



## Fine-Cut Tobacco: Annual TNCO Study using a Monitor Tobacco

Christian Schulz<sup>1</sup> & Franz Peter Marx<sup>2</sup>

1. Imperial Tobacco Group, Reemtsma Cigarettenfabriken GmbH, Albert-Einstein-Ring 7, D-22761 Hamburg, Germany
2. European Smoking Tobacco Association (ESTA), Rond Point Schumanplein 9, 1040 Brussels, Belgium

### INTRODUCTION & BACKGROUND

ISO Standard 15592-3 has been developed to determine nicotine free dry particulate matter ('tar') and nicotine in mainstream smoke of fine cut tobacco smoking articles (FCSAs).

ESTA (European Smoking Tobacco Association) has offered interested laboratories the opportunity to participate in regularly conducted collaborative studies on ISO FCSA smoke analysis using a reference material (ESTA Monitor Tobacco, EMT) in order to assess if the analytical process is in control. Currently the third EMT blend ('EMT03') is in use and has been studied since 2009.

The purpose of these studies is to initially establish 'tar' and nicotine yields of the respective EMT blend and to provide a regular overview on repeatability and reproducibility.

Organisation of the annual EMT studies is supported by the ESTA Products Working Group-Technical Task Force and conducted in a fully anonymous way. Statistical evaluation is done in accordance with ISO 5725. In addition to the study reports provided to each participant, ESTA organises a meeting after each study for laboratory managers to exchange experience and address technical points of relevance observed during the study.

The poster shows results of the 2011 Study and also provides a comparison of results to those obtained from similar studies conducted in 2009 and 2010 using the same reference material.

### EXPERIMENTAL / Study Design

14 laboratories from six countries took part in the 2011 collaborative study. 3 participants delivered data sets from two smoking machines. In total, 17 data sets have been included in the statistical evaluation.

Preparation of the FCSA's and smoke analysis has been conducted according to ISO 15592-2<sup>1</sup> and ISO 15592-3<sup>2</sup> and related ISO standards by the participating labs. CO determination has been carried out according to ISO 8454<sup>3</sup> (although the method is not validated for FCSA smoke analysis)

The following tube type specifications and diameters - specified by ISO 15592 - have been centrally supplied to ensure that only tubes from one production batch are used for the preparation of FCSA's:

- 7.2 mm diameter Paper A tubes (Sample code: A\_7.2)
- 5.2 mm diameter Paper A tubes (Sample code: B\_5.2)
- 7.2 mm diameter Paper B tubes (Sample code: B\_7.2)
- 5.2 mm diameter Paper B tubes (Sample code: B\_5.2)

The reference tobacco blend EMT03 has been produced in 2008 and is supplied by Borgwaldt and Cerulean. The tobacco blend has the following characteristics :

- blend of fire-cured, air-cured and flue-cured tobaccos
- cut width 0.35 mm
- humectant 4 % propylene glycol
- pack moisture 18 % (Karl-Fischer water)



Figure 1

Approximately 500 kg of this tobacco were taken from the middle of a larger run and packed into 40g pouches. Sets of ten pouches were poly-wrapped with paper and foil. These sets of ten poly-wrapped pouches were the basic unit available for purchase from the supplier (see Figure 1).

Participating laboratories have been requested to conduct 5 replicates per paper tube specification. Table 1 provides an overview about the reported data sets, participants and smoking machines used in 2011.

The statistical evaluation of data for this collaborative study followed the methods provided by ISO 5725-2<sup>4</sup>. Especially for outlier testing, the Grubbs and Cochran methods have been used. The protocol 'Harmonized statistical procedure'<sup>5</sup> defined by IUPAC (International Union of Pure and Applied Chemistry) has been applied as well.

	2009	2010	2011
Participants	14	16	14
Countries	10	7	6
Industry/non industry	8 / 6	12 / 4	11 / 3
Smoking machines (Rotary/Linear)	10 R/7 L	14 R/4 L	13 R/4 L
No of replicates per specification	5	5	5
Parameter reported	7	7	7
Data sets complete	15	16	15
Data sets reduced	2	2	2
Data points evaluated	2303	2380	2240

Table 1: study design 2009 - 2011

### RESULTS

Nicotine Free Dry Particulate Matter [mg/FCSA]						
sample	data sets	mean	r	R	CV <sub>r</sub>	CV <sub>R</sub>
A_7.2	15	14,2	1,05	1,87	7,4%	13,2%
B_7.2	16	18,4	2,07	3,43	11,3%	18,6%
A_5.2	15	8,0	1,07	2,29	13,3%	28,5%
B_5.2	14	12,2	1,14	4,09	9,3%	33,5%
Smoke Nicotine [mg/FCSA]						
sample	data sets	mean	r	R	CV <sub>r</sub>	CV <sub>R</sub>
A_7.2	16	1,1	0,11	0,19	10,3%	17,6%
B_7.2	16	1,4	0,14	0,28	10,4%	20,3%
A_5.2	14	0,6	0,07	0,13	12,6%	22,2%
B_5.2	14	0,9	0,12	0,34	13,7%	38,2%
CO - Content [mg/FCSA]						
sample	data sets	mean	r	R	CV <sub>r</sub>	CV <sub>R</sub>
A_7.2	15	10,6	0,91	2,57	8,6%	24,2%
B_7.2	14	12,7	1,35	2,76	10,7%	21,8%
A_5.2	15	6,6	0,79	1,67	12,0%	25,5%
B_5.2	14	9,7	1,22	1,97	12,6%	20,4%

Table 2: Results for TNCO EMT03-2011, outliers excluded

Due to different paper characteristics, amount of tobacco and the diameter of the analysed FCSA's, four mean values for each parameter have been obtained as outlined in Table 2.

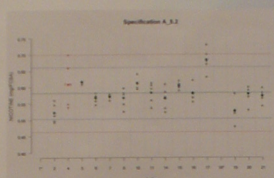


Figure 2: smoke nicotine single values

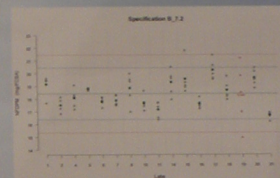


Figure 3: NFDPM single values

Two examples of individual values (2011) are given in figure 2 and 3.

### Comparison repeatability (r) and reproducibility (R) 2009 to 2011 :

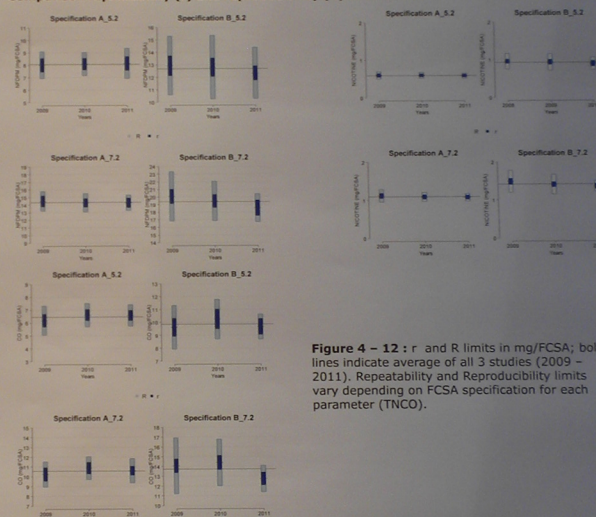


Figure 4 - 12 : r and R limits in mg/FCSA; bolt lines indicate average of all 3 studies (2009 - 2011). Repeatability and Reproducibility limits vary depending on FCSA specification for each parameter (TNCO).

### CONCLUSIONS & OUTLOOK

\*The 2011 study has highlighted repeatability and reproducibility values still above those normally seen for ISO cigarette smoke analysis (see Table 2).

\*In comparison to the studies conducted in 2009 and 2010:

- o CV<sub>r</sub> values (r expressed as a percentage of the overall mean) between 7% and 14% show comparable levels and indicate that analytical processes are under appropriate control within the laboratories taking into account the complexity of the entire analytical procedure (e.g. preparation of FCSA's)
- o CV<sub>R</sub> results of 13% to 38% suggest a trend towards lower figures for the three major parameters NFDPM, NIC and CO. This appears to be more prominent for the B paper configurations

\*Statistically robust mean values for the EMT03 reference material have been established in 2009 through a large international collaborative study involving 14 laboratories and 17 smoking machines. Through annual repetitions with comparable numbers of participants these estimates have been re-evaluated.

\*As shown in figures 4 - 12, it appears that mean values of NFDPM, NIC and CO have remained constant for the A-paper configurations across the 3 studies. This, however, looks different for the B-paper configurations where the mean values suggest a decrease which is more prominent for CO and to a lesser extent also for NFDPM and NIC. This observation might be caused by changes over time of properties inherent to the B paper and requires further investigation.

\*ESTA has received positive feedback for organising a meeting after each study. These meetings provide an excellent opportunity to exchange experience regarding FCSA smoke analysis and serve as a platform to address technical points of relevance observed during each study.

\*An appropriately high number of participants allows a robust estimation of mean values for a reference material which can be used for quality control purposes.

### REFERENCES

- [1] ISO 15592-3 Fine-cut tobacco - Determination of total particulate matter of smoking articles using a routine analytical smoking machine, preparation for the determination of water and nicotine, and calculation of nicotine-free dry particulate matter
- [2] ISO 15592-2 Fine-cut tobacco - Atmosphere for conditioning and testing
- [3] ISO 8454 Cigarettes - Determination of carbon monoxide in the vapour phase of cigarette smoke - NDIR method
- [4] ISO-5725-2 Basic method for the determination of repeatability and reproducibility of standard measurement method. (1994)
- [5] IUPAC Harmonized statistical procedure 1994