Beiträge zur Tabakforschung International/*Contributions to Tobacco Research* Volume 21 · No. 2 · July 2004

DOI: 10.2478/cttr-2013-0775

Measurement of the "Tar" and Nicotine Yield of Fine-Cut Smoking Articles – The ISO/TC 126 Collaborative Study 2001*

by

*Peter I. Adams***

Secretariat ISO/TC 126, Burggrafenstr. 6, 10787 Berlin, Germany

SUMMARY

The Technical Committee ISO/TC 126 "Tobacco and tobacco products" of the International Organization for Standardization set up Working Group 7 to develop and test a method for the determination of "tar" and nicotine yield when fine-cut smoking articles (FCSAs) are smoked by routine-analytical smoking machines. FCSAs are individually made by consumers using tobaccos manufactured for enclosure in paper wrappers (i.e. leaflets and tubes – with or without filters). This paper records the history of method development, defines terms and explains the differences between measurements made on FCSAs and commercially factory made cigarettes. Individual consumer making practices are represented by the specification of four hand-made article types using two wrappers of different physical properties each with either 400 mg or 750 mg tobacco.

ISO Standards require estimates of the repeatability (*r*) and reproducibility (*R*) of analytical methods, so the working group has conducted a collaborative study. The values of *r* and *R* were determined using the definitions given in ISO 5725-2 (18). Twenty laboratories took part and tested three different commercial tobacco blends according to the standard method. The detailed protocol for the study is given in Annex A. Annex B is an illustrated manual used as a training aid for laboratory personnel with little experience of making FCSAs.

After the smoking was completed, the laboratory data were examined for outlying points by the methods of Grubbs and Cochran. It was found necessary to exclude the data from one laboratory prior to statistical analysis otherwise the incidence of outliers was low (1.41%). Values of *r* and *R* were calculated from the results of one-way analyses of variance (within and between laboratories) for smoke nicotine and nicotine-free dry particulate matter (NFDPM). The data for the 19 laboratories are given in Annex C. The quality of the data is high as is shown by the comparisons made between the *r* and *R* values for the articles and those for the monitor test pieces which were used to confirm that smoking conditions run-to-run were satisfactory. The range of NFDPM and smoke nicotine yields resulting from the four designs of articles is large for each of the three blends. The measured NFDPM yields of the four designs are ranked in the same order by all of the laboratories and all pairs are statistically different for every laboratory. For smoke nicotine the ranking order is the same for all laboratories. All pairs are statistically different in all but three laboratories for which only the 400 mg articles were not statistically different.

The *r* and *R* data are given in tables and in graphical representation. The graphs show that *r* and *R* increase with the mean values of NFDPM and smoke nicotine yields. Such a trend has also been found in studies using factory-made cigarettes. A comparison of results shows that *r* values for articles are approximately 40% greater and *R* values about 100% greater. The variability of individual hand-made articles in this study is likely to be much greater than the variability of machine-made matched cigarette samples. The method detailed in ISO 15592-3, therefore, is satisfactory for general adoption even in laboratories where there has been little previous experience.

[Beitr. Tabakforsch. Int. 21 (2004) 139–155]

ZUSAMMENFASSUNG

Die Arbeitsgruppe 7 (WG 7) des Technischen Komitees ISO/TC 126 „Tabak und Tabakerzeugnisse" der Internationalen Organisation für Normung hat eine Methode zur Bestimmung des beim routinemässigen Abrauchen von Feinschnitt-Tabak-Rauchkörpern (FCSAs) in Rauchmaschinen anfallenden Kondensat- und Nikotingehalts erarbeitet und

* Received: 12th June 2003 – accepted: 29th March 2004

** Edited from contributions made by members of the planning sup-group.

getestet. FCSAs werden von den Konsumenten unter Verwendung von Feinschnitttabaken und Umhüllungsmaterialien (Blättchen und Röhrchen – mit oder ohne Filter) selbst hergestellt. In diesem Bericht wird die Geschichte der Erarbeitung der Methode dargestellt, es werden Begriffe definiert sowie die Unterschiede zwischen FCSAs und industriell gefertigten Zigaretten bei der Bestimmung der Abrauchwerte erklärt. Die individuellen Herstellungspraktiken der Konsumenten werden anhand von vier handgefertigten FCSA-Typen unter Verwendung von zwei unterschiedlichen Zigarettenpapierhüllen mit unterschiedlichen physikalischen Eigenschaften und einer Tabakeinwaage von 400 mg beziehungsweise 750 mg dargestellt.
Da ISO Normen eine Bewertung der Wiederholbarkeit (*r*) und Reproduzierbarkeit (*R*) der Analysenmethoden erfordern, führte die Arbeitsgruppe eine Ringuntersuchung durch. Die Werte für *r* und *R* wurden auf der Basis der in ISO 5725-2 (18) angegebenen Definitionen bestimmt. Zwanzig teilnehmende Labors untersuchten nach der Standardmethode drei verschiedene handelsübliche Tabakmischungen. Das ausführliche Studienprotokoll ist im Anhang A wiedergegeben. Anhang B ist eine illustrierte Arbeitsanleitung für Laborpersonal mit geringer Erfahrung in der Herstellung von FCSAs.
Nach Beendigung des Abrauchens wurden die Labordaten mit Hilfe der Grubbs- und Cochran-Methoden nach Ausreißern untersucht. Der Ausschluss der Daten eines Labors vor der statistischen Analyse wurde als notwendig erachtet, ansonsten war die Häufigkeit der Ausreißer gering (1,41%). Die Werte für *r* und *R* wurden auf der Basis der Ergebnisse von Varianzanalysen (innerhalb der Labors und zwischen den beteiligten Labors) für Rauchnikotin und nikotinfreies Trockenkondensat (NFDPM) berechnet. Die Abrauchdaten der 19 Labors sind im Anhang C wiedergegeben. Die Validität der Daten ist hoch, was sich beim Vergleich der Werte für *r* und *R* von FCSAs und Referenz-Zigaretten zeigt. Letztere dienten zur Bestätigung, dass die Rauchbedingungen von Testlauf zu Testlauf zufriedenstellend waren. Die Unterschiede im NFDPM und Rauchnikotingehalt, die sich bei den vier verschiedenen FCSAs ergaben, zeigen für jede der drei Tabakmischungen eine große Varianz. Die Rangfolgen der gemessenen NFDPM-Werte für die vier FCSA-Typen sind in allen Labors gleich und die Unterschiede in jedem Labor statistisch signifikant. Die Rangfolge für Rauchnikotin ist bei allen Labors die gleiche. Der paarweise Vergleich zeigt in allen Labors statistische Signifikanz mit Ausnahme von drei Labors, in denen die FCSAs mit 400 mg nicht statistisch signifikant unterschiedlich sind.
Die Daten für *r* und *R* sind in tabellarischer und grafischer Form dargestellt. Die grafischen Darstellungen zeigen, dass *r* und *R* bei steigenden Mittelwerten von NFDPM und Rauchnikotin zunehmen. Ein solcher Trend hat sich auch in Studien mit industriell gefertigten Zigaretten gezeigt. Ein Vergleich der Ergebnisse mit Fabrikzigaretten zeigt, dass die Werte für *r* für FCSAs etwa 40% und die Werte für *R* etwa 100% höher liegen. Die Varianz handgefertigter FCSAs scheint in dieser Untersuchung viel größer zu sein als bei industriell gefertigten Zigarettenproben. Die in ISO 15592-3 festgelegte Methode ist daher für eine allgemeine Anwendung zufriedenstellend, selbst in Labors, in denen nur über geringe Erfahrungen in Fertigung und Abrauchen von FCSAs verfügt wird. [Beitr. Tabakforsch. Int. 21 (2004) 139–155]

RESUME

Le groupe de travail no. 7 du comité technique ISO/TC 126 « Tabac et produits du tabac » de l'Organisation internationale de normalisation a développé et testé une méthode de détermination des rendements en goudron et nicotine d'objets à fumer à base de tabac à rouler (OFTR), à l'aide d'une machine à fumer analytique de routine. Les fumeurs confectionnent les OFTR en roulant du tabac à rouler dans une enveloppe de papier appropriée (cahiers et tubes – avec et sans filtre). Cet article retrace l'historique de la mise au point de la méthode, définit les différents termes employés et explique les différences entre les mesures réalisées sur des OFTR et celles sur des cigarettes commerciales faites industriellement. Afin de tenir compte des différentes pratiques des consommateurs, quatre objets à fumer sont confectionnés à partir de deux enveloppes ayant des propriétés physiques différentes, et de deux masses de tabac, 400 mg et 750 mg.
La norme ISO nécessitant l'estimation de la répétabilité (*r*) et de la reproductibilité (*R*) des méthodes analytiques, le groupe de travail a mené une étude collective. Les valeurs de *r* et *R* ont été déterminées en utilisant les définitions données dans l'ISO 5725-2 (18). Vingt laboratoires ont pris part à cette étude collective, au cours de laquelle trois mélanges de tabacs commerciaux ont été testés selon la méthode proposée dans la norme. Le protocole détaillé de l'étude est donné dans l'annexe A. L'annexe B est un mode opératoire illustré, pouvant servir de référence à des personnels de laboratoire peu familiarisés avec la méthodologie de confection des OFTR.
Une fois les mesures de fumages achevées, les données des laboratoires ont été analysées afin d'éliminer les valeurs aberrantes selon le test de Grubbs et Cochran. Il s'est ainsi révélé nécessaire d'éliminer les données provenant d'un laboratoire avant d'effectuer l'exploitation statistique. Les valeurs de *r* et *R*, pour la mesure de la nicotine dans la fumée et de la matière particulaire anhydre et exempte de nicotine (NFDPM), ont été calculées à partir d'une analyse de variance à un facteur (intra et inter laboratoire). Les résultats des 19 laboratoires sont donnés dans l'annexe C. Les résultats obtenus sont satisfaisants comme en témoigne la comparaison entre les valeurs de *r* et de *R* obtenues pour les OFTR, et celles obtenues pour les cigarettes de référence (monitor) utilisées lors de l'étude dans le but de vérifier que les conditions de fumage entre chaque série étaient satisfaisantes. Les valeurs des rendements en NFDPM et en nicotine dans la fumée mesurées pour chacun des quatre OFTR, sont comprises dans une large étendue pour chacun des trois mélanges testés. Les rendements en NFDPM mesurés pour chacun des 4 OFTR sont classés dans le même ordre par chacun des laboratoires, et les différents couples sont tous différents pour chacun des laboratoires. Le classement des 4 OFTR pour la nicotine dans la fumée est également identique pour tous les laboratoires. Les différents couples sont tous différents pour chacun des laboratoires à l'exception de trois, pour lesquels il n'est pas observé de différence statistique pour les articles à 400 mg.
Les valeurs de *r* et de *R* sont données dans des tableaux et sous forme de représentation graphique. Ces graphiques montrent que les valeurs de *r* et de *R* augmentent avec la valeur en NFDPM, ainsi qu'avec la nicotine dans la fumée.

Une telle tendance a déjà été mise en évidence dans des études effectuées sur des cigarettes industrielles. Une analyse comparative des résultats montre que les valeurs de *r* sont d'environ 40% plus grandes pour les OFTR, et que les valeurs de *R* sont environ 100% plus grandes. La variabilité de chacun des OFTR préparés manuellement dans cette étude a tendance à être supérieure à celle de cigarettes préparées industriellement. Cependant, la méthode ISO 15592-3 est satisfaisante et peut être adoptée même par des laboratoires ayant peu d'expérience. [Beitr. Tabakforsch. Int. 21 (2004) 139–155]

INTRODUCTION

Hand made smoking articles

Tobacco intended for consumers to make into smoking articles is variously known as fine-cut, hand rolling, rolling, shag, roll-your own (RYO) tobacco etc. Articles made from such tobaccos are referred to here as Fine Cut Smoking Articles (FCSAs). A FCSA is essentially a quantity of tobacco rolled manually in a wrapper normally a piece of paper. In order to produce a standard method many detailed properties of the article must be specified to take account of the way tobacco is used.

When a smoker chooses to smoke hand made articles, he (or she) has to select the type of tobacco and the type of wrapper. It is also a matter of personal preference as to how much tobacco is placed in an article. Tobacco and sometimes a filter are placed in a wrapper, roughly rolled between the fingers and thumb and the paper lap sealed. The shape and diameter of the resulting cylinder can vary greatly according to the mass of tobacco, the width of the wrapper and how the article is made. A variety of hand-held making devices exist to refine the process and make a more uniform product. Some are designed to fill a preformed tube that may or may not be attached to a filter tip.

Standardization

A realistic set of standards for fine-cut tobacco must offer a repeatable and reproducible method to give smoke "tar" and nicotine data relevant to hand-made products that might be made by a consumer taking into account the differences between cigarettes and FCSAs.

For cigarettes the mass of tobacco, the type of cigarette paper, the presence or absence of a filter are fixed once the choice of brand has been made. A set of International Organisation for Standardisation (ISO) standards exists from sampling through to the measurement of "tar" and nicotine as a result of a routine smoking regime (1–7). It is possible to make a proper statistically based sampling of the cigarettes that may be held, lit and smoked in the standard smoking machine to provide the determinations. For FCSAs, different masses of tobacco are used according to the personal choice of the consumer. Studies have been made and are reported in summary in (8) and specifically in DARRALL and FIGGINS (9), KAISERMAN and RICKERT (10) and DYMOND (11,12). Different wrappers are used and the physical make up of the papers employed can lead to different rates of burn characteristics of the finished articles. The cut-width of fine-cut tobaccos is in general less than in factory manufactured cigarettes and tobacco is supplied at higher moisture contents. The article property (mass, wrapper etc.) surveys show that national differences exist. A set of standards therefore must attempt to take account of these differences. Two ISO standards already exist:

- ISO 15592-1 (13) deals with sampling fine-cut tobacco and because tobacco is normally sold in pouches the method differs from that for cigarettes (1). This is because it is not possible to mix tobacco from several pouches without degradation so articles must be made from each pouch and tested as a sampling unit.
- ISO 15592-2 (14) deals with the temperature and relative humidity conditions for tobacco and FCSA conditioning prior to testing. These differ from those specified in (2) because fine-cut tobacco has a considerably higher moisture content than tobacco used in factory made cigarettes.

The work of the Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA) (8) has reported that FCSAs cannot be held reliably in the labyrinth holders specified in ISO 3308 (three rubber diaphragms with holes sized appropriately to the diameter of the cigarette to be tested) without unacceptable leakage during puffing. A holder was specially developed by members of the CORESTA task force to hold a greater length of the article in a thin walled rubber tube. It makes a more effective seal with the less uniform structure of the articles. It is in fact not unlike the cigarette holders of the 1960s primarily used for plain cigarettes. To ensure the rubber tube is not burned, the articles cannot be smoked to butt lengths shorter than 27 mm.

The CORESTA task force did many studies and collaborative tests in order to find ways of representing the wide range of habits of consumers. The report (8) records the use of two types of wrapper of different properties that represent a great proportion of usage (at least in Europe). Also the data from the smoker surveys allowed the selection of two tobacco masses that reasonably cover the range used. These two facts result in the conclusion that a matrix of 4 determinations could reasonably represent the "tar" and nicotine yield of a sample of fine-cut tobacco. It should be noted that the matrix attempts only to represent making practice of different consumers for the purpose of preparing articles for routine smoking analysis. As with factory cigarettes how a consumer smokes a FCSA is another matter concerning personal choice. The analytical data produced (as, indeed, with cigarettes) are valuable for product ranking purposes but not necessarily for estimates of consumer intake of smoke components.

For the reasons above, the use of the smoking machine and method of smoking machine standards (3) and method of smoking standard (4) cannot be used directly. The modifications necessary to apply them to FCSAs became the subject of a new work item in the programme of ISO/TC 126 Tobacco and tobacco products. A working draft ISO/WD 15592-3 was made by the TC 126 Secretariat based on (4) allowing for the cigarette/FCSA differences and some of the findings of (8). A working group of experts (WG 7) was convened at the beginning of 2000 in order to add urgency to the development of the draft.

An ISO working group consists of two experts nominated by any member body e.g. Association française de normalisation (AFNOR), Deutsches Institut für Normung (DIN), Nederlands Normalisatieinstituut (NEN) etc. wishing to take part in the work. The experts so nominated included those with tobacco and wrapper paper interests but also some from regulatory bodies or a laboratory advising a regulator. The group's task was to produce a Committee Draft (CD), the next stage in the ISO process. Consensus was reached for such a draft but not with complete agreement. The CD met with a vote of approval by TC 126 members and the draft reached Draft International Standard (DIS) status in February 2002 and has subsequently been published as an International Standard (15). ISO directives require estimates of repeatability (*r*) and reproducibility (*R*) at the DIS stage prior to publication as an International Standard.

In summary the major changes to the ISO Standard for machine smoking cigarettes are the use of a special longer FCSA holder, a consequent increase in butt length to 27 mm and the matrix of four articles. The values of the machine puffing parameters remain the same; 1 puff per minute of duration 2 s and volume 35 mL Determinations of water and nicotine in smoke condensate (5,6,7) are directly suitable for use.

THE COLLABORATIVE STUDY

A small subgroup of members of WG 7 met with the help of three statisticians in order to plan a study using ISO/CD15592-3 and based on the principles set out in ISO 5725-1 (17). The protocol [a] (somewhat abridged) for the study is given in Annex A. It was agreed that the analysis of the data should in general follow the recommendations embodied in ISO 5725-2 (18). It is recognised, however, that the treatment of outliers (described below) is somewhat different from these recommendations.

The statisticians wished to minimise any rejection of suspect data. By setting the acceptance/rejection limit at 2.5 for the standardised residuals derived from the analyses of variance the rejection rate was lower than that to be expected from the recommended procedure using Cochran's test. The one-way analyses of variance resulted in the rejection of individual data points rather than the rejection of the complete set of data from a laboratory (for a single analyte from a single FCSA variant).

The Cochran and Grubbs tests [as described in ISO 5725-2 (18)] were used after the removal of individual data points using one-way analyses of variance. The outcome of these tests for the main target variables (NFDPM and nicotine) identified only three subsets of data for rejection, and two of these (nicotine; laboratory 7) were subsequently found to be suspect (see below).

Three samples of fine-cut tobacco were supplied. The blends were: 1) A finely cut fire-cured tobacco blend used for rolling and mainly sold in the Netherlands (coded Red); 2) A slightly more coarsely cut blend of roughly equal proportions of fire- and flue-cured tobaccos, mainly sold in Europe (coded White); 3) A blend of flue-cured tobacco with a small proportion of air-cured tobacco somewhat more coarsely cut than either blend 1) or 2) sold mainly in Scandinavian countries (coded Yellow).

Two types of wrapper were supplied as tubes made from paper conforming to the type A and B wrapper specifications given in ISO/CD 15592-3. Each type was used to make articles at each of two tobacco masses, 400 mg and 750 mg, for each of the three blends. Each of the 12 FCSA variants was given a two character alphanumeric code to indicate the blend and configuration used in the design. R, W or Y indicated the use of Red, White or Yellow blend, respectively, and 1, 2, 3 or 4 indicated to which of the four designs (1 = 400 mg of tobacco with A type wrapper tube of 5.2 mm diameter; 2 = 400 mg of tobacco with B type wrapper tube of 5.2 mm diameter, 3 = 750 mg of tobacco with A type wrapper tube of 7.2 mm diameter, 4 = 750 mg of tobacco with B type wrapper tube of 7.2 mm diameter), as defined in ISO 15592-3, the articles were made.

Twenty laboratories took part (see Acknowledgement). Of these 19 were from Europe and one from Australia. Three laboratories had regulatory interests and some had not used the entire procedure before. One of the laboratories compiled a manual to aid the use of the specified making apparatus; this is reproduced in Annex B.

Smoking plans included the use of the CORESTA Monitor 3 (CM3) as a monitor test piece [the subject of ISO 16055 (16)]. It is a filter-tipped cigarette taken from a batch produced under special and controlled manufacturing conditions. It is used to monitor the stability of the analytical processes to determine NFDPM and smoke nicotine yields when using a smoking machine.

Statistical treatment of the data – detection of outlying observations

The data from this experiment arrived for analysis during the period 3rd October 2001 to 7th November 2001, the data from each laboratory arriving as (usually) an Excel worksheet, enabling it to be readily collated with any data sets already received. As the various data sets were successively collated it became apparent that the data from one laboratory showed major discrepancies when compared with the data from other laboratories currently available. The laboratory concerned was contacted and after some investigation, it was agreed that its data should be withdrawn from the analysis. It was also agreed that the results of the final analyses of the study would be made available to assist in the eradication of the problems that gave rise to the observed discrepancies. Thus, although 20 laboratories took part in the experiment, the data from only 19 was used in the final analyses.

The reported value of NFDPM for each smoking was compared with the value calculated as total particulate matter (TPM) – nicotine – water. Differences greater than 0.1 mg represented an error in the reported data. These errors were eliminated by referral to the laboratories.

Two types of smoking machine were used in this study. Linear machines have 20 ports and 20 channels, five FCSAs were smoked through each of these 20 channels during a single smoking run. Rotary machines have one port and 20 channels, 20 FCSAs were smoked through these channels during a single smoking run. In order to

[a] The protocol, as issued to the participants, is available in electronic form from the editorial office of the journal.

Table 1. Linear smoking machines – numbers of outlying points

FCSA	NFDPM	Nicotine
R1	4	2
R2	3	1
R3	3	2
R4	3	2
W1	3	2
W2	2	3
W3	4	3
W4	4	3
Y1	3	5
Y2	1	5
Y3	1	3
Y4	5	3
Total	36	34
Error rate	1.78%	1.78%
Monitor (CM3)	8	5
Error rate	1.68%	1.05%

Table 2. All smoking machines – number of outliers from pooled data averaged over 20 FCSAs

FCSA	NFDPM	Nicotine
R1	1	1
R2	1	1
R3	0	1
R4	2	1
W1	2	1
W2	1	3
W3	1	1
W4	2	0
Y1	2	2
Y2	0	1
Y3	1	1
Y4	3	2
Total	16	16
Error rate	1.41%	1.41%
Monitor (CM3)	5	2
Error rate	1.77%	0.70%

make the results from the two types of machine compatible for a combined analysis, the smoking plans (see Annex A) required that for the linear machines FCSAs of the same specification were smoked in four channels within a single run; thus both linear and rotary machines smoked 20 FCSAs of the same specification within a single smoking run. Individual outliers in the data for each run from the linear smoking machines were detected using one-way analyses of variance (between and within laboratories) for each analyte. The standardised residuals from these analyses were examined, and those whose absolute value was greater than 2.5 were deemed to be outliers. Table 1 shows the number of points detected as outliers in the data from linear machines. Note that these outliers are based upon the means obtained from smoking five FCSAs (or five monitor test pieces).

The data from the linear machines were then averaged over the smoke runs of 20 FCSAs (or 20 monitor test pieces) to reduce the data from the linear machines to the same format as that obtained from rotary smoking machines. These averages were taken over the four channels within a single run using the same FCSAs (or monitor test pieces). The data from the linear and rotary smoking machines were then combined into a single data set and outliers for each smoke run were detected using one-way analyses of variance (between and within laboratories) for each analyte for each FCSA configuration, or the monitor test piece. The standardised residuals from these analyses were examined, and those whose absolute value was greater than 2.5 were deemed to be outliers. Table 2 shows the number of points detected as outliers in the pooled data averaged over 20 FCSAs or monitor test pieces from all machines.

Having removed outlying data points as described above, outlying laboratories (for each analyte for each FCSA/monitor) were detected using the Grubbs and Cochran tests. The Grubbs test is based on laboratory means and is aimed at detecting those laboratories whose mean result differs significantly from the mean results of the other laboratories taking part in the study. It detects any laboratory whose mean is significantly either low or high, any pair of laboratories whose means are both significantly either low or high and any pair of laboratories one of whose means is significantly low and the other significantly high. The Cochran test is based upon within laboratory variances and is aimed at detecting those laboratories whose results are significantly more variable than the results of the other laboratories taking part in the study.

Table 3 shows which laboratories were rejected using the Grubbs test and the only laboratory to be rejected using the Cochran's test. Within the body of the table the appearance of two laboratory codes in the same cell indicates that these laboratories were detected as an outlying pair.

Following the completion of the outlier detection procedures it was noted that the nicotine data for FCSAs Y1 and Y4 from laboratory 7 also came close to the critical point for rejection. Discussions with the laboratory concerned indicated that the nicotine data from all the Y numbers FCSA configurations may all be suspect. It was therefore decided that all these subsets of the data should be removed from the analyses. This decision is reflected by the code for laboratory 7 being italicised in brackets in Table 3.

Table 3. Laboratories omitted from the analyses, Grubb's test

FCSA	Puffs	TPM	Water	Nicotine	NFDPM
R1	13, 17				
R2					
R3	13, 17				
R4	13, 17				
W1			16, 20		
W2					
W3	17				
W4	17 [a]	17	17		
Y1				*(7)* [b]	
Y2				7	
Y3				7	
Y4	17			*(7)* [b]	
Monitor (CM3)					

[a] Laboratory 17 for this table element was also excluded on the basis of the Cochran´s test.

[b] Nicotine data of laboratory 7 were removed from the analyses, see text for further details.

Table 4. NFDPM yield; mean, repeatability (*r*) [a] and reproducibility (*R*) [a] values

Blend type	Tobacco mass (mg)	Parameter	Value (mg)	
			Wrapper A	Wrapper B
Fire-cured (Red)	400	Mean	9.4	12.1
		r	1.41	1.61
		R	2.69	3.74
	750	Mean	15.7	19.8
		r	1.64	1.72
		R	3.13	4.06
Fire-cured/ flue-cured (White)	400	Mean	8.2	10.7
		r	1.02	1.31
		R	2.70	3.55
	750	Mean	14.6	18.4
		r	1.50	1.57
		R	3.07	5.46
Flue-cured/ air-cured (Yellow)	400	Mean	7.7	10.1
		r	0.97	1.27
		R	2.32	3.06
	750	Mean	13.9	17.4
		r	1.53	1.76
		R	3.32	4.52
CORESTA Monitor 3 (CM3)		Mean	15.0	
		r	0.94	
		R	1.98	

[a] For the purpose of calculating *r* and *R* one test result was defined as the mean yield obtained when smoking 20 FCSAs in a single run.

Table 5. Nicotine yield; mean, repeatability (*r*) [a] and reproducibility (*R*) [a] values

Blend type	Tobacco mass (mg)	Parameter	Value (mg)	
			Wrapper A	Wrapper B
Fire-cured (Red)	400	Mean	1.00	1.25
		r	0.136	0.173
		R	0.349	0.453
	750	Mean	1.74	2.09
		r	0.220	0.239
		R	0.402	0.547
Fire-cured/ flue-cured (White)	400	Mean	0.65	0.82
		r	0.083	0.108
		R	0.236	0.334
	750	Mean	1.14	1.39
		r	0.136	0.161
		R	0.286	0.432
Flue-cured/ air-cured (Yellow)	400	Mean	0.51	0.65
		r	0.073	0.080
		R	0.192	0.228
	750	Mean	0.93	1.14
		r	0.103	0.110
		R	0.224	0.300
CORESTA Monitor 3 (CM3)		Mean	1.21	
		r	0.087	
		R	0.216	

[a] See footnote Table 4.

*Calculation of repeatability (*r*) and reproducibility (*R*)*

The data from each of the 19 laboratories after removal of outliers as described above are given in Annex C, Tables C1 to C5.

The values of repeatability (*r*) and reproducibility (*R*) are based on the data after outlier removal, and calculated from the results of the one-way analyses of variance (between and within laboratories) for nicotine and NFDPM, for each FCSA configuration and for the monitor test piece. The values of *r* and *R* are shown in Table 4 for NFDPM and Table 5 for smoke nicotine together with the values of mean yield. These are the tables requested for the *r* and *R* statements in ISO 15592-3.

DISCUSSION

Quality of the data

Tables 1 and 2 show that for NFDPM and nicotine the incidence of outliers was low; 1.78% for the linear machines and 1.41% for the linear plus rotary machine data combined. Table 3 shows that for the target analytes there was only one outlying laboratory (laboratory 7) and that only for smoke nicotine for the blend coded Yellow.

The experimental procedure specified in ISO 15592-3 calls for the use of a monitor test piece. Such monitors are generally available and are the subject of ISO 16055 (16). They are used to confirm that test smoking conditions are satisfactory and the CORESTA Monitor 3 (CM3) was included in the study for this purpose.

It is interesting to compare the *r* and *R* values for the CM3 from this study with those previously reported for factory made cigarettes. The values of *r* and *R* for the CM3 are in line with the collaborative data for conventional factory made cigarettes as reported for NFDPM measurement in ISO 4387 and for smoke nicotine measurement in ISO 10315. Figures 1 to 4 illustrate this. This provides a good indication that the participants in this collaborative had the necessary expertise to perform the analyses.

Review of data against matrix principle

The four designs of article (two wrappers × two masses of tobacco) were chosen to represent a wide range of consumer making practices. For the designs to provide meaningful information to the consumer, they should result in a wide range of NFDPM and smoke nicotine yields, and the yields of any pair should be significantly different. It can be seen from Tables 4 and 5 that the range of NFDPM and smoke nicotine yields is large, the highest yielding design being at least twice that of the lowest, for each of the three blends.

The measured NFDPM yields of each of the four designs are ranked in the same order by all of the laboratories and all pairs are statistically different for every laboratory. Figures C2, C7 and C12 of Annex C illustrate the rank order.

The smoke nicotine yields of each of the four designs are ranked in the same order by all of the laboratories and all pairs are statistically different for all but three laboratories for which only the 400 mg articles were not statistically different. Figures C3, C8 and C13 of Annex C illustrate the rank order.

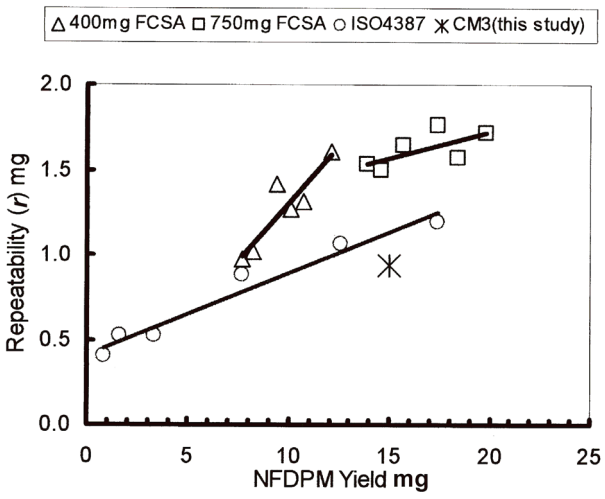


Figure 1. ISO/DIS 15592-3 and ISO 4387 repeatability values for NFDPM

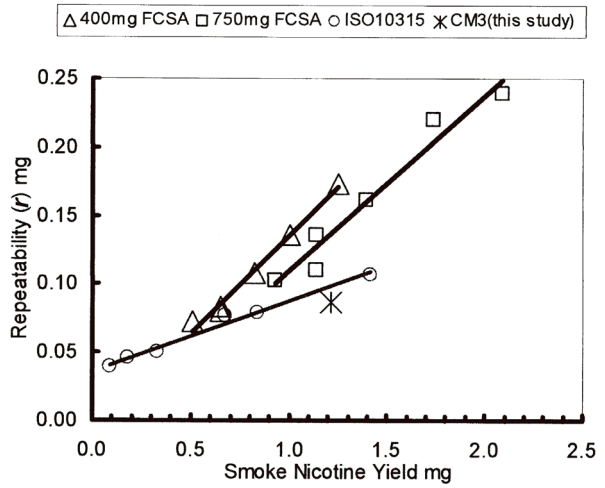


Figure 2. ISO/DIS 15592-3 and ISO 10315 repeatability values for smoke nicotine

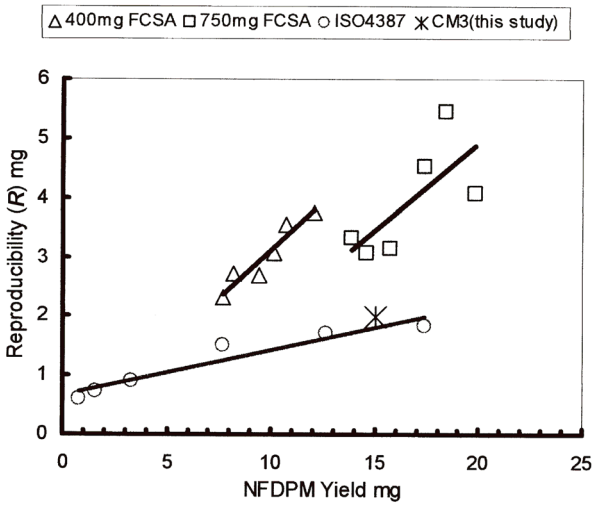


Figure 3. ISO/DIS 15592-3 and ISO 4387 reproducibility values for NFDPM

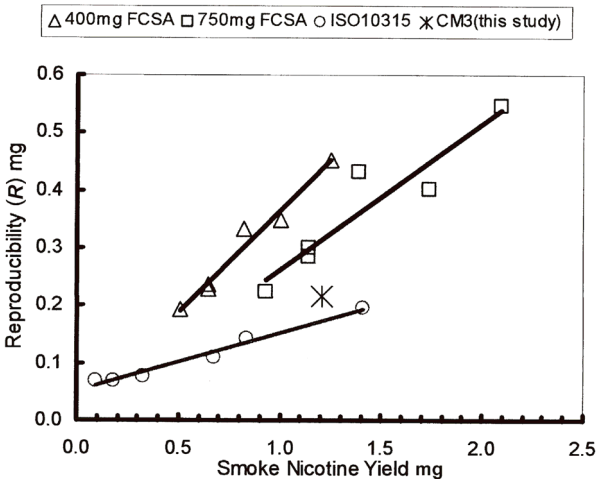


Figure 4. ISO/DIS 15592-3 and ISO 10315 reproducibility values for smoke nicotine

*Graphical presentation of the repeatability (*r*) and reproducibility (*R*) data*

It is obvious, from inspection of the values in Tables 4 and 5, that *r* and *R* are not fixed, but increase with the mean NFDPM and smoke nicotine yields. This is not surprising as the same trend can be seen for the *r* and *R* values provided for factory made cigarettes in ISO 4387 and ISO 10315 (see Figures 1 to 4).

When testing other brands of fine-cut tobacco, it is only possible to relate directly to the *r* and *R* values in the Tables 4 and 5 if the measured NFDPM or smoke nicotine yield is the same value as reported in these tables. In this situation it is often more convenient to derive values from regression equations linking them to the mean NFDPM and smoke nicotine yields.

It was observed that a better estimation of *r* and *R* for a particular smoke nicotine or NFDPM value might be achieved by considering separate regressions for each of the two tobacco masses. This is not surprising when the differences between the two product groups are considered i.e. the 750 mg and 400 mg articles were made with different making devices as well as being smoked in different holders due to the considerable difference in diameter. Plots with regression lines are shown in Figures 1 to 4. These should be regarded as indicative and the extrapolation and use of these lines beyond the mean yields observed in this study should be avoided.

It should be noted that with the exception of the NFDPM repeatability values for the 750 mg and 400 mg FCSAs in Figure 1, the gradients are not dissimilar for the two tobacco masses in Figures 2, 3 and 4. No reason for this exception has been found.

*Comparison with repeatability (*r*) and reproducibility (*R*) values reported in ISO 4387 and ISO 10315*

It is of interest to compare the *r* and *R* values from this study with those previously determined for factory made cigarettes in ISO 4387 and ISO 10315. These comparisons are made in Figures 1 to 4. It is evident from these figures that the *r* and

R values from this study are greater than those reported in both ISO 4387 and ISO10315 for the NFDPM and smoke nicotine yields, respectively. On average the *r* values are approximately 40% greater and the *R* values approximately 100% greater. This is not unexpected as the variability of the hand made articles tested in this study would be much greater than for the matched samples of factory made cigarettes tested and reported in these standards. The values of *r* and *R* would, therefore, be expected to be substantially inflated by this extra product variability. In addition, the within laboratory variability in article making would have been less than between laboratories as the protocol required each laboratory to use a single operator to manufacture the articles. This could explain the relative difference in the increase in the *r* and *R* values.

CONCLUSION

It is clear that the method detailed in ISO 15592-3 is satisfactory for the determination of smoke yields of FCSAs. The data as indicated by the values of *r* and *R* are of good quality within each laboratory and are consistent from laboratory to laboratory.

ACKNOWLEDGEMENT

The editing author wishes to record his thanks to all the members of the working group and all the people who took part in the preparations for and the conduct of the collaborative study. Without their help the work would not have been possible. Members of the planning sub-group were, Mr Peter Adams (United Kingdom), Mr Harry Dymond (United Kingdom), Prof. Wolf-Dieter Heller (Germany), Mr Colin Hill (United Kingdom), Mr Herman Huiskes (Netherlands), Mr Rolf Aage Olsen (Norway), Dr W. David Rowland (United Kingdom), Mr Hans Thomsen (Denmark), Mr Martin Whittaker (United Kingdom), Mr Edward Wilkes (United Kingdom).
The following laboratories took part in the work: Alois Pöschl GmbH & Co KG (Geisenhausen, Germany), Altadis (Fleury-les-Aubrais, France), Austria Tabak (Wien, Austria), British American Tobacco Manufacturing B.V. (Zevenaar, Netherlands), British American Tobacco (Southampton, United Kingdom), British American Tobacco (Sydney, Australia), Central Laboratory of the Tobacco Industry (Kraków, Poland), Chemische Landesuntersuchungsanstalt Sigmaringen (Sigmaringen, Germany), Gallaher Ltd (Ballymena, Northern Ireland), Heinr. Borgwaldt GmbH & Co (Hamburg, Germany), Heintz Van Landewyck S.a.r.l. (Luxembourg, Luxembourg), H.F. & Ph.F. Reemtsma (Hamburg, Germany), House of Prince (Soeborg, Denmark), Imperial Tobacco Ltd (Nottingham, United Kingdom), Imperial Tobacco RYO BV (Joure, Netherlands), Inspector for Health Protection (Hertogenbosch, Netherlands), J. L. Tiedemanns Tobaksfabrik AS (Oslo, Norway), LGC (Teddington) Ltd (Teddington, United Kingdom), LTR Industries (Allonnes, France), Royal Theodorus Niemeyer B.V. (Groningen, Netherlands).

REFERENCES

1. ISO 8243: Cigarettes – Sampling; International Organisation for Standardization, Geneva, Switzerland, 1991.
2. ISO 3402: Tobacco and tobacco products – Atmosphere for conditioning and testing; International Organisation for Standardization, Geneva, Switzerland, 1999.
3. ISO 3308: Routine analytical cigarette-smoking machine – Definitions and standard conditions; International Organisation for Standardization, Geneva., Switzerland, 2000.
4. ISO 4387: Cigarettes – Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine; International Organisation for Standardization, Geneva, Switzerland, 2000.
5. ISO 10362-1: Cigarettes – Determination of water in smoke condensates. Part 1: Gas-chromatographic method; International Organisation for Standardization, Geneva, Switzerland. 1999.
6. ISO 10362-2: Cigarettes – Determination of water in smoke condensates. Part 2: Karl Fischer method; International Organisation for Standardization, Geneva, Switzerland, 1994.
7. ISO 10315: Cigarettes – Determination of nicotine in smoke condensates. Gas-chromatographic method; International Organisation for Standardization, Geneva, Switzerland, 2000.
8. CORESTA Task Force on roll-your-own (fine-cut) tobacco: Roll-your-own report; CORESTA Bulletin 1999-2, pp. 9–12.
9. Darrall, K.G. and J. A. Figgins: Roll your own smoke yields: theoretical and practical aspects; Tob. Control 7 (1998) 168–175.
10. Kaiserman, M.J. and W.S. Rickert: Hand made cigarettes: It's the tube that counts; Am J. Public Health 82 (1992) 107–109.
11. Dymond, H.F: Making habit of roll-your-own smokers in The Netherlands and tar and nicotine yields from the resultant products; Tob. Sci. 40 (1996) 87–91.
12. Dymond, H.F: Physical dimensions and tar and nicotine yields of fine-cut smoking articles rolled by German consumers; Beitr. Tabakforsch. Int. 18 (1999) 165–174.
13. ISO 15592-1: Fine-cut tobacco and smoking articles made from it – Methods of sampling, conditioning and analysis. Part 1: Sampling; International Organisation for Standardization, Geneva, Switzerland. 2001.
14. ISO 15592-2: Fine-cut tobacco and smoking articles made from it – Methods of sampling, conditioning and analysis. Part 2: Atmosphere for conditioning and testing; International Organisation for Standardization, Geneva, Switzerland, 2001.
15. ISO 15592-3: Fine-cut tobacco and smoking articles made from it – Methods of sampling, conditioning and analysis. Part 3: 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; International Organisation for Standardization, Geneva, Switzerland, 2003.

16. ISO 16055: Tobacco and tobacco products – Monitor test piece. Requirements and application; International Organisation for Standardization, Geneva., Switzerland, 2003.
17. ISO 5725-1: Accuracy (trueness and precision) of measurement methods and results. Part 1: General principles and definitions; International Organisation for Standardization, Geneva, Switzerland, 1994.
18. ISO 5725-2: Accuracy (trueness and precision) of measurement methods and results. Part 2: Basic methods for the determination of repeatability and reproducibility of a standard measurement method; International Organisation for Standardization, Geneva, Switzerland, 1994.

Address for correspondence:

Peter I. Adams
Secretariat ISO/TC 126
Burggrafenstr. 6
10787 Berlin
Germany

Annex A. Protocol for the Collaborative Study of ISO/DIS 15592-3

INTRODUCTION

The objective of the study is to determine the between-laboratory variability and within-laboratory variability of the method described in ISO/CD 15592-3 in order to estimate the reproducibility *R* and the repeatability *r* of the method. It should be noted that ISO/CD 15592-3 specifies the making and testing of four fine-cut smoking article (FCSA) configurations (i.e. two wrapper types and two tobacco masses). Current testing experience indicates that the main influence on nicotine -free dry particulate matter (NFDPM) yield is the configuration (wrappers and masses of tobacco) rather than the type of tobacco blend used to make the FCSAs. Therefore, values for repeatability (*r*) and reproducibility (*R*) will be determined for each of the four specified configurations and these values will be entered into section 11 of ISO/CD 15592-3 (or ISO/DIS 15592-3 depending on the progress of the proposed standard).

Note: The values of reproducibility and repeatability obtained from this study will strictly be applicable only to those types of fine-cut tobacco used in this study. It was decided that three would be selected.

WRAPPER TYPES

The following wrapper types have been used:

1) Paper Type A. Wrappers for fine-cut tobacco specified as type A according to ISO/CD 15592-3.
2) Paper Type B. Wrappers for fine-cut tobacco specified as type B according to ISO/CD 15592-3. Annex A.

TOBACCO TYPES

The majority of the blend-styles on the market are composed of three curing types “fire-cured”, “flue-cured” and “air-cured”. Three tobacco blends were selected for this study. These blends contain one or a mixture of these curing types and are sold on the market. The selected blends are:

1) A finely cut fire-cured tobacco blend used for rolling and mainly sold in the Netherlands;
2) A slightly more coarsely cut blend of roughly equal proportions of fire- and flue-cured tobaccos, mainly sold in Europe;
3) A blend of flue-cured tobacco with a small proportion of air-cured tobacco somewhat more coarsely cut than either blend 1) or 2) sold mainly in Scandinavian countries.

The production companies will sample the mid-portion of one batch from their normal production and check that the sample is in accord with their specification for the blend.

Per blend type, the tobacco will be packed in blank pouches (50 g) and individually sealed. The fire-cured blend will be packed in Red pouches, the fire-cured/flue-cured blend will be packed in White pouches and the flue-cured/air-cured blend in Yellow pouches.

Per blend type, the pouches will be randomised and 30 pouches will be made available to each participating laboratory. Twenty pouches are to be used for the study and the extra 10 pouches can be used to practise making the specified articles. This means that each participating laboratory will receive 30 pouches of each of the three blends i.e. 90 pouches in all.

The production companies will make at least 30 laboratory samples. This is more than the number of laboratories participating, but the stock will be retained for use if a laboratory does not receive the first shipment or if additional laboratories wish to be involved.

Each production company will send their laboratory samples by courier to the contact person and postal address of the participating laboratories.

FINE-CUT SMOKING ARTICLES

The tubes and making devices for FCSAs are available from EFKA-Werke Fritz Kiehn GmbH, Germany; the sleeve holder components are available from Filtrona Instruments and Automation Ltd., United Kingdom, and Heinr. Borgwaldt GmbH, Germany.

The FCSAs to be used in this study will be made by each participating laboratory as described in the standard method using the devices manufactured by EFKA-Werke as detailed below:

- Weigh ten of the 7.2 mm paper tubes or 10 of the 5.2 mm paper tubes (as supplied) and record the mean weight. The mean weight of the ten tubes will be required in achieving the overall mean weights of the FCSAs.
- On receipt of the tobacco, keep it in the sealed pouches in an appropriate room, preferably at 22 ˚C and 75% RH.
- Open a sealed 50 g pouch, divide the tobacco into small portions (about 10 g) and place it into airtight containers. Place the airtight containers in store (if possible at 22 ˚C and 75% RH. See ISO 15592-2) until required. Do not open more than one pouch at a time.
- Using one airtight container at a time, weigh sufficient tobacco to end up with 750 ± 10 mg of the tobacco blend in the 7.2 mm diameter FCSAs or 400 ± 10 mg tobacco in the 5.2 mm diameter FCSAs. The overall weight of the FCSAs will be the weight of tobacco plus the mean weight of the paper tube.

Note: The above paragraph is written in this way as different laboratories have different methods for making the FCSAs. This study relies on the fact that the mean weight of the 20 articles used for smoking are within ± 10 mg and that individual articles are within ± 20 mg. Both these weights are gross weights i.e. specified tobacco weight plus the mean weight of the appropriate paper tube.

- Separate the tobacco to avoid lumps as each amount is weighed.
- Keep the weighed tobacco in good condition until required for making.
- Attach a pre-made tube onto the making device and mark the end attached.
- Carefully separate the tobacco from one weighing and spread it evenly onto the making device.
- Using a spatula, carefully introduce the tobacco into the receiving chamber of the making device.
- Partially close the chamber using the lever of the device.
- Reopen the chamber and scrape any remaining strands of tobacco into the chamber.
- Gently close the container completely again using the spatula to ensure that the tobacco is evenly spread and completely in the chamber.
- Using the thumb to hold the tube in place, gently and smoothly rotate the lever to push the tobacco into the tube.
- Open the chamber and ensure that no more than a few short strands of tobacco remain.
- Remove the FCSA from the making device and inspect it for visual defects, e.g. bad shape, uneven filling, etc. Reject poorly made articles.
- Push in any tobacco strands hanging from the ends of the FCSA. Do not cut them off.
- Weigh the individual fine-cut smoking articles and reject any that are outside the limit ± 20 mg.
- Mark the insertion depth and butt mark as specified in ISO CD 15592-3 clause 9.2.3
- Repeat the process to make sufficient FCSAs to produce 110 satisfactory ones for each FCSA configuration.
- Weigh 22 FCSAs and ensure that the mean weight is within ± 10 mg of the specified weight.

- Place the 22 finished articles carefully into a tin or plastic box capable of holding 22 FCSAs.
- Number the tins/boxes sequentially, starting at one, seal the tin and keep it in store if possible at 22 ˚C and 75% RH until required for conditioning prior to smoking.
- Prior to smoking, open the tins of 22 of a batch to be used such that the articles can be conditioned for at least 72 h but not more than 10 days at 22 ˚C and 75% RH as specified in ISO CD 15592-3 clause 9.4.
- Prior to smoking, weigh and record the mass of the 22 FCSAs.
- There will be no subsequent weight or pressure drop selection procedures.

Twelve FCSA configurations will be used in this study, each of the three types of fine-cut tobacco being made into the FCSA configurations according to the matrix shown in the standard method.

SMOKING MACHINES

Two types of smoking machines will be used in this study. 1) 20-channel linear smoking machines smoking 5 articles through each channel on to a 44 mm filter pad. 2) 20-port rotary smoking machines smoking 20 articles on to a 92 mm filter pad.
Note: Since the evaluation of the data from this study is to be based upon the between-laboratory and within-laboratory variability, the data from 4 channels of the 20-channel linear smoking machines must be pooled to provide means from the smoking of 20 FCSAs so as to make the data from the two types of smoking machine comparable. The details of the mode of pooling will be given in the report of the statistical analyses of the data.

VARIABLES

Target variables

The variables to be analysed in order to estimate the between-laboratory and within-laboratory variability (from which *R* and *r* will be calculated) are NFDPM and nicotine. Other analytical measurements will be puff number, total particulate matter (TPM), and water.

Variables to be reported

The variables to be reported fall into two categories; those ancillary to the tests, and those which will form the basis of the statistical evaluation of the study.
The ancillary variables are type of smoking machine used, laboratory temperature during smoking, relative humidity (RH%) in the laboratory during smoking, ambient air flow, weight of 22 FCSAs.
Note that ISO 15592-2 specifies that the Conditioning Atmosphere shall be 22 (± 2) ˚C, 75 (± 3) %RH; the Test Atmosphere shall be 22 (± 2) ˚C, and 60 (±5) %RH.
The variables to be reported for statistical analysis are mean puff number per FCSA, mean TPM per FCSA, mean nicotine yield per FCSA, mean water yield per FCSA, mean NFDPM yield per FCSA.

Dimensions and rounding of test results

Ancillary variables: laboratory temperature (degrees Celsius, ##); laboratory humidity (percent RH, ##); weight of 22 FCSAs (g, ##.##).
Analytical variables: puff number (number, ##.#), TPM (mg per FCSA, ##.##); nicotine (mg per FCSA, #.###); water (mg per FCSA, ##.##); NFDPM (mg per FCSA, ##.##).

Note that the rounding of the data to the formats specified above will take place after any calculations that may be involved. All calculations will use the laboratory data as recorded using the maximum number of digits available.

Internal documentation of tests

Each laboratory will document the method used following (where appropriate) the recommendations of ISO 15592-3, and ISO 4387 for the Monitor CM3. It is requested that each laboratory retains a copy of the internal documentation of this study for at least six months after the completion of the study in order to be able to respond to any queries that might arise from the analysis of the data. To facilitate the exchange of data, all laboratories will be asked to send their results to the co-ordinator for collation prior to the distribution of the data. The validated data from this study will be made available to all the laboratories participating in the study.

DESIGN OF THE STUDY

Numbers of fine-cut smoking articles

The study will use the 12 fine-cut smoking article configurations specified above. The study will also use the CORESTA monitor test piece CM3 as a monitor. Each participating laboratory will be required to smoke 300 monitor test pieces through the standard labyrinth seal holder. Each participating laboratory will be required to smoke 100 pieces of the 12 FCSA configurations through a sleeve holder as specified in ISO 15592-3.

Smoking plans

The smoking plans show 12 FCSA configurations being smoked. The configurations are coded as R1, R2, R3, R4, W1, W2, W3, W4, Y1, Y2, Y3, Y4 and one monitor test piece coded as M (Tables A1, A2).

FCSA codes

R1: 5.2 mm diameter, Paper A, 400 mg fire-cured blend
R2: 5.2 mm diameter, Paper B, 400 mg fire-cured blend
R3: 7.2 mm diameter, Paper A, 750 mg fire-cured blend
R4: 7.2 mm diameter, Paper B, 750 mg fire-cured blend
W1: 5.2 mm diameter, Paper A, 400 mg fire-cured/flue-cured blend
W2: 5.2 mm diameter, Paper B, 400 mg fire-cured/flue-cured blend
W3: 7.2 mm diameter, Paper A, 750 mg fire-cured/flue-cured blend
W4: 7.2 mm diameter, Paper B, 750 mg fire-cured/flue-cured blend
Y1: 5.2 mm diameter, Paper A, 400 mg flue-cured/air-cured blend
Y2: 5.2 mm diameter, Paper B, 400 mg flue-cured/air-cured blend
Y3: 7.2 mm diameter, Paper A, 750 mg flue-cured/air-cured blend
Y4: 7.2 mm diameter, Paper B, 750 mg flue-cured/air-cured blend
M = CORESTA CM3 monitor

For each FCSA configuration, 5 runs will be required when a rotary smoking machine is used, and 20 channels of smoking will be required when a linear smoking machine is used.
For this study, one test result is defined as the mean yield obtained from smoking 20 FCSAs or 20 monitor test pieces

THE INTERPRETATION OF *r* AND *R*

Repeatability (*r*) is a measure of the ability of a single laboratory to repeat the data from a given analysis. Strictly, it says that, under the best possible conditions, a laboratory can expect to get a given value within the limit of the repeatability 95 times in every 100 determinations.

Table A1. Smoking plan for 20-channel machines

	Channel number																			
Run	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
1	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M
2	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M
3	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M
4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4
5	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4
6	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4
7	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3
8	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3
9	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3
10	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2
11	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2
12	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2
13	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1	R2	R3	R4	M	R1
14	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1	Y2	Y3	Y4	M	Y1
15	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1	W2	W3	W4	M	W1

Table A2. Smoking plan for rotary machines

Run	FCSA	Run	FCSA	Run	FCSA	Run	FCSA
1	R1	21	R2	41	Y4	61	Y2
2	R2	22	R3	42	Y2	62	Y4
3	R3	23	R4	43	Y1	63	Y3
4	R4	24	R1	44	Y3	64	Y1
5	M	25	M	45	M	65	M
6	W1	26	Y2	46	W3	66	R2
7	W2	27	Y3	47	W4	67	R4
8	W3	28	Y4	48	W2	68	R3
9	W4	29	Y1	49	W1	69	R1
10	M	30	M	50	M	70	M
11	Y1	31	W4	51	Y3	71	W2
12	Y2	32	W2	52	Y4	72	W4
13	Y3	33	W1	53	Y2	73	W3
14	Y4	34	W3	54	Y1	74	W1
15	M	35	M	55	M	75	M
16	W2	36	R4	56	R3		
17	W3	37	R2	57	R4		
18	W4	38	R1	58	R2		
19	W1	39	R3	59	R1		
20	M	40	M	60	M		

Reproducibility (*R*) is a measure of the closeness of agreement of two laboratories to get the same result. Strictly it says that two laboratories, operating the method under the best possible conditions, can be expected to get results within the limit of reproducibility 95 times in every 100.

However, there are a number of important factors to bear in mind when interpreting these data. The data have been obtained under conditions that are carefully chosen to test the analytical method and they do not take into account that in practice: 1) More than one operator may be involved in the analyses; 2) More than one set of apparatus may be used. These two factors will affect the variability within a single laboratory.

The data for *r* and *R* have been obtained on a limited number of samples, different values may be obtained for different samples. This could affect both within and between laboratory variability. The data for *r* and *R* were obtained from a single batch of product and do not take into account the variability of the product. This could also affect both within and between laboratory variability. Thus, values of *r* and *R* are an indication and should be used as such. They are definitive only at the time they were determined, under the conditions of the test prevailing at the time and for the samples tested, see ISO 5725-1:1994.

GENERAL REMARKS

Each participating laboratory will be asked to use only one smoking machine. It is hoped that at least 16 laboratories will be recruited to take part in this study. It would be advantageous if equal numbers of participating laboratories used the 20-channel linear smoking machine and the rotary smoking machine. If possible, each participating laboratory will use only one operator when operating the smoking machine. If possible, each participating laboratory will use only two operators for the making of the FCSAs, and both these operators should make equal proportions of all products.

The sealed glass fibre filter holder assembly, without the sleeve holder or the labyrinth seal holder, will be weighed both before and after smoking (see ISO/CD 15592-3 Section 9.6.2). The filter holder will be dry-wiped to ensure that any “tar” deposited in the holder is included in the nicotine/water extraction (see ISO/CD 15592-3 Section 9.9.1).

A new slotted washer will be used for each smoke run when using the labyrinth seal holder and care must be taken to use the correct size (see ISO 3308 Section 4.8).

Each laboratory will purchase the CM3 monitor from either Filtrona or Borgwaldt. Each laboratory will purchase the FCSA making device from EFKA. All laboratories will purchase the sleeve holder specified in ISO/CD 15592-3 from Filtrona Instruments & Automation Ltd. All laboratories will purchase the adapter for rotary machines from Heinr. Borgwaldt GmbH.

Annex B. Making fine-cut smoking articles (FCSA) using the FCSA making devices

INSTRUMENTATION – MATERIALS

Analytical balance (accuracy ± 1 mg); FCSA making device, 5.2 or 7.2 mm; spatula (5 mm synthetic material, 30 × 60 mm); tobacco, conditioned for a minimum of 3 days at 22 ˚C and 7% RH; tubes type A and B each with a diameter of 5.2 and 7.2 mm; disk for weighing the tobacco, brush for cleaning the making devices.

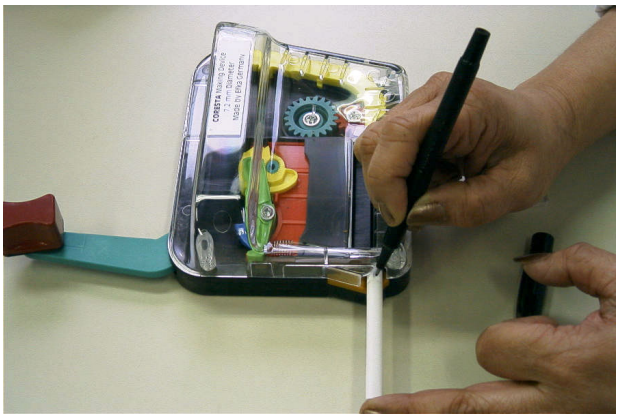
MAKING INSTRUCTIONS

Conditioning

Condition all the tobacco for between 3 and 10 days at 22 ˚C and 75% RH. After conditioning and during making keep the tobacco in as good a condition as possible by storing the tobacco in a closed container or sealed bag.

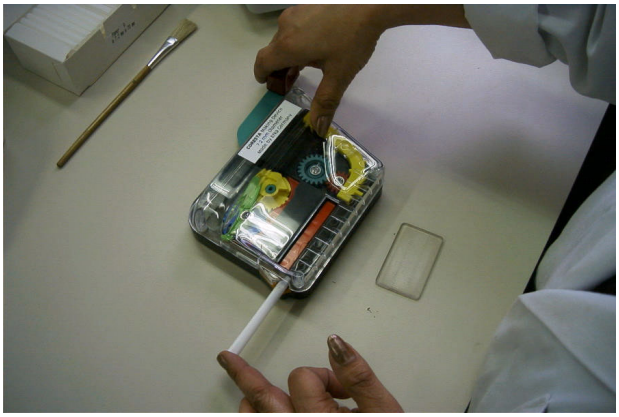
Weighing

Weigh sufficient tobacco to make a smoking article. This will be approx. 770 mg for the 7.2 mm diameter and 420 mg for the 5.2 mm smoking articles. When weighing, separate the tobacco to avoid lumps. If the weighed tobacco is not to be used immediately, store it in a sealed container or bag until it is needed for inserting into the pre-made paper tube.
Attach a pre-made paper tube onto the making device and mark the end attached with a felt tip pen.

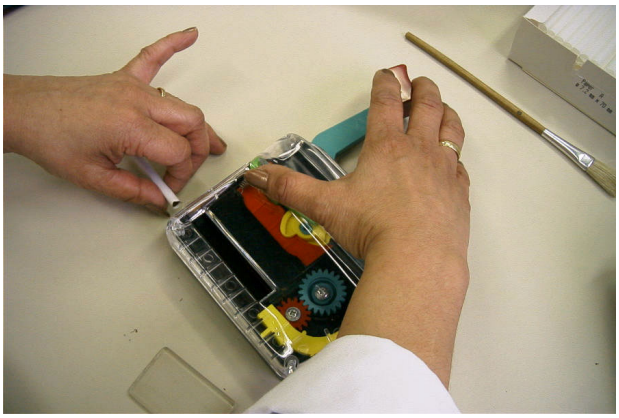


Carefully separate the tobacco, previously weighed, to make a single smoking article and spread evenly onto the making device. Using a spatula, introduce the tobacco into the receiving chamber of the making device.

Partially close the chamber using the lever of the device, then re-open and scrape any remaining strands of tobacco into the chamber.
Gently close the chamber fully to ensure that all the tobacco is inserted into the chamber.



Whilst using a finger to hold the paper tube in place, gently rotate the lever to push the tobacco into the tube. *Note*: This is a delicate procedure and benefits by having a means of supporting the tube without crushing the paper; the device must be held firmly during this process.
Open the chamber and ensure no more than a few short strands of tobacco remain.



Use a pipe cleaner or something similar to gently remove the smoking article from the making device and inspect visually. Reject poorly made articles (e. g. bad shape, uneven filling).
Any tobacco strands hanging from the ends should be pushed into the FCSA, not cut off.
Weigh each smoking article and reject any outside the limit 750 ± 20 mg or 400 ± 20 mg.
The weight of the paper must also be taken into account, see Table B1 for the actual acceptable weights.
After check weighing each article, store those within the acceptable weight range in a tub, or tin, with an airtight lid.
Repeat the process to make 120 satisfactory smoking articles of each type.
Weigh 22 articles together in the tub and ensure the mean tobacco weight is 750 ± 10 mg for the 7.2 mm articles and 400 ± 10 mg for the 5.2 articles.
The weight of the paper must also be taken into account, see Table B1 for the actual acceptable weights.

Table B1. FCSA making devices and accepted weights

Paper type	Diameter (mm)	Wrapper weight (mg) [a]	Acceptable weight range for 1 FCSA (g) [b]	Acceptable weight range for 22 FCSAs (g) [c,d]
A	5.2	35	0.415 – 0.455	9.350 – 9.790
B	5.2	25	0.405 – 0.445	9.130 – 9.570
A	7.2	46	0.776 – 0.816	17.292 – 17.732
B	7.2	32	0.762 – 0.802	16.984 – 17.424

[a] The wrapper weight is calculated from the specification of the paper used to form the wrapper.
[b] The acceptable weight range for 1 FCSA was calculated using the specified tobacco weight, the calculated wrapper weight and a tolerance of ± 20 mg per article.
[c] The acceptable weight range for 22 FCSAs was calculated using the specified tobacco weight, the calculated wrapper weight and a tolerance of ± 10 mg per article.
[d] 22 Articles allow for replacements.

Annex C. Laboratory mean values after outliers removal: Tables C1 to C5, Figures C1 to C15 (Figures C1–C5 fire-cured blend (Red), Figures C6–C10 fire-cured/flue-cured blend (White), Figures C11–C15 flue-cured/air-cured blend (Yellow).

Table C1. Laboratory mean values of TPM yield (mg/FCSA)

		Red blend				White blend				Yellow blend			
Laboratory	Monitor (CM3)	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper
1	18.68	12.58	16.80	19.98	26.97	12.44	16.13	19.65	24.29	11.38	13.89	16.82	22.23
2	18.28	13.14	15.85	21.72	26.82	10.93	14.15	19.23	23.12	10.66	13.95	18.91	23.36
3	18.92	13.78	19.24	21.89	29.96	12.21	16.03	20.00	26.97	10.91	14.98	18.89	25.54
5	19.04	15.37	20.34	20.68	26.68	11.68	16.44	17.98	23.39	11.49	14.93	17.67	22.47
6	18.70	12.19	16.67	20.07	25.28	10.07	13.62	17.60	21.54	8.98	12.25	17.19	21.42
7	19.00	13.98	18.14	22.21	28.03	11.58	15.27	19.78	24.48	11.53	16.33	19.69	25.18
8	18.90	12.94	17.80	21.46	28.04	10.69	15.74	18.31	24.29	10.03	14.13	17.79	22.86
9	19.89	13.25	18.65	21.79	29.21	11.61	15.16	19.53	25.78	10.56	14.15	18.44	24.14
10	18.04	12.55	16.08	20.95	25.41	10.41	13.74	17.56	21.85	10.24	13.55	17.45	22.13
11	18.34	11.86	14.42	21.44	24.69	9.66	12.21	17.57	21.71	9.53	11.55	17.38	20.75
12	17.65	13.90	17.52	22.36	27.67	11.72	14.99	19.81	25.31	11.48	14.84	19.37	25.23
13	19.44	12.85	15.93	22.73	26.67	9.28	10.25	18.63	23.72	8.81	12.68	18.71	23.19
14	18.78	13.73	17.89	21.70	28.71	11.06	15.62	18.64	25.18	10.96	14.53	18.41	24.35
15	19.15	14.59	19.72	23.24	29.03	11.56	16.37	20.85	26.65	11.90	16.12	20.55	26.89
16	18.58	13.17	16.74	22.11	27.41	11.30	14.69	18.54	24.47	10.80	14.32	19.04	23.90
17	18.65	12.33	14.51	19.27	27.21	10.49	11.65	15.69	OV [a]	9.57	12.24	14.57	18.99
18	19.69	13.79	16.75	21.77	27.27	11.42	15.00	19.51	25.49	11.12	13.60	18.44	23.43
19	18.83	15.18	20.06	22.85	28.18	12.69	17.19	19.55	24.98	12.20	16.36	19.40	24.08
20	18.06	9.94	13.21	19.65	27.24	8.18	12.41	17.73	24.04	8.51	11.81	17.21	22.63

[a] OV = outlier value

Table C2. Laboratory mean values of smoke nicotine yield (mg/FCSA)

		Red blend				White blend				Yellow blend			
Laboratory	Monitor (CM3)	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper
1	1.34	1.17	1.45	1.94	2.39	0.84	1.00	1.32	1.53	0.64	0.81	1.03	1.32
2	1.17	0.94	1.11	1.72	2.02	0.58	0.74	1.11	1.25	0.49	0.62	0.92	1.09
3	1.25	1.05	1.44	1.84	2.38	0.74	0.93	1.25	1.64	0.56	0.73	1.01	1.31
5	1.18	1.09	1.36	1.63	2.02	0.69	0.92	1.14	1.43	0.56	0.70	0.90	1.14
6	1.22	0.88	1.17	1.58	1.83	0.51	0.67	0.95	1.14	0.37	0.53	0.82	1.00
7	1.28	1.34	1.57	2.05	2.37	0.68	0.81	1.12	1.32	OV	OV	OV	OV
8	1.27	0.93	1.25	1.69	2.16	0.66	0.92	1.19	1.54	0.50	0.68	0.95	1.17
9	1.23	0.95	1.33	1.71	2.22	0.62	0.79	1.08	1.30	0.49	0.64	0.91	1.15
10	1.15	0.86	1.07	1.55	1.76	0.59	0.77	1.04	1.21	0.45	0.57	0.82	0.99
11	1.22	0.88	1.04	1.66	1.82	0.57	0.71	1.06	1.27	0.45	0.54	0.86	0.97
12	1.15	1.04	1.25	1.78	2.10	0.74	0.89	1.29	1.52	0.55	0.68	0.99	1.19
13	1.23	0.88	1.05	1.63	1.94	0.54	0.61	1.08	1.34	0.42	0.56	0.89	1.09
14	1.24	0.98	1.22	1.73	2.17	0.66	0.89	1.17	1.52	0.52	0.65	0.91	1.16
15	1.23	1.11	1.45	1.91	2.16	0.64	0.88	1.18	1.45	0.59	0.78	1.08	1.29
16	1.13	0.97	1.15	1.72	2.01	0.66	0.81	1.16	1.39	0.56	0.67	0.99	1.15
17	1.27	1.01	1.28	1.66	2.08	0.63	0.67	1.02	1.14	0.51	0.65	0.84	1.05
18	1.24	1.04	1.22	1.78	2.10	0.65	0.84	1.16	1.47	0.54	0.63	0.96	1.17
19	1.25	1.01	1.36	1.70	2.14	0.72	0.98	1.23	1.56	0.53	0.72	0.94	1.18
20	1.01	0.85	1.07	1.76	2.14	0.53	0.73	1.09	1.38	0.48	0.62	0.91	1.10

Table C3. Laboratory mean values of water yield (mg/FCSA)

		Red blend				White blend				Yellow blend			
Laboratory	Monitor (CM3)	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper
1	2.57	2.16	3.28	3.09	4.45	2.35	3.03	3.34	3.78	2.56	3.11	2.91	4.38
2	2.46	2.97	3.67	4.33	5.96	2.21	3.12	3.41	4.33	2.29	3.33	3.54	4.98
3	2.11	2.45	3.74	3.13	4.78	2.02	2.90	2.56	3.76	2.01	3.21	2.85	4.36
5	2.84	3.67	4.97	3.91	5.38	2.62	4.01	2.96	4.13	2.93	3.95	3.69	4.88
6	2.79	2.72	3.79	4.22	5.06	2.41	2.71	3.23	4.19	2.34	3.37	3.65	4.73
7	2.83	2.71	3.79	4.08	5.44	2.50	3.55	3.81	5.22	2.07	3.35	3.35	4.52
8	2.20	2.76	4.20	4.03	5.54	1.80	3.24	2.70	3.70	2.03	3.30	3.05	4.29
9	2.48	2.64	4.07	3.62	5.29	2.19	2.94	2.79	3.72	2.17	3.19	3.09	4.39
10	2.75	2.96	4.02	4.60	5.74	2.10	2.92	3.08	4.08	2.54	3.70	3.83	5.29
11	2.57	2.55	3.28	4.38	5.21	1.91	2.50	3.20	4.20	2.13	2.61	3.57	4.57
12	2.40	2.93	3.94	4.54	5.68	2.02	2.88	3.15	4.21	2.50	3.57	3.95	5.18
13	2.44	2.85	4.13	4.91	5.78	1.98	1.82	3.20	4.37	1.30	2.95	3.59	4.82
14	2.59	3.55	4.80	4.51	5.99	2.63	3.98	3.30	4.63	2.91	4.07	3.62	5.30
15	2.08	3.19	4.49	4.22	5.53	2.31	3.78	3.48	4.33	2.67	4.15	3.77	4.99
16	1.99	1.97	2.61	3.05	4.50	OV	2.04	1.92	3.11	1.81	2.25	2.53	3.84
17	2.29	2.32	2.42	3.54	6.56	1.98	1.87	1.83	OV	2.04	1.78	1.63	3.85
18	3.20	3.12	3.75	4.19	5.49	2.32	3.26	3.59	4.78	2.62	3.35	3.56	4.97
19	2.14	3.53	5.06	4.78	5.89	2.53	3.87	3.29	4.20	2.85	4.43	4.00	5.12
20	3.19	1.58	2.33	3.90	6.22	OV	1.97	2.70	4.04	1.32	2.64	3.08	4.59

Table C4. Laboratory mean values of NFDPM yield (mg/FCSA)

		Red blend				White blend				Yellow blend			
Laboratory	Monitor (CM3)	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper
1	14.78	9.25	12.07	14.96	20.13	9.25	12.10	14.99	18.97	8.17	9.97	12.88	16.53
2	14.65	9.23	11.06	15.66	19.15	8.14	10.29	14.71	17.50	7.88	10.01	14.44	17.30
3	15.55	10.28	14.06	16.90	22.80	9.45	12.20	16.14	21.57	8.35	11.03	15.02	19.87
5	15.01	10.70	13.56	15.11	19.46	8.37	11.41	13.75	17.83	7.84	10.00	13.08	16.44
6	14.69	8.60	12.00	14.27	18.38	7.15	10.23	13.42	16.21	6.27	8.35	12.71	15.68
7	14.88	9.98	12.84	16.08	20.22	8.39	10.91	14.85	17.94	8.69	11.98	15.07	19.15
8	15.43	9.24	12.35	15.74	20.34	8.23	11.58	14.42	19.04	7.50	10.14	13.79	17.41
9	16.19	9.65	13.26	16.47	21.70	9.07	11.29	15.66	20.65	7.91	10.32	14.60	18.60
10	14.13	8.74	10.99	14.80	17.91	7.72	10.05	13.45	16.56	7.25	9.29	12.80	15.86
11	14.56	8.43	10.11	15.45	17.65	7.18	9.01	13.32	16.24	6.95	8.39	12.95	15.21
12	14.00	9.93	12.32	16.05	19.90	8.97	11.21	15.45	19.47	8.43	10.59	14.91	18.36
13	15.62	8.63	10.68	16.16	18.49	6.41	7.82	14.35	17.99	6.60	9.17	14.26	17.32
14	14.93	9.20	11.88	15.50	20.56	7.77	10.76	14.16	19.07	7.53	9.81	13.88	17.89
15	15.85	10.29	13.78	17.04	21.63	8.35	11.68	16.12	20.84	8.64	11.29	15.29	19.94
16	15.46	10.40	12.98	17.33	20.84	9.38	11.57	15.46	19.96	8.58	11.38	15.52	18.32
17	14.95	8.99	10.85	13.64	18.56	7.85	9.11	12.63	13.66	7.01	9.81	11.50	14.32
18	15.20	9.62	11.79	15.79	19.68	8.46	10.90	14.77	19.25	7.96	9.62	13.92	17.29
19	15.47	10.63	13.65	16.37	20.15	9.37	12.26	15.02	19.21	8.69	11.21	14.46	17.78
20	13.78	7.50	10.19	14.28	19.24	6.56	9.71	13.94	18.62	6.50	8.83	13.01	16.66

Table C5. Laboratory mean values of puff number (puffs/FCSA)

		Red blend				White blend				Yellow blend			
Laboratory	Monitor (CM3)	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper	400 mg + A wrapper	400 mg + B wrapper	750 mg + A wrapper	750 mg + B wrapper
1	8.89	5.14	5.72	6.04	7.06	5.22	6.26	6.56	7.56	5.78	6.92	6.44	8.92
2	8.99	4.60	5.28	5.96	7.02	4.82	5.68	6.48	7.36	5.06	6.36	6.86	8.46
3	9.09	4.63	5.72	6.06	7.02	5.02	6.12	6.13	7.43	5.36	6.51	6.90	8.56
5	9.20	5.22	5.94	6.14	7.22	5.31	6.26	7.02	8.20	5.76	6.95	7.18	9.27
6	8.97	4.73	5.83	6.12	6.78	4.83	5.84	6.44	7.35	4.92	6.36	6.62	8.00
7	8.80	5.06	6.04	6.12	7.30	5.78	7.38	7.08	8.88	5.30	6.52	6.64	7.86
8	9.13	4.86	5.79	6.29	7.50	5.25	6.31	6.65	8.33	5.67	7.04	7.54	9.42
9	9.00	4.93	5.84	6.02	7.24	5.14	6.05	6.62	7.92	5.35	6.58	7.16	8.71
10	8.60	4.64	5.34	5.94	6.92	4.70	5.62	6.22	7.48	5.16	6.60	6.94	8.64
11	8.46	4.96	5.58	6.14	7.16	5.14	6.16	6.54	7.98	5.48	6.60	7.24	8.88
12	8.86	4.96	5.58	6.02	7.08	5.08	5.96	6.48	8.02	5.46	6.66	7.26	8.84
13	9.23	OV	6.52	OV	8.54	6.06	7.77	7.68	9.22	6.72	7.59	8.12	9.72
14	8.94	4.74	5.51	5.97	7.17	4.92	6.19	6.56	7.83	5.67	6.56	6.97	8.70
15	8.86	5.10	5.95	6.28	7.18	5.15	6.51	7.11	8.33	5.97	7.06	7.59	8.89
16	8.79	4.56	5.12	5.90	6.74	4.78	5.60	6.26	7.38	5.24	6.32	6.94	8.48
17	9.48	OV	6.56	OV	8.68	6.18	7.14	OV	OV	6.31	7.32	8.56	OV
18	8.69	4.86	5.42	6.06	6.86	5.24	5.94	6.44	7.92	5.32	6.10	7.14	8.72
19	8.86	4.52	5.37	5.66	6.96	4.91	5.88	6.29	7.85	5.35	6.50	7.11	8.68
20	9.23	4.88	5.92	6.02	7.04	5.12	6.08	6.16	7.68	5.50	7.10	6.58	8.74

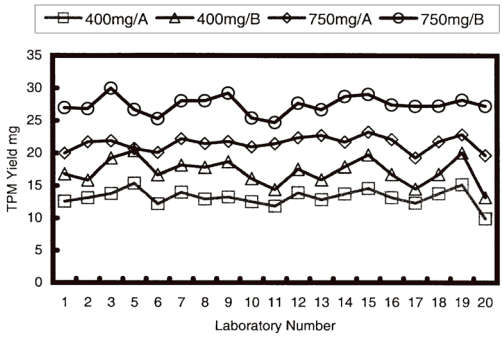


Figure C1. Red blend – TPM yield

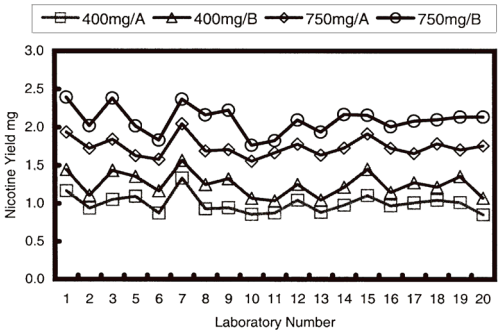


Figure C2. Red blend – NFDPM yield

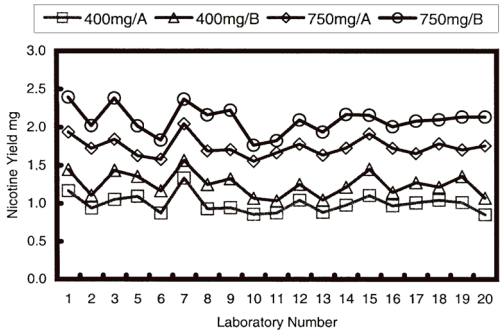


Figure C3. Red blend – nicotine yield

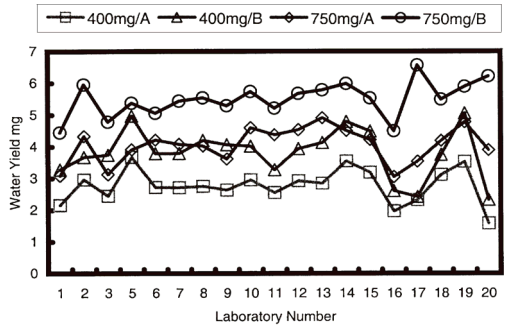


Figure C4. Red blend – water yield

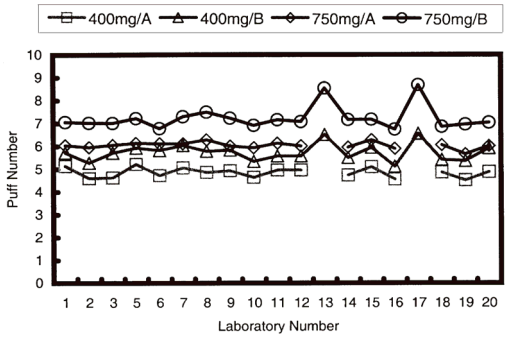


Figure C5. Red blend – puff number

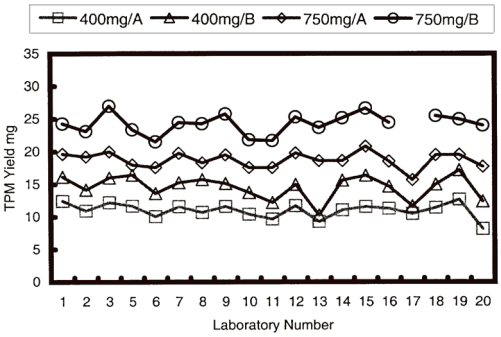


Figure C6. White blend – TPM yield

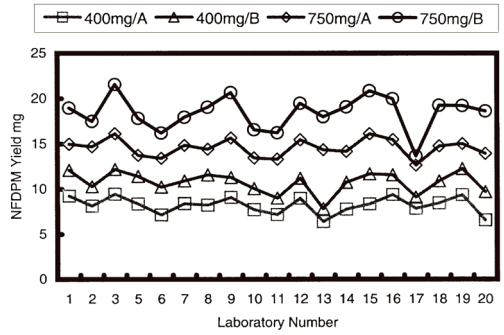


Figure C7. White blend – NFDPM yield

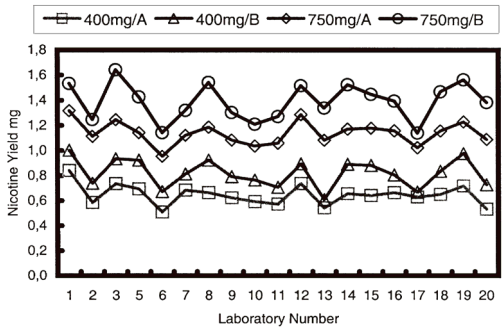


Figure C8. White blend – nicotine yield

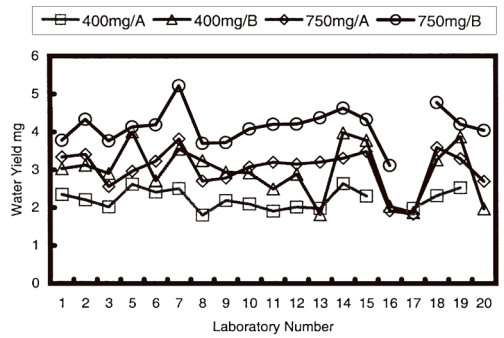


Figure C9. White blend – water yield

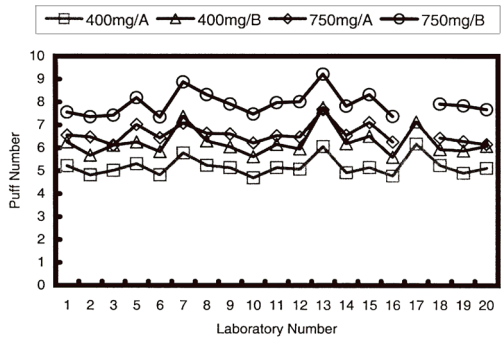


Figure C10. White blend – puff number

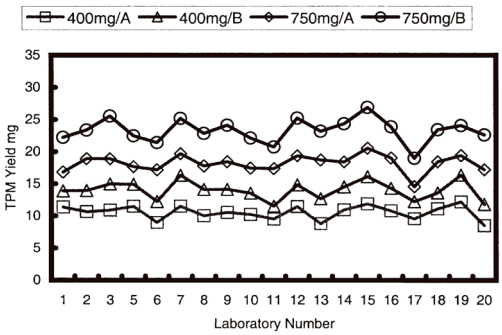


Figure C11. Yellow blend – TPM yield

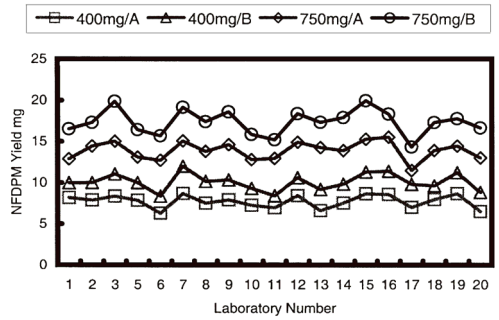


Figure C12. Yellow blend – NFDPM yield

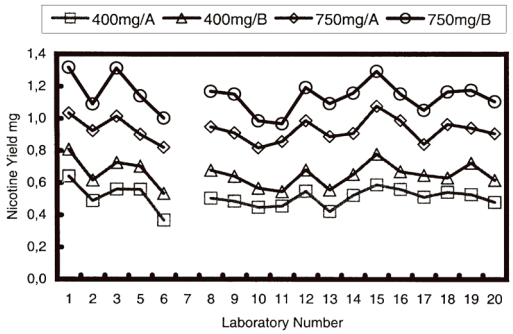


Figure C13. Yellow blend – nicotine yield

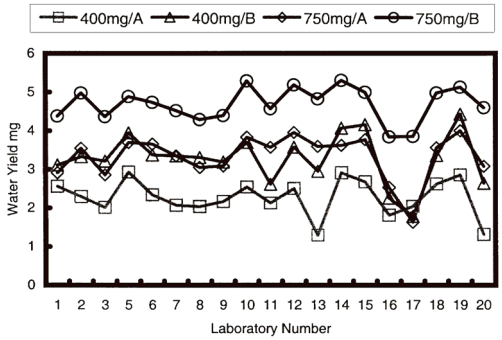


Figure C14. Yellow blend – water yield

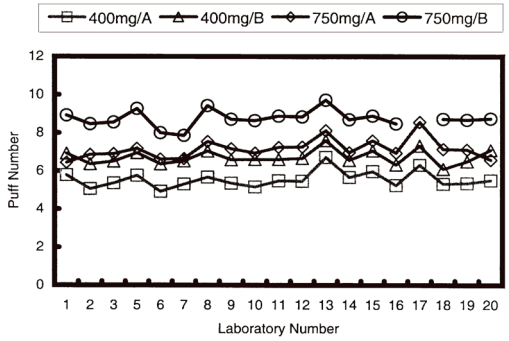


Figure C15. Yellow blend – puff number