

Cigar Smoking Methods Sub-Group

Technical Report

Summary of the 7 Collaborative Studies Results 2006 - 2012

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1. SUMMARY

The following report was requested by the Scientific Commission to summarise the work of the CORESTA Cigar Smoking Methods Sub-Group (CSM SG) between 2006 and 2012.

The CSM SG was established in 2006 with the following objectives:

- 1. To develop and update CORESTA Recommended Methods as requested by the Scientific Commission by investigating the technical problems associated with the mechanical smoking of cigars.
- 2. To conduct periodical collaborative studies in order to improve repeatability and reproducibility in different cigar sizes and types.
- 3. To establish confidence intervals for the smoke yields of all different cigar sizes.

During that time, 7 Collaborative Studies have been conducted and the objectives have been met.

In this Report, the results are reviewed and commented.

2. INTRODUCTION

The former CORESTA Cigars Sub-Group sent a Technical Report (dated 19th April 2005) to the Scientific Commission with a summary of all the Collaborative Studies (CS) carried out by their members from 2002 to 2005 (6th to 9th Collaborative Studies) (see Annex III). These earlier studies led to the issue of the CORESTA Recommended Methods (CRMs) related to Machine Smoking of Cigars except for the later one for the determination of Carbon Monoxide in Cigar Smoke (as shown in Table 1).

The set of CRMs was improved by subsequent work of the Sub-Group and completed by the CRM for measuring CO in the Cigar Smoke. The following list of approved cigar-related CRMs is available on the CORESTA website.

N° 46	Atmosphere for Conditioning and Testing Cigars of all Sizes and Shapes	May 1998
N° 47	Cigars - Sampling	Jan. 2000
N° 64	Routine Analytical Cigar-Smoking Machine - Specifications, Definitions and Standard Conditions	Nov. 2005
N° 65	Determination of Total and Nicotine-Free Dry Particulate Matter using a Routine Analytical Cigar-Smoking Machine – Determination of Total Particulate Matter and Preparation for Water and Nicotine Measurements <i>(Fourth updated edition)</i>	June 2010
N° 66	Determination of Nicotine in the Mainstream Smoke of Cigars by Gas Chromatographic Analysis	Nov. 2005
N° 67	Determination of Water in the Mainstream Smoke of Cigars by Gas Chromatographic Analysis	Nov. 2005
N° 68	Determination of Carbon Monoxide in the Mainstream Smoke of Cigars by Non-Dispersive Infrared Analysis	Jan. 2010

Table 1. Cigar related CORESTA CRMs

In the Technical Report (dated 19th April 2005) it is explained how the smoking regime for cigars was obtained. The working group had started smoking larger cigars around that time in order to fulfil the request of the Scientific Commission to develop a smoking method for all cigar sizes. However those bigger cigars had to be relighted again and again. Therefore it had been decided that the puff volume had to be increased. For comparison reasons it had been decided to apply a constant air velocity for those cigars where the frequent re-lighting occurred, i.e. cigars with a diameter > 12 mm. Keeping a constant air velocity would keep the same burning rate in the burning cone as it would help maintaining the same burning temperature in it through supplying the same amount of oxygen no matter which the cigar diameter was.

With this regime, the cigars with a diameter $\leq 12,0$ mm are smoked with a **puff** volume of 20 ml. For cigars with a diameter > 12 mm the puff volume is continuously modulated in order to obtain a constant air velocity of 11,8 cm/s. The formula to calculate it is: **puff volume (ml) = 0.139*Ø**² where Ø is the diameter in mm. **Puff duration is 1.5s** and **puff interval is 40s** for all the cigars.

The formula for continuously modulating the puff volume in order to obtain a constant air velocity was obtained as follows:

The basic formula for fluid dynamics is:

$$Q(cm^{3}/s) = S(cm^{2}) * V(cm/s)$$

Where Q= Flow, S= Cross Section and V= Velocity

The flow Q inside the cigar is calculated by the following formula:

Q = Puff volume / puff duration = 20 cm^3 / 1.5 s =13.3333 cm³ / s

Then the air velocity inside a cigar of 12 mm of diameter is calculated, which is the limit beyond which the same air velocity is maintained in bigger diameters.

The calculation is the following:

Section S (in cm²) = $\Pi + r^2 = \Pi + (\emptyset/2)^2 = \Pi + (1.2/2)^2 = 1.13097 \text{ cm}^2$

Where r is the cigar radius, Ø the diameter and the values are expressed in cm. The Diameter chosen has been 12 mm = 1.2 cm. Π is used = 3.14159

Substituting the calculated values in the initial formula Q (cm^3/s) = S (cm^2) * V (cm/s), we can obtain the velocity to be maintained in higher diameter cigars:

$13.3333 \text{ cm}^3 / \text{s} = 1.13097 \text{ cm}^{2*} \text{ V} (\text{cm/s})$

Then V (cm/s) = 13.3333 / 1.13097= 11.78926 cm/s (air velocity inside a cigar of 12 mm of diameter)

In the initial formula Q (cm^3/s) = S (cm^2) * V (cm/s):

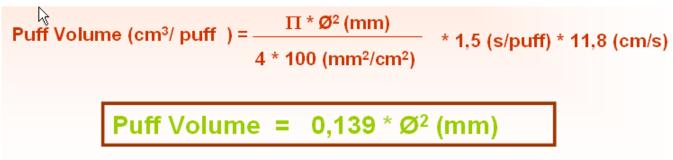
- substitute Q = Puff volume / puff duration (1.5 s/puff)
- Section S is calculated (in cm²) as $\Pi * (\emptyset/2)^2$, but using the measure of the diameter in mm (as measured in the labs) instead of cm
- then calculate the Section S (in cm²) as Π * Ø² (mm) / (2² * 10²) as 1 cm = 10 mm
- and use the calculated air velocity V = 11.78926 cm/s

to obtain the formula for the puff volume calculation (in $ml = cm^3$) with the diameter measured in mm:

Puff volume/1.5 = $\Pi * (\emptyset^2/4*100) * 11.789$

And finally isolating the puff volume, we obtain the formula expressed in Figure 1:





The work for obtaining the mentioned regime was guided by the following reasons:

- 1. Smoking cigars of *all shapes and sizes*, based on one single set of parameters does not provide a credible and useful comparison of their smoke chemistry;
- 2. The Sub-Group is of the opinion that the smoking regime currently used (with a puff volume of 20 ml) is suitable for the machine-smoking of cigars with a diameter up to a maximum of 12 mm. By this limitation, the majority of the brands available on the market were covered (e.g. 81 % in the European market in 2005);

Once the first set of CRMs based on this smoking regime was approved by the Scientific Commission, a new Sub-Group was established in 2006 under the name of Cigar Smoking Methods Sub-Group.

3. GLOBAL RESULTS OF 7 COLLABORATIVE STUDIES 2006 - 2012

Before stating the conclusions of each of the 7 Collaborative Studies (CS) conducted since 2006 until 2012 some summarized information of the global results is given and some basic information about how these results were obtained:

- 1. 9 to 14 laboratories have participated in these CS. Annex I gives the list of the 19 laboratories that have participated through the 7 CS.
- 2. Through all the CS the same 5 cigar products have been smoked. Annex II gives their Product Codes, physical characteristics and smoking parameters applied. Product A has been the CORESTA Monitor: the CM5 version in the 1st and 2nd CS and the CM6 after the 4th CS. It should be noted that although the rest of the products are available brands in the market, the cigars smoked do not come from the same production batch (except for the first two CS) leading to potential temporal variations.
- 3. Statistical evaluation of the data for all the CS have been conducted following the methods provided by ISO 5725-2 (1994) "Basic method for the determination of repeatability and reproducibility of standard measurement method". For outlier testing, the Grubbs and Cochran methods were used before relevant repeatability "r" and reproducibility "R" for all the CS were calculated.

- 4. The smoking methods used were CORESTA approved as described in the Sub-Group Cigars Technical Report (dated 19 April 2005 see Annex III). The CO CRM was not approved until 2010. For this reason, for the 1st CS no statistical analysis for r and R on CO was done while they were calculated for the CO results from the 2nd to the 6th CS. Notwithstanding it should be noted that in the protocol for the 2nd CS no Draft Recommended Method was included. In preparation of the 3rd CS a working group prepared a Draft Recommended Method, and this method was included in the protocol for the 3rd, 4th and 5th. Out of this work, an approved CRM has been used since the 6th study.
- 5. The approved CRM established that smoke yields should be expressed in two ways: mg per cigar and mg per g of tobacco (total cigar weight). The second way allows a better comparison between results of cigars of all sizes and shapes. But for practical reasons the results analyzed on all the CS were expressed in mg per cigar except in the 4th CS where the results appeared in both ways.

Reports on each of the aforementioned CS are available on request to the CORESTA Secretariat.

The global results on averages, repeatability and reproducibility (absolute and relative) obtained in the 5 products through the 7 CS, once the outliers had been removed (except for CO from 1st Collaborative Study) are summarized in Tables 2 to 6:

		overall average *	overall average *	overall average *	overall average *	overall average *	overall average *	overall average *
Para- meter	Product	all labs 1st CS	all labs 2nd CS	all labs 3rd CS	all labs 4th CS	all labs 5th CS	all labs 6th CS	all labs 7th CS
		mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar
ТРМ	A B C D E	12.9 19.8 44.6 67.8 141	12.7 20.3 47.3 73.4 121	21.8 49 80.7 127.4	13.7 21.3 47.5 75.4 138.19	14.1 48.1	14.3 54.3 166.7	14.3 22.02 48.79 57.57
Water	A B C D E	1.06 2.24 4.51 10.03 29.7	0.96 2.45 4.07 11.72 23.57	2.75 4.03 11.21 27.16	0.96 2.62 4.20 12.09 27.39	1.01 3.74	0.81 5.72 43.09	0.8 2.46 3.89 9.18
NFDPM	A B C D E	10.7 16.7 38.3 55.3 107.2	11.23 16.8 40.2 62.4 97.1	18.6 42.1 66.1 97	12.8 18.4 39.7 59.8 103.9	11.9 40.3	12.3 45.0 119.9	12.4 18.53 42.58 46.58
Nicotine	A B C D E	1.03 1.1 2.88 2.35 2.6	0.93 1.08 3.06 1.8 2.13	1.13 3.32 2.87 3.24	1.18 1.13 3.27 2.81 3.76	1.17 2.81	1.15 3.63 3.68	1.15 1.19 3.16 1.57
со	A B C D E	18 50.7 169.2 494.3	9.7 19.3 54.6 175.4 526.4	22.3 60.7 177.6 504.3	11.1 21.1 60.7 190.2 475.4	11.1 63.3	11.3 58.1 545.3	10.5 20.02 61.01 163.18

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		r	r	r	r	r	r	r
Para- meter	Product	1st CS	2nd CS	3rd CS	4th CS	5th CS	6th CS	7th CS
		mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar
ТРМ	A B C D E	1.45 2.88 5.02 13.83 29.03	1.45 2.41 6.22 11.73 25.07	2.82 4.17 10.51 24.16	0.65 4.87 5.67 11.81 36.14	1.29 5.18	1.29 5.74 35.69	1.22 2.75 5.19 12.05
Water	A B C D E	0.45 0.95 1.14 4.98 10.21	0.55 1.2 1.44 3.47 7.43	0.8 1.03 3.33 7.13	0.42 1.37 1.00 3.88 9.27	0.43 1.54	0.28 2.04 13.65	0.28 0.86 1.08 3.30
NFDPM	A B C D E	1.27 2.23 3.53 12.02 21.54	1.44 2.1 5.17 8.61 19.42	2.52 3.42 8.11 18.9	0.85 2.81 4.94 10.01 27.33	1.18 3.89	1.06 4.27 29.27	0.80 2.39 3.70 8.70
Nicotine	A B C D E	0.10 0.13 0.4 0.84 0.98	0.09 0.18 0.52 0.45 0.86	0.13 0.42 0.66 0.93	0.08 0.15 0.50 0.59 1.92	0.09 0.30	0.10 0.44 1.44	0.10 0.16 0.32 0.38
со	A B C D E		1.45 3.87 7.88 28.99 136.19	3.01 5.44 26.27 108.94	1.74 2.83 5.47 27.84 108.86	0.89 6.06	1.68 6.81 108.49	1.14 3.27 5.62 21.78

Table 3. Repeatability r (absolute) from 1st to 7th Collaborative Study

		R	R	R	R	R	R	R
Para- meter	Product	к 1st CS	R 2nd CS	R 3rd CS	ہ 4th CS	к 5th CS	к 6th CS	R 7th CS
		mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar
ТРМ	A B C D E	3.13 8.78 21.85 30.35 101.53	3.53 7.9 13.68 30.08 71.69	9.7 16.57 22.59 56.45	6.29 9.47 19.30 23.93 63.09	3.38 14.38	2.56 9.93 63.18	3.01 4.80 11.91 18.44
Water	A B C D E	1.03 2.58 3.81 10.42 32.4	1.05 2.35 3.7 8.55 31.05	2.95 4.29 7.93 29.8	1.20 2.66 2.61 6.91 25.97	0.74 1.70	0.98 3.03 26.79	0.67 2.27 2.74 5.57
NFDPM	A B C D E	2.11 6.91 13.4 28.18 80.16	1.93 6.3 10.72 23.81 49.82	5.64 13.25 17.3 33.97	1.19 5.53 19.19 20.41 38.43	3.06 10.75	2.63 8.69 46.27	2.93 4.11 9.90 14.08
Nicotine	A B C D E	0.35 0.57 1.31 1.9 2.22	0.31 0.58 0.88 1.07 2.63	0.39 1.06 1.86 2.37	0.21 0.31 0.89 1.09 3.07	0.27 0.96	0.19 1.11 2.36	0.25 0.31 0.93 1.01
со	A B C D E		6.85 14.75 35.04 117.4 348.22	7.26 19.62 45.06 455.15	3.07 14.24 8.60 60.35 175.25	1.91 7.83	2.67 11.64 216.22	3.09 14.85 16.15 56.06

Table 4. Reproducibility R (absolute) from 1st to 7th Collaborative Study

		r %	r %	r %	r %	r %	r %	r %
Para- meter	Product	1st CS	2nd CS	3rd CS	4th CS	5th CS	6th CS	7th CS
		mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar
ТРМ	A B C D E	11 15 11 20 21	11 12 13 16 21	13 9 13 19	5 23 12 16 26	9 11	9 11 21	9 12 11 21
Water	A B C D E	42 43 25 50 34	57 49 35 30 32	29 26 30 26	44 52 24 32 34	43 41	34 36 32	35 35 28 36
NFDPM	A B C D E	12 13 9 22 20	13 13 13 14 20	14 8 12 19	7 15 13 17 26	10 10	9 9 24	7 13 9 19
Nicotine	A B C D E	9 12 14 36 38	10 17 17 25 40	12 13 23 29	7 13 15 21 51	8 11	8 12 39	9 14 10 24
со	A B C D E		15 20 14 17 26	13 9 15 22	16 13 9 15 23	8 10	15 12 20	11 16 9 13

Table 5. Repeatability r % (relative) from 1st to 7th Collaborative Study

		R %	R %	R %	R %	R %	R %	R %
Para- meter	Product	1st CS	2nd CS	3rd CS	4th CS	5th CS	6th CS	7th CS
		mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar	mg/cigar
ТРМ	A B C D E	24 44 49 45 72	28 39 29 41 59	45 34 28 44	46 45 41 32 46	24 30	18 18 38	21 22 24 32
Water	A B C D E	97 115 85 104 109	109 96 91 73 132	107 106 71 110	125 102 62 57 95	73 46	121 53 62	83 92 70 61
NFDPM	A B C D E	20 41 35 51 75	17 38 27 38 51	30 31 26 35	9 30 48 34 37	26 27	21 19 39	24 22 23 30
Nicotine	A B C D E	34 51 45 81 86	33 54 29 59 123	35 32 65 73	18 28 27 39 82	23 34	17 31 64	22 26 29 64
со	A B C D E		71 76 64 67 66	33 32 25 90	28 67 14 32 37	17 12	24 20 40	29 74 26 34

From all these results we can observe that the relative (%) Repeatability "r" and relative (%) Reproducibility "R" have generally improved for the different analytes especially for the 5th to the 7th CS. The ranges of these relative values obtained for the five products obtained are listed in Table 7 coming from the 7th CS except for product E, which values come from the 6th CS.

Table 7. Rela	ative (%) Repeatability "r" a	ana Reproducii	Dility "R"
	repeatability (r) %		reproducibility (R) %
Product A ra	nge		
10%	NFDPM TPM, Nicotine, CO Water	29%	TPM, Nicotine, NFDPM CO Water
Product B ra	nge		
16%	TPM, Nicotine, NFDPM CO Water	74%	TPM, Nicotine, NFDPM CO Water
Product C ra	nge		
	TPM, Nicotine, NFDPM, CO Water	29%	TPM, NFDPM, CO Nicotine Water
Product D ra	nge		
	CO TPM, Nicotine, NFDPM Water		TPM, NFDPM, CO Nicotine, Water
Product E ra	nge		
32%	TPM, NFDPM, CO Water Nicotine		TPM, NFDPM, CO Nicotine, Water

 Table 7. Relative (%) Repeatability "r" and Reproducibility "R"

It is also useful for comparison to have the evolution of average results, repeatability r and reproducibility R obtained with the CORESTA Monitor Test Piece CM6 smoked with the Cigar Method during the 4th to the 7th CS against the results obtained for the CM6 test piece smoked under the ISO 4387 smoking regime (CORESTA CM6 collaborative study 2008). This comparison, given in Table 8, is useful for establishing tolerances. When comparing the results it should be taken into

consideration that the CM6 collaborative study results come from around 60 smoking machines from 40 laboratories while the "cigar" results have been obtained from maximum 13 laboratories and that a single result is obtained in the "cigar" method by smoking 8 cigars while a single result in the ISO cigarette smoking method comes from smoking 20 cigarettes.

After the 6th study, the Nicotine, Water and NFDPM relative r values for CM6 test pieces are from 45 % to 75 % higher when smoked with the cigar smoking method compared to values obtained for ISO cigarette smoking regime" and TPM is 100 % higher and CO is 270 % higher when smoked under the cigar smoking regime. The relative R values on all analytes are roughly 100 % higher when smoked under the cigar smoking regime. These are the results:

Table 8. CM6 – Comparison of results using the "ISO cigarette smoking regime" and the CORESTA recommended methods for cigar smoking

		CIGARS 4th Collaborative Study									
	(mg/cig)	Repe	atability	limits	Reproducibility limits						
CM6	Mean	r SD	r	r (%)	R SD	R	R(%)				
ТРМ	13,67	0,23	0,65	4,73	2,24	6,29	46,00				
Water	0,96	0,15	0,42	43,74	0,43	1,20	125,47				
NFDPM	12,79	0,30	0,85	6,63	0,65	1,19	9,28				
Nicotine	1,181	0,029	0,08	6,94	0,076	0,21	18,12				
CO	11,12	0,62	1,74	15,65	1,10	3,07	27,62				

		CIGARS 5th Collaborative Study									
	(mg/cig)	Repea	Repeatability limits			Reproducibility limits					
CM6	Mean	r SD	r	r (%)	R SD	R	R(%)				
ТРМ	14.08	0.456	1.29	9.2	1.194	3.38	24.0				
Water	1.01	0.152	0.43	42.6	0.263	0.74	73.3				
NFDPM	11.87	0.416	1.18	9.9	1.083	3.06	25.8				
Nicotine	1.17	0.031	0.09	7.7	0.095	0.27	23.1				
CO	11.14	0.316	0.89	8.0	0.675	1.91	17.1				

	CIGARS 6th Collaborative Study										
	(mg/cig)	Repeatab	ility limits	5	Reproducibility limits						
CM6	Mean	r SD	r	r (%)	R SD	R	R(%)				
ТРМ	14.26	0.455	1.29	9.0	0.905	2.56	18.0				
Water	0.81	0.097	0.28	34.0	0.348	0.98	121.4				
NFDPM	12.32	0.376	1.06	8.6	0.93	2.63	21.3				
Nicotine	1.15	0.034	0.10	8.4	0.068	0.19	16.7				
СО	11.28	0.595	1.68	14.9	0.943	2.67	23.7				

	CIGARS 7th Collaborative Study									
	(mg/cig)	Repe	Repeatability limits			Reproducibility limits				
CM6	Mean	r SD	r	r (%)	R SD	R	R (%)			
ТРМ	14.3	0.432	1.22	8.6	1.065	3.01	21.1			
Water	0.8	0.099	0.28	34.9	0.236	0.67	83.4			
NFDPM	12.4	0.283	0.80	6.4	1.037	2.93	23.7			
Nicotine	1.15	0.035	0.10	8.7	0.089	0.25	21.8			
СО	10.5	0.404	1.14	10.9	1.092	3.08	29.4			

	CORESTA CM 6 Collaborative Study 2008 on ISO cigarette smoke *									
	(mg/cig)	Repe	eatability	limits	Reproducibility limits					
CM6	Mean	r SD	r	r (%)	R SD	R	R (%)			
ТРМ	17,54	0,30	0,83	4,73	0,63	1,77	10,09			
Water	1,87	0,15	0,43	22,99	0,40	1,11	59,35			
NFDPM	14,28	0,25	0,71	4,97	0,64	1,80	12,61			
Nicotine	1,390	0,029	0,080	5,75	0,047	0,131	9,42			
CO	14,83	0,29	0,82	5,52	0,59	1,64	11,05			

* Statistical analysis of the analytical results from the 2008 CORESTA CM6 collaborative study in accordance with ISO 5725

4. COLLABORATIVE STUDIES RESULTS

4.1 1st Collaborative Study & Cigar Diameter Cross-Check, April – Sept. 2006

During 2006, 14 labs participated in the 1st Collaborative Study of the newly created Cigar Smoking Methods Sub-Group. For this study four commercial products plus the CORESTA Monitor (CM5) were chosen to be smoked and have been also used for later Collaborative Studies in order to allow the comparison of results and observe trends and lab performance evolution, even if the chosen products have naturally evolved through the years. They were coded as Product A (CORESTA Monitor) B, C, D and E.

The general results are summarized in Tables presented in chapter 3 "Global Results" where a comparison with the subsequent CS results is possible

It should be commented that there were some missing data as some laboratories did not smoke all five products. No single recommended method was available for Carbon Monoxide (CO) in this study. Every laboratory was invited to measure the CO content following their own technique. Not all laboratories were able to participate in this CO analysis, but those laboratories that participated were using very different methodologies. Therefore it was decided not to perform an r & R analysis.

Instead the data received were analysed using graphical descriptive statistics. From this investigation it was obvious that the inter laboratory variance would be extremely high. So it made no sense to perform a repeatability and reproducibility analysis on the measurements of the CO content.

As the Cigar Smoking Method puff volume depend on the cigar diameter it was decided to conduct a cross-check of the different cigar diameter measuring methods used by the participating labs in order to make a recommendation in the CRMs about this issue.

In the report released on 29 June 2006, it is stated that 6 different products were measured with 5 different techniques:

- 1. camera measurement (without rotating the product),
- 2. Charge Coupled Device Measurement (CCD),
- 3. laser measurement,
- 4. tape gauge measurement
- 5. longitudinal cigar slitting. From the cross check it became clear, that CCD and laser measurement generally show the lowest variability.

After some discussion it was agreed that it would be better to incorporate the cigar diameter measurement in paragraph 7.5 of CRM n°65, rather than writing a separate Recommended Method. The following was incorporated in the CRM:

- The measurement should take place at 33 mm of the mouth end;
- The cigar should be rotated;
- An optical measurement system should be used, measuring the silhouette or shadow of the product;
- The measurement technique should be left open, as long as the accuracy is the same as or better than the accuracy of the CCD and laser systems;
- Reference to the cigarette diameter method ISO 2971 should be made;
- The cigar diameter measurement method could result in puff volume differences of ± 1ml.

In 2006 there was no specific CSM SG report to the CORESTA Congress as it was included in a global Product Technology Group Report presented in Paris to the Congress.

4.2 2nd Collaborative Study, October – March 2007

The 2nd Collaborative Study was conducted from November 2006 to January 2007 and was a replica of the first study of April-May 2006. There were 13 participating laboratories and the cigars smoked in the 2nd CS were the same 5 products smoked in the 1st CS but coming from a different production batch than the first one.

For the majority of smoke yields the differences between the two studies are \leq 10 %, as calculated from the overall averages. The higher differences are mainly found in the water and nicotine yields.

In this CS, repeatability either improved or worsened for all 4 smoke analytes. Interestingly, Products D and E (the larger products) show an improved r for all analytes, whereas Product C shows a worse r for all analytes.

On average over all products, reproducibility improved for all analytes. It worsened in only a few individual cases. The "between laboratory precision" was improved over nearly the whole range of products/analytes. For CO, the "r" values are not far from those of NFDPM, but the "R" values are higher than for the other analytes. This is true for products A, B, C and D, with product E as the exception. A Working Group wrote a draft CRM on CO measurements in cigars, based on the experience obtained in the 2nd CS. This draft was discussed and approved during the next SG meeting.

4.3 3rd Collaborative Study, June 2007 - March 2008

The Collaborative Study n°3 (tar, nicotine & CO) was completed on the same 4 cigar products smoked in the 2 previous CS by 11 participating laboratories. The CORESTA Monitor was not available and could not be included.

The purpose of the study was to improve both the repeatability "r" and the reproducibility "R" and to try the first draft of the CO CRM.

For TPM, water, nicotine, NFDPM and CO, repeatability "r" showed a slight improvement from the first to the third collaborative study.

No constant trend could be detected for reproducibility R. A positive exception was the reproducibility of NFDPM for products D and E.

The relative "r" for TPM, nicotine, NFDPM and CO was:

- Products B and C => from 10 to 15%
- Product D => from 10 to 20%
- Product E => around 20%

"r" for water was at a (somewhat) higher level.

The relative "R" for TPM, nicotine, NFDPM and CO was:

- Products B, C and D => from 20 to 40%
- (except for nicotine on Product D)
- Product E => around 40%

"R" for water was at a higher level (up to 100% on Product E).

CO had been measured in all 3 collaborative studies. However "r" and "R" were only analyzed in the second and third study. For the third study a WG had prepared a draft CRM on the determination of CO which resulted in a slightly improved "r" value for all 4 products as compared to the 2nd study;

With the exception of product E, the same was true for the "R" value, even though not all the laboratories had been able to follow the draft CRM completely. The average CO results were relatively close on a per product basis.

From these results a new draft CO method was prepared considering the 3rd CS experience. It was later used in the 4th CS. It was finally approved in January 2010 as the CORESTA Recommended Method n^o 68 after the last amendments done by the Sub-Group.

4.4 4th Collaborative Study, June 2008 - March 2009

The Collaborative Study n°4 (tar, nicotine & CO) was completed on 4 cigars plus the CM6 by 9 participating laboratories. For CO test, only 7 laboratories participated, while for CM6 only 4 results were considered valid after statistical study. Product E was smoked twice (coded as E1 and E2) : one with the puff volume according to the specified cigar diameter and the second one according to lab-measured diameter.

A revision of the CO Analytical Method for Cigars draft was done and the final draft sent to the Scientific Commission for approval, together with a modification of CRM 65 (NFDPM in cigars) in order to reduce the cigar diameter measurement workload and the possible influence of clearing puffs in reproducibility.

A Working Group determined the best bobbins and sleeves construction for the cigar holder in order to reduce the Reproducibility value and establish the constructions to be used in the 5th Collaborative Study.

The purpose of the 4th CS was to improve both the repeatability r and the reproducibility R. The SG used commercial products. The same batch was used for 1st and 2nd CS but different batches were used for 3rd and 4th CS.

The smoke yields were expressed both in mg/cigar and in mg/g and a statistical analysis was conducted for both.

For TPM, water, nicotine, NFDPM and CO, an increase in repeatability was observed with few exceptions in the 4th compared to the 3rd CS.

No constant trend could be detected for reproducibility R. But in the majority of cases the figure has improved and most significantly for CO.

The relative "r" for TPM, nicotine, NFDPM and CO was:

- Products B and C => from 10 to 15%
- Product D => from 15 to 20%
- Product E => around 25%

"r" for water was at a higher level. Also for nicotine in Product E

The relative "R" for TPM, nicotine, NFDPM and CO was:

- Products B, C and D => from 20 to 40%
- Product E => around 40% (except for nicotine)

"R" for water was at a higher level (up to 100% on products B and E) but better than in previous CS.

The smoke yields expressed in both in mg/cigar and in mg/g are given in Table 9.

Table 9. Smoke yields results and relative r&R calculations expressed in mg/g and mg/cigar

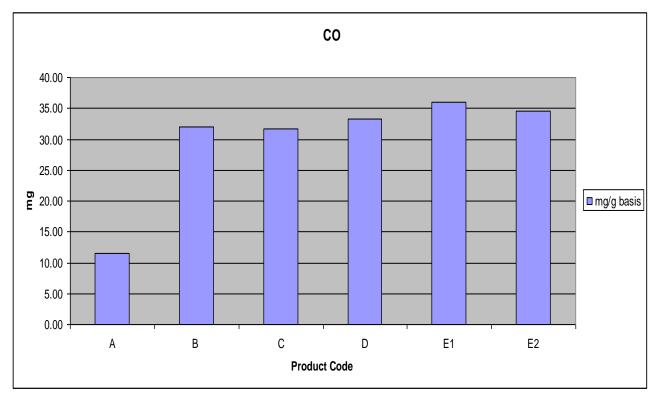
(mg/g basis)			(mg/cigar basis)					
Product A	Mean	r(%)	R(%)	Mean	r(%)	R(%)		
TPM	14,15	6,74	43,37	13,67	4,73	46,00		
Water	1,47	66,99	165,19	0,96	43,74	125,47		
NFDPM	12,17	12,00	36,05	12,79	6,63	9,28		
Nicotine	1,197	7,88	21,89	1,181	6,94	18,12		
CO	11,50	15,22	26,96	11,12	15,65	27,62		
	·			·	•			
Product B	Mean	r(%)	R(%)	Mean	r(%)	R(%)		
TPM	29,65	23,02	45,22	21,28	22,87	44,49		
Water	3,65	52,83	102,53	2,62	52,27	101,64		
NFDPM	25,68	15,61	30,89	18,41	15,26	30,04		
Nicotine	1,593	9,38	26,03	1,130	13,01	27,61		
CO	29,39	12,61	66,69	21,13	13,37	67,39		
-								
Product C	Mean	r(%)	R(%)	Mean	r(%)	R(%)		
TPM	24,92	13,72	40,59	47,54	11,93	40,60		
Water	2,26	30,59	63,05	4,20	23,72	62,17		
NFDPM	21,34	14,91	16,80	39,70	12,45	48,33		
Nicotine	1,671	16,45	17,63	3,271	15,29	27,15		
CO	31,66	8,09	16,26	60,66	9,01	14,17		
Product D	Mean	r(%)	R(%)	Mean	r(%)	R(%)		
TPM	13,25	16,47	31,59	75,43	15,66	31,73		
Nicotine	0,493	21,61	39,15	2,806	21,06	38,88		
Water	2,12	32,98	57,20	12,09	32,08	57,19		
NFDPM	10,50	17,54	33,61	59,82	16,73	34,11		
CO	33,33	14,99	30,82	190,16	14,64	31,73		
Product E1	Mean	r(%)	R(%)	Mean	r(%)	R(%)		
TPM	10,62	33,04	48,87	138,19	26,15	45,66		
Nicotine	0,293	62,18	91,68	3,758	51,15	81,70		
Water	2,09	34,44	91,51	27,39	48,71	110,52		
NFDPM	7,98	33,30	41,93	103,91	26,30	36,98		
CO	35,93	26,36	39,84	475,42	22,90	36,86		
		-						
Product E2	Mean	r(%)	R(%)	Mean	r(%)	R(%)		
TPM	9,84	37,09	60,12	128,56	29,64	55,17		
Nicotine	0,261	45,70	70,23	3,371	37,63	63,86		
Water	2,32	49,03	104,70	30,36	9,31	46,90		
NFDPM	7,26	39,84	56,59	94,83	32,61	52,55		
CO	34,58	24,51	34,96	453,49	19,40	33,35		

In general, the relative r and R values are slightly higher in mg/g than in mg/cigar

For CO, the improvement of "R" in the 4 products was outstanding compared to the 2nd CS prior to the CO draft, demonstrating the value of CRMs.

After this CS, some modifications were introduced in the draft CRM and a final draft was sent to the Scientific Commission for approval.

The average CO results in mg/g are stable around 35 mg/g all along the different products and weights, as shown in the Figure 2.





The cigar holder construction shows a great disparity among different laboratories. A WG was established in order to study the way to avoid this disparity and reduce this source of yield variability and to improve consequent yield reproducibility.

Products E1and E2 come from the same batch but smoked one with a puff volume fixed in the protocol and the other with the calculated puff volume after measuring the cigar diameter, as established in the CORESTA CRM. Results are given in Table 10.The difference between the average yields, whether expressed in mg/cigar or mg/g, are always lower than the r and R values so the different puff volumes used do not give results that could be considered as different.

Table 10. Comparison of results of Product E smoked with fixed and calculated puff volume, expressed in mg/g and mg/cigar

Δνοτα	ges (mg/cig.)	E1	E2	E1	E2	E1	E2	E1	E2	E1	E2	E1	E2
Atera	geo (mg/olg.)	Puff											
Lab.code	Puff volume dif.	volume	Puff volume	TPM	TPM	Nicotine	Nicotine	Water	Water	NFDPM	NEDPM	co	co
	(E1-E2) (ml)	(ml)	(ml)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)	(mg/cig.)
1	-1	46	47	105,32	104,12	2,278	2,792	17,068	16,036	85,97	85,287	389,22	378,884
10	-1	46	47	130,81	156,64	3,521	4,519	25,73	34,651	101,559	117,47	425,484	498,841
6	1	46	45	118,03	101,9	4,572	3,29	16,603	23,451	96,85	75,159	100 550	170.010
8	3	46	43	154,02	146,77	3,11	2,875	41,72	40,332	109,186	103,559	490,559	478,948
14 5	3	46 46	43 42	139,71 161,15	127,27 134.67	3,111 4,826	3,039 3,713	27,803 47,421	26,829 40,862	108,794 108,904	97,405 90,095	495,192 495,559	466,184 444,605
5	4	40	42	161,15	134,67	4,020	3,713	47,421	40,002	108,904	90,095	495,559	444,605
			Average										
	2		(mg/cig.)	134,8	128,6	3,57	3,37	29,39	30,36	101,88	94,83	459,20	453,49
			r (mg/cig.)	36,1	38,1	1,92	1,27	9,27	13,34	27,33	30,92	108,86	87,98
			R (mg/cig.)	63,1	70,9	3,07	2,15	25,97	30,27	38,43	49,83	175,24	151,24
1			r (%)	27	30	54	38	32	44	27	33	24	19
			R (%)	47	55	86	64	88	100	38	53	38	33
Aver	ades (ma <i>l</i> a)	F1	F2	F1	F2	F1	F2	F1	F2	F1	F2	F1	F2
Avera	ages (mg/g)	E1 Puff	E2	E1	E2	E1	E2	E1	E2	E1	E2	E1	E2
Avera Lab.code		E1 Puff volume	E2 Puff volume	E1 TPM	E2 TPM	E1 Nicotine	E2 Nicotine	E1 Water	E2 Water	E1 NFDPM	E2 NE DPM	E1 CO	E2 CO
		Puff											
	Puff volume dif.	Puff volume	Puff volume	ТРМ	ТРМ	Nicotine	Nicotine	Water	Water	NFDPM	NEDPM	со	со
Lab.code 1 10	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46	Puff volume (ml) 47 47	TPM (mg/g) 8,00 9,76	TPM (mg/g) 8,09 11,91	Nicotine (<i>mg/g)</i> 0,176 0,265	Nicotine (mg/g) 0,220 0,347	Water (mg/g) 1,288 1,916	Water (mg/g) 1,254 2,641	NFDPM (mg/g) 6,537 7,576	NFDPM (mg/g) 6,619 8,924	CO (mg/g)	CO (mg/g)
Lab.code 1 10 6	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46 46 46	Puff volume (ml) 47 47 45	TPM (mg/g) 8,00 9,76 9,72	TPM (mg/g) 8,09 11,91 7,46	Nicotine (mg/g) 0,176 0,265 0,381	Nicotine (<i>mg/g)</i> 0,220 0,347 0,241	Water (<i>mg/g)</i> 1,288 1,916 1,358	Water (mg/g) 1,254 2,641 1,717	NFDPM (mg/g) 6,537 7,576 7,981	NF DPM (mg/g) 6,619 8,924 5,506	CO (mg/g) 29,355 31,56	CO (mg/g) 28,879 37,817
Lab.code 1 10 6 8	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46 46 46	Puff volume (ml) 47 47 45 43	TPM (mgig) 8,00 9,76 9,72 11,54	TPM (mg/g) 8,09 11,91 7,46 10,90	Nicotine (mg/g) 0,176 0,265 0,381 0,237	Nicotine (mg/g) 0,220 0,347 0,241 0,217	Water (mg/g) 1,288 1,916 1,358 3,108	Water (mg/g) 1,254 2,641 1,717 2,979	NFDPM (mg/g) 6,537 7,576 7,981 8,197	NF DPM (mg/g) 6,619 8,924 5,506 7,708	CO (mg/g) 29,355 31,56 36,352	CO (mg/g) 28,879 37,817 35,361
Lab.code 1 10 6 8 14	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46 46 46 46	Puff volume (ml) 47 47 45 43 43	TPM (mg/g) 8,00 9,76 9,72 11,54 10,49	TPM (mg/g) 8,09 11,91 7,46 10,90 9,66	Nicotine (mg/g) 0,176 0,265 0,381 0,237 0,239	Nicotine (mg/g) 0,220 0,347 0,241 0,217 0,234	Water (mg/g) 1,288 1,916 1,358 3,108 2,079	Water (mg/g) 1,254 2,641 1,717 2,979 2,021	NFDPM (mg/g) 6,537 7,576 7,981 8,197 8,174	NFDPM (mg/g) 6,619 8,924 5,506 7,708 7,409	CO (mg/g) 29,355 31,56 36,352 36,912	CO (mg/g) 28,879 37,817 35,361 35,052
Lab.code 1 10 6 8	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46 46 46	Puff volume (ml) 47 47 45 43	TPM (mgig) 8,00 9,76 9,72 11,54	TPM (mg/g) 8,09 11,91 7,46 10,90	Nicotine (mg/g) 0,176 0,265 0,381 0,237	Nicotine (mg/g) 0,220 0,347 0,241 0,217	Water (mg/g) 1,288 1,916 1,358 3,108	Water (mg/g) 1,254 2,641 1,717 2,979	NFDPM (mg/g) 6,537 7,576 7,981 8,197	NF DPM (mg/g) 6,619 8,924 5,506 7,708	CO (mg/g) 29,355 31,56 36,352	CO (mg/g) 28,879 37,817 35,361
Lab.code 1 10 6 8 14	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46 46 46 46	Puff volume (ml) 47 47 45 43 43 43 42	TPM (mg/g) 8,00 9,76 9,72 11,54 10,49	TPM (mg/g) 8,09 11,91 7,46 10,90 9,66	Nicotine (mg/g) 0,176 0,265 0,381 0,237 0,239	Nicotine (mg/g) 0,220 0,347 0,241 0,217 0,234	Water (mg/g) 1,288 1,916 1,358 3,108 2,079	Water (mg/g) 1,254 2,641 1,717 2,979 2,021	NFDPM (mg/g) 6,537 7,576 7,981 8,197 8,174	NFDPM (mg/g) 6,619 8,924 5,506 7,708 7,409	CO (mg/g) 29,355 31,56 36,352 36,912	CO (mg/g) 28,879 37,817 35,361 35,052
Lab.code 1 10 6 8 14	Puff volume dif. (E1-E2) (ml)	Puff volume (ml) 46 46 46 46 46	Puff volume (ml) 47 47 45 43 43	TPM (mg/g) 8,00 9,76 9,72 11,54 10,49	TPM (mg/g) 8,09 11,91 7,46 10,90 9,66	Nicotine (mg/g) 0,176 0,265 0,381 0,237 0,239	Nicotine (mg/g) 0,220 0,347 0,241 0,217 0,234	Water (mg/g) 1,288 1,916 1,358 3,108 2,079	Water (mg/g) 1,254 2,641 1,717 2,979 2,021	NFDPM (mg/g) 6,537 7,576 7,981 8,197 8,174	NFDPM (mg/g) 6,619 8,924 5,506 7,708 7,409	CO (mg/g) 29,355 31,56 36,352 36,912	CO (mg/g) 28,879 37,817 35,361 35,052
Lab.code 1 10 6 8 14	Puff volume dif. (E1-E2) (ml) -1 1 3 3 3 4	Puff volume (ml) 46 46 46 46 46	Puff volume (ml) 47 47 45 43 43 43 42 Average	TPM (<i>mg/g</i>) 8,00 9,76 9,72 11,54 10,49 12,51 10,3 3,5	TPM (<i>mg/g</i>) 8,09 11,91 7,46 10,90 9,66 11,01	Nicotine (<i>mg/g</i>) 0,176 0,265 0,381 0,237 0,239 0,376	Nicotine (mg/g) 0,220 0,347 0,241 0,217 0,234 0,307	Water (mg/g) 1,288 1,916 1,358 3,108 2,079 3,676	Water (<i>mg/g</i>) 1,254 2,641 1,717 2,979 2,021 3,307	NFDPM (mg/g) 6,537 7,576 7,981 8,197 8,197 8,174 8,454 7,82 2,66	NFDPM (mg/g) 6,619 8,924 5,506 7,708 7,409 7,399	CO (mg/g) 29,355 31,56 36,352 36,912 38,06 34,45 9,47	CO (mg/g) 28,879 37,817 35,361 35,052 35,773
Lab.code 1 10 6 8 14	Puff volume dif. (E1-E2) (ml) -1 1 3 3 3 4	Puff volume (ml) 46 46 46 46 46	Puff volume (ml) 47 45 43 43 43 42 Average (mg/cig.) r (mg/cig.) R (mg/cig.)	TPM (mg/g) 9,76 9,72 11,54 10,49 12,51 10,3 3,5 5,2	TPM (mg/g) 8,09 11,91 7,46 10,90 9,66 11,01 9,8 3,7 5,9	Nicotine (mg/g) 0,176 0,265 0,381 0,237 0,239 0,239 0,239 0,239 0,239 0,239 0,239 0,239 0,239 0,218	Nicotine (mg/g) 0,220 0,241 0,217 0,234 0,234 0,307 0,26 0,12 0,18	Water (mg/g) 1,288 1,916 1,358 3,108 2,079 3,676 2,24 0,72 1,91	Water (mg/g) 1,254 2,641 1,717 2,979 2,021 3,307 2,32 1,14 2,43	NFDPM (mg/g) 6,537 7,576 7,981 8,197	NFDPM (mg/g) 6,619 8,924 5,506 7,708 7,409 7,399 7,26 2,89 4,11	CO (mg/g) 29,355 31,56 36,352 36,912 38,06 34,45 9,47 14,32	CO (mg/g) 28,879 37,817 35,361 35,052 35,773 34,58 8,48 12,09
Lab.code 1 10 6 8 14	Puff volume dif. (E1-E2) (ml) -1 1 3 3 3 4	Puff volume (ml) 46 46 46 46 46	Puff volume (ml) 47 45 43 43 43 43 42 Average (mg/cig.) r (mg/cig.)	TPM (<i>mg/g</i>) 8,00 9,76 9,72 11,54 10,49 12,51 10,3 3,5	TPM (<i>mg/g</i>) 8,09 11,91 7,46 10,90 9,66 11,01 9,8 3,7	Nicotine (mg/g) 0,176 0,265 0,381 0,237 0,239 0,376 0,28 0,18	Nicotine (mg/g) 0,220 0,347 0,241 0,217 0,234 0,307 0,26 0,12	Water (mg/g) 1,288 1,916 1,358 3,108 2,079 3,676 2,24 0,72	Water (mg/g) 1,254 2,641 1,717 2,979 2,021 3,307 2,32 1,14	NFDPM (mg/g) 6,537 7,576 7,981 8,197 8,197 8,174 8,454 7,82 2,66	NF DPM (mg/g) 6,619 8,924 5,506 7,708 7,409 7,399 7,26 2,89	CO (mg/g) 29,355 31,56 36,352 36,912 38,06 34,45 9,47	CO (mg/g) 28,879 37,817 35,361 35,052 35,773 34,58 8,48

4.5 5th Collaborative Study, June 2009 - March 2010

The final revision of the CO Analytical Method for Cigars draft was approved by the Board and published as CRM 68.

CRM 65 (NFDPM in cigars) was revised for allowing smoking only 1 cigar per filter pad (for diameter <12mm) when filter trap maximum load is exceeded.

The Collaborative Study n°5 (tar, nicotine & CO) was completed on 1 cigar plus the CM6 by 10 participating laboratories. For CO test, only 9 laboratories participated.

The purpose of the study was to improve both the repeatability r and the reproducibility R. Commercial products were used. While the same batch was used for 1st and 2nd CS, the samples for the 3rd, 4th and 5th Collaborative Study were drawn from different batches.

In the protocol it was requested to smoke the same Product C that was selected for the previous studies, plus the CM6 named as product A. The holder constructions for both products were established by a WG prior to the CS and were fixed in the protocol. Volunteer laboratories smoked Products A and C with all the possible bobbin, sleeve and end seal combinations and proposed then one combination for each product to be used by all labs during the 5th CS as shown in Table 11.

Table 11. Bobbin, sleeve and end seal specifications

	Product Code A	Product Code B
Bobbin	10.5 mm	12.5 mm
Sleeve	5.5 mm	7.0 mm
End seal	8.0 mm	10.0 mm
Puff volume	20 mL	20 mL

The results were expressed in mg/cigar and a statistical analysis for r and R was conducted on them.

The comments on the results were the following:

- Average yields : The variation observed is lower than the r value.
- Relative Repeatability:
 - for Product A there is a great improvement achieved in CO, while a slight worsening can be observed in TPM, Nicotine and NFDPM. The level for the analytes TPM, nicotine, NFDPM and CO is situated in the range of 10%, and for Water is of 45 %.
 - for Product C there is an improvement in TPM, Nicotine and NFDPM, with a slight decline in CO and a worsening in Water. The level for the analytes TPM, nicotine, NFDPM and CO is situated in the range of 10%, and for Water is of 40 %.
- Relative Reproducibility:
 - for Product A there was a great improvement in TPM, Water and CO, while a worsening can be observed in Nicotine and NFDPM. The level for the analytes TPM, nicotine, NFDPM and CO is situated in the range of 20-25%, and for Water is of 75 %.
 - for Product C there is an improvement in all the analytes except Nicotine, that has a slight decline. The level for the analytes TPM, Nicotine and NFDPM is situated in the range of 30-35%, for Water is of 45 % and for CO is of 12 %.
- CO results of Product A (CM6 CORESTA Monitor):
 - 8 labs have been able to provide valid results.
 - The average CO results are very similar in the 2 studies in which the CM6 has been smoked.
 - The r and R values have had an outstanding improvement in the 5th CS compared to the 4th, with a reduction of almost 50 % of the relative values.
 - CO results of Product C:
 - 8 labs have been able to provide valid results.
 - The average CO results increased from 50.7 to 63.3 mg/cigar through the 5 studies, but it is stabilised around 61 since the introduction of a Draft Recommended Method in the 3rd study.

- Introduction of a Draft Recommended Method in the 3rd study lead to an improved "r" value, even if in the 5th CS the figure obtained has been slightly higher than in the 3rd and 4th CS.
- The introduction of Draft Recommended Method in the 3rd study led to a significant improvement of the "R" value as compared to the 2nd study, with another significant improvement in the 4th study and a further improvement again in the 5th study.

4.6 6th Collaborative Study, June 2010 - March 2011

The Collaborative Study n°6 (tar, nicotine & CO) was completed on 2 cigar plus the CM6 by 9 participating laboratories. The products were coded A, C and E.

The purpose of the study was to improve both the repeatability r and the reproducibility R. The samples of the 1st and 2nd Collaborative Study were part of one and the same batch, whereas the samples for the 3rd to the 6th Collaborative Study were drawn from different batches.

In the Protocol it was requested to smoke the same Products C and E that were selected for the previous studies, plus the CM6 named as Product A. A statistical analysis of repeatability and reproducibility (r and R) has been conducted on a mg/cigar basis.

This study had fixed the bobbin, sleeve and end-seal combination but the results do not show a real improvement out of this decision.

	Product Code A	Product Code B	Product Code E
Bobbin	10.5 mm	12.5 mm	20.5 mm
Sleeve	5.5 mm	7.0 mm	14.0 mm
End seal	8.0 mm	10.0 mm	14.0 mm
Puff volume	20 mL	20 mL	49 mL

Table 12. Cigar holder constructions fixed in the CS

Three labs volunteered to determine the TPM maximum load in Cambridge filters. It was agreed that no further test was needed. Past studies have shown that the maximum load depends on the filter diameter, and lies around 200/250mg. The results agreed with that conclusion.

For Product A, the results have been stable except for a slight reduction in water. For Products C and E, all the analytes have grown except nicotine in Product E. The variation observed between the 4th and the 6th CS is similar to the repeatability (r) value calculated.

The variation of the r and R values (absolute and relative) with respect to the 5^{th} CS is expressed in the Table 13.

The repeatability and reproducibility ranges of the analytes of the different products are expressed in Table 14.

(with respect to the	e 5th CS)	repeatabi	lity (r	.)	(with respect to	the 5th CS)	reproduc	ibility (R)
Product A	improve	stable	W	orse	Product A	improve	stable	worse
TPM Nicotine Water NFDPM CO	Water NFDPM	TPM Nicotine	со		TPM Nicotine Water NFDPM CO	TPM Nicotine NFDPM		Water CO
Product C	improve	stable	w	orse	Product C	improve	stable	worse
TPM Nicotine Water NFDPM	Water	ТРМ	Nico	tine	TPM Nicotine Water NFDPM	TPM Nicotine NFDPM		Water
CO			0	0	co			со
Product E	improve	stable	W	orse	Product E	improve	stable	worse
TPM Nicotine Water NFDPM CO	TPM Nicotine Water NFDPM CO				TPM Nicotine Water NFDPM CO	TPM Nicotine Water		NFDPM CO
Table 14 Product A	. Range o repeatab		es in	the d	ifferent produc	cts reproducib	ility (R) %	٦
range 10% 15% 35%	CO	ine, NFDPM			range 17% - 20% 25% 120%	TPM, Nicotine CO Water	e, NFDPM	
Product C range					Product C range			-

range 10% - 12% 35% TPM, Nicotine, NFDPM, CO Water Product E

range 20% - 25 % TPM, NFDPM, CO 32% Water 40% Nicotine ange 18% - 20% TPM, NFDPM, CO 30% Nicotine 55% Water

Product E	
range	
39% - 40%	TPM, NFDPM, CO
65%	Nicotino Water

65% Nicotine, Water

9 labs have provided valid CO results. The comparison of the average data of the studies and the r & R data of the 2nd to the 6th study lead to the following conclusions:

Product A (CM6 CORESTA Monitor): The average CO results were very similar in the 3 studies which smoked the CM6. After significant improvement in the 5th CS compared to the 4th, the relative r and R values were stable around a 15 % and 25 % value respectively.

Products C and E: The average CO for product C is stabilised around 60 mg/cigar since the introduction of a Draft Recommended Method AA in the 3rd study. For product E, a big cigar, it fluctuates around 500 mg/cigar since the beginning. It has also lead to a stable repeatability value in both products and to a big improvement of the reproducibility value as compared to the 2nd study, with an apparent stabilisation after the significant improvement from the 4th study on.

Comments on CM6

A comparison between the r and R absolute and relative values of CM6 smoked as cigarettes (results from the 2008 survey in accordance to ISO 5725) and as cigars (CORESTA CRMs) in the 4th to the 6th Collaborative Studies was done.

While in the 4th study only 4 laboratories obtained results, in the 6th study from 6 to 9 laboratories have obtained results against around 40 laboratories for the "cigarette" results.

After the 6th study, the relative R values on all the analytes are roughly 100 % higher than in "cigarettes".

These results are worse in water and CO than in the 5th CS. The "cigarette" results for 2008 have improved and for "cigars" have worsened, probably due to changes in the number and name of the participating labs.

When comparing the CM6 r and R results between the Cigar and Cigarette methods it should be realized that the cigar method is more variable due to the lower number of cigars smoked to obtain one result (8 cigars vs. 20 cigarettes).

Main conclusions:

- There were no consistent improvements in r and R results in the last CS.
- Even for the CM 6, there were unstable results.
- Poor cigar conditioning may have occurred as cigar weight reported by labs had a high variation.
- Labs need to observe their own coefficient of variation result to know how they perform.
- Another source of variation is the continuous change in the participating laboratories.

It had been suggested in 2009 that all labs should systematically smoke CM6 each time they undertake smoking tests so they can control the smoking process by building their own control chart and ensure their yields are in line with reference values before more lengthy smoking of cigar samples. In 2009 a Control Chart Template according to ISO 8258 was distributed to all the SG members for this purpose.

The higher r and R in cigars may be explained by Production and Product related reasons.

4.7 7th Collaborative Study, June 2011 - March 2012

The Collaborative Study n°7 (tar, nicotine & CO) was completed on 3 cigars by 13 participating laboratories. The products were coded B, C and D. Product A (CM6) was smoked every three months in subsequent Collaborative Studies just for this CORESTA Monitor and separate reports were issued for each of them. The results were reviewed in the CSM SG meeting held on 17 April, 2012 Madrid.

The 7th CS analysed Products B, C and D in order to try to improve repeatability "r" and reproducibility "R" as much as possible. Labs decided on the bobbin, sleeve, end seal and puff volume themselves again following the CRM instructions.

A CS on the CM6 monitor was carried out 3 times in a year to monitor improvements of the performance of participating laboratories: smoking 5 replicates with 8 CM6 products in each replicate.

Comments on TPM, Water, Nicotine, NFDPM, CO :

Average values:

- For product B the results were stable except for a slight reduction in water.
- Product C presents now average values closer to those obtained previously to the 6th CS.
- Product D shows a clear reduction in all the smoke analytes maybe due to a reduction in weight since the first study. This product has a closed end that need to be cut for smoking it.

Repeatability "r":

• Products C and D have clearly improved. B is stable

Product B	improved	stable	worse
TPM		TPM	
Nicotine		Nicotine	
Water		Water	
NFDPM		NFDPM	
CO		CO	
Product C	improved	stable	worse
TPM	TPM		
Nicotine	Nicotine		
Water	Water		
NFDPM	NFDPM		
CO	CO		
Product D	improved	stable	worse
TPM		TPM	
Nicotine	Nicotine		
Water	Water		
NFDPM	NFDPM		
CO	CO		
-			

Table 15. Variation of repeatability "r" with respect to the previous CS

Reproducibility "R":

• Product D improved in all analytes. Being the biggest product, it may indicate a better performance of labs with big cigars.

In general, there is a majority of analytes in the three products with improvements

Product B	improved	stable	worse
TPM Nicotine Water NFDPM CO	TPM Water NFDPM	Nicotine	со
		1	
Product C	improved	stable	worse
TPM Nicotine Water NFDPM CO	Nicotine Water		TPM NFDPM CO
Product D	improved	stable	worse
TPM Nicotine Water NFDPM CO	TPM Nicotine Water NFDPM CO		

Table 16. Variation of Reproducibility "R" with respect to the previous CS

The repeatability and reproducibility ranges of the analytes of the different products are expressed in Table 17.

Table 17. Range of r & R relative values in the different products

	repeatability (r) %		reproducibility (R) %
Product B ra	nge		
16%	TPM, Nicotine, NFDPM CO Water	22-26% 74% 92%	
Product C ra	nge		
9-11% 28%	TPM, Nicotine, NFDPM, CO Water	23-26% 29% 70%	
Product D ra	nge		
13% 19-24% 36%	TPM, Nicotine, NFDPM		TPM, NFDPM, CO Nicotine, Water

Between 11 and 13 labs have provided CO valid results for Products B, C and D. The comparison of the results with the previous Collaborative Studies lead to the following conclusions:

- The average CO for Product B is stable around 20 mg/cigar and for Product C is stabilised around 60 mg/cigar since the introduction of a Draft Recommended Method in the 3rd study. For product D, the bigger 100 % natural tobacco cigar, CO has been reduced to 160 mg/cigar. This product has a closed end that need to be cut for smoking.
- Introduction of Draft Recommended Method in the 3rd study has led to a stable repeatability value in Products B, C and D.
- The Reproducibility has also improved for Products C and D while Product B has a result similar to the 2nd CS.

5. DISCUSSION

The Collaborative Studies have shown high yield repeatability "r" and reproducibility "R", both in absolute as well as in relative terms, when compared with cigarette yields in equivalent collaborative studies with the relevant methodology. This is partly caused by the fact that we consider one result to be the average of 8 cigars smoked, whereas the cigarette method uses 20 cigarettes for one result, making the result an average of a greater number of units, thus reducing its variation. In addition, due to the wide range of products, cigars cannot be smoked in a fully automated manner.

More importantly however, the high cigar variability is mainly caused by reasons related to the cigar production system and to the inherent characteristics of the product itself:

5.1 PRODUCTION RELATED REASONS:

There is a very great dispersion of the weight, density, pressure drop, length and circumference of the products, due to:

- Different and relatively low technology machine making systems;
- The production often takes place in small batches, especially in the higher price-classes;
- Some cigars are handmade;
- Difficulties in controlling the porosity and gluing of the natural binder and wrapper giving rise to additional variability.

5.2 PRODUCT RELATED REASONS

There is a wide range of the tobaccos used for cigar filler, wrapper and binder:

- Tobacco is a natural product. The thickness, texture, porosity and combustibility of the leaves vary substantially;
- For binder and wrapper sometimes there are only a few bales available because of the sorting according to harvesting by grade, colour and length. Subsequent batches may also differ significantly;
- The filler tobaccos for most cigars are threshed. For long-filler cigars, the filler tobaccos are not threshed at all. The consequence is a different size of the

particles and therefore non-homogeneity of the filler within a cigar. This influences the pressure drop and the smoking characteristics of the cigar;

- The spirally rolled wrapper may allow air inflow;
- Headed cigars present a special problem, as the operator must cut them manually (as in Product D).

6. CONCLUSIONS AND FUTURE WORK

The objectives of the CORESTA Sub-Group "Cigar Smoking Methods" are the following:

- 1. To develop and update CORESTA Recommended Methods as requested by the Scientific Commission by investigating the technical problems associated with the mechanical smoking of cigars.
- 2. To conduct periodical collaborative studies in order to improve repeatability and reproducibility in different cigar sizes and types.
- 3. To establish confidence intervals for the smoke yields of all different cigar sizes.

The Sub-Group Cigars has conducted several Collaborative Studies by which the CRMs have been improved and the last CRM on CO measurement has been finally approved. These methods provide a credible and useful comparison of the smoke chemistry of cigars of all shapes and sizes, and take into consideration the practical, time consuming work of cigar smoking in laboratories.

These methods for the machine smoking of cigars are a technical convention, only to be used for comparison between different cigar brands.

Repeatability and Reproducibility have been improved through the different collaborative studies but there are signs in the last CS that due to previous modifications, further significant improvement may not be possible. The improvement of operation in the participating labs and the continuation of their participation in the CSs should help in improving the r&R results. However, the production and product related reasons mentioned in the discussion in addition to the lower number of cigars smoked per result and the smoking technology available make it impossible to reach the same reproducibility levels obtained for cigarettes.

The first two objectives of the CORESTA Sub-Group Cigar Smoking Methods have been met.

Future collaborative studies will be carried out periodically to improve yield repeatability and reproducibility and to help establish realistic tolerances for cigar smoke yields.

ANNEXES

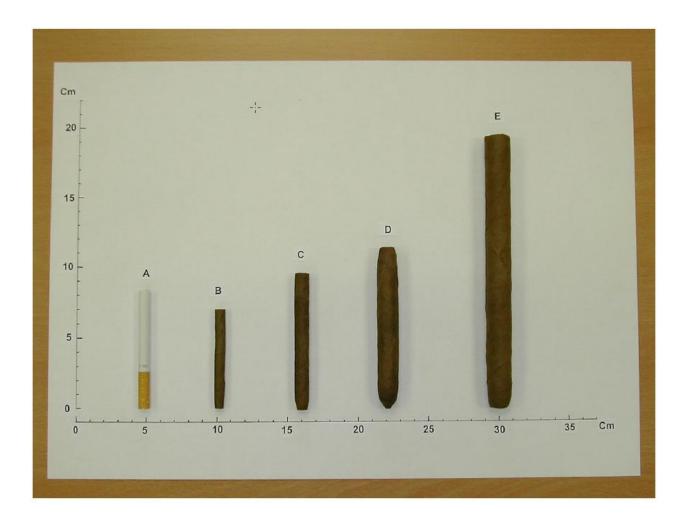
- I. Participating laboratories
- II. Products smoked and smoking parameters
- III. CORESTA Sub-Group Cigars Technical Report of 19 April 2005
 - Available upon request from the CORESTA Secretariat.
- IV. Cigar CORESTA Recommended Methods
 - As listed in Table 1, available on the CORESTA website. www.coresta.org

ANNEX I: Participating laboratories

	CS1	CS2	CS3	CS4	CS5	CS6	CS7
Agio Cigars	Х	Х	х	х	х	Х	Х
Altadis France		Х	Х	Х	Х		
ITG Altadis Spain	Х	Х	Х	Х	Х	Х	Х
Arnold André	Х	Х	Х	Х	Х	Х	Х
MST Vectis (BAT)	Х	Х		Х	Х	Х	Х
Burger		Х	Х	Х	Х		
JTI UK (Gallaher) (CITA)	Х	Х			Х	Х	Х
STG (Henri Wintermans)	Х	Х	Х	Х	Х	Х	Х
Schweitzer-Mauduit	Х						
STG (Swedish Match Cigars)	Х	Х	Х	Х	Х	Х	Х
Swedish Match North America	Х						
Tabacofina Vander Elst	Х	Х	Х		Х		
Villiger	Х	Х	Х		Х	Х	Х
Austria Tabak		Х	Х	Х	Х		
Reemstma ITG		Х	Х		Х		Х
CNTC Zhengzhou Tobacco Research Ir	nst.				Х	Х	
CNTC Chuanyu							Х
CNTC Shandong							Х
FILTRONA Technology Centre							Х
TOTAL	14	13	11	9	10	9	13

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ANNEX II Products smoked and smoking parameters

Product Codes, Characteristics and Smoking Parameters				
Codes	approx. weigh g	length mm	diameter mm	Puff Volume ml
А	0,97	84,0	7,9	20
В	0,73	70,0	7,5	20
с	2 DO	96,0	10,3	20
D	6 DO	114,5	15,7	34
E	13.3	194 ₍ D	18,1	46

For all Product Codes:

Puff Frequency = 40 sPuff Duration = 1.5 sButt Length = 33 mm