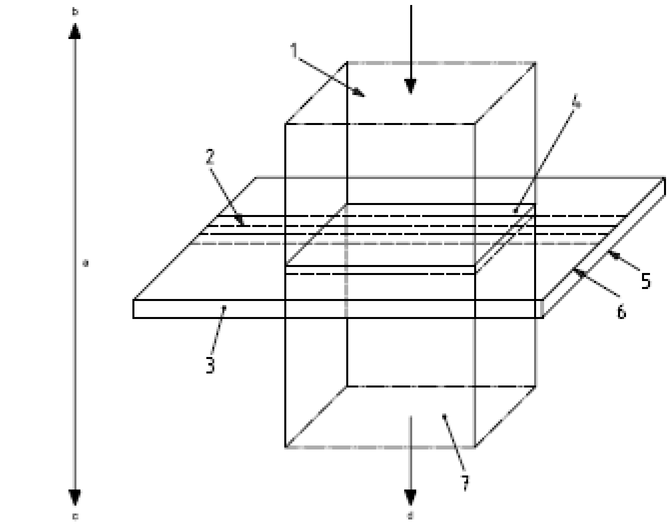
- Note: other standards related to fabrics, paper and board, as well as polymeric materials and cellular flexible have been developed for "air permeability measurement": ASTM D737, ISO 4638, ISO 5636-1 to 5, ISO 7231, ISO 9237.

Introduction

Principle of ISO 2965 method

- The paper sample is fixed in an appropriate device.
- A fixed pressure drop (1.00 kPa) is applied between the two faces of the samples.
- The resulting flow rate is measured.

- Note: in some cases, the function of flow with pressure is non-linear, it is required to carry out a second measurement under a pressure drop of 0.25 kPa.



Key

1 air stream
2 perforated zone (if present)
3 test piece
4 test surface of area (2 cm²)
a Pressure difference.
b High pressure.
5 inner surface
6 outer surface
7 air stream
c Low pressure.
d Direction of air flow.

Figure 1 — Principle of measurement

NF ISO 2965:2009

Introduction

<u>Tobacco regulation</u>: cigarette fire ignition propensity standard
⇒ ***Banded papers or LIP papers***
Such papers are characterized by **narrow paper bands** presenting **lower** air permeability.

The air permeability of the band, which is correlated to the ignition propensity of cigarettes, is therefore a **key quality parameter**.

⇒ <u>ISO 2965:**2009** - *Determination of air permeability - suitable for the materials used as cigarette papers, filter plug wrap and filter joining paper, including material having a discrete or oriented permeable zone* ***and materials with bands of differing permeability***</u>

Introduction

- « Weaknesses » of ISO 2965 method:

  – Need of a pressure regulator
  – Need of a flow meter
  – Need of flow calibration (permeability standards)

  ⇒Expensive method

- For LIP papers , the measurement of air permeability is difficult due to the narrow width and the particularly low level of the measured airflow.

Leak-based method for low air permeability

Objectives

- An alternative method to overcome the drawbacks: no pressure regulator, no flow meter and no permeability standards.
- This new method would be particularly suitable for low permeability levels, so mainly useful for LIP papers.

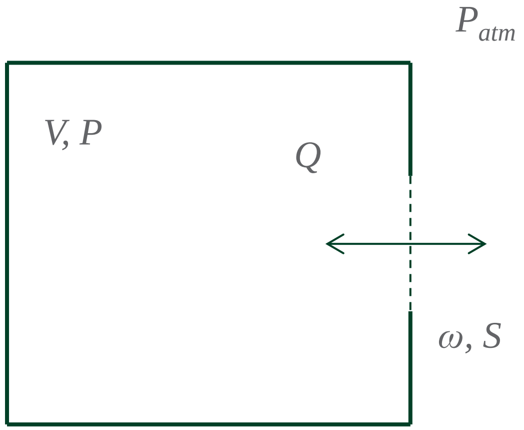
⇒ Based on the measurement of the ***evolution of the pressure over time when an initial difference of pressure is applied between the two faces of a sample.***

⇒ Theoretical aspects were developed. A first experimental investigation was conducted with banded papers and a cigarette paper.

Theory

Through cigarette papers, the airflow is the addition of a viscous and an inertial component (Baker, 1989)*. Thus, the relationship between airflow and the difference of pressure is:

$$Q = \omega_v \times S \times \Delta P + \omega_i \times S \times \Delta P^k$$



Q = flow rate
ω_v = the permeability of the paper related to the viscous flow
ω_i = the permeability of the paper related to the inertial flow
ΔP = difference of pressure between the two faces of the paper
S = area through which the airflow occurs
k = a constant comprised between 0.5 and 1.0

Baker, R.R., The viscous and inertial flow of air through perforated papers; Beitr. Tabakforsch. Int. 14 (1989) 253-260

Theory: viscous flow

Low permeability levels are usually associated with small paper pore size.

⇒ The airflow through such papers is essentially **viscous**, and then simply proportional to the difference of pressure.

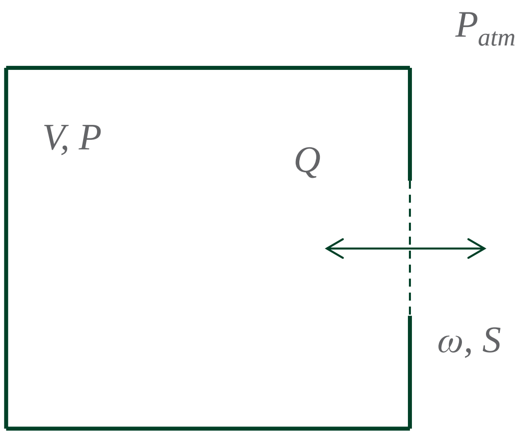
$$Q = \omega \times S \times \Delta P = \omega \times S \times |P_{atm} - P|$$

Theory: viscous flow

The volumetric airflow *Q* exiting or entering the measuring chamber is also a function of the number of mole *n* entering or exiting per unit of time.

From the perfect gas law, the following differential equation can be derived:

$$Q = \frac{dn}{dt} \times (Molar\ Volume) = \frac{dn}{dt} \times \frac{RT}{P}$$



T = Gas temperature in the measuring chamber
P = Gas pressure in the measuring chamber
R = Universal gas constant (8.314 $Pa.m^3.mol^{-1}.K^{-1}$)

Theory: viscous flow

By combining the previous equations, the air permeability can be linked to the evolution of the molar flow and the change in pressure:

$$\frac{dn}{dt} = \frac{1}{RT} \times \omega \times S \times P \times \Delta P$$

In the measuring chamber, the change of the number of mole *n* over time can be linked to the change of pressure and temperature by the perfect gas law:

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

By combining both equations (through the equalization of the derivative of *n)*, the following differential equation can be obtained:

$$\frac{d\Delta P}{dt} = \frac{(P_{atm} - \Delta P)}{V} \times \left[-\omega \times S \times \Delta P - \frac{V}{T} \times \frac{dT}{dt}\right]$$

Theory: viscous flow

Assuming that the temperature is constant in the measuring chamber as this is usually the case in laboratory conditions (ISO 3402), the equation becomes:

$$\frac{d\Delta P}{dt} + \frac{\omega \times S \times P_{atm}}{V} \times \Delta P - \frac{\omega \times S}{V} \times \Delta P^2 = 0$$

The analytical solution of this differential equation is:

$$\Delta P(t) = \frac{\Delta P_{init} \times P_{atm}}{(P_{atm} - \Delta P_{init}) \times e^{\frac{\omega \times S \times P_{atm}}{V} \times t} + \Delta P_{init}}$$

Air permeability determination – Approach 1

The first approach consists in the measurement of ΔP_1 at the time t_1 and ΔP_2 at the time t_2.

$$\Delta P_2 = \frac{\Delta P_1 \times P_{atm}}{(P_{atm} - \Delta P_1) \times e^{\frac{\omega \times S \times P_{atm}}{V} \times \Delta t} + \Delta P_1}$$

$\Delta t = t_2 - t_1$ with $t_2 > t_1$

The permeability ω can be easily deduced:

$$\omega = \frac{V}{S \times P_{atm} \times \Delta t} \times ln\left(\frac{\Delta P_1 \times P_{atm} - \Delta P_1 \times \Delta P_2}{\Delta P_2 \times (P_{atm} - \Delta P_1)}\right)$$

Air permeability determination – Approach 2

The first approach is related to the use of only two sets of data subject to variability.

The estimation of ω can be improved by recording the change of pressure over time.

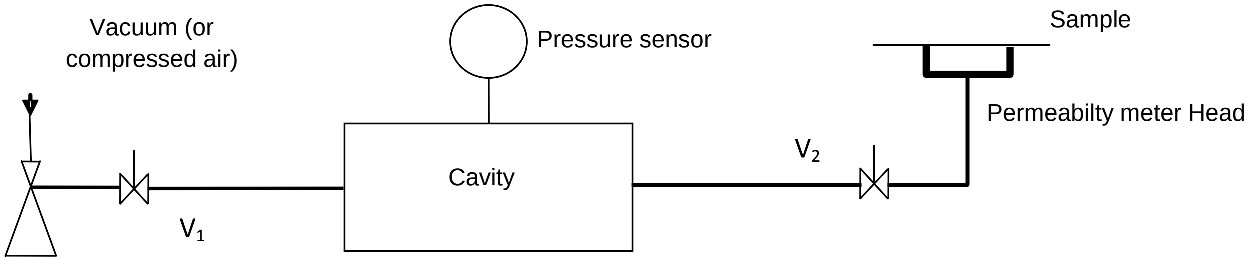
From the measurement of ΔP over time, the following function *f* can be calculated:

$$f(t) = \ln\left(\frac{\Delta P_{init} \times \left(P_{atm} - \Delta P(t)\right)}{\Delta P(t) \times \left(P_{atm} - \Delta P_{init}\right)}\right) = \frac{\omega \times S \times P_{atm}}{V} \times t$$

$\Rightarrow$ The estimation of the slope of *f versus time* by linear regression, provides an estimation of the permeability ω.

Experimental results

Based on the principle presented previously, a prototype has been developed.



⇒ Protocol:

- Valve V1 is opened and valve V2 is closed.
- The cavity is put under vacuum up to the targeted pressure.
- V1 is closed.
- The sample is inserted in the measurement head.
- V2 is opened: the pressure inside the cavity increases progressively (through the sample).

Experimental results

The AP of the sample can be calculated from the recording of the evolution of pressure over time in the cavity.

⇒ Material:

- 2 types of samples have been tested:
  - LIP papers (2)
  - Cigarette paper (1)

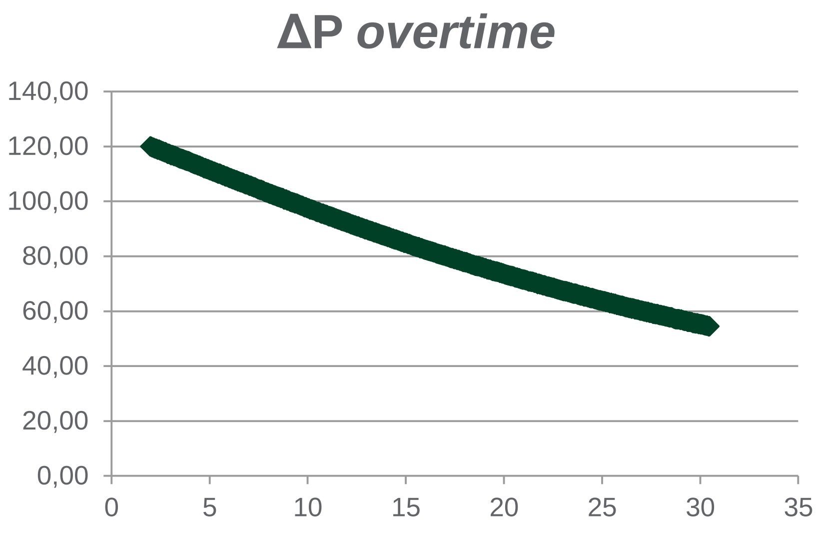
- Measuring head: 0.3 cm^2 (0.2cm x 1.5cm)
- Circuit volume (cavity and pipes) : 377ml
- Depressure inside the cavity : ≈ 1kPa
- Approach 2 for calculation of ω

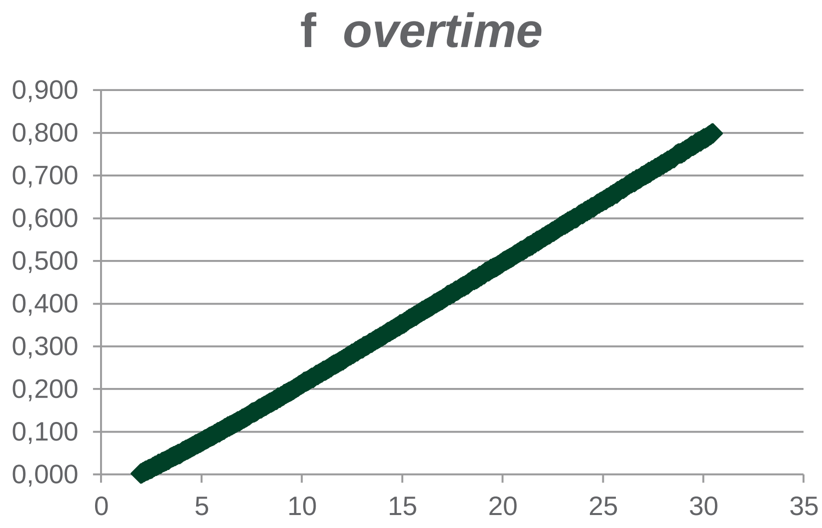
Experimental results

The results obtained with the described alternative method were compared to results measured on a permeameter P3 using ISO 2965 method.

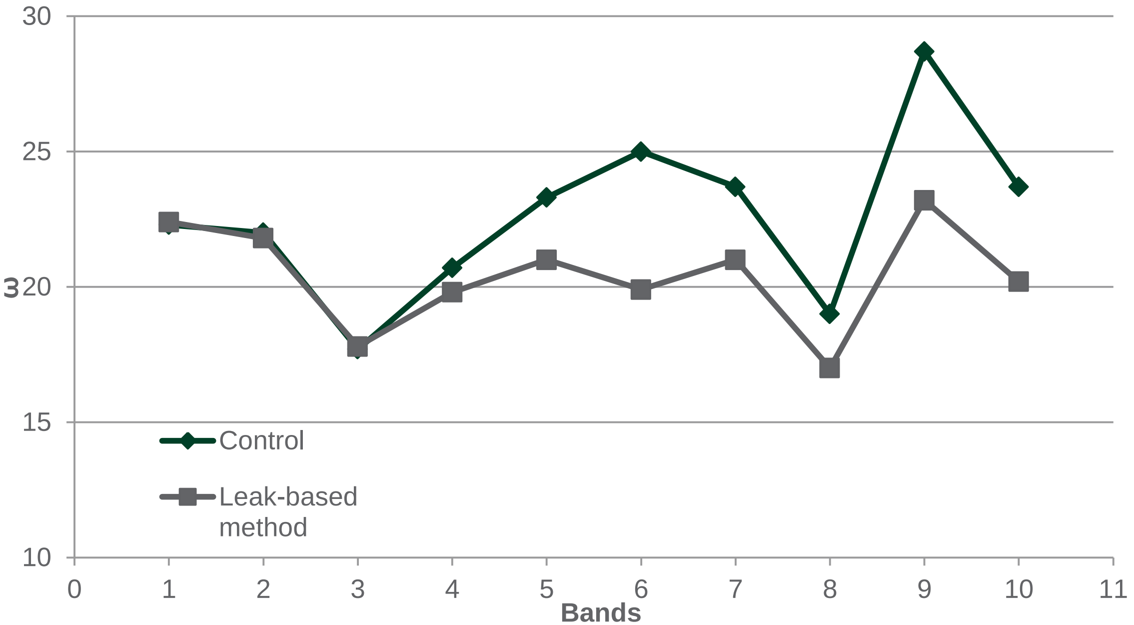
Banded (LIP) paper

LIP papers are well-known for their low permeability levels. *ΔP* overtime and function *f* were plotted:





Banded (LIP) paper ω ($cm^3.min^{-1}.cm^{-2}$)

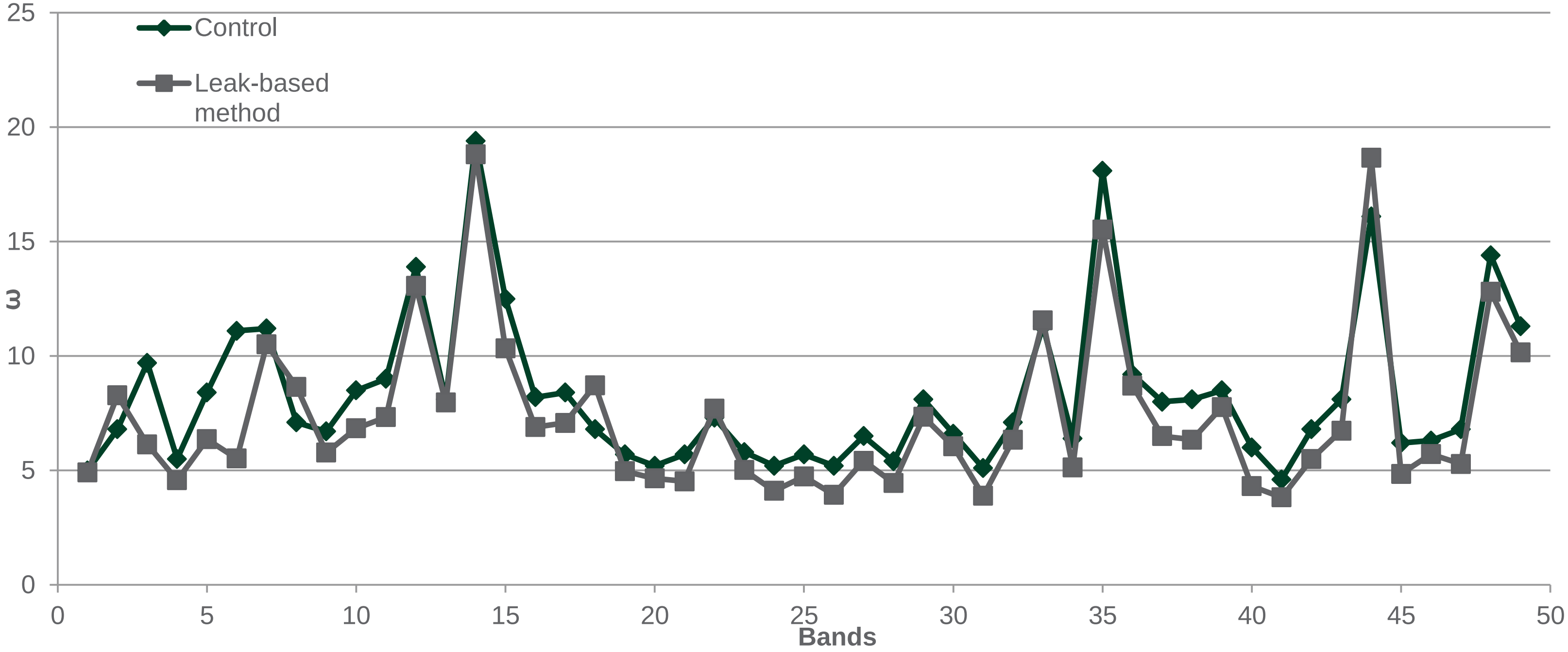


Repeatability on 10 different bands	Control	Leak-based method	Difference	Difference (%)
MEAN	22,6	20,4	2,2	9,7
SD	3,11	1,93		

*Repeatability on one band (*10)*	Control	Leak-based method
CoV (%)	2,9	0,3

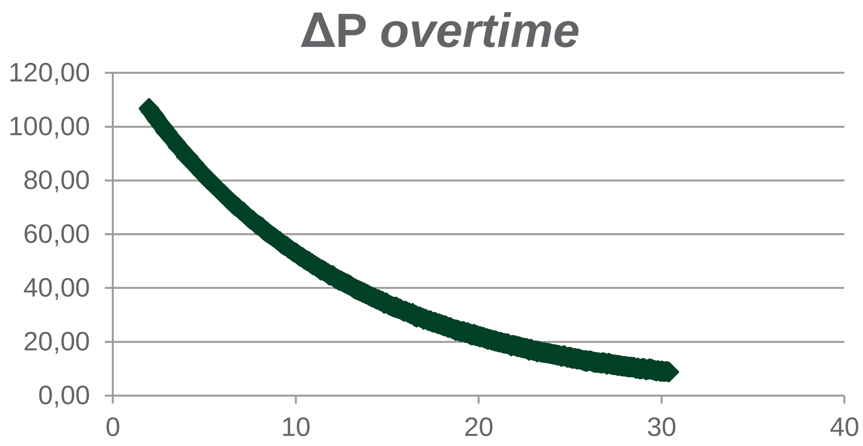
Note: all statistics have been calculated without outliers detection

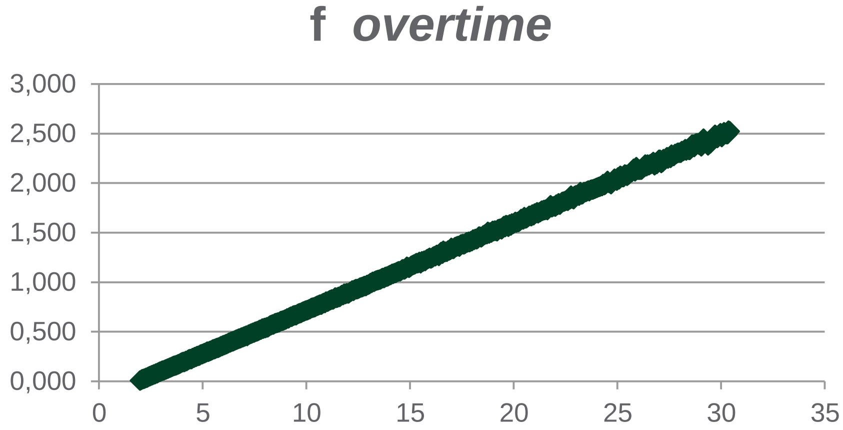
Banded (LIP) paper ω ($cm^3.min^{-1}.cm^{-2}$)



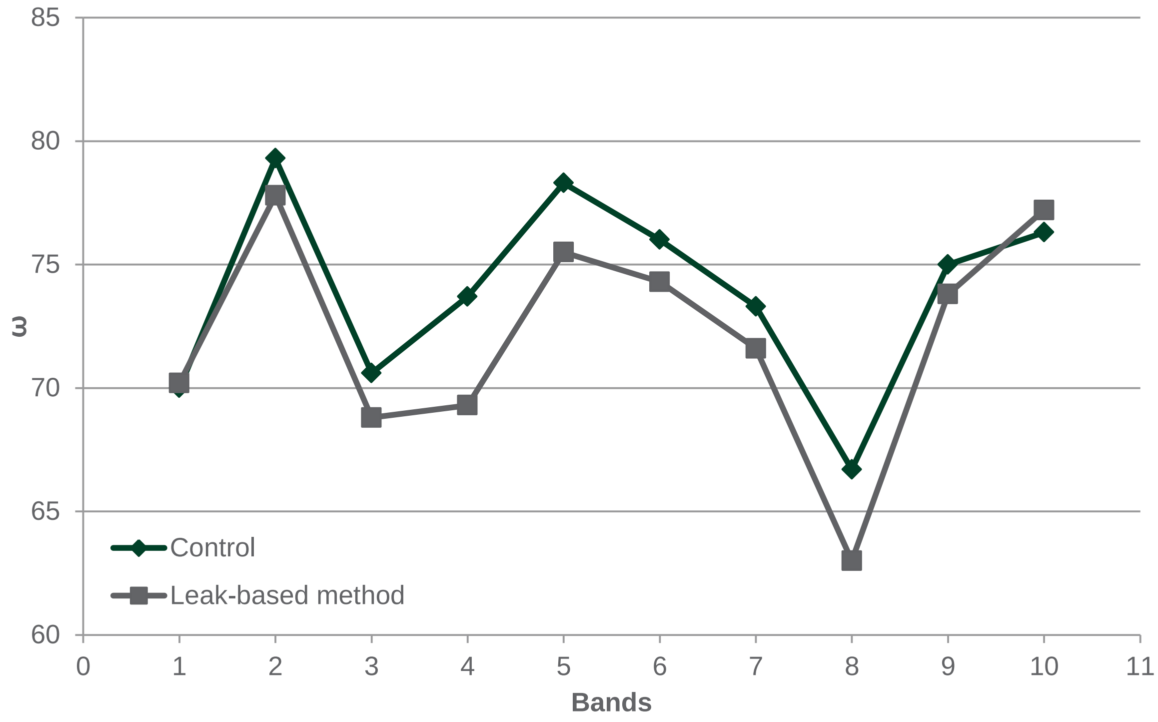
5 Repeatability on 10 different bands	**Control Mean**	*Control SD*	**Leak-based method Mean**	**Leak-based method SD**	**Difference**	**Difference (%)**
1	7,9	2,2	6,7	2,0	1,1	14,5
2	10,1	4,1	9,2	4,0	0,8	8,4
3	6,0	1,0	5,2	1,3	0,8	13,7
4	8,8	3,7	7,8	3,4	1,1	12,0
5	8,7	3,9	7,8	4,7	0,9	10,1

Cigarette paper





Cigarette paper ω ($cm^3.min^{-1}.cm^{-2}$)



Repeatability on 10 different positions	Control	Leak-based method
MEAN	73,9	72,2
SD	3,92	4,51
CoV (%)	5,3	6,3

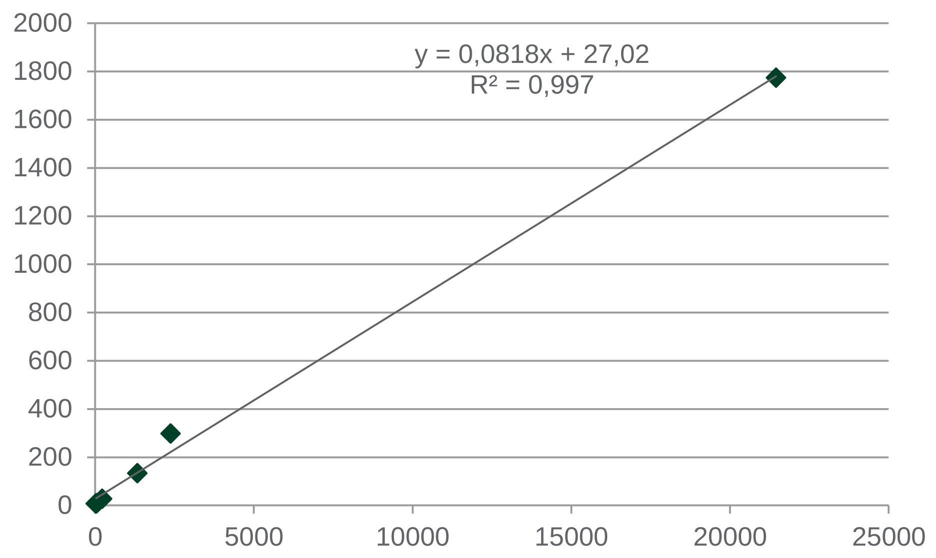
*Repeatability on one position (*10)*	Control	Leak-based method
CoV (%)	0,7	0,1

Cigarette paper R versus « AP level »

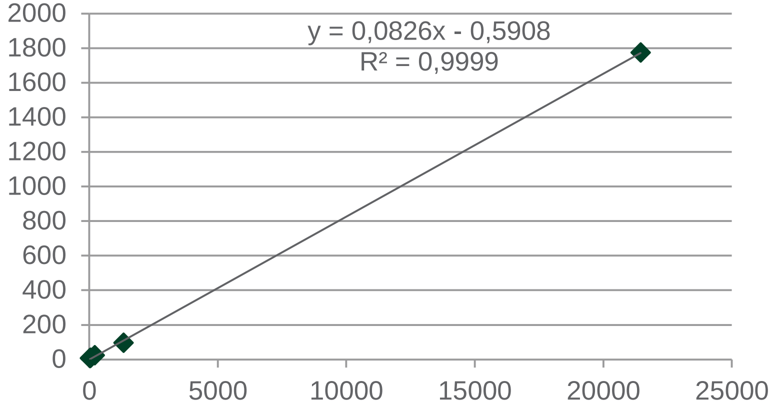
Table 2 — Alternative repeatability and reproducibility limits for Study 1

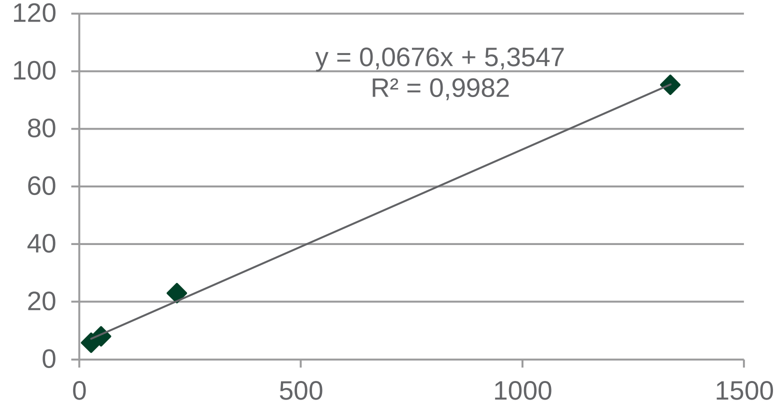
Mean air permeability [$cm^3 \cdot (min^{-1} \cdot cm^{-2})$ at 1 kPa]	Repeatability limit r	Reproducibility limit R
26,9	1,57	5,72
49,2	3,12	7,89
221	11,7	22,9
1 334	45,2	95,1
2 376	249[a]	297
21 449	519	1 773

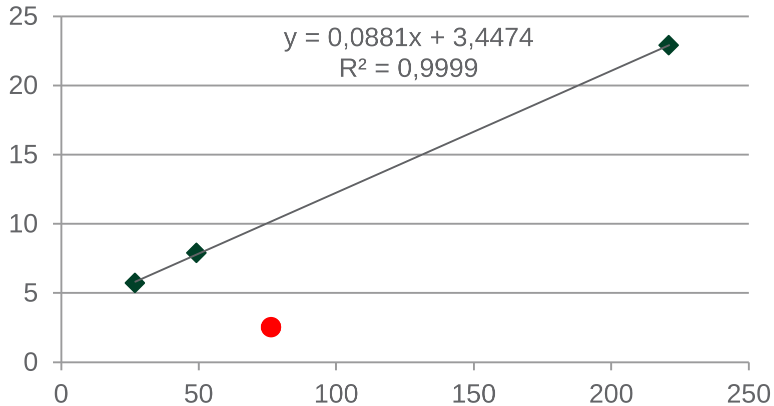
[a] See 9.4.



Cigarette paper R versus « AP level »







Control	Leak-based method	Difference	Difference (%)	Estimated R
73,9	72,2	1,8	2,4	10,0

Conclusion

- The leak-based method seems to be a possible alternative to ISO 2965 method for:
  - LIP paper
  - Cigarette paper
- Further tests on different papers would be needed to validate it.
- Another mathematical model for viscous and inertial flow has also been studied with consistent experimental results as well.
- Simple, reliable & cheaper method

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