STANDARDIZATION AND METROLOGICAL RELIABILITY OF CIGARRETE FIRMNESS RESULTS

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Abstract. *Cigarette quality can be evaluated by physical and chemical parameters. Characterizing most of these parameters is essential to defining the indices cigarette manufacturers use while monitoring the product on the market. Cigarette firmness is one of the parameters used for defining these indices and evaluating cigarette degree of rigidity. Generally, it can be defined as the ratio of the final cigarette diameter, following a compression test, to its initial diameter. Firmness is a function of three variables: cigarette upright deformation, as produced by applying a standard load; specified tobacco moisture, and cigarette mean circunference. With a view to enabling comparison between several products, the measured firmness is corrected to a previusly specified reference moisture value by having use of a model. In spite of this model having been validated for Souza Cruz products and considered as acceptable for the determination of the corrected firmness, the uncertainties associated with its use have not been evaluated. The tests described in this paper aim to qualify metrologically the measurements presently carried out to determine the cigarette firmness, the causes of errors and uncertainty of measurement. The conclusion was that product characteristics contribute to increasing the associated uncertainty, which results in poor repeatability. Finally, a methodology was developed to estimate the uncertainty of corrected firmness without the need of many measurements.*

Keywords*: Metrology, Cigarettes, Firmness, Uncertainty*

1. INTRODUCTION

The cigarette quality can be evaluated by means of several chemical and physical parameters. The chemical parameters are responsible for identifying and quantifying the tobacco components and the cigarette smoke. The physical parameters characterize the cigarette dimensions (length and circumference), mass, moisture, paper permeability, pressure drop, side loss, ventilation and firmness. Their measured values can be used to calculate quality indices, being used to monitor the product performance in the market.

Firmness is the rigidity of the cigarette, and relates the final to the initial diameter of the cigarette after a compression test, where a standard load is applied to the cigarette. The produced strain is a function of three variables: cigarette upright deformation; tobacco moisture, and cigarette mean circumference. Former studies indicate that moisture is the parameter that mostly influences the cigarette firmness. When comparing the performance of several cigarettes, the measured firmness must be corrected to a standard moisture value, which characterizes the commercial product. Souza Cruz follows (ISO 3402,1999) standard for conditioning the product at (60 ± 3) % relative humidity and (22 ± 1) °C temperature for a 48 h period before introducing it into the market. For process control, however, it is not possible to interrupt the production and store a sample for 48 h. A mathematical model must be used to estimate the firmness that could be obtained if the test was performed at the standard conditions.

The objective of this paper is to validate the experimental procedure used (Nogueira, 2005), and to estimate the uncertainty of the results, with 95,45 % confidence level.

2. EXPERIMENTAL METHODS

2.1 Firmness measurement

Two BORGWALDT firmness stations, respectively D-37 and DD-60A) models, were used to measure the cigarette firmness. A standard force is applied to the sample during a given time interval, resulting in its deformation along the vertical direction. Ten (10) cigarettes are placed horizontally side by side between two parallel plates. A standard load is applied over the upper plate, thus compressing the sample. The vertical displacement of the upper plate is thus measured, thus defining the final average cigarette upright dimension, which is divided by the cigarette initial diameter to give a percent value for its firmness.

2.2 Circumference measurement

A CTS circumference station, manufactured by CERULEAN/ FILTRONA, and a SODIMAT circumference station, manufactured by SODIM Instrumentation, are used by Souza Cruz to measure the average cigarette circumference, besides cigarette mass, pressure drop and ventilation, which do not influence directly the firmness measurement.

In the SODIMAT circumference station, the cigarettes are placed inside a test chamber, where each one is rotated. The mean cigarette circumference is measured 1024 times by a laser beam, as the cigarette makes a complete turn, with a resolution of 0,0025 mm. Average values and standard deviation are calculated. In the CTS circumference station, about 100 circumference measurements are performed in each cigarette, with a resolution of 0,01 mm, and repeatability of 0,05 mm.

Stainless steel circular cylinders are measured by a Laboratory accredited by the Brazilian Calibration Network, and used to calibrate the circumference stations.

2.3 Tobacco moisture measurement

An oven, manufactured by G.H. Bowen, is used by Souza Cruz to measure the amount of volatile compounds in the tobacco sample. Air, heated by an electric resistance, after having its temperature homogenized by a fan, is blown through five (5) vertically packed trays, each one containing twenty (20) cans filled with tobacco to be dried out. It is then released to the atmosphere, carrying the volatile compounds, mainly moisture, which are measured by weighting the samples before and after the test.

2.4 Tobacco conditioning

Souza Cruz has a conditioning room, where the dried tobacco samples are stored before going the next test. Its temperature is kept to within (22 ± 1) °C. Its humidity, to within (60 ± 3) %, according to (ISO 3402, 1999) standard. However, another chamber, manufactured by Binder GmbH, with a setting temperature resolution of ± 0,1 °C, and setting humidity resolution of ± 1 % RH, was used in these test to measure the time required for the tobacco samples to achieve equilibrium conditions.

3. QUALIFICATION OF THE MEASUREMENT PROCEDURE

3.1 Time required for conditioning tobacco samples

As a first step to qualify the measurement procedure, twelve (12) sets of ten (10) cigarettes, each, were placed in the conditioning chamber. The same amount of cigarettes was placed in the conditioning room. The same initial conditions were set for both experiments, (22 ± 1) °C for temperature, and (60 ± 3) % for relative humidity. The tobacco was weighted every 2 h over a three (3) day period.

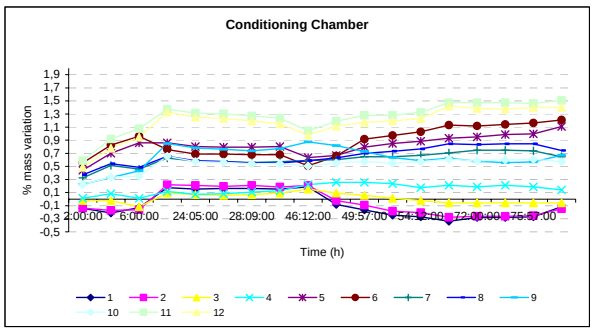

Figure1: % mass variation in conditioning chamber

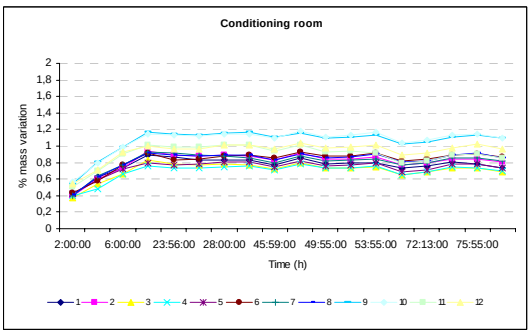

Figure 2: % mass variation in the conditioning room.

The objective of the tests was to evaluate the influence of the conditioning time on the moisture content of the sample, and also to compare the conditioning process for both conditioning chamber and room, including the influence of the sample position on the moisture absorption by tobacco. The tests were used to determine when there is no more moisture absorption by the tobacco, meaning that the samples are fully conditioned to the required temperature and relative humidity conditions.

Figure 1 shows the absorption of moisture as a function of time for each one of the twelve (12) trays placed in the conditioning chamber. It is shown that there is a trend towards absorbing more moisture in the upper trays, and that the central trays achieve quicker the equilibrium conditions.

Figure 2 shows that the stability in the conditioning room is higher than in the conditioning chamber. Even though, for operational reasons, it was decided to make the experiments in the conditioning chamber. Thus, it was decided to condition the tobacco in later tests for a period between 24 and 30 h, because the mass variation is less than 0,2 %, which meets the (ISO 3402, 1999) criterium for sample conditioning, that also suggests a 48 h conditioning time interval.

3.2 Tobacco moisture content after conditioning

The influence of the conditioning time of the sample on the tobacco moisture content was examined using six (6) samples containing six (6) sets of 20 cigarettes, each one. Three (3) samples were conditioned in the conditioned chamber for 24 h at, respectively, (75 ± 3) %, (60 ± 3) % and (40 ± 3) % relative humidity and (22 ± 1) °C temperature. Another three (3) samples were conditioned for 48 h, under similar temperature and relative humidity condtions. No significant differences were found in the 40 to 75 % relative humidity range.

3.3 Tobacco moisture content measurement

3.3.1 Influence of sample mass on moisture content measurement

Several experiments were conducted to determine the influence of the sample mass on the tobacco moisture content measurement. One hundred (100) cans filled with tobacco were placed in an oven, each one having previously specified a nominal tobacco mass of 8, 10 or 12g. The following parameters were measured for each can.

Table 1: Measured parameters for moisture determination

Symbol	Unit	Paramter
P_1	g	Mass of empty can
P_2	g	Mass of can filled with humid tobacco
P_3	g	Mass of humid tobacco
P_4	g	Mass of can filled with dry tobacco
P_5	g	Mass of dry tobacco
P_6	g	Mass of water and volatile products

The mass of water and volatile products (P_6) can be calculated as:

$$P_6 = P_3 - P_5 = (P_2 - P_1) - (P_4 - P_1) \tag{1}$$

Considering that the tobacco moisture content is not the same for each of n cans, together with the non uniformity of the drying process, an average value $(\overline{X})$ must be calculated using the mass of water and volatile products for each can (X_i), calculated from Eq. (1). Also, the standard uncertainty (u), according to (ISO GUM, 1995) can be calculated as its standard deviation, because the uncertainty of mass measurement is much smaller than the data dispersion due to non uniformity.

$$\overline{X} = \frac{1}{n}.\sum_{i=1}^{n} X_i \tag{2}$$

$$u = \sqrt{\frac{1}{n-1}.\sum_{i=1}^{n}(X_i - \overline{X})^2} \tag{3}$$

$$U = t.u \tag{4}$$

Table 2: Tobacco moisture content after having been dried for 3,5 h at 110 °C

Parameter	Nominal tobacco mass (g)					
	8		10		12	
	$\overline{X}$	u	$\overline{X}$	u	$\overline{X}$	u
Humid Tobacco (g)	8,000	0,0007	10,000	0,0007	12,000	0,0006
Water and Volatile Products (g)	1,123	0,0049	1,397	0,0101	1,671	0,0091
Tobacco moisture content (%)	14,038	0,0610	13,972	0,1011	13,926	0,0751

Using as a definition of the tobacco moisture content (H) the percent ratio between the mass of water and volatile products (P_6) and the mass of humid tobacco (P_3), Tab. 2 shows the measured values considering that each can has a nominal tobacco mass of 8, 10 and 12 g, , after having dried the samples in a oven for 3,5 h at 110 °C.

Table 2 indicates that to within the uncertainty of measurement the measurement of the tobacco moisture content does not depend on humid tobacco mass to be dried. Thus, an average value for the tobacco content can be calculated using all the measured values, independently of the humid tobacco mass to be dried. Using Eq. (4), the expanded uncertainty can be calculated for 95,45 % confidence level.

$$H = (13{,}98 \pm 0{,}18)\ \% \tag{5}$$

3.3.2 Influence of the drying time in the oven and use of a dehumidifier to complement the tobacco drying.

A similar experiment was conducted with 99 cans filled with tobacco to be dried during a 3 h time interval in an oven at 110 °C. Then, a dehumidifier was used to complement the drying process until the samples achieved a temperature of 50 °C, as suggested by a used methodology. The tobacco moisture content was slightly reduced and the uncertainty slightly increased.

$$H = (13{,}15 \pm 0{,}25)\ \% \tag{6}$$

The use of the normalized error (E) statistical test (Orlando, 2009) for comparing the two values (zero average statistical test) resulted in a value of 2,7. This value is well above 1 (95,45 % confidence level), indicating that the two procedures are different and systematic errors may be occurring. Possibly the samples are not completely dried in the last test.

$$E = \frac{13{,}98 - 13{,}15}{\sqrt{0{,}18^2 + 0{,}25^2}} = 2{,}7 \tag{7}$$

3.3.3 Influence of drying temperature on tobacco moisture content measurement

Three (3) experiments were conducted, each one with 20 cans filled with tobacco, in the oven temperature range around the set point of (110 ± 1) °C , during a 3,5 h time interval, simulating a drift in the set point.

Table 3 shows that there is a trend towards determining a larger tobacco moisture content when the temperature increases. However, when comparing the results at 109 °C and 111 °C, the normalized error is equal to 0,99, meaning that the results are still statistically meaningful (less than 1). Physically, this difference indicates that the tobacco is not completely dry at lower temperatures and needs to be in the oven during a larger time interval.

$$E = \frac{11{,}95 - 11{,}74}{\sqrt{0{,}15^2 + 0{,}15^2}} = 0{,}99 \tag{8}$$

Table 3: Tobacco moisture content $(\overline{X})$ determination at different oven temperatures

Oven temperature (°C)					
109		110		111	
$\overline{X}$	U	$\overline{X}$	U	$\overline{X}$	U
11,74	0,15	11,82	0,14	11,95	0,15

3.3.4 Influence of the amount of tobacco in the oven on its moisture content determination

Keeping the oven temperature (110 °C) and drying time (3,5 h) as constants, the influence of the amount of tobacco in the oven on the moisture content determination was investigated, by emptying some of the 100 cans placed inside the oven. Five (5) experiments were conducted, respectively, for 20, 25, 50, 75 and 100 cans filled with tobacco.

Table 4 indicates that all differences are smaller than the uncertainty of measurement, which means that the amount of tobacco in the oven does not seem to be important for tobacco moisture content determination. The normalized error between any two configurations is much smaller than 1, meaning that the results are statistically meaningful.

Table 4: Influence of the amount of tobacco in the oven on its moisture content determination $(\overline{X})$

Number of cans filled with tobacco									
20		25		50		75		100	
$\overline{X}$	U	$\overline{X}$	U	$\overline{X}$	U	$\overline{X}$	U	$\overline{X}$	U
12,87	0,24	12,90	0,29	12,80	0,26	12,76	0,29	12,87	0,24

3.3.5 Influence of drying time in the oven on the tobacco moisture content determination

The drying time of the tobacco in the oven was varied from 1 to 7 h to check its influence on the tobacco moisture content determination. It can be concluded from Tab. 5 that the tobacco needs to stay longer than 3,5 h in the oven to be completely dry and the systematic errors be reduced. As a conclusion, it was assumed that the uncertainty of measuring the tobacco moisture content is ± 0,29 %,

Table 5: Influence of drying time in the oven on the tobacco moisture content determination $(\overline{X})$

Parameter	Drying time (h)									
	1,0	1,5	2,0	2,5	3,0	3,5	4,0	5,0	6,0	7,0
$\overline{X}$	10,93	11,95	12,50	12,92	13,15	13,38	13,61	13,89	14,16	14,32
U	0,79	0,45	0,30	0,28	0,31	0,22	0,24	0,26	0,24	0,31

3.4 Circumference measurement

A CTS circumference station and a SODIMAT circumference station are used by Souza Cruz to measure the average cigarette circumference. They are calibrated using stainless steel cylinders, which are measured by the stations and thus calibrated for cigarette diameter measurement.

3.4.1 Calibration of the stainless steel cylinders used as reference materials for calibrating the stations.

Seven (7) stainless steel cylinders were manufactured in different diameters, ranging from 5 to 8 mm, in 0,5 mm steps. Ten (10) diameter measurements were made for each cylinder by a digital caliper, calibrated by a laboratory in the Brazilian Calibration Network to within U_p = ± 0,01 mm (95,45%). The standard uncertainty is thus $u_p = U_p/2$ = 0,005 mm. The average diameter ($\overline{d}$) was calculated using Eq. (2) and n = 10. The standard deviation (u_r) was calculated using Eq. (3) and n=10. Thus the combined uncertainty (u_d) can be calculated by Eq. (9). *The* expanded uncertainty (U_d) can be calculated using Eq. (4), with t= 2,32 (9 degrees of freedom). Average circumference ($\overline{C}$) and expanded uncertainty (U_C) are calculated using, respectively, Eq. (10) and (11).

$$u_d = \sqrt{u_p^2 + u_r^2} \tag{9}$$

$$\overline{C} = \pi.\overline{d} \tag{10}$$

$$U_C = \pi.U_d \tag{11}$$

Table 6 : Calibration of the stainless steel cylinders (CTS circumference station position)

Parameter	Cylinder nominal diameter (mm)						
	5,00	5,50	6,00	6,50	7,00	7,50	8,00
u_p	0,005	0,005	0,005	0,005	0,005	0,005	0,005
u_r	0,00000	0,00000	0,00000	0,00000	0,00000	0,00000	0,00516
u_d	0,005	0,005	0,005	0,005	0,005	0,005	0,007
$\overline{d}$	4,990	5,500	6,000	6,500	7,000	7,500	8,014
$\overline{C}$	15,677	17,279	18,850	20,420	21,991	23,562	25,177
U_C	0,036	0,036	0,036	0,036	0,036	0,036	0,052

Table 6 shows the results of the calibration of the CTS circumference station. Diameters were measured at the same position along the cylinder length that the CTS test station measures the cigarette diameter. Similar measurements were made also at the same position along the cylinder length that the SODIMAT circumference station measures the cigarette diameter. The normalized error test shows that there is no statistical difference between the results. Even though, a different calibration curve was used for the SODIMAT circumference station position.

3.4.2 Calibration of the circumference stations

During the calibration for circumference measurement, seven (7) stainless steel cylinders with different diameters, used as reference materials, were measured ten (10) times, each one, by both test station and digital caliper. A correction (e) was calculated as the difference between the average values indicated by the test station $\left(\overline{C}\right)$ and by the digital caliper (C). However, it was assumed that no correction should be added to the indicated value by the test station. Therefore, this systematic error was considered as a type B one, with standard uncertainty (u_e) expressed as :

$$u_e = \frac{|e|}{\sqrt{3}} \tag{12}$$

The combined uncertainty of measuring the circumference by the circumference station was estimated with the help of the following components.

- Standard uncertainty of measuring circumference by the digital caliper (u_C), Tab. 6 and 7
- Repeatability of measuring circumference by the circumference station (u_r), using Eq. (3), and n=10.
- Standard uncertainty of reading the circumference by the circumference station (u_l), calculated as the ratio between the reading resolution of the circumference station and $\sqrt{3}$.

The combined uncertainty of measuring the circumference by the circumference station (u_{ET}) can thus be calculated by Eq. (13) and (14), where u_σ is the uncertainty component due to data dispersion (Orlando, 2009).

$$u_\sigma = \sqrt{u_l^2 + u_r^2} \tag{13}$$

$$u_{ET} = \sqrt{u_\sigma^2 + u_e^2 + u_C^2} \tag{14}$$

The expanded uncertainty (U_{ET}) can be calculated using Eq. (4). Table 7 shows the results of the calibration of the CTS circumference station, using seven (7) stainless steel cylinders with different diameters. The same procedure was used for the SODIMAT circumference station. The normalized error test shows that there is no statistical difference between the results. Even though, a different calibration curve was used for the SODIMAT circumference station.

Table 7: Calibration of CTS circumference station

Parameter	Cylinder nominal diameter (mm)						
	5,00	5,50	6,00	6,50	7,00	7,50	8,00
u_C	0,018	0,018	0,018	0,018	0,018	0,018	0,026
u_l	0,00577	0,00577	0,00577	0,00577	0,00577	0,00577	0,00577
u_r	0,00707	0,00516	0,00667	0,00471	0,00316	0,00422	0,01059
C	15,677	17,279	18,850	20,420	21,991	23,562	25,177
$\overline{C}$	15,715	17,286	18,860	20,420	22,011	23,558	25,147
e	-0,038	-0,007	-0,010	0,000	-0,020	0,004	0,030
u_e	0,022	0,004	0,006	0,000	0,011	0,002	0,017
u_{ET}	0,030	0,020	0,021	0,020	0,023	0,020	0,034
U_{ET}	0,070	0,047	0,049	0,046	0,052	0,046	0,078

3.4.3 Uncertainty of measurement of cigarette circumference using circumference stations

The cigarette uniformity and repeatability of the measurement procedure was evaluated by substituting the stainless steel cylinders for three types of cigarettes (F,H,B), with different brands and diameters. Each of the ten (10) cigarettes was measured six (6) times in each test station, using the same methodology as before. Table 8 shows the cigarette diameter and circumference measurement with a digital caliper for each test station. Table 9 shows similar results as measured by each circumference station.

From Tab. 6 and 8 it can be concluded that the uncertainty of measuring the average circumference with a digital caliper is much smaller for the rigid cylinders than for the cigarette, probably due to non-rigidity of the cigarette, what makes the measurement pressure an important and non repeatable parameter. This fact justifies why, for the digital caliper, the measured circumference is smaller and data dispersion is larger. As a conclusion, it was assumed that the uncertainty of measurement of the cigarette circumference by the circumference station was equal to the largest value in Tab. 9, that is, ± 0,25 mm.

Table 8 : Cigarette diameter measurement with a digital caliper (mm)

Parameter	CTS circumference station			SODIMAT circumference station		
	F	H	B	F	H	B
u_p	0,005	0,005	0,005	0,005	0,005	0,005
u_r	0,163	0,144	0,067	0,109	0,106	0,102
u_d	0,163	0,144	0,068	0,109	0,106	0,102
$\overline{d}$	7,451	7,069	5,301	7,481	7,119	5,340
C	23,41	22,21	16,65	23,50	22,36	16,78
U_C	1,04	0,92	0,43	0,70	0,68	0,65

Table 9 : Cigarette circumference measurement with the circumference stations (mm)

Parameter	CTS circumference station			SODIMAT circumference station		
	F	H	B	F	H	B
C	23,41	22,21	16,65	23,50	22,36	16,78
$\overline{C}$	24,30	22,94	17,03	24,28	23,00	16,98
e	-0,89	-0,73	-0,37	-0,77	-0,63	-0,20
u_l	0,00577	0,00577	0,00577	0,00577	0,00577	0,00577
u_r	0,059	0,056	0,098	0,094	0,065	0,122
u_σ	0,059	0,056	0,098	0,094	0,065	0,122
U_σ	0,12	0,12	0,20	0,19	0,13	0,25

3.5 Firmness measurement

3.5.1 Calibration of the reference cylinders

The calibration of the firmness stations was made using, as transfer reference materials, six (6) 80 mm long stainless steel cylinders. Each cylinder diameter was measured along its axis by a coordinate measuring machine. An average diameter (d) was calculated as the arithmetic mean between the maximum (d_{max}) and minimum (d_{min}) diameters. The type A uncertainty of diameter measurement was selected as the maximum value (U_{max}) among those in the calibration certificate for different diameters along the cylinder axis. The diameter uniformity (U_h) was defined as a type B uncertainty and calculated as the half the difference between the maximum (d_{max}) and minimum (d_{min}) diameters. Thus, the combined (u_d) and the expanded (U_d) uncertainties for the cylinder diameter can be expressed (Orlando, 2009) by, respectively, Eq. (15) and (16) and shown in Tab. 10.

$$u_d = \sqrt{u_{\max}^2 + u_h^2} \tag{15}$$

$$U_d = 2.u_d \tag{16}$$

Table 10 : Calibration of the reference cylinders (mm) for firmness measurement.

Parameter	Reference cylinder (mm)					
	A	B	C	D	E	F
d	7,9934	7,9940	6,000	6,003	4,003	4,002
u_{max}	0,0022	0,0020	0,0015	0,0015	0,0015	0,0015
u_h	0,0003	0,0005	0,0000	0,0003	0,0000	0,0000
u_d	0,0022	0,0021	0,0015	0,0015	0,0015	0,0015
U_d	0,0044	0,0042	0,0030	0,0030	0,0030	0,0030

3.5.2 Uncertainty of vertical displacement measurement using the reference cylinders

Using the same methodology for calibrating the circumference stations, the D-37 and DD-60A Borgwaldt firmness stations were calibrated with the reference cylinders, indicating that the uncertainty of the final cigarette diameter after the compression test is in the ± 0,014 to ± 0,021 mm range. Thus, a value of ± 0,021 mm was assumed for the uncertainty of final cigarette diameter, with is approximately twice its resolution. Equation (2) is used to calculate the average value of the measured diameter by the firmness meter ($\overline{d}$), using reference cylinders A,B,C,D,E and F with diameter d and its combined uncertainty (u_d) , given in Tab.10.The combined uncertainty of the final cigarette diameter (u_F) is given by Eq. (1), together with its expanded uncertainty (U_F), calculated by Eq. (4).

$$u_F = \sqrt{u_l^2 + u_r^2 + u_e^2 + u_d^2} \tag{17}$$

Table 11 : Calibration of the firmness stations for vertical displacement measurement (mm)

Parameter	BORGWALDT-1 D-37			BORGWALDT-2 DD-60A			BORGWALDT-3 DD-60A		
	E,F	C,D	A,B	E,F	C,D	A,B	E,F	C,D	A,B
$\overline{d}$	4,01	6,00	8,00	4,00	6,00	7,99	4,00	6,00	7,99
d	4,00	6,00	7,99	4,00	6,00	7,99	4,00	6,00	7,99
u_e	0,0014	0,0009	0,0031	0,0014	0,0009	0,0021	0,0026	0,0032	0,0027
u_l	0,0058	0,0058	0,0058	0,0058	0,0058	0,0058	0,0058	0,0058	0,0058
u_r	0,0053	0,0000	0,0032	0,0000	0,0000	0,0000	0,0042	0,0052	0,0032
u_F	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01
U_F	0,019	0,014	0,018	0,014	0,014	0,015	0,018	0,020	0,017

3.5.3 Firmness measurement

Firmness is the rigidity of the cigarette, and relates the final (L) to the initial diameter ($d=C/\pi$) of the cigarette, with circumference C, after a compression test, where a standard load is applied to the cigarette. Therefore, the indicated firmness value (F_i) at the test conditions can be calculated as ,

$$F_i = \frac{L.\pi}{C} \tag{18}$$

3.5.4 Comparison of firmness results at different cigarette moisture contents

Comparing different products with different moisture contents is made at Souza Cruz by estimating the value of the firmness that would be measured by the firmness station if the moisture content were at a reference condition of 13,5 %. It is called corrected firmness (F). Several experiments were performed, and the following empirical conversion expression (Baridó, 2002), Eq. (19), is presently used by Souza Cruz, starting from the measured values of vertical displacement (L), circumference (C) and moisture content (H).

$$F = 100 - 100.\left(1 - \frac{L.\pi}{C}\right).\left(\frac{13{,}5}{H}\right)^{1,6} \tag{19}$$

3.5.5 Influence of the non uniformity of the product on the cigarette firmness measurement

As seen from Tab. 11, the firmness station measures the vertical displacement to within ± 0,021 mm, using reference stainless steel cylinders. When measuring the product firmness, however, due to the fact that the cigarette properties vary, there is a need to determine an effective value for the firmness uncertainty that takes into account the product non uniformity. Two samples of 50 cigarettes each for each brand (D and K, respectively) were tested in two (2) Souza Cruz quality control laboratories (CPD and UDI, respectively). Table 12 shows the average measured values of vertical displacement ($\overline{L}$), circumference ($\overline{C}$), moisture content ($\overline{H}$) and corrected firmness ($\overline{F}$), together with, respectively, their standard deviation s_L , s_C, s_H and s_F. It can be seen that due to non uniformity of the product the data dispersion is much larger than its uncertainty of measurement .Therefore, in order to take into account the non uniformity of the product, it was decided to use an effective value of u_L = ± 0,09 mm or U_L = ± 0,18 mm for the effective vertical displacement uncertainty.

Table 12: Influence of product non uniformity on cigarette corrected firmness measurement

LAB	Cigarette	$\overline{L}$	s_L	$\overline{C}$	s_C	$\overline{H}$	s_H	$\overline{F}$	s_F
CPD	D	5,51	0,09	24,35	0,08	13,72	0,14	71,78	1,12
UDI	D	5,32	0,09	24,30	0,08	13,37	0,19	68,20	1,19
CPD	K	5,63	0,06	24,35	0,05	13,48	0,13	72,53	0,88
UDI	K	5,46	0,07	24,32	0,06	13,05	0,22	68,84	1,38

4. ESTIMATING THE UNCERTAINTY OF THE CORRECTED FIRMNESS MEASUREMENT

Without considering the uncertainty of the empirical conversion expression, the uncertainty of estimating the firmness (u) at the reference moisture content of 13,5 % can be calculated.

$$c_L = \frac{\partial F}{\partial L} = \frac{100.\pi}{C}.\left(\frac{13{,}5}{H}\right)^{1,6} \tag{20}$$

$$c_C = \frac{\partial F}{\partial C} = -\frac{100.L.\pi}{C^2}.\left(\frac{13{,}5}{H}\right)^{1,6} \tag{21}$$

$$c_H = \frac{\partial F}{\partial H} = -100.\left(1-\frac{L.\pi}{C}\right).1{,}6.\left(\frac{13{,}5}{H}\right)^{0,6}.\left(-\frac{13{,}5}{H^2}\right) \tag{22}$$

$$u = \sqrt{\left(c_L.u_L\right)^2 + \left(c_C.u_C\right)^2 + \left(c_H.u_H\right)^2} \tag{23}$$

Estimating the uncertainty of the corrected firmness measurement can be made by using the same average values of L, C , H and F from Tab. 12. However, to take into account the non uniformity of the product, rather than using their uncertainty of measurement, a maximum value in all situations were used, that is, u_L = 0,09 mm (item 3.5.2), u_C = 0,125 mm (item 3.4.3) and u_H = 0,145 % (item 3.3.5). The combined uncertainty of corrected firmness (u) can be calculated from Eq. (23).

An analysis of Tab. 12 and 13 shows that the calculated values of the standard uncertainty of the corrected firmness measurement is slightly larger than the standard deviation. The advantage of using this procedure is that there is no need of measuring several times to determine the data dispersion and thus the uncertainty of measurement. Also, it can be concluded from Tab. 13 is that largest contribution to the corrected firmness uncertainty of measurement is due to vertical displacement measurement, followed by moisture content, and, finally, circumference.

Using Eq. (4) the expanded uncertainty of the corrected firmness measurement (*U*) can be estimated in ± 2,6 , without considering the uncertainty of the correlation, Eq. (19), as developed by Souza Cruz (Baridó, 2002).

Table 13: Uncertainty of the corrected firmness measurement (u)

LAB	Cigarette	$\overline{L}$	$c_L.u_L$	$\overline{C}$	$c_C.u_C$	$\overline{H}$	$c_H.u_H$	$\overline{F}$	u
CPD	D	5,51	1,13	24,35	0,35	13,72	0,46	71,78	1,30
CPD	K	5,63	1,16	24,35	0,37	13,48	0,46	72,53	1,30

5. CONCLUSIONS

As a first part of this study, it was shown that the cigarette samples to be measured can be stored in a conditioning chamber between 24 to 30 h, so that the tobacco moisture content varies less than 0,2 % over a 3 h period, meeting (ISO 3402, 1999) standard.

As a second part of this study, several experiments were conducted to qualify the presently used methodology for measuring the cigarette firmness as a function of its moisture content and circumference, identifying the sources of errors and estimating the uncertainty of measurement. The calibration of the equipments was important for this analysis. A careful check of the results indicate that the instruments are measuring correctly all the parameters, without the need of corrections. Their uncertainties are low, showing that the differences are due to the properties of the tobacco, which increase the data dispersion. Therefore, a methodology was developed to take them into account, after having analyzed tests with many cigarettes in two Souza Cruz quality laboratories. As a result, the effective uncertainty of measurement, with 95,45 % confidence level, was estimated in ± 0,25 mm for circumference, ± 0,29 % for moisture content and ± 0,18 mm for vertical displacement measurement in the firmness station.

Finally, a methodology was developed to determine the uncertainty of the corrected firmness measurement to a reference moisture content of 13,5 %, using the above uncertainties that takes into account the tobacco non uniformity. The mean cigarette circumference was found to have the smallest contribution to the corrected firmness uncertainty. The vertical displacement was found to have the largest contribution. The expanded uncertainty of the corrected firmness measurement (U) can be estimated in ± 2,6 , without considering the uncertainty of the correlation, Eq. (19).

As a result of this work, it was found that each type of cigarette must be examined separately because they have different properties. Then the dispersion can be smaller than the one that can be obtained including all of them together.

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7. REFERENCES

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