REPORT OF THE CORESTA TASK FORCE ON ROLL-YOUR-OWN (FINE-CUT) TOBACCO (Published in the Special CORESTA Bulletin 1999-2)

CONTENTS

Executive Summary

This report provides details of the technical studies carried out by the Task Force established by CORESTA, to meet the following defined objectives:

- To document how RYO smokers in different countries make their cigarettes.
- To establish the effects of materials and cigarette design on particulate matter and nicotine yields of RYO cigarettes.
- To make recommendations to the Smoke Study Group on the relevance to the consumer of proposed standard procedures.
- To consider the establishment of a recommended method to give meaningful comparisons among fine-cut tobaccos or fine-cut smoking articles (FCSAs).

The above objectives are quoted from the original wording. The term fine-cut smoking article was adopted very early in the work of the Task Force as the term "cigarette" is used for a manufactured product.

The work carried out meets the first three of these objectives, and provides the basis for a standard method in line with the fourth. The following are the key outputs and conclusions of the work.

Consumer practices for making their smoking articles differ widely both between and within markets. There are wide variations in weight, diameter and shape as well as a range of wrappers. The choices made by the consumer are crucial in determining smoke yields.

Current and proposed national procedures for measuring NFDPM and nicotine yields from fine-cut smoking articles (FCSAs) are inadequate, and do not provide consumers with sufficient information about the effect of the choices available when making their fine-cut tobacco products.

Consumers should be advised of the effect of their choices on smoke yields and a 2x2 matrix (reflecting the effects of tobacco quantity and wrapper used) provides the simplest approach to do so. A wrapper classification system is defined and recommended.

Making devices to provide satisfactory articles from fine-cut tobaccos and wrapper materials have been developed for both of the diameters required, to accommodate the two different tobacco weights, and have been evaluated successfully. Articles produced using these devices, following a defined protocol, seem to be sufficiently consistent for a standard method. However the ends of the articles are neither as firm nor as cylindrical as those of manufactured cigarettes. This is considered to be a major cause of the high variability found in the results from smoking tests.

Smoking of laboratory-made articles results in considerable variation in smoke yields, even when the articles have been made under controlled conditions, and attention is required to reduce this in the smoking procedure adopted. Consumer-made articles are still more variable, and require additional care in smoking.

Practical difficulties were identified by the majority of the participating laboratories, and higher levels of variability were experienced when smoking FCSAs using the Labyrinth Seal holder specified in ISO 3308:1991. An alternative holder has been developed that reduces this variability, especially when used with an increased insertion depth to allow for the irregular and under-filled end of the article.

Even with the improvements established by the work set out in this report, variability is inevitably greater for data from laboratory-produced FCSAs than that from manufactured cigarettes.

This report does not attempt to define what level of variability is necessary to provide a satisfactory standard for FCSAs that could be used for regulatory purposes. CORESTA considers that this requires the input of regulatory laboratories, and is willing to make its documented experimental information available to ISO to allow the method to be completed under its auspices.

CORESTA and its member companies will offer full co-operation in finalising a standard, based on the technically sound principles arising from the practical studies detailed in this report. All of the data reported here are available from CORESTA for further analysis.

1. Introduction

Very little information existed on the use, consumer making habits or analysis of fine-cut tobacco before 1989. CORESTA therefore considered establishing a Task Force to study the issues associated with the determination of smoke particulate matter and nicotine arising from the use of fine-cut tobacco.

The Task Force began its work in November 1989, when 29 people representing a wide range of interests met to consider the issues associated with the determination of yields from fine-cut tobacco or smoking articles made from fine-cut tobacco. It was fully inaugurated in October 1990 when the objectives were defined as follows:

1. To document how RYO smokers in different countries make their cigarettes.

2. To establish the effects of materials and cigarette design on particulate matter and nicotine yields of RYO cigarettes.

3. To make recommendations to the Smoke Study Group on the relevance to the consumer of proposed standard procedures.

Initially, the first two objectives were studied simultaneously whilst the third was examined considering current knowledge from objective 2 and other external publications.

From the beginning it was clear that the term “cigarette” was not appropriate for articles made, in the laboratory or by consumers, by combining a wrapper with fine-cut tobacco, as this term is used specifically for machine-made products. It was decided to use the term “fine-cut smoking article”.

As a result of work performed, a further objective of the Task Force was added in 1991:

4. To consider the establishment of a recommended method to give meaningful comparisons among fine-cut tobaccos or fine-cut smoking articles.

As well as the work performed under the auspices of CORESTA, a considerable amount of work was undertaken in parallel by those companies involved in the manufacture and marketing of fine-cut tobacco. The data from these companies have been made available to the Task Force.

Previous data on fine-cut tobacco usage from the main markets, for weight and diameter of the smoking article, method of making, filter use and wrapper type had shown that consumer making behaviour varies considerably both among countries and with different consumers; even individual making behaviour is variable. This was reflected in the very wide range of weights of tobacco used to make a smoking article, a very wide range of diameters, different densities and a variety of shapes. In the main markets there are a wide range of papers available and in some markets non-specialist papers are used, such as book paper or packing paper. Some consumers use pre-made tubes as wrapper for the fine-cut smoking articles or include a loose filter.

Since condensate is produced from the finished article and not from the tobacco alone, it was considered that these making habits would substantially affect the yields from the finished product. Furthermore, it was anticipated that different wrapper types would affect the yield of NFDPM and nicotine.

The Task Force attendance averaged almost fifty from over thirty different organisations. The attendees represented manufacturers of fine-cut tobacco, cigarette manufacturers, paper manufacturers, paper converters, suppliers to the tobacco industry and manufacturers of testing equipment.

During the course of the work to establish a CORESTA Recommended Method for the determination of smoke yields, several other studies were performed that led to the development of CORESTA Recommended Method 42 (conditioning) and method 43 (sampling). These methods were submitted to ISO in 1997 as part of CORESTA's contribution to the ISO work item. At the time of writing, these have the status of "ISO Committee Draft". It is not the intention of this report to include full details of the work leading up to the development of these methods. However, section 8.1 provides the essential information that led to CORESTA Recommended Method 42.

The major problem associated with the development of a method was the poor end quality of fine-cut smoking articles and the high variability of the smoke yield measurements caused by the variable nature of the hand-made products. To overcome this, the Task Force had to set about a systematic appraisal of the factors contributing to the variability. To date, 30 Task Force meetings have been held (these are listed in Appendix 1). In addition, there have been approximately 140 meetings of various subgroups. These were established to:

- Determine the protocol of the major collaborative studies.
- Examine the effect of the wrapper (paper).
- Examine the design of the holder to be used in conjunction with the fine-cut smoking article.
- Design a suitable making device.
- Investigate the special problems associated with pre-made filter tube wrappers.
- Evaluate the statistical data from the major collaborative studies.

Several other *ad hoc* meetings were also held to discuss specific problems or issues.

The early studies were conducted to guide the Task Force in the correct direction for more detailed corroborative and collaborative work. As such the Task Force did not produce detailed reports for these early studies but full reports, agreed by the Task Force, were prepared for studies 11 onwards. A list of the co-operative studies is given in Appendix 2. Appendix 3 lists the other major studies. This report highlights the results from the early studies and the full, detailed reports for studies 11-16 are appended.

The initial studies were reported in September 1991 (1) and a further interim report was produced in May 1995 (2).

2. Definitions

For the purposes of this report, the following definitions apply:

Fine-cut tobacco (FCT)

> Tobacco produced to be used by consumers for making into their own smoking articles with a wrapper specially prepared for this purpose.

Wrapper (for fine-cut smoking articles)[1]

> Material specially prepared and supplied in a form suitable for enclosing fine-cut tobacco so as to produce a fine-cut smoking article. These wrappers are normally sold in booklet form, in the form of a roll, or as pre-made tubes with or without a filter.

Fine-cut smoking article (FCSA)

> An article, suitable for smoking, produced by combining fine-cut tobacco with a wrapper.

Dutch Blend

> This term is used in this report to identify a blend of fine-cut tobacco, representative of a blend of virginia and fire cured tobacco, typical of a blend used for the most popular brand of fine-cut tobacco on sale in Europe.

3. General Considerations

Many of the studies had inter-acting objectives. However, for clarity of reading, this report is written in the sequence of the objectives. The reporting sequence is not necessarily chronological but chosen to assist understanding.

From the initial work, done before the Task Force was created, it was clear that fine-cut tobacco is made into fine-cut smoking articles in two main ways:

1. Using a machine to insert the tobacco into a tube (tubing).
2. Hand-rolling including the use of a hand held making device.

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[1] **Note:** Conscious of the possibility that in the future, material other than paper designated for use with fine-cut tobacco may be used to make fine-cut smoking articles and need to be tested, the Task Force decided to use the term "wrapper" rather than paper. This term also encompasses the use of pre-made filter tubes.

Smoking articles made by hand-rolling fall into two broad categories differentiated by tobacco weight and associated diameter.

After some early studies it was decided that work should be done at two diameters. The diameters were chosen as 5.2 mm and 7.2 mm; 5.2 mm is closely representative of the thin articles, typically made in the United Kingdom, and is near the mid range of the diameters made by these consumers; 7.2 mm is typical of the diameter made in mainland Europe. Wrapper tubes are now available at these diameters for laboratory making of fine-cut smoking articles.

Because, at the beginning of the studies, it was easier to make fine-cut smoking articles in the laboratory at 7.2 mm diameter, and because it was believed that the effects at a smaller diameter would be similar, the majority of the work was done at this diameter. The fine-cut smoking articles made at 5.2 mm were examined in detail at the end of the studies. The studies with these smaller articles are reported in section 8.3.

At an early stage in the consideration of Objective 4, it became apparent that a single figure to describe the yields from fine-cut smoking articles would be insufficient and could be misunderstood by the consumer. It was considered that a method should be developed that would give an indication of the effect of wrapper type and making practice. It was proposed that the yields should be expressed in the form of a matrix of four values per constituent. This concept influenced many of the subsequent studies. For clarity of understanding the matrix concept is described first.

International standard methods require that there is an estimate of the within-laboratory variability and the between-laboratory variability. These estimates are normally expressed as r, repeatability and R, reproducibility. CORESTA has decided to follow this principle and recent CORESTA Recommended Methods include estimates of r and R.

Values of r and R could not be calculated for the data determined in any of the studies described in this report. The studies were designed to lead to the development of a recommended method. In accordance with the terms laid down by ISO 5725, r and R can only be determined when the study is performed in a very controlled way. In particular r and R should only be determined for the final method. However an indication of the within-laboratory and between-laboratory variability can be extremely useful in the development of a method. [Where within-laboratory and between-laboratory variability has been calculated the notation v and V have been used in place of r and R to emphasise the fact that the study was not set up to estimate r and R.]

4. The matrix concept

Having considered the effects of packing density and of the wrapper and comparing the magnitude of these effects to that of the tobacco itself, the Task Force decided that a single figure to describe the yields from fine-cut smoking articles would be insufficient and could be misunderstood by the consumer.

Following the studies described in later sections, the Task Force decided that the yields should be expressed in the form of a matrix of four values per constituent. The four values will be derived from smoking four types of fine-cut smoking articles, made with two models (tobacco weight and diameter) and two wrapper types. This would give the consumer guidance on the effect of making behaviour and of the other components of the finished article.

An example of a matrix is given below.

	Wrapper Type A	Wrapper Type B
Thin Roll O (400 mg tobacco)	NFDPM Yield from Article 1 Nicotine Yield from Article 1	NFDPM Yield from Article 2 Nicotine Yield from Article 2
Thick Roll O (750 mg tobacco)	NFDPM Yield from Article 3 Nicotine Yield from Article 3	NFDPM Yield from Article 4 Nicotine Yield from Article 4

Fine-cut tobacco is sometimes sold with a clear indication that it may be used with a specified special wrapper, pre-made filter tube wrapper, or with a loose filter. The Task Force proposed that in this instance, it might be appropriate to provide additional information in the expression of results. This additional information should relate to the range of weights of fine-cut tobacco that might be used with the wrapper.

This approach implies that all wrappers intended to be used with fine-cut tobacco will have to be classified into one of two groups.

5. Objective 1 - Variation in making habits by Country

5.1 Market Information

All delegates were asked to gather information from published data, in-house data or consumer research. Data were gathered for approximate fine-cut tobacco consumption, weight and diameter of the smoking articles, methods of making, filter use and paper type.

These data have been updated from time-to-time throughout the period of the study. The group regarded this objective as an ongoing requirement and continually updated the tables. The latest available data are summarised in Tables 1-4.

Table 1
Fine-cut tobacco sales by country 1997 data
(tonnes per year approximate)

AUSTRIA	190	ITALY	259
BELGIUM	9,234*	NETHERLANDS	13,790
CANADA	3,969	NORWAY	2,737
CZECH REPUBLIC	236	POLAND	25
DENMARK	1,057	SPAIN	718
FRANCE	5,270	SWEDEN	827
FINLAND	996	SWITZERLAND	95
GERMANY	14,123	UK	1,875*
HUNGARY	87	USA	2,375

*The figures for Belgium and the UK are known to be heavily distorted by cross-border trade. It is estimated that approximately 5,000 tonnes are bought in Belgium but consumed in the UK.

The table contains the latest data available to the task force.

Table 2
Consumer making method
(%)

	Hand-rolling	Machine-rolling	Tubing
BELGIUM/ LUXEMBOURG	51	15	34
CANADA	Exact figures not available. Mainly tubing		
DENMARK	8	0	92
FINLAND	0	90	10
FRANCE	Mainly by hand. Figures not available for machine		<1
GERMANY	70	10	20
EIRE	>95	<5	0
NETHERLANDS	82	3	15
NORWAY	88	1	11
SWEDEN	18	22	60
UK	95	5	0

The table contains the latest data available to the task force.

Table 3
Use of loose filters
(%)

FINLAND	70	SWEDEN	20
GERMANY	10	UK	5

The table contains the latest data available to the task force.

Table 4
Branded Wrappers for the Fine-Cut Tobacco Market
(%)
Data provided by the European Confederation of Paper Converting Industries

Porosity CU	Europe %	World-wide, incl. Europe %	Additive
0-9	13	45	NONE
10-29	40	30	NONE
30-50	14	7	NONE
50+/NON-PERFORATED	3	1	NONE
50+/PERFORATED	30	17	CITRATE

CU = CORESTA Units of Air Permeability
The table contains the latest data available to the task force.

5.2 Consumer Studies

When the Task Force was established, many of the participants had consumer data available. In addition to this historical data, several consumer studies were performed and made available to CORESTA.

Tobacco weights given in the following sections are determined at the relevant moisture content or as a result of conditioning to the quoted RH.

Studies were conducted in Germany in 1977,1988 and 1994 and in Netherlands in 1978, 1987 and 1994. In each of these studies consumers used their own brand of tobacco and wrapper. They were not supplied with free tobacco. The data for these studies are included in the summary table given in section 5.4. Further studies were also conducted in other countries and reported to the Task Force. These are summarised below.

Additional data were collected for smoking articles made from pre-made filter tubes. These data are discussed in a section 8.5.

5.2.1 The French Study

In 1992 SEITA commissioned an independent research company in France to conduct a consumer survey of the making parameters of fine-cut smoking articles. Two hundred smokers were asked to roll 10 FCSAs using their own tobacco and wrapper and then to answer a questionnaire. These FCSAs were examined by the research company at the time of interview and returned to the consumers. They were then asked to make a further 12 FCSAs that were sent to SEITA for physical analysis and smoking. The physical test results were given to a task force meeting.

At the time of analysis, SEITA noted that, for convenience, the FCSAs were conditioned to 13 % moisture content. For comparative purposes, the figures have been reported corrected to 18 % moisture content.

The physical characteristics were analysed by age of the consumers. The mean weight of the FCSAs was 722 mg. The wrapper weight is about 50 mg. The range was not quoted but was given as a standard deviation of each of the age groups. These values indicate that the range is at least 510 to 910 mg. The mean diameter was 7.8 mm. Calculation of the range suggests it was at least 6.7 to 8.5 mm. SEITA noted a significantly lower weight of tobacco used by the smokers under 35 years (approximately mean 690 mg) compared with those over 50 years (approximate mean 870 mg). The mean diameter was correspondingly smaller (7.3 versus 8.3). There was also “a suggestion” that the younger smokers used wrappers with a higher porosity.

5.2.2 The Dutch Study

The European Smoking Tobacco Association (ESTA) made available the results from an independent consumer study conducted in the Netherlands in 1994. The data from this study have been reported in the scientific literature. (3). The study of the making habits of consumers of fine-cut-tobacco was conducted by a leading market research agency. The objective was to study the making habits of roll-your-own consumers and to determine the nicotine free dry particulate matter (NFDPM) and nicotine yields of the products made.

A total of 140 consumers, representative of users of the leading Dutch fine-cut tobacco and paper brands, were asked to roll smoking articles using this tobacco and paper. Using tobacco and paper supplied, they each made 22 articles, applying their normal rolling procedures and marking the mouth end. These smoking articles were placed in protective tins and collected by the research agency for analysis. An independent analytical laboratory in Germany measured the diameter at three positions along the length. After conditioning at 22°C and 75% RH, the weight and NFDPM and nicotine yields of each individual article were also determined.

The Labyrinth Seal holder described in ISO 3308 and ISO 4387 was considered unsuitable for fine-cut smoking articles because of a tendency for visible leakage around the soft, non-cylindrical, ends of consumer-made, hand-rolled, smoking articles. Consequently, the holder described in ISO 3308 and ISO 4387 was modified to allow a greater insertion depth to be used to reduce leakage on the article. A longer cap with 4 labyrinth seals but without the bumper/washer was used. This resulted in an insertion depth of 13 mm which is longer than the insertion depth of 9 mm specified in ISO 4387. As a result of the longer insertion depth and the variable burning rate of the articles, it was considered necessary to smoke to a 27 mm butt length. Using a rotary smoking machine, the condensate from individual fine-cut smoking articles was collected onto a 44 mm glass fibre filter pad for analysis.

After outlier detection and removal, 2588 sets of results were obtained from 133 consumers. The results show that Dutch consumers make smoking articles that vary in shape, weight, diameter and uniformity of packing. On average, the weight of tobacco used in making was 784 mg (range487 to 1065), the diameter 7.6 mm (range 6.2 to 9.2), the number of puffs 9.4 (5.8 to 12.1), the NFDPM yield 13 mg (range 6 to 24), and the nicotine yield 1.2 mg (range 0.6 to 1.8). These data are summarised in section 5.4.

5.2.3 The German Study

In 1997 ESTA commissioned a similar study in Germany involving fine cut smokers. This paper has also been published (4). An independent market research agency in Germany recruited known smokers of the most popular brand of fine-cut tobacco and the most popular brand of booklet paper. An independent laboratory undertook the smoking and analysis using the same conditions and methods of analysis that were used in the Dutch study.

After outlier detection and removal, a total of 2575 sets of results were obtained from 131 consumers. This study shows that an average of 830 mg (range 448 to 1594) tobacco was used at an average diameter of 7.6 mm (5.7 to 10.0). German booklet paper is 2 mm shorter than Dutch paper. German products are more cylindrical than Dutch products and this probably accounts for the reduced variability of German products compared with Dutch products. The mean NFDPM yield of these articles was 12 mg (range 5 to 18) and the mean nicotine yield was 0.9 mg (range 0.4 to 1.4). The puff number was 7.7 (range 5.4 to 13.1).

5.2.4 The Norwegian Study

In 1995 JL Tiedemanns commissioned a reputable market research company (Scanfact) to conduct a consumer study. They were asked to find persons, on a demographic basis, who used the combination of the market-leader of tobacco and wrapper. A total of 40 consumers were recruited. They were given free tobacco and wrappers. The study was performed exactly as the Dutch study except that all analyses were conducted by Tiedemanns, rather than by an external laboratory. For the smoke analyses, an early version of the Sleeve holder was used with a butt length of 27 mm.

The mean weight of the FCSA was 929 mg, (range 578-1362). These weights include the weight of the wrapper which is about 50 mg. The mean NFDPM was 17 mg, (range 12-21), nicotine 1.4 mg, (range 1.0-1.8) and puff number 12.8, (range 10.3-17.0).

5.2.5 The Imperial Tobacco UK Study

In 1995 Imperial Tobacco asked an independent research company in the UK to conduct a consumer study of smoking articles made in the UK. In this study, consumers were smokers of the company’s leading brand of fine-cut tobacco who also used the leading brand of paper sold in the UK. They were asked to use their own tobacco and were not supplied with free tobacco. Sixty consumers were recruited and each was asked to make 12 FCSAs.

The fine-cut smoking articles were conditioned at 22°C and 75% RH. In this study the laboratory used an early version of the Sleeve holder (see later) to smoke 2 channels of 5 FCSAs per consumer to a butt length of 27 mm using a linear smoking machine. The mean weight of tobacco was 400 mg (range 185-680 mg per consumer, 140-920 per FCSA). The diameter was estimated from photographs taken of the FCSAs to be 5.0, (range 3.6-6.7). The mean NFDPM yield was 16 mg; the mean nicotine in smoke yield was 1.4 mg; the mean puff number was 9.9. There was a clear relationship between yield of NFDPM and weight, i.e. the yields increased as the weight increased.

5.2.6 The Gallaher UK Study

In 1996 Gallaher also conducted a study in the UK. They recruited 106 smokers of their leading brand of tobacco blend and the leading paper on sale in the UK. Each consumer was given a free supply of tobacco and paper and asked to make 12 FCSAs marking the mouth end. Gallaher conducted the sample testing and statistical analysis.

The samples were conditioned at 75% RH and 22°C. The articles were smoked to a 27 mm butt length using a linear smoking machine incorporating the standard Labyrinth Seal holder. They smoked 2 channels each of 5 FCSAs per consumer. The average weight of tobacco used, 456 mg range (170-930 mg per FCSA), was higher in this study than in the Imperial Tobacco study. The mean NFDPM yield was 15 mg with a standard deviation of 3.1 mg; the nicotine yield was 1.1 mg with a standard deviation of 0.23 mg; the mean puff number was 9.5. There was a relationship between yield of NFDPM and weight, although poor compared with the Imperial Tobacco data, possibly due to the different holders used.

5.2.7 The Laboratory of the Government Chemist UK Study

In 1998 Darrall and Figgins (5) reported on a study conducted in the United Kingdom. In this study, 26 consumers were provided with free tobacco and paper and asked to make 20 fine-cut smoking articles at one time. The products were stored at 22° and 75% RH until smoking. They smoked 2 ports each of 5 FCSAs per consumer using the Labyrinth Seal holder at a butt length of 23 mm. Using a popular brand of tobacco and paper on sale in the United Kingdom, the following values were reported:

- Mean weight of tobacco used was 505 mg (range 328 to 818 mg per consumer, 226- 919 mg per FCSA).
- Average yield of NFDPM was 15.7 mg (range 9.9 to 21.0).
- Average yield of nicotine was 1.3 mg (range 0.9 to 1.8).
- Mean puff number was 12.3.

As with the Gallaher study, the relationship between yield of NFDPM and weight is poor compared to the Imperial Tobacco data, possibly owing to the different holders used and to the smaller number of consumers. The mean tobacco weight was greater than that found by Imperial Tobacco.

5.2.8 ESTA UK Study

In an attempt to determine whether consumers use different amounts of tobacco when it is provided free compared to when they are asked to use their own, ESTA commissioned a research agency to conduct a small study in the UK. The study was undertaken in 1998.

The consumers were recruited as two sample populations, each of 30 respondents, aged 18 years and over, from smokers of the leading brand of fine-cut tobacco in the UK. One population was asked to use their own tobacco whilst the other was provided with free tobacco. Each population was asked to make 22 articles the way that they would for normal smoking and to return them in the tin provided.

The articles were conditioned at 22°C and 75% RH on arrival at the laboratory. The conditioned articles were weighed and an allowance was made for the average weight of the wrapper. The diameter was determined at three points along the length using an electronic camera and repeated after rotating the articles through 90 degrees.

The following data were obtained:

	Using "own" Tobacco		Using "free" tobacco	
	Articles	Respondents	Articles	Respondents
Number	593	27	579	26
Mean tobacco weight (mg)	405	404	450	450
Mean Diameter (mm)	5.8	5.8	5.9	5.9

This study indicates that the mean tobacco weight of consumer-made smoking articles in the UK is very close to the 400 mg chosen for use in the matrix. The difference in weight when tobacco is provided free for the study is almost 50 mg. This may help to explain some of the differences found among other UK studies (see 5.2.9). These products were not smoked.

5.2.9 General comments on the consumer studies

It is interesting to compare the three earlier UK studies and to speculate on the possible reasons for some of the differences both in terms of smoke yields and weight of tobacco used.

There is a better correlation derived from the data of the Imperial Tobacco study compared with that obtained by both Gallaher and the Laboratory of the Government Chemist (LGC). Figure 1 is a graph showing the NFDPM yield against tobacco weight. It can be seen that the relationship is much better with the Imperial Tobacco data. The coefficient of determination (R^2) value, that measures the proportion of variation in yield that is explained by weight, was 0.64 for that data compared with 0.32 for the Gallaher data and 0.21 for the LGC data. Imperial Tobacco used the Sleeve holder, combined with a longer insertion depth and butt length. At any given tobacco weight, many of the lowest NFDPM yields on the graph come from the LGC data. This could be explained by leakage of the holder.

The Imperial Tobacco study was the only study in the UK in which the consumers were not supplied with free tobacco. For this reason, it may be assumed that the data on weight gathered by Imperial Tobacco are closer to normal usage than the weights found in the other UK studies. This hypothesis is supported by the ESTA study.

The studies in Germany, Holland and Norway were conducted on the basis of freely issued tobacco and wrappers. For this reason the data for average weight of tobacco used, as determined by these studies, may be an overestimate of the true position.

5.3 CORESTA Making Study (Study 5)

The Task Force considered that terms such as weight, diameter and density probably have little meaning to the consumer when applied to their smoking article. Since variation of these parameters is required to control the yield of particulate matter and nicotine from smoking articles, it was important to know if consumers can relate to this. Because the task force was considering the adoption of a matrix approach (see section 4), it was important to study this in a range of countries to determine if consumers could correctly follow written instructions.

The Task Force undertook a study in 1994 in five countries to determine the reaction of consumers when given simple instructions concerning the making of the smoking article. In each country, 40 consumers were asked to make 10 fine-cut smoking articles, as they would do normally. Half of them were then asked to roll 10 articles using "considerably less tobacco"; the other half were asked to roll 10 articles "considerably thinner". The consumers were also asked to complete a questionnaire.

Tobacco weight, diameter at three positions and length were determined. Data from the "normal articles" are shown in Table 5, where it can be seen that the original consumer data are largely replicated.

About a third said it was difficult to comply with the instruction given to make the FCSA in a different way from their normal practice (in Holland almost half said it was difficult). However, despite this perceived difficulty, the measured data showed that the instruction had been followed. It was concluded that consumers were able to follow the instructions. The results were remarkably consistent across all countries. The concept of "rolling thinner" produced more consistent change, and was obviously easier to follow than the concept of "using less tobacco".

The conclusions were:

Consumers are able to follow simple instructions.
Similar reductions in weight were achieved from both instructions.
The average weight reduction was 25%.
Density is maintained remarkably constant.
Shape is maintained.
Diameter as represented by "thinner" is an easier concept for consumers.

This CORESTA study was intended only to be a study to determine whether consumers could follow simple instructions. It was not designed to produce definitive data on consumer making practice. Therefore making data from the CORESTA study must not be taken as representative. However, they do confirm the wide ranges of weight diameter and shape.
These data are included in the summary table in section 5.4.

5.4 Summary of consumer data

Table 5
Typical weight and diameter (range)

		General Data			1994 CORESTA Study Data (40 consumers sampled in each country)		
COUNTRY Year general data obtained		Number of consumers	Weight (mg)	Diameter (mm)	Weight (mg)	Diameter (mm)	Shape
FINLAND	1991		800 (600-1000)	8 (6-8)			
FRANCE	1990		760 (350-1150)	7.8 (6.0-9.5)	810 (500-1430)	7.1 (6.0-9.2)	17% Trumpet^
	1992	200	722˜ (510-910)	7.8 (6.7-8.5)			
GERMANY	1977	600	822	7.6	860 (580-1230)	7.7 (6.6-9.0)	10% Trumpet^
	1988	612	800	7.5			
	1994	621	780 (470-1200)	7.5 (6.2-8.9)			
	1997	131	830* s = 178	7.6* s = 0.6			
NETHERLANDS	1978	400	870	7.6	800 (580-1120)	7.1 (6.3-8.2)	38% Trumpet^
	1987	412	780	7.2			
	1994	133	784* s = 131	7.6* s = 0.6			
	1994	597	770 (490-1500)	7.6 (5.4-10.8)			
NORWAY	1990		900 (800-1000)	7 (6-8)	1070 (740-1390)	7.5 (6.5-8.4)	Cylindrical
	1995	40	880 (540-1310				
UK	1995	60	400* (140-920)	5.0 (3.6-6.7)	410 (280-690)	5.6 (4.7-6.6)	Cylindrical
	1996	106	456* s = 152				
	1998	26	505* (230-920)				
	1998	27	404*	5.8			

* Figures taken from the studies described above.
^ Trumpet indicates that the lit end is larger than the mouth end by "x" %.
˜ FCSA weight including approximately 50 mg wrapper weight.

From the studies it can be concluded that making behaviour and weight, diameter and shape of the fine-cut smoking articles vary significantly by country and consumer.

5.5 Validation of the matrix concept

The data shown above clearly demonstrates the validity of using a matrix of data. At an early stage in the work of the task force it was concluded that the two diameters should be 5.2 and 7.2 mm. The density was chosen to be approximately 260 mg/cm^3. This density is representative of a large part of the European consumer making habit and, on the basis of the main density study (Study 8), is within the range where density affects yield (see 6.2). This was achieved using weights of tobacco of 400 and 750 mg respectively for diameters of 5.2 mm and 7.2 mm. For this reason the weights of 400 and 750 mg were used for the majority of the work of the Task Force. For simplicity, it can be stated that over 95% of the wrappers currently on sale fall into two broad categories leading to differences in constituent yield. These are best represented by wrappers typically used in the UK (limited combustibility) and in Germany (combustible). These two wrapper types were chosen for later work. (See 6.3)

6. Objective 2 - The effects of materials and fine-cut smoking article design

6.1 Initial work (Studies 1 & 2)

Initial work was organised to evaluate the extent of the issues. The first experimental work was performed to assess the inherent variability of the yields derived from the fine-cut smoking articles. Even under controlled conditions, the overall variation was approximately twice that from manufactured cigarettes.

[Note: These comparisons were made before 1991 when the introduction of the new ISO standard methods for cigarettes reduced the between-laboratory variability for manufactured cigarettes. Even so the variability for FCSAs was much too great for a recommended method.]

Somewhat surprisingly, tube-made products were slightly more variable than hand-rolled products. This was almost certainly due to the fact that the assistants involved in making the products were experienced hand-rollers whereas they were not experienced with the use of a tubing machine. It was decided to continue with tubing as it was considered this type of product would be easier to use for the larger scale studies that were going to be required and ultimately for a standard method.

This study also demonstrated the difficulty in working with these hand-made products. Even with skilled operators, using the making devices available at the time, it was very time consuming to make the number of products required for testing.

To establish the overall magnitude of the effects of materials and fine-cut smoking article design, and reduce the workload of the participating laboratories, the second major study was subdivided into six parts. Since only three laboratories undertook each of the parts the data can not be interpreted quantitatively. Nevertheless, the following observations allowed the Task Force to decide further work.

1. Tobacco weight, density and product diameter are interrelated and affect smoke yields.

2. A comparison of tubed versus hand-rolled products indicated minor differences in yield that, due to the large variability, were not significant. These small differences may be due to the different density produced as a result of tubing.

3. The effect of the extent of the overlap of the paper was smaller than anticipated. The data were probably confounded by other factors that were not kept constant.

4. Tobaccos of very different type, physical characteristics and nicotine level, when made into a similar fine-cut smoking article, lead to very similar yields of particulate matter. Nicotine yields were in keeping with the blend nicotine levels.

5. A study of paper porosity did not provide conclusive evidence of its effect on smoke yields but work with manufactured cigarettes suggested that a clear effect should have been observed. As this was such an important area of research it was decided to repeat the work using a more controlled experimental design.

6. A study on paper burn additives was inconclusive owing to the small differences in the papers under study.

It was clear that consumer-making habits (i.e. the design of the fine-cut smoking article) played the most important part in determining the yield of particulate matter from smoking articles. The Task Force decided that further work was required; in particular there was a need to undertake detailed studies of the paper effect and the weight/diameter/density interaction.

6.2 The effect of tobacco weight/density and diameter (Studies 3, 8 and 9)

Previous knowledge from manufactured cigarettes indicates that the yields of NFDPM and nicotine are related to the amount of tobacco. However it is also known that diameter and density affect pressure drop and rod filtration and hence influence yield. In this respect, model studies with fine-cut tobaccos are unlikely to reveal additional information. Nevertheless, it is known that consumers use different amounts of tobacco when making fine-cut smoking articles. Three companies conducted individual research to study the effect of the tobacco weight of hand-rolled fine-cut smoking articles using consumer panels to make the articles. A detailed statistical analysis of the data revealed the following:

Survey 1 using a single tobacco type:

There was a positive correlation between diameter and yield.
This correlation was stronger when the effect of shape was considered.
There was a negative correlation between moisture content at smoking and yield.
Although there was no correlation with weight or density, this can not be interpreted as independence of those variables owing to the experimental design and high variability.
Other factors (not identified) accounted for much of the variation in yield of NFDPM but not for nicotine.

Survey 2 using two tobacco-types:

> Yield was positively correlated with weight.
> Tobacco type did not influence yield of NFDPM but did influence yield of nicotine in line with the blend nicotine.
> Other factors (not identified) accounted for some of the variation in yield of NFDPM and nicotine.

Survey 3 using eleven tobacco-types:

> Tobacco type had a small influence on yield of particulate matter and a much greater effect on nicotine yield.
> Tobacco weight was positively correlated with nicotine yield.
> Weight, density, diameter and paper porosity were not correlated to yield of NFDPM.

Statistical analysis revealed that the absence of a correlation could not be interpreted as no correlation since the data were very variable. The fact that the data were pooled also contributed to this lack of correlation since individual consumer making behaviour varies considerably. Further attention to identify the factors was required.

A further study was performed. This was a fully factorial study to examine two weights of tobacco, represented by two diameters, by four densities using a single paper type.

Nine laboratories participated. The diameters chosen were 5.2 mm and 7.2 mm. The densities chosen were 190, 240, 290 and 340 mg/cm^3. These cover the range of densities commonly made by European consumers. The wrapper was a wrapper commonly used in the Netherlands, which is one of the largest hand rolling market in Europe.

The wrappers were made into tubes 70 mm long and to the chosen diameter. The participating laboratories made fine-cut smoking articles using a tubing device frequently used in Canada. These devices were modified to allow them to be used at the chosen diameters. The tobacco was a blend, representative of a major brand sold in Europe, which was conditioned, packed and sealed. The tobacco was reconditioned at 22°C and 75% RH before making into fine cut smoking articles.

The fine-cut smoking articles were reconditioned at 22°C and 75% RH for three days. They were weight selected to mean weight ± 20 mg and stored in airtight containers. Weight selection was performed in order to minimise the variability of the product that might otherwise conceal the effect of the parameters under investigation. One hundred articles were smoked using a smoking machine complying with CORESTA Recommended Methods 22 and 25, to a butt length of 27 mm. CORESTA Recommended Methods 7 and 8 were used for the analysis of water and nicotine in total particulate matter.

Full details of the experimental protocol, detailed results and statistical analysis were distributed to all members of the Task Force and constitute part of the records of CORESTA.

The results were highly variable and individual laboratories produced significantly different absolute values. However, the trend of the mean results was very clear. The results are given in Table 6.

Table 6.
Effect of density on yield at two different diameters

DENSITY mg/cm^3	NFDPM (mg / FCSA)		Nicotine (mg / FCSA)	
	5.2 mm diameter	7.2 mm diameter	5.2 mm diameter	7.2 mm diameter
190	7.2	11.1	0.57	0.98
240	8.6	13.7	0.68	1.16
290	9.4	14.9	0.78	1.24
340	9.5	15.3	0.82	1.28

The findings from this study apply for both NFDPM and nicotine and show that both behave in a similar manner. It can be seen that there is a clear difference in the data from the two different diameters. There is no overlap between the two density versus diameter lines even at the extreme positions.

Within a given diameter, NFDPM values increase with density up to a density of about 290 mg/cm^3. However, between 290 mg/cm^3 and 340 mg/cm^3 there is no significant difference.

> **Note:** A questionnaire was issued to all participating laboratories. They reported a major problem with leakage during the smoking process using a holder designed for manufactured cigarettes, described in CORESTA Recommended Method 22. This was particularly apparent for the 7.2 mm diameter article at the two lower densities. This is referred to later in this report under “the holder used”.

6.3 The effect of paper parameters (Studies 3 & 9)

A major co-operative study was undertaken to examine the effect of wrappers. In this study tubed products with filters were used to minimise problems during smoking. Two subsets were formed each involving 8 laboratories. The first group studied the effect of porosity at different packing densities (Study 3A) and the second studied the effect of burn additive at different porosities (Study 3B). The following findings were reported:

> A logarithmic relationship exists between yield and porosity for both NFDPM and nicotine. The effect was greater for NFDPM.
>
> At a density of 260 mg/cm^3, the yield was lower than at a density of 310 mg/cm^3. However, increasing the density to 360 mg/cm^3 did not result in a further increase in yield; *i.e.* increased weight is balanced by increased rod filtration at the higher pressure drop.
>
> The presence of citrate reduces the yield of both NFDPM and nicotine. However there is an interactive effect between porosity and citrate.

A further study was designed as a fully factorial study to examine two diameters by four types of wrapper at a single density (Study 9).

Nine laboratories participated in the study. The four wrappers were chosen as they have the highest market share with a substantial proportion of the total market in France, Germany, The Netherlands and the United Kingdom/Scandinavia. The French wrapper has a very low chalk level and a low porosity whereas the UK wrapper has a moderate level of chalk and a slightly higher porosity; neither of them contains a burn additive. The Dutch and German wrappers both contain burn additives and have a higher level of chalk; the major difference between them is the balance between natural porosity and perforation.

The wrapper specifications are given in Table 7.

Table 7
Wrapper specifications

Wrapper type	Substance g/m^2	Chalk ($CaCO_3$) %	Porosity (CU)	Perforation (CU)	Total Permeability (CU)	Additive
French	15	<5.0	7	-	7	-
UK / Scandinavian	18	12.5	10	-	10	-
Dutch	20	20.0	13	52	65	1% Citrate
German	25	25.0	40	-	40	0.6% Citrate + 0.6% Acetate

CU = CORESTA Units of Air Permeability

As reported in section 5.5, the density was chosen to be approximately 260 mg/cm^3. This was achieved using weights of tobacco of 400 and 750 mg respectively for diameters of 5.2 mm and 7.2 mm.

The wrappers were made into tubes 70 mm long and to the chosen diameter. The tobacco was a blend, representative of a major brand sold in Europe, which was conditioned, packed and sealed prior to despatch. It was reconditioned at 22°C and 75% RH before making into fine cut smoking articles.

Smoking was performed at a butt length of 27 mm using the standard Labyrinth Seal holder.

The data are shown in Table 8.

Table 8
Mean values averaged across all laboratories
(mg/FCSA)

Wrapper Type	NFDPM		Nicotine	
	400 mg model 5.2 mm diameter	750 mg model 7.2 mm diameter	400 mg model 5.2 mm diameter	750 mg model 7.2 mm diameter
(German)	8.4	13.6	0.71	1.17
(French)	12.6	17.1	1.05	1.38
(UK/Scandinavian)	11.2	17.6	0.95	1.45
(Dutch)	8.6	13.6	0.76	1.23

It is clear from this table that these wrappers, which represent over 95% of the wrappers currently on sale, fall into two broad categories leading to differences in constituent yield.

Individual results again varied considerably. There was greater variation of data with the French and Dutch wrappers than there was from the German and UK/Scandinavian wrappers. Fine-cut smoking articles made from the French wrapper fail to stay alight during the smoking process and much care is required in the laboratory. The Dutch wrapper is electrostatically perforated and this may be the reason fine-cut smoking articles made with this wrapper gave rise to more variable data.

These data support the concept of providing data in the form of a matrix.

7. Objective 3 - Relevance of proposed standard procedures

Currently, only Canada has imposed a standard procedure for the measurement of NFDPM and nicotine yields from fine-cut tobacco (6). In this method, a smoking article is made to a standard specification using a pre-made filter tube. The smoking article is then conditioned and smoked using the standard procedures and methods used for manufactured cigarettes. However, if a manufacturer sells a tube in combination with the tobacco, which is common in Canada, he is permitted to quote figures pertaining to tests using that tube. This may lead to problems of interpretation for the consumer when attempting to rank fine-cut tobaccos.

It is interesting to note that an article published by a testing laboratory in Canada entitled "It's the tube that counts" (7) demonstrated that the influence of variation of the tobacco is minor compared to the influence of the wrapper. (In the case of Canada, the wrapper is likely to be a pre-made filter tube.)

The German Standardisation Institute DIN has a draft method based on producing a smoking article to a standard specification using a pre-made plain tube of 8.0 mm diameter with a tobacco weight of 820 mg (8). The smoking article is conditioned at (66±3)% RH and then smoked using the standard procedures and methods used for manufactured cigarettes; except for the butt length that is specified as 27 mm. A Commission of the Federal Department of Health (BGA) has published another method, similar to the DIN method (9). The weight of tobacco is 1000 mg and, at the time of publication, the butt length was 27 mm. Both methods specify that they should only be used for ranking fine-cut tobaccos and that data from these methods are not comparable to data produced for manufactured cigarettes. Neither method is currently in use for regulatory purposes.

The Dutch Ministry of Health has discussed proposals to use a range of yields but nothing has been produced officially. It is understood that the authorities are awaiting the outcome of the CORESTA Task Force.

Data have been produced in various other countries e.g. Norway (10) but these data have not been produced as a result of agreed methods, nor were details of the procedures given.

The Task Force has reviewed all of these methods and has serious reservations about all of them. In particular, none of the methods makes any attempt to inform the consumer about the effect of making behaviour. The influence of tobacco type is minimal for NFDPM. The wrapper and the making parameters used have a far greater effect on yields and these effects should be incorporated into any declaration of yields. Only in the case of the Canadian method is any attempt made to do so; even in this case only the wrapper effect is considered, and then only if it is sold with the tobacco.

8. Objective 4 - To consider the establishment of a recommended method

In order to develop a Recommended Method there are a number of issues that have to be studied. These are detailed below.

8.1 The effect of moisture content (Studies 4, 6 & 7)

Fine-cut tobacco is manufactured, sold and smoked at higher moisture content than tobacco used in manufactured cigarettes. Figures quoted depend on the method of determination but as an approximate guide the following figures apply:

Tobacco for manufactured cigarettes	12 to 13 %
Tobacco intended for tubing	14 to 16 %
Tobacco intended for hand-rolling	
(Mainland Europe)	18 to 21 %
(UK and Republic of Ireland)	25 to 27 %

Attempting to roll smoking articles at low moisture content results in tobacco degradation and badly produced products. In addition it was expected that the moisture content would affect smoke yields. In an attempt to quantify the effect, a co-operative study was established in which nine laboratories analysed tubed products at three different moisture contents. The products were made at one level and subsequently conditioned to the other levels. The three conditioning atmospheres were chosen as:

ISO standard conditions used for manufactured cigarettes, viz. 60% RH
Conditions used in continental Europe for packaging of fine-cut tobacco, viz. 75% RH
Conditions used in the United Kingdom and the Republic of Ireland for packaging of fine-cut tobacco, viz. 85% RH

The results from this study show an effect of moisture content of the tobacco on yields.

A further study was planned in which the tobacco was conditioned before making the fine-cut smoking articles. In order to obtain maximum sensitivity, nine laboratories made fine-cut smoking articles at a nominal tobacco weight of 1000 mg. One set of articles was conditioned at 60% RH, another at 75% RH and a third at 85% RH. Five separate determinations from 20 FCSAs were made at each of the moisture levels.

The conclusions were:

Increasing relative humidity resulted in a significant decrease ($p<0.05$) in NFDPM, a highly significant decrease ($p< 0.001$) in nicotine and a highly significant increase in puffs ($p< 0.001$).

The differences between the results of the samples could be accounted for by differences in moisture content.

There was a highly significant difference in yields between laboratories.

The results are summarised in Table 9.

Table 9
Effect of relative humidity

Conditioning Humidity %	NFDPM (mg/FCSA)	Nicotine (mg/FCSA)	Puff Number
60	16.0	1.49	6.7
75	15.3	1.30	7.9
85	14.7	1.13	9.5

A further study (Study 7) was established to further examine the effect of moisture content but this time concentrating on 60 and 75 % RH. The objective of the study was to determine the effect of moisture content of fine-cut tobacco prior to making and of the moisture content of FCSAs. Nine laboratories participated in the study, each smoking 100 FCSAs.

The data is shown in Table 10

Table 10
Yields at various moisture content conditions

<table>
<tr><th rowspan="3">Tobacco Condition (RH)</th><th colspan="6">Conditioning of the FCSA (RH)</th></tr>
<tr><th colspan="3">60</th><th colspan="3">75</th></tr>
<tr><th>NFDPM (mg)</th><th>Nicotine (mg)</th><th>Puffs</th><th>NFDPM (mg)</th><th>Nicotine (mg)</th><th>Puffs</th></tr>
<tr><td>60</td><td>18.1</td><td>1.75</td><td>6.9</td><td>16.8</td><td>1.49</td><td>8.2</td></tr>
<tr><td>75</td><td>16.8</td><td>1.66</td><td>6.4</td><td>15.8</td><td>1.40</td><td>7.5</td></tr>
</table>

The effect of conditioning is significant ($p<0.05$) on the yield of NFDPM both before and after making the FCSAs. In the case of nicotine, conditioning of the FCSA is more significant ($p<0.01$) than conditioning of the tobacco ($p<0.1$). Puff number is affected by conditioning of the tobacco and of the FCSA.

8.2 The holder used to hold fine-cut smoking articles on the smoking machine (Studies 12, 14 and 15)

8.2.1 Background

The problem of using the existing Labyrinth Seal holder for holding fine-cut smoking articles was first raised at the second meeting of the Task Force in April 1990, following the pilot studies. It caused considerable concern throughout the studies. However, it was not possible to address this issue in detail until near the end of the work, as it was necessary to reduce the variability in other aspects of the procedures before being able to address this aspect.

At the 15th meeting of the roll-your-own task force held in London on 10 May 1995, a number of comments were made concerning the need to establish a separate set of methods for the analysis of fine-cut tobacco. Several of the participants were concerned that the holder specified in CORESTA Recommended Method No. 22 was inadequate for the determination of NFDPM and nicotine from fine-cut smoking articles. There had been lengthy discussions on the problems concerning holding and sealing of fine-cut smoking articles and the practical use of the existing Labyrinth Seal holder for smoking fine-cut smoking articles. It was agreed that the method for the determination of NFDPM and nicotine from fine-cut tobacco must be applicable to products made in the laboratory. In addition, it was agreed that it should also give guidance on the analysis of hand rolled products made by consumers as legislators may wish to undertake such work, and some have already done so.

Using the specification for butt length and the holder described in CORESTA Recommended Method 22, only minor problems have occurred with machine smoking of manufactured cigarettes and these have been confined to plain cigarettes. However, in the CORESTA studies on fine-cut smoking articles, some laboratories have experienced difficulties using the holder containing labyrinth seals.

In the CORESTA studies preceding the meeting that examined products made at different diameters and different densities, the problems were very evident and many laboratories experienced considerable difficulty using the Labyrinth Seal holder. The problems were greatest when attempting to smoke fine-cut smoking articles made at low packing density and/or low firmness, or with a small diameter, or both.

Several laboratories reported almost total failure to insert the fine-cut smoking article into the standard Labyrinth Seal holder despite using the prescribed labyrinth seals. Other laboratories reported damage to the smoking article. In an attempt to overcome the problem a simple metal “sleeve insert” was developed which first penetrated the labyrinth seals allowing the fine-cut smoking article to be placed through the metal tube and into the seals without initial damage. The tube was then withdrawn to allow the labyrinth seals to grip the smoking article. Even using this device, some distortion of the product was apparent and leakage still occurred.

The problems described apply to products made in the laboratory by inserting tobacco into paper tubes using a making device operated by a technician. The products are therefore are as good as can reasonably be achieved. With consumer-made fine-cut smoking articles, even bigger problems may be expected. It is unlikely that there will be sufficient improvement in the quality of the FCSAs to nullify the insertion problem of using labyrinth seals.

8.2.2 SEITA Study

At the Task Force meeting held in Paris on 4 September 1995, SEITA presented the findings of a study conducted in their laboratories. As part of the presentation a video was produced to support the findings. This study can be summarised as follows:

> The purpose of the study was to reduce the variability of the yields and to compare the yields of FCSAs with the existing holder and a modified holder with 6 seals, using the latter at a longer insertion depth and with a longer butt length.
>
> The effect of density was also studied. Two densities, 240 &340 g/cm^3 were studied.
>
> SEITA concluded that it was necessary to maintain a free smoking space of "at least 14 mm" between the butt mark and the insertion depth. Based on the values given for the Labyrinth Seal holder this gave a butt length of 27 mm for the "tested" holder with 6 seals.

The video showed a limited number of samples but showed leakage with both the Labyrinth Seal holder and the modification with 6 seals.

The conclusions of this study, drawn by SEITA, were:

> The increase in butt length results in a decrease of NFDPM of 11% at both densities.
>
> The two holders show little effect between them although the longer insertion depth gave some reduction in leakage.
>
> Especially at low density there is a reduction in the coefficient of variation.
>
> Labyrinth seals cause distortion of the FCSAs.
>
> Creases in the paper caused extra leakage probably caused by empty ends and the high moisture content.

8.2.3 ESTA Presentation

The European Smoking Tobacco Association was aware of the problem of holder leakage and made a presentation to the Task Force meeting in Paris. The specification of the Labyrinth Seal holder was considered in detail. It was concluded that it is likely that much of the specification may not be relevant to fine-cut smoking articles. Some of the detail of the specification is not appropriate and may cause some of the problems listed above.

The problem of holder design was put to one of the leading manufacturers of smoking machines. The company recommended that an insertion depth of at least 15 mm was required in order to ensure leak-free smoking of such products. They produced a holder that was tested by a laboratory experienced in the smoking of fine-cut tobacco products.

The distance between the holder and the butt termination point of the product must be great enough to allow an operator to remove the product before the coal damages the seal. This distance must be sufficient to allow for the smoulder effect during the inter-puff periods between the fastest and slowest burning products. For plain manufactured cigarettes, the distance is 14 mm.

The minimum possible distance between the insertion depth and the butt mark was calculated to allow removal of the smoked articles without damaging the seals. Given the variability of puff number (7-14), caused at this stage of the work by a lack of a suitable making device, this translates to 3-6 mm tobacco burnt between puffs. Allowing for a minimum of two puffs, this means that the length between insertion and butt mark was taken as 12 mm in this study.

Although determined by one laboratory only, the data showed that the yields may be a little higher using the “special” style holder. This could be for two reasons:

1. The longer insertion depth is affecting the air dilution

2. There is less leakage using the “special” holder.

The paper used in the study was chosen to have a very low porosity in order to minimise the first possibility and it seems likely that reduced leakage is the main reason for the difference in yields. Visual observation during the smoking process confirmed that there was less leakage.

The investigators concluded that:

> Theoretical and experimental considerations both indicate that a longer insertion depth is required to achieve adequate smoking of fine-cut smoking articles compared with manufactured cigarettes.
>
> A “special” style of holder is required to accommodate fine-cut smoking articles.
>
> The “special” style holder can be used without distortion of the fine-cut smoking article.
>
> An insertion depth of 15 mm would appear adequate.
>
> At this insertion depth, a butt length of at least 27 mm will be required.
>
> Yields using this holder are expected to be higher than using the holder designed for manufactured cigarettes (at an equivalent butt length).
>
> A major advantage of the “special” holder is that the standard deviations for all data are lower than those obtained using the Labyrinth Seal holders.

The full paper presented to the Task Force is attached as appendix 4.

8.2.4 Holder Sub Group

As a result of the two presentations given at the Paris Task Force meeting, a small group was formed to examine the data already available and to decide on the requirements for a "special" holder for fine-cut tobacco. The two manufacturers of smoking machines were asked to contribute.

They defined two interrelated issues:

Viz. Holding and sealing.

A survey was conducted of all previous holders and a review of the patent literature was undertaken.

The sub-group presented the following observations to the task force:

The holder described in ISO 3308 can cause damage to a fine-cut smoking article during insertion, resulting in subsequent leakage.

Labyrinth seals should not be used as sealing devices (at least in their present form).

A design of holder must be capable of holding soft, irregular products.

The holder should not incorporate a "bumper"/"washer" as this also damages the fine cut smoking article.

The design should displace the holding/sealing pressure over a larger area/length without significant distortion.

It will probably be necessary to separate the holding and sealing function.

A Sleeve (vacuum) holder appears to be the most practical design.

8.2.5 Density profiles of FCSAs

In an attempt to define the problem, six different groups of fine-cut smoking articles were made in different ways:

1. Hand rolled by an experienced roller (1).
2. Hand rolled by an experienced roller (2).
3. Box-type making device.
4. Tubing device used for CORESTA studies 7.2 mm diameter.
5. Tubing device used for CORESTA studies 5.2 mm diameter.
6. Hand rolled by an inexperienced roller.

Five of each of these were taken at random and the density was measured at 3 mm increments along the length. Density profiles for the 5 individual articles were produced for the 6 different types.

A mean value for the density of each article (using the middle 40 mm to produce the mean value) was calculated and then each of the individual readings was plotted relative to the mean. This produced graphs that were easy to interpret. From the normalised data it was concluded that an insertion depth of 9 mm for fine-cut smoking articles would be totally inadequate due to the softness of the ends. It appeared that an insertion depth of about 15 mm is required to hold these soft fine-cut smoking articles to ensure leak-free smoking.

A second study was done to measure density along the length of fine-cut smoking articles. Nine companies produced 100 fine-cut smoking articles at each of two diameters using a tubing device. Ten of each batch were chosen at random for measurement of density along the length. Plain and filtered cigarettes were examined for comparison. Again the data from the FCSAs showed the great variability in density and hence making. The data also showed that approximately the first 15 mm are soft compared to the mean density. The data from the two density studies were reported in a paper presented in Yokohama, Japan in November 1996 and are published in the CORESTA Bulletin (11). For convenience the paper is also given as Appendix 5.

8.2.6 Study 12

A co-operative study (No. 12) was organised. This was designed to compare the Labyrinth Seal holder with a new design of holder that used a sleeve of 4.8 mm diameter with an insertion depth of 15 mm. Eleven laboratories were recruited to undertake 5 smoking runs each using the existing (Labyrinth Seal) and new Sleeve holders. Six of these laboratories used a rotary smoking machine, 5 used a linear smoking machine.

Two statisticians examined the data independently.

Data for NFDPM are shown in Table 11

Table 11
NFDPM for two holder types
(mg)

	Holder type	
Laboratory	Labyrinth Seal 27 mm butt	Sleeve (vacuum) 4.8 mm sleeve 27 mm butt
1	14.4	15.1
2	14.5	14.5
3	12.9	13.8
4	17.6	16.5
5	10.4	11.4
6	14.9	16.3
7	14.1	11.7
8	14.4	14.5
9	15.7	14.8
10	15.4	14.8
11	15.0	16.0

The data showed that the difference between the two holders is negligible compared to the difference between laboratories. The reports from the laboratories suggested that the sleeve was too small as several of the FCSAs were creased. The full report is shown as appendix 6.

8.2.7 Study 14a and 14b

Since Study 12 had been inconclusive as the variability between laboratories eclipsed any holder effect, the Task Force decided to undertake Study 14. The objective of this study was to evaluate further the “special” holder for fine-cut smoking articles.

Study 14a was designed to compare:

> The Labyrinth Seal holder at a butt length of 23 mm.
> The Labyrinth Seal holder at a butt length of 27 mm.
> A “modified” Sleeve holder with a sleeve diameter of 5.5 mm. The smoking was conducted at a butt length of 27 mm. (This diameter was chosen because it is the size of the hole in the labyrinth seals used for cigarettes at a diameter of 7.2 mm).

Eleven laboratories were recruited to conduct smoking tests, each providing smoking data based on 100 FCSAs. The fine-cut smoking articles were made using a blend of fine-cut tobacco previously used with a German style paper, to a specification of 7.2 mm diameter, 70 mm length, 750 mg tobacco, conditioned at 75% RH and 22 ^{o}C.

A second related study (Study 14b) was conducted using a plain manufactured cigarette, at physical dimensions as close as possible to the dimensions used to produce FCSAs. The cigarettes were smoked under standard ISO conditions, i.e. conditioning to 60% RH at 22 ^{o}C, standard Labyrinth Seal holder, 9 mm depth of insertion and 23 mm butt length. Thirteen laboratories took part in this part of the study, including all 11 laboratories that participated in the first part. The purpose of this study was to see how laboratories performed when analysing a machine-made product.

Two statisticians examined the data independently. Each statistician examined Study 14a and Study 14b separately, but after a discussion within the Task Force it was agreed that the report should be based on a combined analysis of the two parts. Appendix 6 contains the full report.

As a result of applying a statistical test, 17 individual data points were identified as outliers. Of these only 2 were obtained with the modified Sleeve holder.

The data from this study are shown in Table 12.

Table 12
NFDPM from the Labyrinth Seal holder and Sleeve holder (mg)

Laboratory	FCSA: Labyrinth Seal Holder 23 mm butt	FCSA: Labyrinth Seal Holder 27 mm butt	FCSA: Sleeve Holder (5.5 mm sleeve) 27 mm butt	Cigarette: Labyrinth Seal Holder 27 mm butt
1	16.6	14.6	14.5	12.6
2	13.9	12.1	15.3	9.6
3	17.8	15.2	14.9	13.4
4	15.7	15.0	16.1	14.0
5	17.3	15.0	15.4	14.6
6	12.7	10.6	14.6	12.0
7	14.7	12.8	15.1	10.3
9	16.9	15.1	16.3	14.3
10	15.9	14.5	16.3	13.1
11	18.0	15.7	14.8	12.4
Mean	16.0	14.1	15.3	12.6
‘v’	1.6	1.5	1.6	1.2
‘V’	5.0	4.6	2.4	4.6

Note Laboratory 8 failed to comply with the protocol.

It can be concluded that:

The data are very variable. In particular the between-laboratory variability was in all cases greater than obtained in a CORESTA collaborative study by laboratories analysing manufactured filter cigarettes.

The within-laboratory variability was greater for FCSAs than for a manufactured cigarette.

> Although much greater than desirable, the between-laboratory variability for the Sleeve holder was smaller than the value using the Labyrinth Seal holder, even for a cigarette. The statistical evaluation was so affected by the overall variability of the data that this conclusion could not be substantiated as a statistically genuine effect.

It was considered that the existing Labyrinth Seal holder used to smoke manufactured cigarettes is inappropriate for smoking fine-cut smoking articles owing to the damage caused during insertion. Feedback from most of the laboratory operators suggested a strong preference for a Sleeve holder as opposed to a Labyrinth Seal holder.

The important question of depth of insertion and/or butt length could not be answered by this study.

The two statisticians proposed that after the laboratory representatives had made their recommendations and after such had been implemented, a study with clearly defined objectives and measures of success should be undertaken to provide more robust data. This was agreed by the Task Force and as many laboratories as possible were encouraged to participate.

The study report is given in full in Appendix 7.

8.2.8 Study 15

8.2.8.1 Study Design

An experts group met and agreed a common approach to the design and the statistical analysis of the study. Eighteen laboratories participated in the study. The study plan called for the use of the standard Labyrinth Seal holder with labyrinth seals and of four newly developed Sleeve holders with different insertion depths.

The objective of the study was defined as follows:

> To determine the appropriate holder design and insertion depth required to smoke fine-cut smoking articles for the determination of NFDPM and nicotine in smoke.

It was agreed that the results would be evaluated primarily on the basis of within-laboratory variability. In particular, the data from this study could not form the basis of the calculation of r and R.

Because of the fragile nature of fine-cut tobacco, it was decided that an attempt to randomise the FCSAs would severely damage the articles, and jeopardise the study. As a result, it is not permissible, formally, to estimate the between-laboratory variability.

8.2.8.2 FCSA Making

All fine-cut smoking articles used in this study were made centrally in one laboratory with a modified Canadian tubing device and distributed to the participating laboratories.

The specification of the fine-cut smoking articles used was as follows:

Length: 70 mm
Diameter: 7.2 mm
Tobacco weight: 750 mg
Tobacco type: Dutch blend
Paper: German type.

8.2.8.3 Smoking

The Labyrinth Seal holder was used according to ISO Standard 3308:1991. For this reason only one insertion depth (9 mm) could be used with this holder.

The Sleeve holder was supplied as four separate designs in such a way that 4 insertion depths could be used. Using the Sleeve holder, the fine-cut smoking articles were smoked using nominal insertion depths of 9 mm, 11 mm, 13 mm, and 15 mm. A butt length of 29 mm was used for all smoking of the fine-cut smoking articles to allow valid comparisons between holder configurations.

The CORESTA test piece (CM2) was smoked using the standard Labyrinth Seal holder. A butt length of 30 mm was used for the smoking of the test piece in line with the protocol given for its use.

On receipt in the laboratory, the fine-cut smoking articles were conditioned according to CORESTA Recommended Method 42.

The 20-channel linear smoking machines were used to smoke 5 articles through each channel on to a 44 mm filter pad.

The 20-channel rotary smoking machines were specially equipped with a trap designed to allow the smoking of 5 articles on to a 44 mm filter pad. Since the evaluation of the data from this study was to be based upon the within-laboratory variability, the 20-channel rotary smoking machines were adapted in this way so as to make the variability of the data from the two types of smoking machine directly comparable. Additionally, by reducing the number of fine-cut smoking articles smoked per filter pad, the sensitivity of the data is greatly enhanced.

If leakage at the holder occurs for some individual fine-cut smoking articles, then this may be reflected in an increased puff number. Since suspected leakage at the holder was one of the reasons for the design of the Sleeve holder, analysis of the puff numbers of individual fine-cut smoking articles was considered necessary in order to provide important insight into this aspect of the design of the holder. Laboratories were therefore asked to record individual puff numbers.

8.2.8.4 Statistical analysis

Three statisticians agreed the protocol for statistical analysis. It was performed in the following way:

Early on, one laboratory (number 7) was completely eliminated from the analysis because it was unable to follow the experimental protocol.

The statisticians applied a one-way analysis of variance within and between holders for each laboratory as the basis for detecting outlying data points separately for both NFDPM and nicotine. These points were then referred to the originating laboratories to allow the data to be checked.

Any points whose standardised residual remained > 3.0 after checking were deemed to be outliers and were removed from the analysis.

The data, excluding the one laboratory, contain 2040 records. There were 16 outliers for both NFDPM and nicotine, giving an error rate of 0.78%.

The statistical techniques of analyses of variance rely upon the assumption that the data (or more strictly, the residuals after fitting the main effects and interactions) are Normally distributed. In this study, the target variable is the standard deviation and it is a well-known fact that standard deviations are not Normally distributed. In order to deal with this the statisticians used a standard technique known as a Box and Cox variance stabilising transformation. The same transformation was used for both NFDPM and nicotine.

If data are Normally distributed, then a plot of the data versus the Normalised score of the data should show a straight line. A plot of transformed SD (NFDPM) versus its Normalised score is virtually straight, with no systematic deviations from expectation at either end of the line showing that the transformation was correct.

Applying analysis of variance techniques, the effects of differences (in smoke yields) between laboratories and of differences between smoking machines (which are completely confounded with the effect of laboratories) were eliminated as these were of no intrinsic interest in this study. The remaining residuals are now essentially only determined by potential holder effects and general residual variance.

Whether differences in within-laboratory variability exist between the various holders is now examined by a further analysis of variance of the residual transformed standard deviations. This results, on the one hand, in an F-value (representing the overall differences between the holders) and in pairwise comparisons of the different holders.

The measurement variations of the CORESTA test piece, using the Labyrinth Seal holder (see Table 13), and of the fine-cut smoking articles with the Labyrinth Seal holder and the four Sleeve holders (see Tables 14 to 16) were examined separately.

The full detail including the statistical report on Study 15 is given as Appendix 8.

<u>8.2.8.5 CORESTA Test Piece Data</u>

The CORESTA test piece CM2, smoked through the Labyrinth Seal holder with 9 mm insertion depth, was included in this study as a monitor of the performance of the participating laboratories.

Repeatability and reproducibility were calculated and expressed as v and V (as defined in Chapter 3). It must, however, be remembered that the values of NFDPM and nicotine recorded in this study are based upon smoking 5 products, rather than 20 as is usually the case. In order to make v and V comparable with earlier CORESTA studies using the test piece, the data were pooled over four results (representing 20 products smoked).

The pooled values of v and V for NFDPM and nicotine for the test piece (Table 13) indicate that the participating laboratories performed well during the course of this study. This is concluded by comparing the pooled values obtained in the study with the values of v and V calculated from the data given in CORESTA Report 91-1 (12). The values of v for NFDPM (1.0) and V for nicotine (0.16) are in fact somewhat less than the values expected from the information given in CORESTA Report 91-1. The value of v for nicotine (0.12) is the same as the expected value and the value of V for NFDPM (2.0) is only slightly higher than expected.

Table 13
v and V for the CM2 test piece
(Laboratory 7 omitted)

	NFDPM					Nicotine			
	v	v calc.	V	V calc.		v	v calc.	V	V calc.
After pooling	1.0	1.1	2.0	1.8		0.12	0.12	0.16	0.22

8.2.8.6 FCSA Data - Within-Laboratory Variability

It must be remembered that the data obtained in CORESTA studies or published in CORESTA papers are generally based upon the smoking of 20 products through each filter pad, whereas in this study only 5 products were smoked through each filter.

Table 14 gives the values for NFDPM and standard deviations for each laboratory after individual outlier removal but before transformation. Table 15 gives the corresponding values for nicotine. Table 16 gives the corresponding values for puff number.

Table 14
Mean NFDPM data with corresponding within-laboratory standard deviations
(mg)

Laboratory Number	Holder									
	Labyrinth Seal		Sleeve 9 mm		Sleeve 11 mm		Sleeve 13 mm		Sleeve 15 mm	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
1	13.28	1.06	10.90	1.30	12.06	1.32	12.77	1.11	13.49	0.73
2	11.76	0.88	11.46	0.81	11.63	0.69	12.07	0.46	12.50	0.49
3	11.13	1.36	11.69	0.98	13.09	1.08	14.00	1.14	14.44	0.77
4	12.51	1.09	11.39	1.64	12.54	1.49	13.52	1.17	14.11	0.85
5	13.88	1.09	13.36	0.82	13.41	0.98	13.65	1.08	13.70	1.09
6	13.45	0.83	13.03	0.60	13.84	0.74	14.41	0.93	14.93	0.79
8	11.47	0.74	10.83	0.69	11.67	0.70	12.34	0.59	12.70	0.48
9	13.62	0.81	11.19	1.02	12.54	1.07	12.84	0.94	13.01	0.77
10	12.88	1.02	12.08	0.75	13.14	0.71	13.41	0.55	13.81	0.86
11	14.32	0.94	13.10	1.53	14.04	1.29	15.02	1.21	15.06	0.96
12	13.23	0.98	11.78	0.70	12.34	0.89	13.81	0.79	14.29	0.80
13	12.39	0.84	11.57	1.30	13.16	0.69	13.35	0.93	13.88	0.76
14	14.08	1.60	13.30	1.18	14.33	1.26	14.80	1.03	14.47	1.79
15	11.50	1.05	11.42	1.32	11.87	1.10	12.76	0.92	12.81	0.79
16	8.82	1.62	11.57	1.59	11.93	1.40	13.06	1.36	12.66	1.28
17	12.34	1.11	11.28	0.69	12.12	0.95	12.77	0.91	13.20	0.74
18	14.49	0.57	12.58	0.54	13.50	0.57	13.85	0.46	14.23	0.66
All	12.65	1.07	11.91	1.09	12.78	1.04	13.43	0.95	13.72	0.91

Table 15
Mean nicotine data with corresponding within-laboratory standard deviations
(mg)

Laboratory Number	Holder									
	Labyrinth Seal		Sleeve 9 mm		Sleeve 11 mm		Sleeve 13 mm		Sleeve 15 mm	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
1	1.004	0.053	0.906	0.057	0.954	0.063	0.995	0.051	1.011	0.039
2	1.060	0.102	0.980	0.090	0.993	0.064	1.013	0.063	1.047	0.066
3	0.878	0.077	0.919	0.072	0.999	0.066	1.022	0.072	1.061	0.053
4	0.899	0.065	0.838	0.080	0.916	0.074	0.963	0.078	0.971	0.050
5	1.261	0.093	1.259	0.069	1.249	0.093	1.270	0.052	1.249	0.110
6	1.095	0.057	1.048	0.069	1.127	0.064	1.149	0.076	1.160	0.078
8	0.915	0.055	0.877	0.045	0.926	0.042	0.952	0.039	0.971	0.041
9	1.038	0.054	0.914	0.053	0.967	0.067	0.987	0.056	0.975	0.037
10	1.003	0.081	0.973	0.069	1.012	0.066	1.002	0.055	1.047	0.083
11	1.131	0.095	1.098	0.120	1.115	0.103	1.144	0.083	1.151	0.079
12	1.027	0.056	0.955	0.060	0.991	0.059	1.058	0.045	1.064	0.041
13	0.968	0.064	0.923	0.082	1.005	0.070	1.013	0.068	1.046	0.077
14	1.065	0.114	1.025	0.071	1.071	0.088	1.106	0.100	1.063	0.123
15	0.935	0.056	0.954	0.068	0.975	0.052	0.996	0.061	1.018	0.051
16	0.782	0.095	0.916	0.048	0.920	0.040	0.984	0.059	0.986	0.058
17	0.998	0.070	0.938	0.042	0.979	0.053	1.005	0.064	1.031	0.055
18	1.054	0.048	0.981	0.042	1.007	0.050	1.014	0.041	1.039	0.047
All	1.008	0.075	0.971	0.070	1.012	0.068	1.036	0.064	1.052	0.068

Table 16
Puff Number data with corresponding within laboratory standard deviations

Laboratory Number	Holder									
	Labyrinth. Seal		Sleeve 9 mm		Sleeve 11 mm		Sleeve 13 mm		Sleeve 15 mm	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
1	5.91	0.28	6.20	0.23	6.13	0.20	6.03	0.25	5.90	0.18
2	5.90	0.29	5.88	0.20	5.86	0.21	5.81	0.26	5.71	0.25
3	6.25	0.33	6.37	0.32	6.24	0.27	5.97	0.22	5.88	0.24
4	6.18	0.27	6.22	0.29	6.24	0.17	6.11	0.27	6.02	0.16
5	4.57	0.29	4.72	0.23	4.75	0.25	4.84	0.43	4.68	0.21
6	5.77	0.26	6.20	0.40	5.74	0.21	5.74	0.23	5.64	0.28
8	5.74	0.34	5.99	0.26	6.11	0.25	5.85	0.20	5.71	0.32
9	5.22	0.17	5.69	0.28	5.54	0.31	5.32	0.26	5.37	0.26
10	5.71	0.16	5.63	0.24	5.67	0.23	5.52	0.31	5.50	0.26
11	5.39	0.40	5.59	0.42	5.51	0.36	5.45	0.29	5.42	0.48
12	5.81	0.21	5.84	0.23	5.80	0.24	5.72	0.21	5.53	0.22
13	5.87	0.28	5.83	0.28	5.74	0.26	5.65	0.20	5.65	0.28
14	5.85	0.42	6.12	0.32	6.13	0.34	6.15	0.47	5.87	0.57
15	5.83	0.33	5.76	0.27	5.84	0.28	5.81	0.23	5.68	0.25
16	6.82	0.45	6.30	0.44	6.17	0.25	6.25	0.20	6.02	0.23
17	6.17	0.17	6.27	0.30	6.16	0.26	6.12	0.24	6.00	0.29
18	5.75	0.20	5.90	0.21	5.78	0.19	5.79	0.27	5.77	0.23
All	5.81	0.30	5.91	0.30	5.85	0.26	5.77	0.28	5.66	0.29

The laboratory coded as 5 (as well as laboratory 7) was omitted[2] because the mean levels of the data from laboratory 5, especially the determinations of nicotine, are much higher than expected, and these high means would have had an undue influence on the calculations of V.

As the result of the basic statistical analysis (validation of data, outlier treatment), the values v (as defined in Chapter 3) for within-laboratory variability were obtained and are shown in Table 17:

Table 17
Values of v (based on 5 products per pad; laboratories 5 and 7 omitted)

	NFDPM	Nicotine
Labyrinth Seal 9 mm	3.0	0.21
Sleeve 9 mm	3.1	0.20
Sleeve 11 mm	2.9	0.18
Sleeve 13 mm	2.9	0.19
Sleeve 15 mm	2.5	0.18
Test piece	1.7	0.17

Puff numbers were collected on the basis of individual FCSAs, unlike NFDPM and nicotine data that were collected per trap. Thus, it is not realistic to quote a value for v in this table.

[2] As a result of the analysis performed in study 16, (see 8.2.9) a principle components analysis was performed and confirmed that laboratory 5 was an outlier laboratory (see 8.2.10).

In terms of variability of NFDPM, based on the values of v, the ranking of the holders, used for the smoking of fine-cut smoking articles, is as follows: Sleeve 9 mm exhibits the highest variability, followed by Labyrinth Seal 9 mm, Sleeve 11 mm, Sleeve 13 mm and Sleeve 15 mm which shows the lowest variability.

Upon completion of a variance stabilising transformation and elimination of machine and laboratory effects (see Chapter 8.2.8.4), analysis of variance was used to examine the differences in within-laboratory variability between the holder types. The F-value, testing the global difference between the five holders, was significant at 3.23 ($p = 0.018$) (see Table 2.1 of Appendix 8). Pairwise comparison of the holders showed that the differences between Labyrinth Seal and Sleeve 13 mm, Labyrinth Seal and Sleeve 15 mm, Sleeve 9 mm and Sleeve 15 mm, Sleeve 11 mm and Sleeve 15 mm, were all significant ($p<0.05$).

In terms of variability of nicotine, the holders were ranked in the order Labyrinth Seal 9 mm, Sleeve 9 mm, Sleeve 13 mm, Sleeve 11 mm, Sleeve 15 mm; the Labyrinth Seal holder gave results with the largest variability and the Sleeve 15 mm holder gave results with the lowest variability. The F-value of 1.74 ($p = 0.151$) indicates no significant overall difference between the five holders (see Table 3.1 of Appendix 8). Pairwise comparisons of the holders showed that the differences between Labyrinth Seal and Sleeve 13 mm and Labyrinth Seal and Sleeve 15 mm, were both significant ($p<0.05$).

In this study, the individual puff number of every FCSA smoked was recorded. By recording the individual puff numbers the power of the study to detect any effect of the holder configurations was substantially improved.

In terms of variability of puff numbers, the holders were ranked in the order Sleeve 9 mm, Labyrinth Seal 9 mm, Sleeve 11 mm, Sleeve 13 mm, Sleeve 15 mm; the Sleeve 9 mm holder gave results with the largest variability and the Sleeve 15 mm holder gave results with the lowest variability. The F-value of 11.53 ($p < 0.001$) indicates a highly significant overall difference between the five holders (see Table 4.1 of Appendix 8). Pairwise comparisons of the holders showed that the differences between Labyrinth Seal and Sleeve 11 mm, Labyrinth Seal and Sleeve 13 mm, Labyrinth Seal and Sleeve 15 mm, Sleeve 9 mm and Sleeve 11 mm, Sleeve 9 mm and Sleeve 13 mm, Sleeve 9 mm and Sleeve 15 mm, Sleeve 11 mm and Sleeve 15 mm, were significant ($p<0.05$).

To analyse the effects of insertion depth, regression analyses have been performed for the various configurations of the <u>Sleeve</u> holder. It was shown (see Tables 2.2, 3.2 and 4.2 of Appendix 8) that:

- There is a significant reduction in the variability of the NFDPM data as the insertion depth is increased ($p<0.01$).
- There is no significant reduction in the variability of the nicotine data as the insertion depth is increased ($p>0.05$).
- There is a highly significant reduction in the variability of the puff number data as the insertion depth is increased ($p<0.001$).

8.2.8.7 FCSA Data - Mean Values

It was not an objective of the study to focus on the absolute values for NFDPM and nicotine or for puff numbers. However, it is interesting to note the following results of the regression analysis of the data from the Sleeve holders, as insertion depth is increased, at a constant butt length of 29 mm, from 9 to 15 mm.

- There is a highly significant increase in mean NFDPM (by 1.8 mg; $p<0.001$).
- There is a highly significant increase in mean nicotine (by 0.08 mg; $p<0.001$).
- There is a highly significant decrease in mean puff number (by 0.24; $p<0.001$).

8.2.8.8 FCSA Data – Between Laboratory Variability

The prime focus of the analyses of this study was the variability within laboratories. It is however of interest to consider the variability between laboratories as far as it is possible considering the experimental design. To that end, values of V (as defined in Chapter 3) were estimated. The values for NFDPM and nicotine are shown in Table 18.

Table 18
Values of V (Laboratories 5 and 7 omitted)

	NFDPM	Nicotine
Labyrinth Seal 9 mm	5.0	0.32
Sleeve 9 mm	3.7	0.26
Sleeve 11 mm	3.7	0.25
Sleeve 13 mm	3.5	0.24
Sleeve 15 mm	3.4	0.24
Test Piece	2.3	0.21

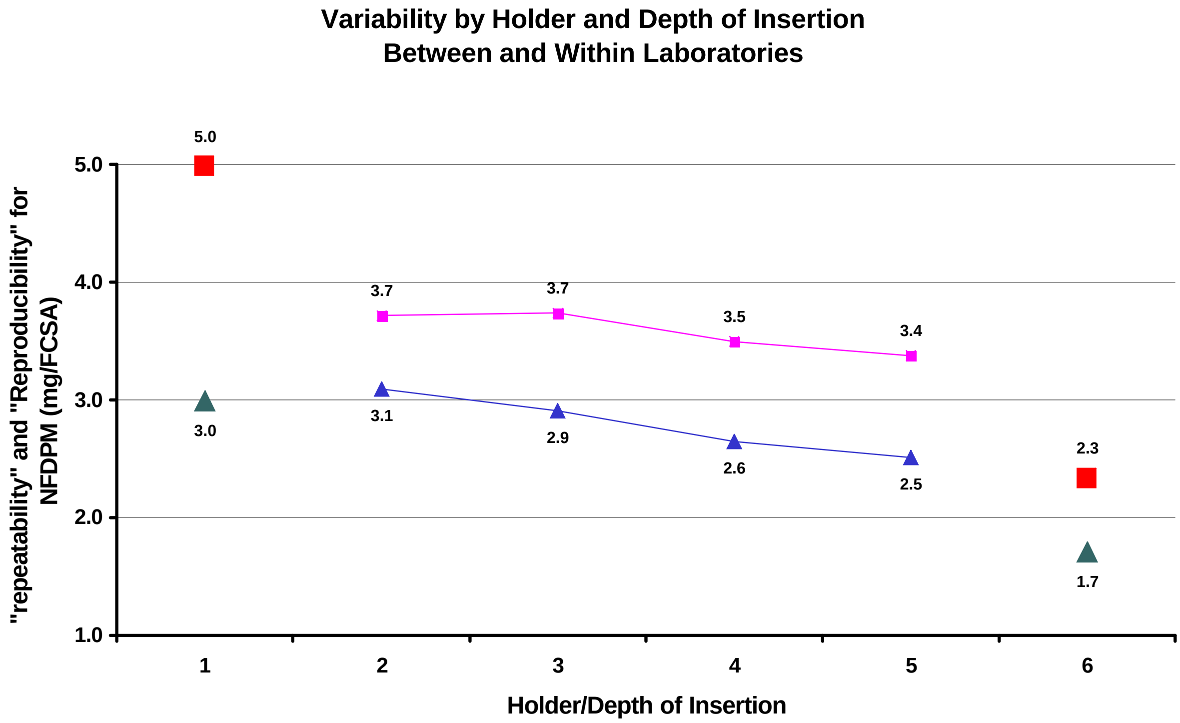
Puff numbers were collected on the basis of individual FCSAs, unlike NFDPM and nicotine data that were collected per trap. Thus, it is not realistic to quote a value for V in this table.

It can be seen that when considering the NFDPM results, the value of V (an indicator of between-laboratory variability) is at a maximum when the Labyrinth Seal holder is being used, whereas all versions of the Sleeve holder give lower values of V.

When considering the nicotine results, the value of V is at a maximum when the Labyrinth Seal holder is being used, whereas all versions of the Sleeve holder give lower values of V. The difference between the Labyrinth Seal and Sleeve holders is less marked than is the case when considering the NFDPM data.

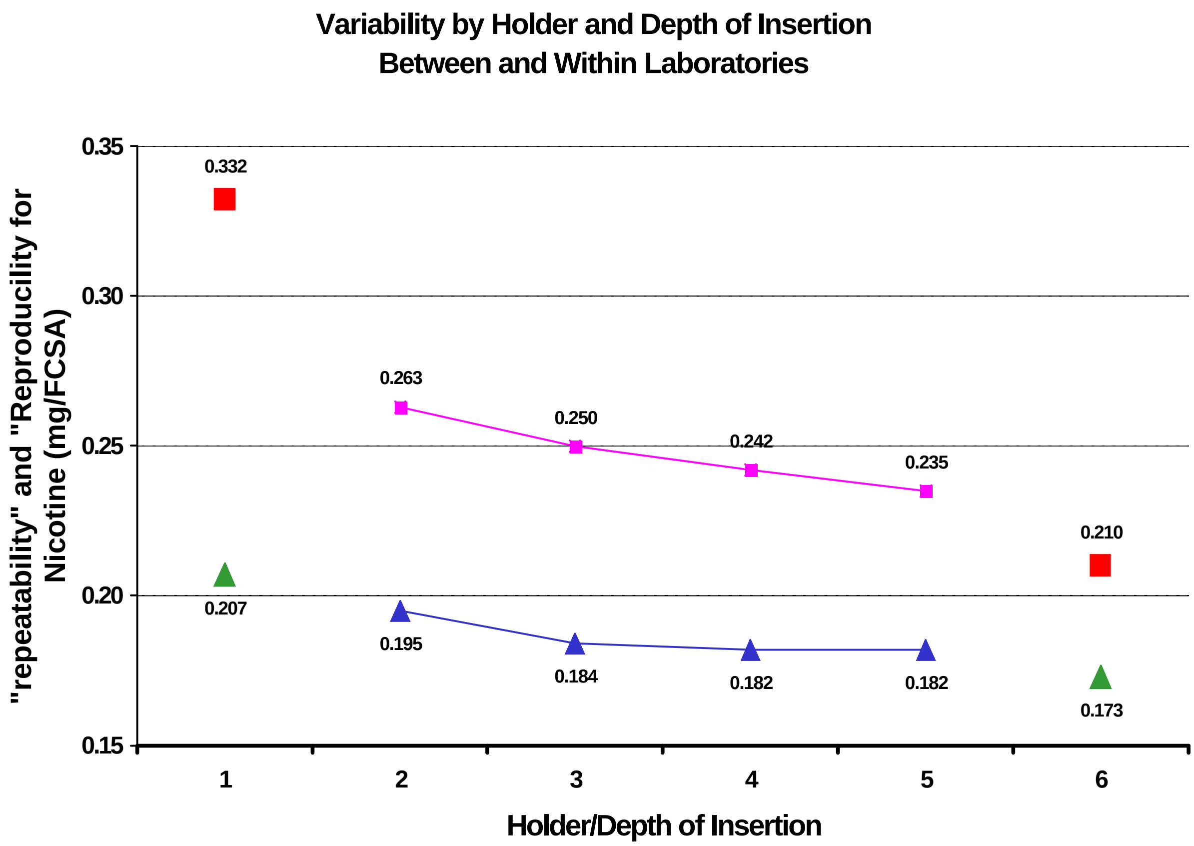
The data for within-laboratory and between-laboratory variability were presented to the Task Force in graphical form.

Appendix 8 contains the full report of Study 15.



In these graphs code 1 = the Labyrinth Seal holder; 2 = Sleeve 9 mm; 3 = Sleeve 11 mm; 4 = Sleeve 13 mm; 5 = Sleeve 15 mm; 6 = CM2

The upper line represents the between-laboratory variability and the lower line represents the within-laboratory variability.



8.2.8.9 Laboratory Comments

As well as the statistical evaluation, the comments of the laboratory staff, relevant to the conduct of the method were considered to be important. These are shown in Table 19.

Table 19
Laboratory Comments

Lab. No.	Remark
1	No relights. No need for spare FCSA using the correct labyrinth seals.
2	Good filling better than made ourselves. Small folds from Sleeve holder. Vacuum hole not always round. Labyrinth Seal holder very difficult to insert FCSAs - mostly crushed. Almost impossible to achieve 9 mm insertion. Crush marks from lab seals. Sleeve holders smoke seen.
3	Front seal bulged otherwise no problem with the Sleeve holder.
4	Labyrinth Seal holder - FCSAs must be inserted with utmost care time consuming. Ends mostly crushed. Some tearing. 9 mm sleeve some leakage.
5	No particular problems with regard to user friendliness. When asked directly they reported no problem with Labyrinth Seal holder insertion.
6	Labyrinth Seal holder- 4 spare FCSAs used to get proper insertion. Sleeve 9 mm some spares used. Other sleeves no problems.
7	Samples not easy to handle. FCSAs rather soft irregularly filled especially ends. No vacuum available on rotary smoking machine.
8	No special problems.
9	Experienced operators had no problems with either holder.
10	Aligning bobbin caps to prevent distortion of washer difficult. Washer bulges cap difficult to remove. Key needed Airline location awkward. Pump tone good indication of good seal. Modified traps much easier than using Labyrinth Seal.
11	No comment received.
12	Products well made. Labyrinth Seal assembly easier than vacuum holder. Less smoke leakage with Labyrinth Seal holder than with short insertion depth on Sleeve holder. With Sleeve holder, visible smoke less with increasing insertion. Products with poorly filled ends more easily inserted using the Sleeve holder though may snag the front washer.
13	Slight bulging noticed. Sleeve holders stiff to use at first but loosened. More time consuming than Labyrinth Seal holder. Labyrinth Seal holders cause damage.
14	No comments received.
15	Slight bulging. FCSAs difficult to insert with Labyrinth Seal holder. Crushing occurred. Some visible leakage.
16	Very difficult to insert FCSAs into Labyrinth Seal holder.
17	Labyrinth Seal holder - to prevent damage FCSAs turned and inserted needed to reform to reduce deformed products.
18	No comments received.

One laboratory (number 7) did not use vacuum on the Sleeve holder and the data from this laboratory was not included in the analysis.

Of the remaining 17 laboratories:

Three made no comment.

Ten laboratories reported difficulties when using the Labyrinth Seal holder or reported a preference for the Sleeve holder.

Four laboratories stated that there were no problems with either holder.

One laboratory stated that the visible leakage was more noticeable at a 9 mm insertion depth using the Sleeve holder but decreased with insertion depth.

Five laboratories commented that the Sleeve holders were more time consuming or that the design could be improved.

8.2.9 Study 16

It will be seen (8.2.9.1) that the objectives of this study involved both 5.2 mm and 7.2 mm diameter FCSAs. For clarity of reading the data from the 7.2 mm diameter products are dealt with in this section and those from the 5.2 mm diameter products are dealt with in section 8.3.

8.2.9.1 Study Design

As with Study 15, an experts group met and agreed a common approach to the design and the statistical analysis of the study. Seventeen laboratories participated in the study. As a result of experience gained in Study 15, the Sleeve holder design was again modified slightly. The study plan called for the use of the standard Labyrinth Seal holder with labyrinth seals and of three modified Sleeve holders with different insertion depths.

It is also important to note that further modifications were made to the Sleeve holder. An aluminium cap replaced the plastic cap, the hole front face was very thin and the hole was just large enough to allow the FCSA to enter. In this way the holder cap acted as the positioner. As a result, the front seal also required to be modified.

The objectives of the study were defined as follows:

- To determine the between-laboratory and within-laboratory variability of fine-cut smoking articles made under “normal” conditions and at two diameters.

- To evaluate the effect of insertion depth on the smoke yields and between-laboratory and within-laboratory variability of 5.2 mm diameter FCSAs.

- To compare the between-laboratory and within-laboratory variability of 5.2 mm diameter FCSAs with the between-laboratory and within-laboratory variability of 7.2 mm diameter FCSAs.

- To evaluate the effect of insertion depth on the smoke yields of 7.2 mm diameter FCSAs when these articles are made under "normal" conditions and to contrast these data with those obtained from Study 15.

- To evaluate the local making of FCSAs, and to determine if there are any problems arising from articles being made locally or during the smoking (especially the 5.2 mm diameter FCSAs).

The following limitations were also noted:

As the analytical parameters of the experimental method are not yet fully specified it must be noted that, although the statistical analysis of this study will be founded upon the between-laboratory and within-laboratory variability, this study cannot be used to estimate the repeatability (r) or the Reproducibility (R) of the method.

Extreme caution must be used when making any comparisons between the data arising from this study and that from Study 15 for the following reasons:

The fine-cut smoking articles were made by a central laboratory in Study 15 but were made by the participating laboratories in Study 16.
In this study the means are based upon the smoking of 20 FCSAs whereas in Study 15 the means were based upon the smoking of 5 FCSAs.
The design of the Sleeve holder for use in this study has been modified from that used in Study 15.
The mix of participating laboratories is different for the two studies.

<u>8.2.9.2 FCSA Making</u>

All fine-cut smoking articles used in this study were made in the laboratory conducting the analysis using tubing devices supplied to the design required by the Task Force and manufactured by a wrapper converting supplier company in Germany.

The specification of the fine-cut smoking articles used was as follows:

<u>7.2 mm diameter articles</u>	<u>5.2 mm diameter articles</u>
Length: 70 mm	Length: 70 mm
Diameter: 7.2 mm	Diameter: 5.2 mm
Tobacco weight: 750 mg	Tobacco weight: 400 mg
Tobacco type: Dutch blend	Tobacco type: Dutch blend
Wrapper: German type.	Wrapper: German type.

The fine-cut smoking articles were made using tobacco straight from sealed pouches of tobacco and conditioned according to CORESTA Recommended Method 42 after making.

<u>8.2.9.3 Smoking</u>

The Labyrinth Seal holder was used according to ISO Standard 3308:1991. For this reason only one insertion depth (9 mm) could be used with this holder.

The Sleeve holder was supplied as three separate designs at each diameter in such a way that 3 insertion depths could be used at each diameter. Using the Sleeve holder, the fine-cut smoking articles were smoked using nominal insertion depths of 9 mm, 13 mm, and 15 mm. A butt length of 29 mm was used for all smoking of the fine-cut smoking articles to allow valid comparisons between holder configurations.

The CORESTA test piece (CM2) was smoked using the standard Labyrinth Seal holder. A butt length of 30 mm was used for the smoking of the test piece in line with the protocol given for its use.

The 20-channel linear smoking machines were used to smoke 5 articles through each channel on to a 44 mm filter pad. The 20-channel rotary smoking machines were used to smoke 20 articles on to a 92 mm filter pad. Individual puff numbers were not recorded in this study but mean puff numbers, per smoking run, were recorded.

8.2.9.4 Statistical analysis

Laboratories 9 and 10 did not participate in Study 16 but an additional laboratory (number 19) took part.

The design of this study, although similar in many ways to that of Study 15, is sufficiently different in respect of the method of making, smoking plan and number of variants, that the agreed statistical procedures for the two studies were slightly different especially in the treatment of outliers.

It is important to note that the treatment of outlying data points and particularly the elimination of laboratories as outliers is crucial to the subsequent conclusions. This is especially true for the between-laboratory variability since removing an outlying laboratory could materially reduce the between-laboratory variability (V).

Three statisticians agreed the protocol for statistical analysis. It was performed in the following way:

The statisticians applied a one-way analysis of variance within and between holders for each laboratory as the basis for detecting outlying data points separately for both NFDPM and nicotine. These suspicious data points were then referred to the originating laboratories to allow the data to be checked and corrected if necessary.

The statistical procedure for the removal of outliers followed 3 stages

1. The data from the linear machines are available as means of 5 FCSAs, and it was agreed that these data should be scanned for outliers prior to the averaging over four channels. Any points whose standardised absolute residual exceeded 2.75 after validation by the laboratory were deemed to be outliers and were removed from the data. This procedure resulted in the removal of 28 individual data points for NFDPM and 18 data points for nicotine. This corresponded to 1.75% and 1.12% respectively.

After the removal of these outliers, the data from the linear machines were averaged over four channels, or the number of channels remaining per run per holder configuration. This allows the linear and rotary machines to be evaluated on the same basis of 20 FCSAs.

2. The analytes of interest in the analysis of this study are NFDPM and nicotine. It was therefore decided that the (NFDPM, nicotine) pairs of observations should be scanned for outliers, rather than scanning the data for each analyte separately. A 2-variate analysis of variance of the form

(NFDPM, nicotine) = Laboratory effects + error

was used for each holder configuration.

This can be written as

$$\underset{(n \times 2)}{[\text{NFDPM, nicotine}]} = \underset{(n \times 17)}{[\text{Laboratory contrasts}]}*\underset{(17 \times 2)}{[\text{Coefficients}]} + \underset{(n \times 2)}{[\text{Error}]}$$

where [NFDPM, nicotine] is an (n x 2) matrix of observations, [Laboratory contrasts] is an (n x 17) matrix which represents the mean differences between the 17 laboratories which participated in this study, and [Error] is an (n x 2) matrix of residual errors. The coefficients of this model were then calculated in the usual way i.e. by making the determinant of the $[E^{/}E]$ matrix a minimum.

Outliers were then detected by an examination of the plots of the (NFDPM, nicotine) elements of the [E] matrix.

In order to eliminate any subjectivity that might arise during the examination of these plots a principal component decomposition of the [E] matrix was used, and the resulting principal component scores were Normalised. The decomposition has the effect of removing the correlation known to exist between the (NFDPM, nicotine) residuals, and the Normalisation allows the presence of outliers to be detected by reference to a contour line drawn on the plot. For this study, any data point lying outside the 3.25 SD contour line was deemed to be an outlier.

Whenever an outlier was detected, the corresponding (NFDPM, nicotine) data point was removed from the [NFDPM, nicotine] matrix and the [E] matrix was recalculated. This cycle was repeated until no further points appeared outside the 3.25 SD contour line on the plots of residuals.

Illustrative graphs are included in Appendix 9. This process led to a total of 17 data points out of 761 or 2.23% being removed.

3. After the removal of outlying data points as described above, the data were examined for the presence of outlying laboratories. For this examination the data from the 5.2 mm FCSAs and the 7.2 mm FCSAs were treated separately. The correlation matrices arising from data were subjected to principal component analysis, and the first three principal component scores were used to compute the distance of each laboratory from the centre of the 3-dimensional space represented by the three principal components.

At the first pass laboratory 5 was deemed to be an outlier within the data from the 7.2mm FCSAs and was therefore deleted from all subsequent analyses of these data.

At the second pass (after the deletion of the data from laboratory 5) laboratory 2 was also deemed to be an outlier within the data from the 7.2 mm FCSAs and was therefore deleted from all subsequent analyses of these data.

A third pass of the principal component analysis after the deletion of the data from laboratories 5 and 2 revealed no other outlying laboratory.

8.2.9.5 CORESTA Test Piece Data

A comparison of these data (see Appendix 9, Tables 1 to 6) with that from the CORESTA study reported in CORESTA Report 91-1, again shows that Study 16 was very well performed.

8.2.9.6 FCSA Data - Within-laboratory Variability

Tables 20 and 21 show the means, within-laboratory standard deviations, and the pooled within-laboratory standard deviations, after the removal of outliers and the deletion of the outlying laboratories as described in the previous sections. Note data are not produced for puff number from Study 16 since this data was collected normally and not for each individual FCSA as was done for study 15.

TABLE 20
7.2 mm FCSAs, OUTLIERS REMOVED, LABORATORIES 2 and 5 DELETED

Nicotine – MEAN (mg)

LAB	B	F	G	H	M
1	1.0854	1.0252	1.1052	1.1036	1.5600
3	1.2035	1.0592	1.1645	1.1650	1.5705
4	1.0789	1.0040	1.0281	1.0535	1.6146
6	1.0036	1.0196	1.1181	1.1120	1.5341
7	1.2068	1.0840	1.1544	1.1320	1.5442
8	1.0076	1.0026	1.0492	1.0602	1.5706
11	0.9660	0.9620	1.0640	1.0020	1.5160
12	1.0240	1.0735	1.0530	1.0968	1.5440
13	1.0997	1.1296	1.1366	1.1429	1.5906
14	0.8497	0.9370	0.9760	1.0195	1.5815
15	0.7830	1.0315	1.0780	1.0750	1.5634
16	1.1802	1.2049	1.2096	1.2744	1.6038
17	1.0940	1.0500	1.1880	1.1525	1.5680
18	0.9296	1.0046	1.0720	1.1044	1.5978
19	1.2106	1.0556	1.1382	1.1448	1.6960
ALL	1.0470	1.0407	1.1013	1.1064	1.5770

Nicotine - STANDARD DEVIATIONS

LAB	B	F	G	H	M
1	0.0471	0.0275	0.0412	0.0521	0.0306
3	0.0190	0.0236	0.0411	0.0399	0.0199
4	0.0645	0.0546	0.0603	0.0695	0.0354
6	0.0429	0.0463	0.0763	0.0509	0.0493
7	0.0208	0.0205	0.0399	0.0302	0.0224
8	0.0458	0.0388	0.0311	0.0551	0.0205
11	0.0747	0.0259	0.0305	0.0303	0.0288
12	0.0476	0.0468	0.0158	0.0787	0.0221
13	0.0563	0.0676	0.0379	0.0284	0.0419
14	0.0600	0.0286	0.0432	0.0078	0.0175
15	0.0518	0.0141	0.0209	0.0611	0.0387
16	0.0259	0.0309	0.0496	0.0477	0.0358
17	0.0498	0.0534	0.0512	0.0419	0.0646
18	0.0600	0.0658	0.0215	0.0262	0.0229
19	0.0915	0.0627	0.0531	0.0664	0.0534
ALL	0.0544	0.0441	0.0439	0.0495	0.0362

TABLE 21
7.2 mm FCSAs, OUTLIERS REMOVED, LABORATORIES 2 and 5 DELETED

NFDPM – MEAN (mg)

LAB	B	F	G	H	M
1	13.968	12.834	13.868	14.130	15.652
3	15.671	12.760	14.577	14.403	15.563
4	13.634	12.467	13.015	13.295	15.199
6	13.193	13.703	14.804	14.982	15.769
7	15.484	13.726	14.738	14.078	15.846
8	12.808	12.246	13.028	13.370	15.260
11	11.916	11.456	13.610	13.228	15.616
12	13.356	13.842	13.824	14.716	15.840
13	13.323	13.548	14.181	14.182	15.767
14	10.582	12.286	13.171	13.620	16.087
15	9.265	13.084	14.093	14.090	16.077
16	14.492	14.402	14.815	15.782	16.357
17	12.508	11.748	13.696	13.307	15.136
18	10.916	11.554	13.068	13.226	15.514
19	15.110	12.460	13.982	14.224	15.698
ALL	13.061	12.786	13.897	14.028	15.692

NFDPM - STANDARD DEVIATIONS

LAB	B	F	G	H	M
1	0.556	0.716	0.624	0.687	0.325
3	0.395	0.524	0.614	0.533	0.225
4	0.702	0.265	0.474	0.804	0.341
6	0.650	0.602	0.560	0.399	0.408
7	0.317	0.496	0.447	0.188	0.184
8	0.601	0.564	0.233	0.569	0.246
11	1.297	0.536	0.305	0.361	0.425
12	0.523	0.615	0.549	0.674	0.334
13	0.534	0.940	0.725	0.794	0.229
14	1.135	0.388	0.984	0.343	0.443
15	0.710	0.580	0.633	0.672	0.258
16	0.306	0.791	0.456	0.356	0.262
17	0.541	0.561	0.556	0.628	0.407
18	1.173	0.662	0.095	0.342	0.282
19	0.700	0.754	0.252	0.470	0.431
ALL	0.743	0.617	0.550	0.553	0.331

Table 22 shows the calculated values of the within-laboratory variability (v)

Table 22
Values of v (based on 20 products per pad; laboratories 2 and 5 omitted)

	Holder	NFDPM	Nicotine
Labyrinth Seal 9 mm	B	2.1	0.15
Sleeve 9 mm	F	1.7	0.12
Sleeve 13 mm	G	1.5	0.12
Sleeve 15 mm	H	1.6	0.14
Test piece	M	0.9	0.10

Thus it can be seen that in terms of v the 9 mm Labyrinth Seal holder produces the more variable data when used to smoke 7.2 mm FCSAs. In terms of v for nicotine the data suggests that using a Sleeve holder with 15 mm insertion gives rise to more variable data compared to that obtained when using either a 9 mm or a 13 mm insertion depth. In terms of v for NFDPM there is a downward trend as Sleeve holder insertion depth is increased from 9 mm to 13 mm with no further decrease on going to a 15 mm insertion depth.

There are no significant differences between the variability of nicotine yields when smoking 7.2 mm FCSAs through any of the holder variants used in this study.

There is a statistically significant difference ($p<0.05$) between the variability of NFDPM yields of the data from the B and G holders (Labyrinth Seal and Sleeve using 13 mm insertion). No other comparisons of the holder variants showed a significant difference.

These data are plotted with those of the 5.2 mm FCSAs in Section 8.3.2.

8.2.9.7 FCSA Data - Mean Values

There are no statistically significant differences between the puff numbers arising from the use of the various holder configurations when smoking 7.2 mm FCSAs. The means are shown plotted in figure 10 in Appendix 9.

The mean nicotine yields from holders B and F are not significantly different, and the mean nicotine yield from holders G and H are not significantly different. There is however a statistically significant difference between the mean nicotine yields of these two pairs (B and F, G and H). The means are shown plotted in figure 11 in Appendix 9.

The mean NFDPM yield from holders B and F are not significantly different and the mean NFDPM yields from holders G and H are not significantly different. There is however a statistically significant difference between the mean NFDPM yields of these two pairs (B and F, G and H). The means are shown plotted in figure 12 in Appendix 9.

8.2.9.8 FCSA Data – Between Laboratory Variability

These data are shown in Table 23

Table 23
Values of V (Laboratories 5 and 7 omitted)

	Holder	NFDPM	Nicotine
Labyrinth Seal 9 mm	B	5.5	0.39
Sleeve 9 mm	F	2.9	0.21
Sleeve 13 mm	G	2.3	0.21
Sleeve 15 mm	H	2.4	0.22
Test piece	M	1.3	0.15

It can be seen, when considering the NFDPM results, that the value of V (an indicator of between-laboratory variability), for the data from the Labyrinth Seal holder are more variable than that from any of the Sleeve holder variants. There is a reduction in variability as insertion depth into the Sleeve holder is increased from 9 mm to 13 mm but a slight rise on further increasing the insertion depth to 15 mm. When smoking 7.2 mm FCSAs the data from the Labyrinth Seal holder are more variable than those from any of the Sleeve variants but there is little difference between the Sleeve variants.

These data are plotted with those of the 5.2 mm FCSAs in Section 8.3.2.

8.2.9.9 Laboratory comments

Laboratory comments made on this study are given in Table 24. Note that these comments also refer to the 5.2 mm FCSAs referred to in Section 8.3.2.

Table 24
Comments of the Task Force Members

Laboratory Number	Comment
1	No comments received.
2	5.2 mm insertion depth with the Labyrinth Seal holder was almost impossible to insert as it was the sleeve holder. The 7.2 mm Labyrinth Seal holder was very difficult with some FCSAs crushed the 9 mm Sleeve holder was OK but the 13 mm and 15 mm Sleeve Holder resulted in crushing.
3	Nothing to report.
4	Sleeve holders preferred. 9 mm Sleeve holder gave problems too much leakage. The black seals in the Sleeve holder need improving. The 'O'-seals are too thin. Vacuum difficult to apply. Caps good but screw caps would be better. 13 mm Sleeve holder is the best. 15 mm too difficult to use.
5	Great care needed to insert the FCSAs, especially those at 5.2 mm. However no "wrecks" occurred. Difficult to light FCSAs. 2 operators were used to ensure that the butts could be removed before burning the sleeves.

6	Both sizes gave problems with the FCSAs. We had to modify the procedure to insert them. The 5.2 mm diameter FCSAs were compressed by the 15 mm holder sleeve. The Sleeve holder for 7.2 mm diameter FCSAs at 9 mm insertion gave leaks. Standard Deviation of CM2 higher than normal probably due to the interference of the airflow when handling the FCSAs. Likely to have affected the Standard Deviation of the FCSAs. More deposit on holder using the Sleeve holder, it may be necessary to examine this in more detail. Sleeve holder at 13 mm behaved the best at both diameters.
7	No comments received.
8	Huge problems to insert the 5.2 mm diameter products into the Labyrinth Seal holders. Generally the thinner FCSAs were more difficult to handle
9	No comments received.
10	Holder A (the 5.2 mm diameter Labyrinth Seal holder) especially resulted in damage to the FCSAs. Some other FCSAs went out and had to be re-lit. No issues arose with the Sleeve holder.
11	Analysing the FCSAs that failed to complete smoking for the 5.2 mm diameter, there were 40 in total of which 35 were from the Labyrinth Seal holder. For the 7.2 mm diameter holder, there were 14 in total of which 10 were from the Labyrinth Seal holder. Sleeve holder not as easy to assemble as in Study 13. Front seal too loose and leaks may occur or sleeve not open with the vacuum. A front seal similar to that in Study 15 is preferable.
12	Labyrinth Seal holder was used with tweezers as the holder caused damage if they were not used. Sleeve holder sometimes failed to operate properly. Protocol should have included a check on the sleeve operation before starting to smoke. All holders showed visible leakage. This worsened with use.
13	Smoking easier than with Study 15. Latex sleeves were better quality. The only difficulty was in loading the Labyrinth Seal holder. This caused the FCSAs to “crumple”. Used a pair of tweezers to insert them. 7.2 mm were easier than the 5.2 mm in the Labyrinth Seal holder because of their size. Two technicians operated the smoking machine to ensure no difficulties in smoking.
14	No comments received.
15	By using “less stiff” Labyrinth Seals in the holders, it is a little easier than before but still the ends are deformed and requires great care. The assembly of the holders needs to be done carefully. Some leakage can occur and the FCSAs may not be inserted properly.
16	Insertion of both 5.2 and 7.2 mm into the Labyrinth Seal holder caused damage to the FCSAs. No difficulty inserting with the Sleeve holder but the holder must be assembled very carefully otherwise leakage will occur. The 15 mm insertion depth sometimes caused leakage. It seems that the ‘O’-ring do not always keep in the correct position.
17	No serious problems but insertion of the 5.2 mm diameter products was difficult into the Labyrinth Seal holders.

8.2.10 Reconsideration of Study 15

Following the statistical testing for outlier removal in Study 15, the expert group asked the statisticians to conduct a similar principal components analysis to that conducted for Study 16 to establish which, if any, laboratories would be detected as outliers. If it was determined that there were a different number of outlying laboratories, the statisticians were asked to recalculate the final data.

This analysis was performed as requested and, using the correlation matrix to perform the test, laboratory 5 was determined to be an outlier. Consequently, the data given for Study 15 did not require to be recalculated since the outcome from this more rigorous statistical approach coincided with the original decision.

8.2.11 Comparison of studies 15 and 16

The protocol for Study 16 makes it clear that there would not be a comparison of the data with that of Study 15. Nevertheless, providing all the restrictions are clearly understood, as laid down in the protocol for Study 16, some useful observations can be made.

In terms of mean nicotine yield, there is close agreement between the results from Studies 15 and 16. In terms of mean NFDPM yield, there is also close agreement between the results from Studies 15 and 16, although the agreement is perhaps not quite as close as that shown by the nicotine data.

In terms of the within-laboratory standard deviation of nicotine yields there is some disparity between Studies 15 and 16. It is to be expected that Study 16 would show greater variability than Study 15 given the fact that for Study 15 the FCSAs were made centrally and might therefore be expected to be more uniform than the locally-made FCSAs used in Study 16. This might be one of the factors that could explain, at least in part, the difference in general level between the two studies, but this does not explain the difference between the performance of the Sleeve holder using 15 mm insertion depth. In Study 15 this version of the Sleeve holder gave the lowest variability, whereas in Study 16 it gave the highest variability amongst the Sleeve holders.

In terms of the within-laboratory standard deviations of NFDPM yields there is some disparity between Studies 15 and 16. In Study 15 the Labyrinth Seal holder gave results which were slightly less variable than the Sleeve holder using 9 mm insertion depth, whereas in Study 16 the reverse is the case. Also, in Study 16 the results from the Sleeve holder using 15 mm insertion depth are as variable as the results from the Sleeve holder using 13 mm insertion, whereas in Study 15 the results from the Sleeve holder using 15 mm insertion depth were less variable than those from the Sleeve holder using 13 mm insertion.

In terms of between-laboratory variability, Study 16 showed even more pronounced differences than Study 15 with both NFDPM and nicotine being very considerably less variable using the Sleeve holder.

All these data are shown graphically in Appendix 9.

Overall, the data tend to indicate that the Sleeve holder at 13 mm insertion depth gives the least variable results for both NFDPM and nicotine, for both between and within-laboratory variability.

8.3 5.2 mm Diameter Articles (Studies 8 &16)

8.3.1 Effect of Density

Study 8 has already been discussed with respect to the effect of tobacco weight/density and diameter in section 6.2. For easy reference the relevant values are given in Table 25 from which it can be seen that yield increases with density for both NFDPM and nicotine. In this respect 5.2 mm diameter articles behave in a similar way to 7.2 mm diameter articles.

Table 25
Effect of density on yield (5.2 mm diameter articles)

DENSITY mg/cm^3	NFDPM (mg / FCSA)	Nicotine (mg / FCSA)
190	7.2	0.57
240	8.6	0.68
290	9.4	0.78
340	9.5	0.82

8.3.2 Study 16

Study 16 was designed to evaluate the effect of holder type and depth of insertion at constant butt length. Section 8.2.9 gives the details. In practice the statistical analysis was performed at the same time and the same outlier treatment was used.

<u>8.3.2.1 Outlying Laboratories</u>

At the first pass of the principal components analysis, Laboratory 2 was deemed to be an outlier within the data from the 5.2 mm FCSAs and was therefore deleted from all subsequent analyses of these data. A repeat of the principal component analysis after the deletion of the data from Laboratory 2 revealed no other outlying laboratory.

<u>8.3.2.2 FCSA Data - Within-laboratory Variability</u>

Tables 26 and 27 show the means, within-laboratory standard deviations, and the pooled within-laboratory standard deviations, after the removal of outliers and the deletion of the outlying laboratory as described previously.

TABLE 26
5.2 mm FCSAs, OUTLIERS REMOVED, LABORATORY 2 DELETED

Nicotine – MEAN (mg)

	A	C	D	E	M
1	0.5976	0.5922	0.5916	0.6080	1.5600
3	0.5640	0.5975	0.5681	0.5794	1.5705
4	0.5594	0.5696	0.5675	0.5628	1.6146
5	0.5730	0.4745	0.5105	0.5455	1.5460
6	0.5939	0.6115	0.6551	0.6532	1.5341
7	0.6524	0.6204	0.6306	0.6448	1.5442
8	0.5654	0.5930	0.6178	0.5754	1.5706
11	0.4760	0.5700	0.5780	0.5225	1.5160
12	0.6000	0.6365	0.6470	0.6502	1.5440
13	0.5802	0.6455	0.6424	0.6753	1.5906
14	0.5355	0.6180	0.6115	0.6360	1.5815
15	0.5265	0.6305	0.6225	0.6437	1.5634
16	0.7027	0.6998	0.7229	0.7006	1.6038
17	0.5980	0.6340	0.6560	0.6820	1.5680
18	0.5080	0.5628	0.6014	0.5892	1.5978
19	0.5622	0.6330	0.6080	0.5948	1.6960
ALL	0.5747	0.6053	0.6151	0.6181	1.5751

Nicotine - STANDARD DEVIATIONS

	A	C	D	E	M
1	0.0329	0.0353	0.0433	0.0335	0.0306
3	0.0129	0.0167	0.0069	0.0461	0.0199
4	0.0289	0.0416	0.0391	0.0416	0.0354
5	0.0330	0.0476	0.0421	0.0612	0.0624
6	0.0595	0.0440	0.0364	0.0886	0.0493
7	0.0188	0.0323	0.0138	0.0319	0.0224
8	0.0380	0.0229	0.0244	0.0275	0.0205
11	0.0513	0.0524	0.0268	0.0395	0.0288
12	0.0352	0.0147	0.0331	0.0363	0.0221
13	0.0480	0.0223	0.0274	0.0486	0.0419
14	0.0323	0.0239	0.0320	0.0258	0.0175
15	0.0540	0.0187	0.0555	0.0312	0.0387
16	0.0460	0.0497	0.0459	0.0300	0.0358
17	0.0517	0.0167	0.0219	0.0130	0.0646
18	0.0629	0.0335	0.0188	0.0207	0.0229
19	0.0340	0.0300	0.0225	0.0393	0.0534
ALL	0.0422	0.0340	0.0334	0.0420	0.0384

TABLE 27
5.2 mm FCSAs OUTLIERS REMOVED LABORATORY 2 DELETED

NFDPM – MEAN (mg)

	A	C	D	E	M
1	8.084	7.624	7.760	7.934	15.652
3	7.042	7.827	7.706	7.925	15.563
4	7.250	7.071	7.492	7.400	15.199
5	7.235	6.295	6.695	7.300	15.695
6	8.011	8.368	8.894	8.938	15.769
7	8.648	8.090	8.376	8.220	15.846
8	7.284	7.302	7.758	7.260	15.260
11	5.238	6.748	7.174	6.483	15.616
12	8.061	8.396	8.718	8.784	15.840
13	7.534	7.852	8.225	8.658	15.767
14	6.718	8.039	8.251	8.558	16.087
15	6.338	8.235	8.248	8.530	16.077
16	9.075	8.476	9.049	8.759	16.357
17	6.832	7.274	7.706	8.060	15.136
18	6.372	6.994	7.554	7.514	15.514
19	7.710	7.782	7.713	8.066	15.698
ALL	7.339	7.644	7.964	8.045	15.692

NFDPM - STANDARD DEVIATIONS

	A	C	D	E	M
1	0.228	0.198	0.479	0.236	0.325
3	0.213	0.300	0.243	0.410	0.225
4	0.463	0.681	0.506	0.260	0.341
5	0.169	0.574	0.632	0.714	0.401
6	0.635	0.657	0.536	0.960	0.408
7	0.318	0.171	0.401	0.481	0.184
8	0.417	0.317	0.139	0.341	0.246
11	0.502	0.324	0.416	0.480	0.425
12	0.677	0.441	0.150	0.587	0.334
13	0.647	0.816	0.347	0.600	0.229
14	0.489	0.358	0.387	0.374	0.443
15	0.552	0.215	0.530	0.158	0.258
16	0.563	0.740	0.350	0.539	0.262
17	0.713	0.210	0.188	0.409	0.407
18	0.530	0.280	0.259	0.287	0.282
19	0.472	0.693	0.407	0.440	0.431
ALL	0.501	0.484	0.401	0.495	0.336

Table 28 shows the calculated values of the within-laboratory variability (v)

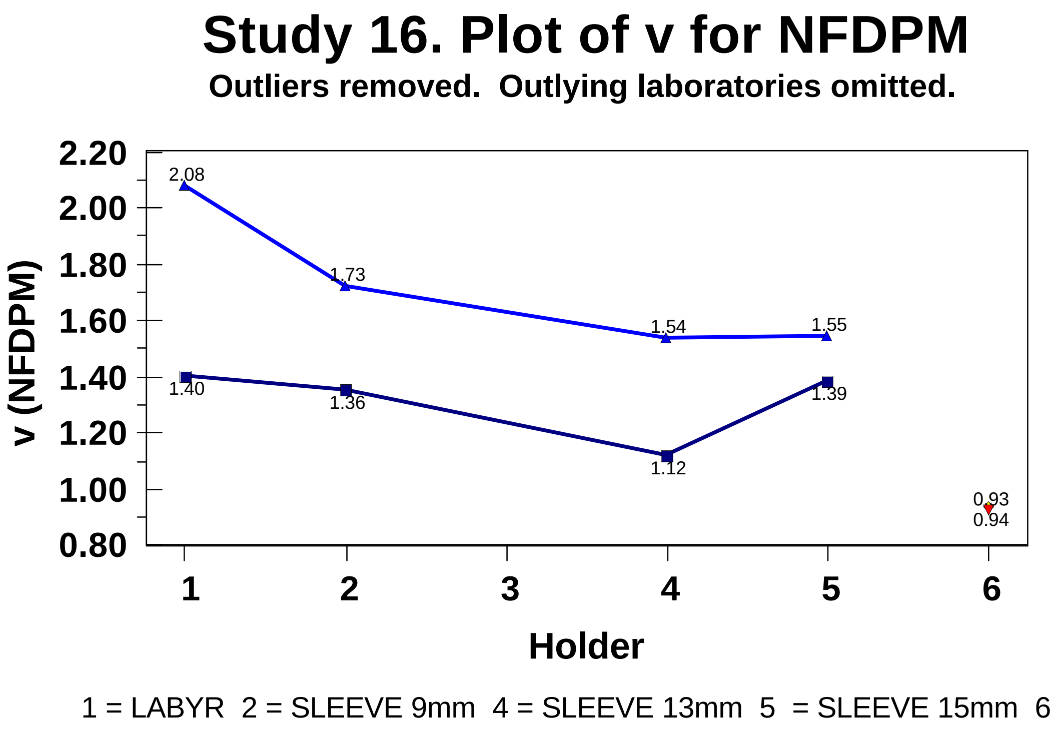
Table 28
Values of v (based on 20 products per pad; Laboratory 2 omitted)

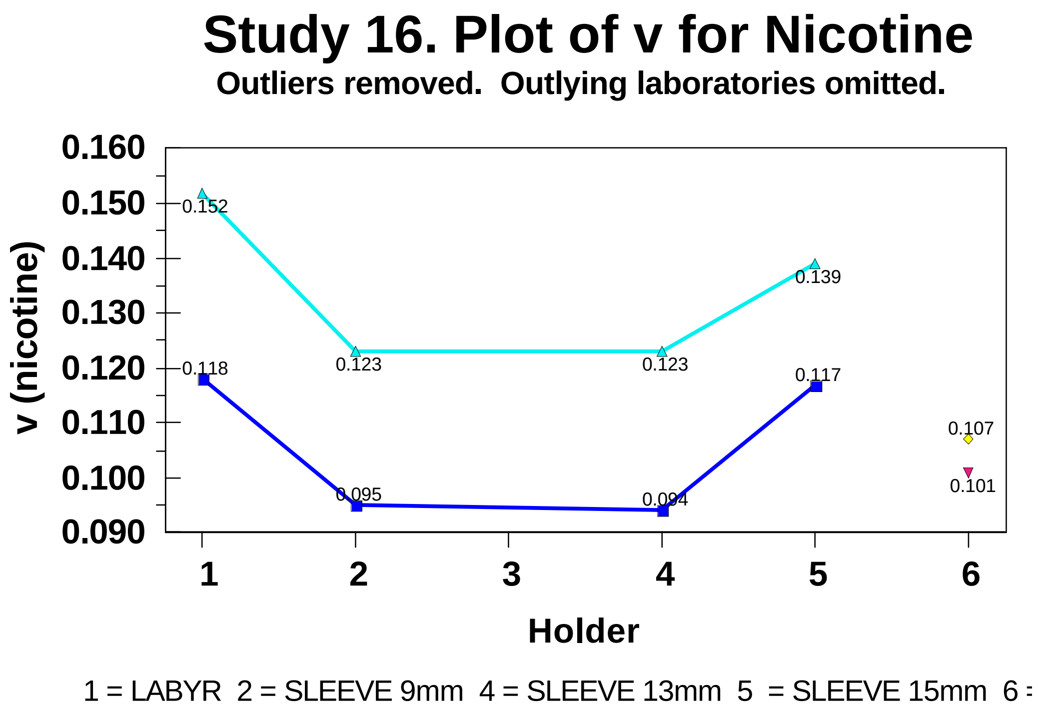
	Holder	NFDPM	Nicotine
Labyrinth Seal 9 mm	A	1.4	0.12
Sleeve 9 mm	C	1.4	0.10
Sleeve 13 mm	D	1.1	0.09
Sleeve 15 mm	E	1.4	0.12
Test piece	M	0.9	0.11

In terms of v for NFDPM, when smoking 5.2 mm FCSAs, the Sleeve holder using 13 mm insertion is less variable than any of the other combinations.

In terms of v for nicotine the data suggests that, when smoking 5.2 mm FCSAs, using a Sleeve holder with 13 mm insertion depth and the 9 mm insertion depth, gives rise to less variation compared to that obtained when using the other holders.

These data are shown graphically below. The graph also shows the values for v for the 7.2 mm diameter articles. In the following graphs, the upper line and the points identified with a triangle are from the 7.2 mm FCSAs whilst the lower line is from the 5.2 mm FCSAs. It is also worth noting that, in some instances there are two points for the monitor data. This is because only 1 laboratory data set was removed from the 5.2 mm FCSA data, whereas, 2 laboratory data sets were removed from the 7.2 mm FCSA data. Thus, the mean value of the monitor may not be the same for the 5.2 mm study and the 7.2 mm study.





There are no significant differences between the variability of NFDPM yields of any of the holder variants used in this study when smoking 5.2 mm FCSAs.

There is a statistically significant difference between variability of nicotine yields of the data from the Labyrinth Seal and Sleeve using 13 mm insertion. No other comparisons of the holder variants showed a significant difference.

There are no significant differences between the variability, as measured by transformed standard deviations, of puff numbers when smoking 5.2 mm FCSAs through any of the holder variants used in this study.

8.3.2.3 Analysis of means.

There are no statistically significant differences between the puff numbers arising from the use of the various holder configurations when smoking 5.2 mm FCSAs. The mean nicotine yield from the Labyrinth Seal holder is significantly lower than that from the Sleeve holders. The mean NFDPM yield from the Labyrinth Seal holder is significantly lower than that from the Sleeve holders.

8.3.2.4 FCSA Data – Between Laboratory Variability

These data are shown in Table 29.

Table 29
Values of V (Laboratory 2 omitted)

	Holder	NFDPM	Nicotine
Labyrinth Seal 9 mm	A	2.9	0.18
Sleeve 9 mm	C	2.2	0.16
Sleeve 13 mm	D	2.0	0.16
Sleeve 15 mm	E	2.4	0.18
Test piece	M	1.2	0.15

The Labyrinth Seal holder produces more variable data when used to smoke 5.2 mm FCSAs.

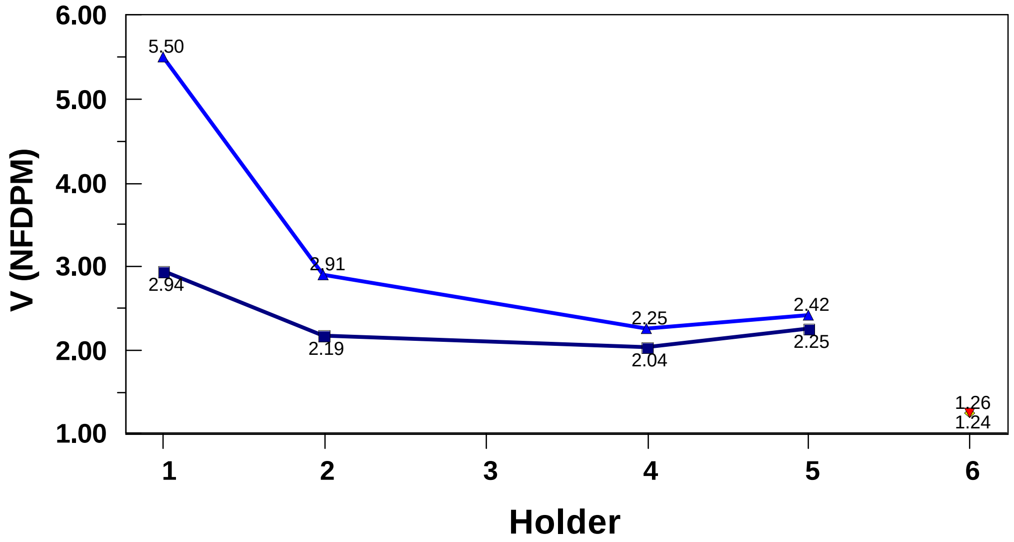
In terms of V for NFDPM, when smoking 5.2 mm FCSAs, the data from the Labyrinth Seal holder is more variable than that from any of the Sleeve holders. The Sleeve holder using 13 mm insertion depth is less variable than the other Sleeve holders.

In terms of V for nicotine, when smoking 5.2 mm FCSAs, there is little difference between the Labyrinth Seal holder and any of the Sleeve variants although the 9 mm and 13 mm Sleeve holders give the lowest values.

These data are shown graphically below. The graph also shows the values for V for the 7.2 mm diameter articles. In the following graphs, the upper line and the points identified with a triangle are from the 7.2 mm FCSAs whilst the lower line is from the 5.2 mm FCSAs. It is also worth noting that, in some instances there are two points for the monitor data. This is because only 1 laboratory data set was removed from the 5.2 mm FCSA data, whereas, 2 laboratory data sets were removed from the 7.2 mm FCSA data. Thus, the mean value of the monitor may not be the same for the 5.2 mm study and the 7.2 mm study.

Study 16. Plot of V for NFDPM

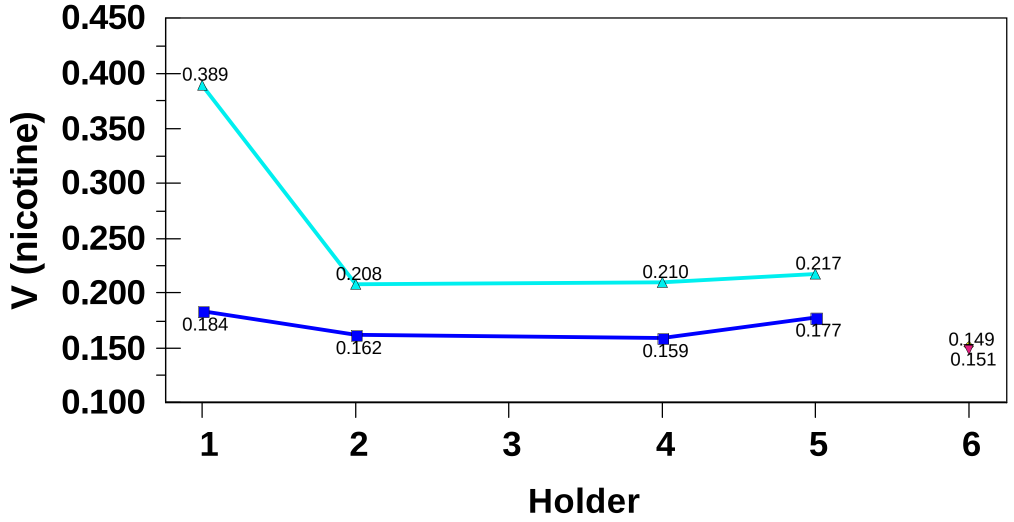
Outliers removed. Outlying laboratories omitted.



1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = C

Study 16. Plot of V for Nicotine

Outliers removed. Outlying laboratories omitted.



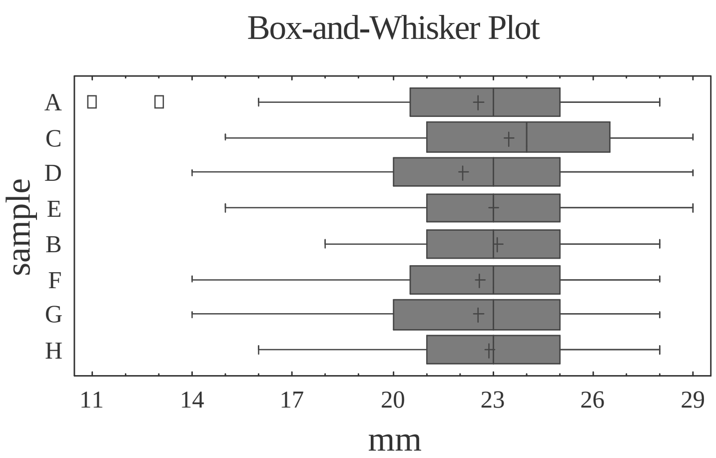
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 =

8.4 Distance from the Butt mark to the Insertion Depth

The distance between the butt-mark and the front of the holder is important when smoking FCSAs because of the variable burning quality if FCSAs. When performing Study 16, one laboratory made an attempt to determine the required “safety zone”. A linear smoking machine was used in this laboratory.

The FCSAs from each holder-type were regarded as a group. On all smoking runs the groups of FCSAs were monitored separately and they were first extinguished when the smoking procedure of the last FCSA in that group was terminated. This led to one butt with a length of approximately 29 mm and 3 others with shorter butt lengths that depended on the speed of burn. Theoretically, 100 butts should have been collected in each group (a total of 800). However, if it was obvious that the coal would burn into the holder, or extremely close to it, before the last FCSA in a group was terminated, it was extinguished. As a result a total of 798 butts were collected.

The data are shown as a “box and whisker” plot of the tobacco butt remaining after coal extinction.



The right hand side represents the actual butt length of 29 mm, or what was measured, whilst the left side shows the resulting butt length of the quickest burning FCSA in each group. The two extreme values of the A-group are not considered to be outliers.

The data are also shown in Table 30

Table 30
Length of tobacco burned

Holder type	FCSA diameter	Length of tobacco burned by the fastest burning FCSA	
		Maximum	Mean
A (Labyrinth Seal 9 mm)	5.2	18	6.4
C (Sleeve 9 mm)	5.2	14	6.6
D (Sleeve 13 mm)	5.2	15	6.9
E (Sleeve 15 mm)	5.2	14	5.8
B (Labyrinth Seal 9 mm)	7.2	11	5.8
F (Sleeve 9 mm)	7.2	15	6.4
G (Sleeve 13 mm)	7.2	15	6.4
H (Sleeve 15 mm)	7.2	13	6.1

Data for the maximum amount of tobacco burned after the butt mark suggest that an operator will have to take extreme care to ensure that damage the latex holding the FCSA does not occur. Certainly the "safety zone" allowed in ISO 4387:1991, i.e. 14 mm, for manufactured cigarettes must, at least, be maintained for a smoking standard for FCSAs. This conclusion, reached by experimentation, was strongly supported by comments from other laboratory personnel who took part in Study 16.

8.5 Classification of wrappers (Studies 10 & 13)

8.5.1 Study 10

Study 10 was established to produce a simple method that could be used to classify papers into two main groups that would correlate with the yield of NFDPM. The European Cigarette Paper Converting Industry proposed a combustion method known as the Louisville Combustibility Test (LCT). Nine papers were tested:

A typical UK paper	UK
As above but perforated	UKPer
A typical Dutch paper	NL
As above before perforation	NLNP
A low porosity paper from an Austrian supplier	WAT1
A high porosity paper from an Austrian supplier	WAT2
A typical French paper	F
A typical Canadian paper	CND
A typical German paper	D

As in other studies, a single blend of tobacco was used and the fine-cut smoking articles were made using 750 mg tobacco at 7.2 mm diameter and 70 mm paper length. For this study, 60 (3 x 20) FCSAs only were smoked compared with the normal 100 used previously. Just 5 laboratories performed the smoking tests. Eight laboratories provided data from a series of paper tests; these were the Louisville Combustibility Test (LCT), the static burn test, a suction test and air permeability.

The suction test was too variable to be of any value and the data were not analysed further. Fine-cut smoking articles made with the French paper self-extinguished and could not be included in the analysis for those tests that required smoking or smouldering. For the papers tested, the Louisville Combustibility Test was able to separate the papers into two distinct groups. The data from the LCT are given in Table 31.

Table 31
Louisville Combustibility Test results
(seconds)

Laboratory Paper	1	2	3	4	5	6	7	8	Paper Mean	Standard Deviation
UK	56.0	56.8	55.0	60.9	64.5	63.5	68.4	55.9	60.1	5.0
UKPer	67.4	70.8	71.5	74.8	75.9	72.4	83.3	65.1	72.7	5.6
NL	33.5	37.9	43.1	42.9	41.6	40.9	45.9	35.2	40.1	4.2
NLNP	34.3	37.7	41.2	42.7	39.9	42.5	46.3	34.5	39.9	4.2
WAT1	32.2	43.2	41.5	39.6	43.4	38.7	42.6	36.0	39.6	3.9
WAT2	47.3	61.8	64.0	56.1	68.3	61.7	63.8	57.7	60.1	6.4
F	Did not burn under the conditions of the test.									
CND	59.1	59.0	65.2	64.0	66.6	66.7	66.7	59.5	63.3	3.5
D	31.5	36.3	34.9	39.7	36.0	40.1	51.6	31.1	37.6	6.5

The table shows large differences between laboratories and this was of concern to the Task Force. Additionally, the papers that were tested tended to congregate at the extremes of the range. It was therefore decided to standardise further the LCT test used by the laboratories and to investigate how the test performs on papers near the middle of the combustibility range.

8.5.2 Study 13

Although it was known that the air permeability on its own is not a good indicator of NFDPM yield, it was decided to examine the permeability as well as the LCT in an attempt to determine if there is a simple relationship between these two parameters that will predict NFDPM.

The objectives of Study 13 were:

- To evaluate a range of fine-cut smoking articles made with different wrappers for yields of NFDPM and nicotine.
- To investigate further, the relationships between these paper parameters and smoke yields.
- To determine whether one or more test procedures can be used in a simple manner to classify papers into two categories.

A revised protocol, based on that used for Study 10, was developed for the application of the LCT method and a large number of laboratories performed this test and also determined air-permeability by the standard CORESTA method.

Seven wrappers were investigated:

1. NL : Typical Dutch paper (also in Study 10)
2. D : Typical German paper (also in Study 10)
3. UK : Typical UK paper (also in Study 10)
4. CND : Typical Canadian paper (also in Study 10)
5. WAT3 : Test Paper 3: 20-25 CU, no burn additive
6. WAT4 : Test Paper 4: 10 CU, up to 2% burn additive
7. WAT5 : Test Paper 5: Mid range base porosity, with additional perforation

Fine-cut smoking articles were made using a blend of fine-cut tobacco previously used in other studies. Eight laboratories conducted smoking tests, 12 laboratories the Louisville Combustibility Tests and 10 the air permeability at 0.25 kPa and 1 kPa. In all, 16 laboratories were involved in some part of the analysis.

Two statisticians examined the data independently. A full report of this study is given in Appendix 10.

Statistician 1 used a stepwise multiple regression procedure. This analysis resulted in regressions for NFDPM, nicotine and puff number fitted on 56 observations, $R^2 = 0.89$.

The equations generated were used to 'predict' the outcome for NFDPM, nicotine and puff number for a combination of paper parameters. The predictions for NFDPM and nicotine are shown in Table 32.

Table 32
Predicted Values for NFDPM and nicotine by LCT and air permeability

LCT (sec.)	Air Permeability (CU)	NFDPM (mg)	Nicotine (mg)
40	5	15.7	1.29
40	60	13.6	1.16
60	5	17.5	1.41
60	60	15.3	1.28
80	5	19.3	1.52
80	60	17.1	1.39

This analysis suggests that data from the LCT may be an important factor in the prediction of NFDPM and nicotine yields for the papers used in this study. However, the above table also shows that data from an air permeability test (in particular air permeability measured at 1 k Pa) are also required.

Given the ability of a system based on two parameters to predict NFDPM or nicotine, it is important to determine how much worse the predictions become if one parameter, e.g. air permeability, is ignored.

In terms of variation, loss of information on air permeability of the paper increased the unexplained variation ($1-R^2$) by 50% (from 0.13 to 0.20 i.e. from 13% to 20%). What is more relevant is the effect on individual papers as measured by the average residual across laboratories. See Table 33.

Table 33
Average residuals for NFDPM under 2 models

	Average Residual Under Regression	
Paper	With Air Permeability	Without Air Permeability
WAT3	0.20	0.54
WAT4	-0.33	0.62
WAT5	-0.58	-1.66
D	0.24	0.35
NL	0.52	-0.04
UK	0.19	0.05
CND	-0.24	0.14

WAT5 is poorly specified under the regression that excludes air permeability. All eight laboratories obtained a negative residual for this paper (ranging from -0.21 to -3.66). This demonstrates that, for these data, air permeability must be included.

Statistician 2 first examined the data for outliers. The tables of means with the outlying values removed provided the data for the regression analysis of the smoke data. These regressions show that, based on overall means of the data, neither of the single paper parameters can be used on its own but that there is a clear relationship between the smoke data (NFDPM and nicotine) and the paper data combined (Permeability and LCT).

It is tempting to believe that the regression equations derived from these data could be used as general predictive equations for smoke yield if the values of Permeability and LCT were known for any paper. This is most unlikely to be the case. The report contains tables that show the results of using these regressions to predict the NFDPM and nicotine data from the paper measurements for the laboratories that participated in this study.

The difference in the sign of ERROR in passing from one laboratory to another, coupled with the magnitude of the ERROR, shows that, even for the laboratories that participated in this study, the predictive equations perform poorly. It would be expected that if these predictors were to be used by laboratories that did not participate in this study then their performance is likely to be worse.

The poor performance of the predictive equations is due to the fact that for this study there was considerable variation between the laboratories for both the paper measurements and the determination of smoke yields.

Since the data from one paper-based test alone are not sufficient to classify paper for constituent yields, it must be concluded that the classification should be done using another procedure. A direct method based on the determination of smoke yields is more appropriate than an indirect method and based on these data, the Task Force agreed that a direct method would be used to classify wrappers as A or B as required for the matrix described in Section 4. Thus it is proposed that wrappers will be tested in a standard manner to determine the yield of NFDPM and to compare this value with those obtained using two specimen wrappers.

However, the statistical studies carried out in this study demonstrate that the LCT method has potential as a quality control tool for paper wrappers. This is confirmed in Table 34 where the LCT results are shown by Paper Type and by Laboratory.

Table 34
Louisville Combustibility Test by paper type and laboratory
(secs)

Paper type	D	WAT4	NL	WAT5	WAT3	CND	UK
Lab 1	45.6	52.0	52.2	53.3	59.2	68.6	81.9
Lab 2	42.0	48.5	50.7	53.6	55.4	69.8	78.3
Lab 3	48.5	52.7	54.1	54.7	62.8	70.5	76.5
Lab 4	44.4	57.8	55.2	59.8	62.7	69.8	72.7
Lab 5a	44.8	52.8	54.1	55.5	60.3	70.0	76.5
Lab 5b	44.3	48.8	50.1	51.4	58.6	66.7	73.6
Lab 6	38.8	44.0	47.7	44.1	46.1	56.5	63.8
Lab 7	45.6	51.7	56.7	53.1	58.5	71.0	82.3
Lab 8	41.8	47.8	48.2	51.9	55.8	61.2	74.6
Lab 9	51.5	55.9	58.7	59.2	64.5	76.4	84.0
Lab 10	48.8	34.4	47.1	47.8	40.2	84.6	70.5
Lab 11	44.2	50.3	53.5	56.0	58.8	71.0	86.2
Overall	45.0	49.7	52.4	53.4	56.9	69.7	76.7

The Task Force recommended that further work be undertaken to develop the LCT method so that it can be used as a quality control tool within the paper and paper converting industries.

8.6 The Making Device

Early in the course of this work it became apparent that the success of the method would be very dependent on the development of a device capable of making fine-cut smoking articles. An investigation of the devices available on the market revealed only those normally used for personal use by consumers. Only one device, an instrument made in Canada, was robust enough to be used in a laboratory situation. As this device was not available to handle tubes of the correct diameter for the CORESTA studies, one of the fine-cut tobacco manufacturers undertook the modification to produce machines capable of tubing at 5.2 and 7.2 mm diameters.

An approach was made to the Canadian company to see if they were able to undertake the manufacture. Unfortunately, they were not.

A leading manufacturer of wrapper tubes undertook to produce an automatic laboratory device capable of making FCSAs at both diameters and at a "reasonable speed". They commenced a major development involving an integral balance and operating on a principle of rolling before tubing. This would simulate, as closely as possible, the action of a smoker making an FCSA. The trials demonstrated that the short-term reproducibility was satisfactory but the maintenance required to keep it operational was excessive. After several months of development the machine was abandoned.

The same company was then asked to produce a simple device based on the commercially available design. This development was completed late in 1998 and the device was made available in quantity for the first time for laboratories to use in Study 16. Two machines are available, one at 5.2 mm and one at 7.2 mm.

These machines are now endorsed by CORESTA and can be ordered from equipment suppliers.

8.7 Articles made using pre-made filter tubes (Study 11)

8.7.1 Consumer Study

Studies were performed to estimate the making behaviour of consumers who use pre-made filter tubes to make fine-cut smoking articles.

The use of pre-made filter tubes is most common in Canada, Germany, Sweden and Denmark. A survey was conducted in Sweden, Denmark and Germany in 1995. 210 respondents were recruited, each making approximately 20 fine-cut smoking articles (4204 fine-cut smoking articles were collected).
The data shown in Table 35 were determined.

Table 35
Weights of tobacco used with pre-made filter tubes
(mg)

	Denmark	Germany	Sweden
Relative humidity	75%	70%	75%
Average weight mg	929	964	990
Range by respondent	751-1235	516-1402	686-1337
Range - all articles		248-1650	506-1489

8.7.2 Study 11

A study (Study 11) was established to investigate the effect of packing density on smoke yields when fine-cut tobacco is used with different pre-made filter tubes. Eight laboratories took part. Five smoking runs (i.e. 100 FCSAs) were completed for each of 6 products. The study comprised a fully factorial 2 (filter tubes) x 3 (densities) design, with pre-made filter tubes A (a non-ventilated tube) and B (a ventilated tube) packed at densities of 220, 260 and 300 mg/cm^3. The 8 laboratories were split evenly between users of 20-channel rotary smoking machines and users of 20 channel linear machines. The tubes were 84 mm long with a filter length of 15 mm. The full report is given as Appendix 11.

Two statisticians examined the data independently. The two modes of analysis produced virtually identical outcomes. Table 36 provides averages for NFDPM across all laboratories for both filter tubes at each density. This shows that with tube A, NFDPM increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With tube B there is no significant density effect.

Table 36
Mean NFDPM across all laboratories
(mg/piece)

	Density (mg/cm^3)		
Filter tube	220	260	300
A	15.2	17.6	18.6
B	10.4	11.1	10.9

Table 37 shows the averages for nicotine in smoke across all laboratories for both filter tubes at each density. This shows that for tube A, nicotine increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With tube B there is little evidence of a density effect.

Table 37
Mean nicotine across laboratories
(mg/piece)

	Density (mg/cm^3)		
Filter tube	220	260	300
A	1.33	1.60	1.73
B	1.10	1.13	1.13

From the data it is clear that a tubed product can be tested at two densities and that densities of 220 and 300 mg/cm^3 are appropriate. However, with highly ventilated tubes it is possible that there will be very little difference in yield as a result of the two densities. In these circumstances, it is recommended that a single figure, based on the average, be used to inform the consumer. The Task Force concluded that a single figure should be quoted if the difference in yields is less than 15% or where the difference in yields is less than 1 mg NFDPM or less than 0.1 mg nicotine.

These findings will be used in the compilation of the experimental method. It is proposed that the method will be based on any tobacco sold where there is no indication what wrapper to use. However, when there is an indication that the tobacco should be used with a particular pre-made tube, it is recommended that additional information be generated with the particular wrapper.

References

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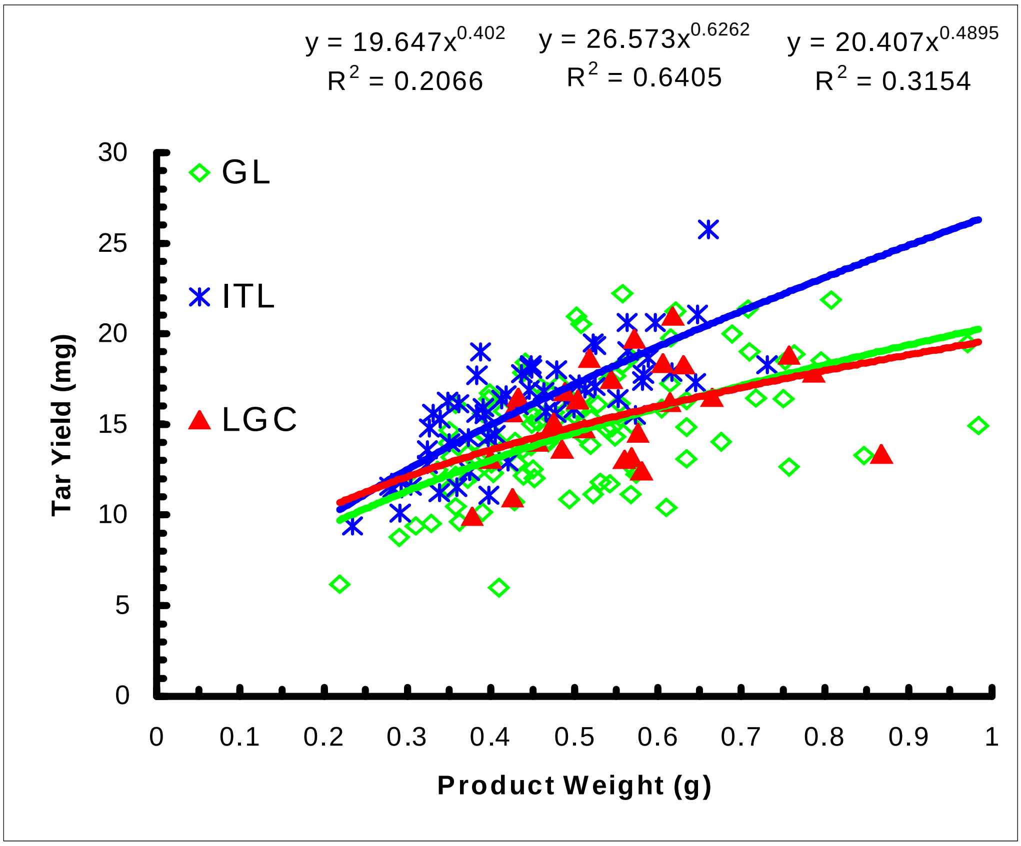
10 Memorandum from the secretariat - Measurements of toxin levels in hand-rolling tobaccos. National Council on Tobacco and Health, Norway. August 1994.

11 Dymond, H.F and Camm, B.J. Measurement of density along the length of fine-cut smoking articles made by various methods. CORESTA Information Bulletin, Yokohama 1996.

12 CORESTA Report 91-1, CORESTA Information Bulletin 91-3 Special Smoking Methods.

Figure 1 Comparison of UK Study data



Appendix 1. List of meetings held

Meeting	Date	Place
Set-up	07.11.1989	Bristol UK
1	17.01.1990	London UK
2	19.04.1990	Düsseldorf Germany
3	25.09.1990	Amsterdam The Netherlands
4	26.03.1991	Innsbruck Austria
5	08.05.1991	Amsterdam The Netherlands
6	20.06.1991	Düsseldorf Germany
7	30.01.1992	London UK
8	02.06.1992	Paris France
9	24.11.1992	Luxembourg
10	16.03.1993	Bonn Germany
11	07.09.1993	Bristol UK
12	01.03.1994	Landshut Germany
13	07.09.1994	Rotterdam The Netherlands
14	17.01.1995	Munich Germany
15	10.05.1995	London UK
16	04.09.1995	Paris France
17	17.01.1996	Brussels Belgium
18	23.04.1996	Barcelona Spain
19	24.07.1996	Innsbruck Austria
20	23.10.1996	Amsterdam The Netherlands
21	12.02.1997	Paris France
22	15.04.1997	Stockholm Sweden
23	25.06.1997	Copenhagen Denmark
24	04.09.1997	Frankfurt Germany
25	23.04.1998	Brussels Belgium
26	15.06.1998	London UK
27	18.08.1998	Brussels Belgium
28	30.09.1998	Brussels Belgium
29	02.12.1998	Brussels Belgium
30	06.05.1999	Brussels Belgium
31	07.06.1999	Brussels Belgium

Appendix 2. List of Co-operative Studies

No.	Date	No. of Laboratories	Study
1	1990	Mostly one	Pilot studies involving weight, diameter, paper type, making method,
2	1990	3 Laboratories per study	Parameter studies - 5 sub studies Weight and density Tubing vs. hand-rolling Effect of paper overlap Effect of tobacco blend Paper porosity and burn additives
3a 3b	1992	8 6	Interaction of density (3 levels) and paper porosity (2 levels) Effect of citrate in paper (2 loadings) at different porosities
4	1994	7	Effect of moisture content on yields
5	1994	5	Physical dimensions of consumer made articles - Made as a result of giving them instructions
6	1994	7	Effect of moisture content on yields
7	1994	9	Effect of moisture content on yields
8	1995	9	Influence of density and diameter
9	1995	9	Paper and density
10	1996	8	Various tests to attempt to classify paper
11	1996	8	Study of density in pre-made filter tubes
12	1996	10	Fine-cut smoking article holder
13	1996	9 Smoking 12 Paper tests	Paper classification
14	1997	10	Fine-cut smoking article holder
15	1998	18	Fine-cut smoking article holder - Labyrinth Seal vs. Sleeve holder
16	1999	17	Fine-cut smoking article holder - Labyrinth Seal vs. Sleeve holder. 7.2 and 5.2 mm diameter holders

Appendix 3. Other Major Studies

Year	Study description
1994	Yield of consumer made products - 3 separate in-company studies
	Independent consumer research agency report on making parameters in The Netherlands and Germany
1995	Independent consumer research agency report on making parameters and tar and nicotine yields in The Netherlands
1995/1996	2 Company studies on consumer making practices in the UK
	Pilot studies on pre-formed filter tubes
1997	Independent consumer research agency report on making parameters and tar and nicotine yields in Germany

Appendix 4. Technical paper considering butt length presented by ESTA to the Task Force

The machine smoking of fine-cut smoking articles (Technical and other considerations)

Scope

This paper considers the factors which are pertinent to the machine smoking of hand-rolled smoking articles and other smoking articles made from fine-cut tobacco fabricated with the aid of a making device.

It does not consider the factors which constitute the sampling, conditioning, making or analysis of the condensate produced from smoking.

This paper assumes that the smoking parameters already described for the machine smoking of manufactured cigarettes can be applied to fine-cut smoking articles. i.e. puff volume 35 ml; puff duration 2 sec.; puff frequency 1 per min.

Background

In considering the smoking of fine-cut smoking articles it is reasonable firstly to consider the equipment and practices utilised in the smoking of manufactured cigarettes.

The holder for manufactured cigarettes is specified in ISO 3308 as follows:

> The standard cigarette holder shall cover 9 mm ±0.5 mm from the butt end of a cigarette and shall be impermeable to smoke components and to air. The standard cigarette holder shall ensure that the leakage between the cigarette and the cigarette holder is not greater than 0.5 % of the puff volume.
>
> Either the cigarette holder or the smoke trap shall be equipped with a perforated disc (washer) of plain expanded synthetic rubber, closed cell sponge grade, which partly obstructs the butt end of the cigarette. The synthetic rubber shall have a density of 150 kg/m^3 to 170 kg/m^3, low swell oil resistance and compression-deflection range of 35 kPa to 63 kPa. Four labyrinth seals shall be used; the one closest to the butt end (back seal) shall be reversed. The dimensions of the washer and labyrinth seals are given in figure 1. The washer shall be supported by a structure with a hole in its centre of 4 mm diameter. The assembly shall be constructed so that the cigarette shall be in contact with the washer when the cigarette is inserted to a depth of 9 mm from the sealing annulus of the front labyrinth seal.

Typical consumer practice in relation to the butt length of a manufactured product formed the basis for the original CORESTA / ISO methods and hence for the present International Standard ISO 4387:1991. This specifies the butt length as follows

The standard butt length to which cigarettes shall be marked shall be the greatest of the following three lengths:

- 23 mm
- length of filter + 8 mm
- length of overwrap + 3 mm

where the overwrap is defined as any wrapper applied to the mouth end of the cigarette and the length of the filter is defined as the total length of the cigarette minus the length of the tobacco portion.

Since the development of the cigarette smoking standard these butt lengths have been accepted as the norm for manufactured products.

Using this specification for butt length and the holder described in ISO 3308:1991, only minor problems have occurred with machine smoking of manufactured cigarettes and these have been confined to plain cigarettes.

In the early CORESTA studies on fine-cut smoking articles, some laboratories experienced difficulties using a cigarette holder containing labyrinth seals. In the recent CORESTA studies, examining products made at different diameters and different densities, the problems were very evident and many laboratories experienced considerable difficulty using the cigarette holder described in ISO 3308:1991. The problems are greatest when attempting to smoke products made at low packing density and / or low firmness or with a small diameter, or both. In many cases laboratories reported almost total failure to insert the product into the standard cigarette holder despite using the prescribed labyrinth seals. At the least, laboratories reported damage to the smoking article. In an attempt to overcome the problem a simple metal "sleeve insert" was developed which first penetrated the labyrinth seals allowing the fine-cut smoking article to be placed through the sleeve and into the seals without initial damage. The sleeves were then withdrawn to allow the labyrinth seals to grip the smoking article. Even using this device, some distortion of the product was apparent and leakage still occurred.

It is apparent that the design of a holder using 4 labyrinth seals is inappropriate for fine-cut smoking articles.

Discussion

The problems described above apply to products made in the laboratory using a making device and the products are as good as can be expected , and therefore the problems are the least that might be expected. As stated above, some problems are experienced when inserting plain manufactured cigarettes into the standard cigarette holder but it is clear that the problems associated with fine cut products are much more severe, even when dealing with smoking articles produced by a making device.

When considering an analytical method for fine-cut smoking articles in general, it should also be applicable to hand-rolled products made by consumers. This is evident from the studies recently conducted on behalf of official agencies in some European countries where smoking articles were produced by hand-rolling.

Because of the nature of fine-cut tobacco, consumers do not make smoking articles from it like plain manufactured cigarettes:

> Fine-cut tobacco is used at a much higher moisture content than that used for manufactured products and this results in a product which softens more than a manufactured cigarette at the mouth end during smoking.
> The tobacco is unevenly distributed through the length of the fine-cut smoking article giving rise to variable density and soft points along the length of the article.
>
> There is often poor packing at the end of the fine-cut smoking article.
>
> Consumers make products with a wide variety of shapes.

In summary, consumer making practices usually result in a very soft, often thinner, end that is frequently devoid of tobacco.

Clearly, the problems described above for laboratory made products are going to be even more apparent with consumer made products.

It is important to analyse the cigarette holder specification to understand which aspects might apply to a holder designed for the analysis of smoking articles made from fine-cut tobacco and which may be causing some of the problems listed above.

It is clear that a holder designed for use with smoking articles made from fine-cut tobacco should be impermeable to air and that the leakage should not be greater than 0.5 %;

However the remaining specification may not be relevant for the following reasons:

A. The perforated disc is only relevant to filtered products and is not relevant to hand rolled fine-cut smoking articles. Indeed the presence of the washer may cause distortion of the hand-rolled product which is generally softer than a manufactured cigarette.

B. The number of seals required to be used may be more than 4 in order to avoid leakage. Alternatively the method of sealing the smoking article should be more applicable to a softer, less even product.

C. The reason for reversing the fourth seal is to ensure a tight seal between the cigarette and the washer. Since there is no need for the washer there is no need to reverse the fourth seal. Indeed the use of a reversed seal is likely to cause damage to the fine-cut product.

D. In order to effect a good seal for asymmetric and / or uneven smoking articles, it is probable that the insertion depth of hand rolled smoking articles made from fine-cut tobacco will have to be greater than 9 mm.

As discussed above there are considerable differences between hand rolled smoking articles made from fine-cut tobaccos and manufactured cigarettes. These have an influence on the selection of a specific holder for fine-cut products.

In turn, these also have a major influence on the smoking process. In particular they give rise to uneven burning and differences in puff count for products of nominally the same specification.

In order to take these factors into account it was necessary to consider the options :

> Insertion depth / leakage. Either the product could be treated in some way to minimise the possibility of leakage or the insertion depth would have to be increased.
>
> The distance between the holder and the butt end of the product. This must be great enough to allow an operator to remove the product before the coal damages the seal. With a variable product the distance must be sufficient to allow for the smoulder effect during the inter-puff periods between the fastest and slowest burning products. For plain manufactured cigarettes, the distance is 14 mm. For filter cigarettes the distance is even greater depending on filter tip length. If a longer insertion depth were needed for smoking articles made from fine-cut tobacco, this would necessitate a longer distance between the holder and the butt end. (i.e. a longer butt length.)

When considering the butt length for smoking of a tobacco product, whether manufactured, tubed or hand-rolled, it is necessary to consider the technical difficulties involved in smoking the product on a mechanical smoking machine.

Experimental evidence

An early attempt was made to increase the number of labyrinth seals to 6. Whilst this resulted in reduced leakage the problem of insertion and damage remained.

The problem was put to one of the leading smoking machine manufacturers who were asked to design a holder which could be used on products as follows:

> Diameter from 5.2 mm upwards
>
> Irregular diameter and product shape
>
> Low packing density
>
> Ends devoid of tobacco, or at best at lower packing density than the rest of the smoking article
>
> Able to be used on existing smoking machines, both rotary and linear.

After careful research they recommended that an insertion depth of at least 15 mm was required in order to ensure leak-free smoking of such products. They produced a holder of a design shown in Figures 1 and 2.

This holder was tested by a laboratory experienced in the smoking of fine-cut products. The distance between the insertion depth and the butt mark was set at the minimum possible length to allow removal of the finished articles without damaging the seals. This distance was established as 12 mm.

Their data set is shown Table 1. Note:

This data set was produced using high tobacco weight and low paper porosity in order to produce data which would maximise any effect. At the same time any problems associated with the cigarette holder would be minimised.

Of necessity, the butt length using the "special" holder was 27 mm and for comparison the Labyrinth Seal holder was also used at a butt length of 27 mm.

In both cases, conditioning was to normal fine-cut moisture levels
(i.e. 22°C, 75% RH)

Table 1
Comparison of data from the "special" holder and a cigarette holder

HOLDER TYPE		Weight mg	Puffs	TPM mg	Water mg	Nicotine mg	NFDPM mg
LABYRINTH SEAL	Mean	1,045	12.1	21.1	2.37	1.42	17.3
	SD	5	0.5	3.1	0.96	0.16	2.2
	Max.	1,055	12.9	28.8	5.30	1.75	21.7
	Min	1,035	11.0	16.2	1.10	1.07	13.0
SPECIAL	Mean	1,051	11.9	24.3	3.63	1.52	19.2
	SD	5	0.6	1.9	0.69	0.13	1.4
	Max.	1,059	12.6	28.5	4.90	1.81	22.5
	Min	1,035	10.6	20.8	2.10	1.36	16.9

Concerning the "special" holders, the operators concluded that:

They observed no leakage.

They observed no distortion of the smoking articles.

Their use was "extremely practical" when compared with the Labyrinth Seal holder

The holder diameter was 55 mm. They considered that a 44 mm design would be preferable

Even at a 15 mm insertion depth and a high tobacco weight product, there was some drooping indicating that the insertion depth could not be reduced.

A minor technical difficulty was experienced with the "O" ring seal which could be overcome in a production batch.

It can be seen from the results, that the yields may be a little higher using the "special" style holder. This could be for two reasons:

1. The longer insertion depth is affecting the air dilution

2. There is less leakage using the "special" holder.

The wrapper used in the study was chosen to have a very low porosity in order to minimise the first possibility and it seems likely that reduced leakage is the main reason for the difference. Visual observation during the smoking process confirmed that there was less leakage.

Conclusions

Theoretical and experimental considerations both indicate that a longer insertion depth is required to achieve adequate smoking of fine-cut smoking articles compared with manufactured cigarettes.

A "special" style of holder has been developed which can accommodate designs of fine-cut smoking articles as small as 5.2 mm diameter; even smaller may be possible.

The holder can be used with both existing smoking machines without the need for conversion.

The "special" style holder can be used without distortion of the fine-cut smoking ` article.

An insertion depth of 15 mm would appear adequate.

At this insertion depth, a butt length of at least 27 mm will be required. (i.e. a tobacco smoulder length of just 12 mm compared with 14 mm allowed for manufactured cigarettes.)

Yields using this holder will be expected to be higher than using the holder designed for manufactured cigarettes (at an equivalent butt length).

A major advantage of the "special" holder is that the standard deviation for all data using it is considerably lower than that obtained using the cigarette holders.

Adoption of a holder specially designed for use with smoking articles, made using a hand held device and hand-rolled by consumers, will contribute greatly to the satisfactory smoking of these products. A holder is central to the development of a smoking method and if this holder is adopted for fine-cut smoking articles it will open up the development of the remainder of the analytical method.

Appendix 5. Measurement of density along the length of fine-cut smoking articles made by various methods.

H.F.Dymond and B.J.Camm

ABSTRACT

Smoking articles made from fine-cut tobacco are generally very soft compared to manufactured cigarettes. This leads to a number of problems when these articles are smoked using the holder described in CORESTA Recommended Method No. 22. The product is frequently damaged and even if this can be avoided, leakage from the labyrinth seals is readily apparent. As an initial step to designing a new holder for fine-cut smoking articles, density was measured at increments of 3 mm along the entire length of the fine-cut smoking article. Various methods were adopted to make the fine-cut smoking articles including hand rolling. The data showed that the end 10 - 15 mm of fine-cut smoking articles, however made, were very loosely filled, accounting for the fact that there was leakage with the labyrinth seal cigarette holder. The results clearly demonstrated that 9 mm is inadequate as an insertion depth for fine-cut smoking articles and that a minimum of 15 mm is required. In turn, this demonstrates that the butt length for smoking fine-cut smoking articles needs to be 27 mm.

BACKGROUND

In 1991 CORESTA produced Recommended Method No. 22 that gives the specifications, definitions and standard conditions for a routine analytical smoking machine for manufactured cigarettes. This Recommended Method includes a specification for the holders to be used with these machines.

The CORESTA Task Force on fine-cut tobacco (Roll Your Own) was inaugurated in October 1990. One of the objectives of the task force was to “establish the effects of materials and fine-cut smoking article design on nicotine free dry particulate matter (NFDPM) (“tar”) and nicotine yields of fine-cut smoking articles when smoked according to a standardised smoking regime”.

The early experiments, performed by the Task Force, demonstrated a number of problems associated with the holder and this was confirmed in detailed experiments performed during 1995 and 1996. At one of the Task Force meetings, two separate reports were presented both detailing difficulties due to leakage when using the standard holder described in CORESTA Recommended Method No. 22. One of the reports included a video recording showing very obvious leakage when using the holder. Simple experiments to try and overcome the leakage by increasing the number of labyrinth seals and increasing the insertion depth failed to resolve the problem.

A sub group was established reporting to the Task Force, with the objective to investigate the problems associated with the holder described in CORESTA Recommended Method No. 22 for fine-cut smoking articles and to make proposals for an alternative holder.

INTRODUCTION

Initial experiments confirmed the difficulties associated with the existing holder described in CORESTA Recommended Method No. 22 when used with fine-cut smoking articles.

The problems are greatest when attempting to smoke products made at low packing density and / or low firmness, or with a small diameter, or both. In many cases, laboratories reported almost total failure to insert the product into the standard cigarette holder despite using the prescribed labyrinth seals. At the least, laboratories reported damage to the smoking article. In an attempt to overcome the problem a simple metal “sleeve insert” was developed which first penetrated the labyrinth seals allowing the fine-cut smoking article to be placed through the sleeve and into the seals without initial damage. The sleeves were then withdrawn to allow the labyrinth seals to grip the smoking article. Even using this device, some distortion of the product was apparent and leakage still occurred.

Because of the nature of fine-cut tobacco, the fine-cut smoking articles made from it are not like plain manufactured cigarettes:

> Fine-cut tobacco is used at a much higher moisture content than that used for manufactured products and this results in a product that softens at the mouth end during smoking more than a manufactured cigarette.
>
> The tobacco is unevenly distributed through the length of the fine-cut smoking article giving rise to variable density and soft points along the length of the article.
>
> There is often poor packing at the end of the fine-cut smoking article.

The problems described above apply to products made in the laboratory using a making device and the products are as good as can be expected, and therefore the problems are the least that might be expected.

When considering an analytical method for fine-cut smoking articles in general, it should also be applicable to hand rolled products made by consumers. In addition to the problems described above, consumers make products with a wide variety of shapes.

In summary, consumer making practices usually result in a very soft, often thinner, end that is frequently devoid of tobacco. The problems described above for laboratory made products are even more apparent with consumer made products.

It is clear that the design of a holder using 4 labyrinth seals is inappropriate for fine-cut smoking articles.

EXPERIMENTAL PRINCIPLE

This problem of holding and sealing was explored by attempting to measure the density of the fine-cut smoking article along its length. In this way it was hoped it would be possible to determine at what insertion depth reasonable holding and sealing might be achieved.

A moisture and density microwave measurement system developed and patented by Tews-Electronik, Hamburg and distributed by Filtrona Instruments and Automation Ltd, as part of their quality testing module (QTM) range, was described at the CORESTA congress in Zimbabwe. The QTM module produced by Filtrona Instruments and Automation Ltd is equipped with a system to allow the tobacco rod to step sequentially in increments whilst the moisture and density measurements are made at each step. This offers a unique way of determining density along the length of a product in very small increments. Figure 1 is a reproduction of the figure shown by Barry Camm at the Zimbabwe conference and demonstrates the independence of moisture measurements with density. The particular figure shown also illustrates the dense ending effect used with some cigarette products.

Measurement of density of fine-cut smoking articles made in different ways:

This instrument was used to measure the density profile of a number of products made from fine-cut tobacco. Six different products were tested. These products were made in different ways as follows:

1. Hand-rolled by an experienced roller.
2. Hand-rolled by an experienced roller.
3. Made-using a box type making device.
4. Made using a tubing device, commonly used in Canada and adapted for use in CORESTA studies at 7.2 mm diameter.
5. Made using a tubing device, commonly used in Canada and adapted for use in CORESTA studies at 5.2 mm diameter.
6. Hand rolled by an inexperienced roller.

The fine-cut smoking articles were all made using the same tobacco and the same wrapper type. Twenty smoking articles were made of each design.

Five of each of the fine-cut smoking articles were taken at random and the density was measured at 3 mm increments along the length, with the first measurement being at 3mm from one end.

These results are shown in Figures 2 - 7. The Y axis shows the normalised density. This was produced by taking the individual values of density for the middle 36 mm of the product, determining the mean value and normalising all other values of density to this mean value. This enables different products to be compared.

The data show quite clearly that hand-rolling smokers with experience of hand-rolling tend to produce products that have 2 denser spots, presumed to be at the position at which the hand roller uses the thumb and forefinger to roll the product. The inexperienced hand roller, although still producing 2 peaks where the fingers and thumbs have been placed, has a much greater range of density throughout the product. The use of a box type making device shows no improvement over hand-rolling and again there is evidence of 2 denser spots at either end of the fine-cut smoking article. The 5.2 mm diameter product made with a tubing device used for the CORESTA studies showed a tendency for 3 peaks of density; 2 at each end and 1 in the middle. The 7.2 mm diameter product tends to show some uniformity of density in the middle 50 mm of the product.

Figure 8 shows the mean value of the 5 smoking articles for the 6 different products on common axes.

It can be seen from these graphs that the first 9 to 12 mm and the last 9 to 12 mm of fine-cut smoking articles made up by these 6 different procedures are extremely soft due to poor filling of the wrapper tubes.

Measurement of density of fine-cut smoking articles made using the same making device in different laboratories

In order to pursue this investigation further, 9 laboratories each made 100 fine-cut smoking articles using the device specified for making fine-cut smoking articles for the CORESTA studies. Articles were made at 7.2 mm diameter using the same tobacco blend and the same paper type as used previously. Companies were told the quantity of tobacco to be used with the making device (750 mg). They were also told to condition tobacco before making to 22º C / 75% RH. They then made a further 100 fine-cut smoking articles of 5.2 mm diameter using 400 mg of tobacco.

Looking at the data for the 7.2 mm diameter smoking articles it was seen that there were similarities. The data from just 3 laboratories are shown as examples. It can be seen from Figure 9 that company C produced a product where the density rises to a maximum near the middle of the product but that at least 15 mm at each end of the product is very much softer than the middle. Company E, on the other hand, produced a product that exhibits the tendency to produce 2 firm points, presumably where the fingers have been used to compress the tobacco into the hopper prior to the tubing action of the machine. It can be seen that the density is greatest at approximately 15 mm from the plunger end of the device. Once again however, it is clear that the first 9 to 12 mm of the product is very much softer than the remainder (Figure 10). The results from company I (Figure 11) show that they have produced a product that exhibits a much more uniform density across the entire product. Nevertheless, the density of the first and last 9 mm of the product, shows that there is much less tobacco present at the ends of the product.

An examination of the data for the 5.2 mm product shows that there is no consistency between the laboratories for this product compared with the 7.2 mm product. Looking at just 3 of the examples, Figures 12 shows the data for company A and demonstrates that the products made in this laboratory at 5.2 mm diameter are extremely irregular with the maximum density almost double that of the mean, being present at about 20 mm from the free end of the tube. There is a tendency for there to be 2 maxima, presumably where the fingers are used to compress the tobacco into the chamber. The data from company F, (Figure 13) show that the products made in this laboratory rise to maximum density at the middle of the product and in this case uniform density is not reached until 15 mm from the beginning of the product and again falls 10 mm before the plunger end of the product. The data from company H, (Figure 14) show that they are making a fairly consistent product and that the product reaches mean density after 10 mm but again begins to fall away from normalised density at approximately 10 mm from the plunger end.

The mean data from the 5 fine-cut smoking articles for each of the laboratories at 5.2 mm diameter are plotted in Figure 15 and the equivalent data at 7.2 mm diameter are plotted in Figure 16. From this it can be seen very clearly that the density along the length of the 7.2 mm product is marginally better than the mean density along the length of the 5.2 mm diameter product. The data show quite clearly that the density towards the plunger end of the making device is slightly higher than it is at the free end and that the length of “loosely filled” wrapper is slightly shorter at the plunger end. Nevertheless, with the 7.2 mm product the length at the plunger end is approximately 7 - 10 mm and at the free end approximately 12 mm, whereas with the 5.2 mm device the length is approximately 8 mm compared to almost 15 mm at the free end.

In order to put these data in context two machine-made, plain cigarettes of 70 mm length were put through the same process. These products were conditioned at 22º C / 60% RH, which is the standard for factory-made cigarettes. The data are shown individually in Figures 17 and 18 and show quite clearly that the 2 plain cigarette products show no tendency to produce any uneven filling at the ends of the products and that the spread of density along the length of the cigarette is very much smaller that the spread for fine-cut smoking articles. The normalised data for the mean of the 5 articles from the 2 products are shown in Figure 19 which demonstrate the above points quite clearly.

CONCLUSIONS

The data from the initial experiments with different making devices and the subsequent data using the same making device in different laboratories, show very clearly that the density at the end of a fine-cut smoking article, even when made by an experienced operator using the best available tubing device, is extremely low.

An outstanding feature of all of the measurements is that an insertion depth of 9 mm for fine-cut smoking articles is quite unrealistic and the problems encountered using the smoking machine holder described in CORESTA Recommended Method No. 22 are accounted for by this very soft product. This applies to smoking articles made on a making device, when articles made by consumers are analysed this problem will be greatly exaggerated.

It is apparent that a special holder needs to be constructed for fine-cut smoking articles to allow for this. It is quite clear that any holder design must seal effectively and uniformly over a minimum of 15 mm from the end of the product. This in turn has an effect on the butt length at smoking, since a minimum distance of 12 mm from the end of the holder to the butt termination device is required for practical purposes.

A new design of holder has been evaluated by the sub group that overcomes these problems.

NOTE: The figures referred to in this appendix are not attached. To obtain them, please contact the authors or CORESTA.

Appendix 6. Study 12

Study of a potential Holder for Fine-Cut Smoking Articles

Executive Summary

The objective of this study was to evaluate the holder developed by one supplier as a replacement for the current Labyrinth Seal holder.

Experiments in CORESTA, including video evidence, showed that the Labyrinth Seal holder, used for the smoking of manufactured cigarettes, is unsuitable for the smoking of fine-cut smoking articles. The ask Force engaged the services of the two major equipment suppliers to design a replacement holder.

Eleven laboratories were recruited to undertake five smoking runs each using the existing Labyrinth Seal and new Sleeve holders.

Two statisticians examined the data independently.

The entire data set is highly variable. For this reason no clear statement can be made in response to the original objective. It is recommended that this study be repeated as part of the continued development of a replacement for the existing ISO holder.

Objective

To evaluate the Sleeve holder developed as a replacement for existing Labyrinth Seal holder.

Background

Experiments in CORESTA, including video evidence, have shown that the ISO holder, used for the smoking of manufactured cigarettes, is unsuitable for the smoking of fine-cut smoking articles. The Task Force engaged the services of the two major equipment suppliers to design a replacement holder.

Protocol

A copy of the protocol as distributed to the participants is attached as Appendix 1.

Study Design

Eleven laboratories were recruited. Five smoking runs (i.e. 100 FCSAs) were completed each using the existing Labyrinth Seal and new Sleeve holders. Six laboratories used a 20 channel rotary smoking machine while five used a 20 channel linear machine.

Statistical Methods

Two statisticians examined the data independently. One statistician used stepwise regression and two-way analysis of variance, with holder type and laboratory (laboratory type) as main factors. Here particular attention was paid to laboratory x holder interactions, to investigate whether different laboratories, using different machines, interacted differently with the different holders. The other statistician used one way analysis of variance for outlier detection. The tabulated mean data were then analysed.

Results

Table 1 gives mean results (generally based on 100 FCSAs) by laboratory for puff number, NFDPM and nicotine. One laboratory manager decided to repeat the analysis, and the second set of data is given in Table 1. Because of the uncertain nature of the data from this laboratory, the analysis was repeated without these results. Since the conclusions were not materially affected either way, the results have been included.

Table 1
Average Results by Laboratory and Holder

LAB	Machine Type	NFDPM (mg)		Nicotine (mg)		Puffs	
		Labyrinth Seal	Sleeve	Labyrinth Seal	Sleeve	Labyrinth Seal	Sleeve
1	R	14.9	16.3	1.15	1.26	5.6	5.6
2	L	14.4	15.1	1.19	1.21	6.2	6.0
3	R	14.5	14.5	1.25	1.24	6.8	6.9
4	R	12.9	13.8	1.12	1.19	6.4	6.6
5	R	10.4	11.4	0.91	0.94	7.9	7.7
6	L	17.6	16.5	1.41	1.30	6.8	6.9
7	L	14.4	14.5	1.15	1.09	6.4	6.6
8	R	14.1	11.7	1.17	0.95	6.2	6.7
9	R	15.7	14.8	1.33	1.22	6.3	6.3
10	L	15.4	14.8	1.17	1.08	6.6	7.4
11	L	15.0	16.0	1.34	1.33	6.3	6.9
Grand Mean		14.5	14.5	1.20	1.17	6.5	6.7

Overall there is no evidence of any difference in the yields of NFDPM or nicotine between the two holders.

<u>NFDPM</u>

The range of NFDPM yields varies from a minimum of 10.4 mg to a maximum of 17.6 mg (i.e. 7.2 mg). Using the Labyrinth Seal holder the range is 10.4 to 17.6 (7.2 mg), and using the Sleeve holder the range is 11.4 to 16.5 (5.1 mg). The range using linear machines is 14.4 to 17.6 (3.2 mg), and using the rotary machines the range is 10.4 to 16.3 (5.9 mg). The difference between the two holders (Sleeve minus Labyrinth Seal) for each laboratory ranges from -2.4 to +1.4 mg. Both types of machine show negative and positive differences.

Nicotine

The range of nicotine yields recorded varies from a minimum of 0.91 mg to a maximum of 1.41 mg (0.50 mg). Using the Labyrinth Seal holder the range is 0.91 mg to 1.41 mg (0.50 mg), and using the Sleeve holder the range is 0.94 to 1.33 (0.39). Using linear machines the range is 1.08 mg to 1.41 mg (0.33 mg), and using the rotary machines the range is 0.91 mg to 1.33 mg (0.42 mg). The difference between the two holders (Labyrinth Seal minus Sleeve) for each laboratory ranges from -0.22 to +0.11 mg. Both types of machine show negative and positive differences.

Puffs

The range of puff numbers recorded varies from a minimum of 5.6 to a maximum of 7.9 (2.3 puffs). Using the Labyrinth Seal holder the range is 5.6 to 7.9 (2.3 puffs), and using the Sleeve holder the range is 5.6 to 7.7 (2.1 puffs). Using linear machines the range is 6.0 to 7.4 (1.4 puffs), and using the rotary machines the range is 5.6 to 7.9 (2.3 puffs). The difference between the two holders (Labyrinth Seal minus Sleeve) for each laboratory ranges from -0.2 to +0.8. Both types of machine show negative and positive differences.

Conclusions

It must be emphasised that the entire data set is highly variable. For this reason no clear statement can be made in response to the original objective. It is recommended that this study be repeated as part of the continued development of a replacement for the existing ISO holder.

Appendix 1 – protocol for Study 12

Tobacco	: Dutch blend
Tobacco-weight	: 750 mg
Diameter	: 7.2 mm
Paper	: Germany
Making device	: By DEVN, modified Premier-device for diameter 7.2 mm.
Butt length	: 27 mm

Holder	Insertion depth (mm)	Butt length (mm)	Code
Labyrinth Seal (4 seals + washer)	9	27	A
Sleeve holder : 99512 (14 mm) with sleeve 99514 (4.8 mm) Front seal 99518 (7.5 mm internal diameter) Rear seal 99516 (5.0 mm internal diameter)	15	27	B

*) See Annex A2

- Make and smoke the cigarettes according to the protocol used for Co-operative Study 9 (See Annex A1).

- There will be 2 samples (2 types of holders, 1 butt length and 1 type of paper).

- For each sample smoke 5 x 20 cigarettes.

- Results should be sent to:

Douwe Egberts Van Nelle Tob. Int.
Dept. Tabak/I.M.R./Research
Att.: A. de Vries
P.O. Box 3
8500 AA JOURE
Netherlands
Fax: ..31 - (0)513 - 488833

Before the 27th of September

Annex A1

1. PRODUCT SPECIFICATION

1.1 One level of relative humidity (RH) will be used in this study.

This will be: (75 ± 5) % RH.

2. SMOKING ARTICLE MAKING

2.1 The tobacco will be a brand produced in Holland taken from a single batch production, already conditioned, packed, and sealed, dispatched by the manufacturer (1 bundle, 10 packs each).

2.2 Before making, the tobacco will be conditioned at one moisture level. For this the tobacco must be unpacked and conditioned for 4 days at:

(22 ± 2) °C and (75 ± 5) % RH.

Keep the tobacco at this conditioning level until just before making the FCSAs. When removing the tobacco from conditioning for making the FCSAs, keep the tobacco, as much as possible, in a well sealed container to avoid moisture loss during making.

2.3 The test products shall be made from pre-formed tubes in order to minimise the total variability and to increase the sensitivity. The tubes will be provided and sent together with the tobacco. For tubing, a tubing device will also be sent to you.

2.4 Participating laboratories will be required to make their own FCSAs using tobacco, tubes and device supplied.

2.5 300 Tubes will be supplied to each participating laboratory.

2.6 From the conditioned tobacco for each holder at least 150 FCSAs should be made.

2.7 Weigh the amount of tobacco required (750 mg) for each FCSA. Carefully introduce this tobacco into the tubing device, spread the tobacco evenly over the total length of the chamber and press it carefully in place. Transfer the tobacco into the tube until it presses against the end of the tube. Remove the product from the tubing machine. Carefully cut off any tobacco hanging out of the ends. Mark the end fixed to the tubing device as the butt-end.

2.8 Store the FCSAs in well sealed containers to avoid the FCSAs drying out.

2.9 All FCSAs are restored at the conditioning level, 22 °C and 75 % RH, for 3 days.

2.10 After re-conditioning weight-select out of each lot by mean weight ± 20 mg. (See 3.5).

3. OPERATIONAL DETAILS

3.1 Select at random 1 pouch out of the bundle. Determine the moisture content preferably by using the Karl Fischer method according to ISO 6488 : 1992 rev.
If an alternative method is used, please specify.

3.2 Open the pouches and condition at (22 ± 2) °C and (75 ± 5) % RH (Tobacco before making, and FCSAs). This to be achieved by using a conditioning cabinet or as follows:

Mix a quantity (generally about 1 kg) of sodium chloride, NaCl, (of at least GPR quality) with water to form a slurry. Ensure that there is always an excess of free salt. Pour the slurry into a desiccator. Place the desiccator in a conditioning cabinet and allow the air to circulate over the slurry.

3.2.1 For those laboratories not able to comply with one of these procedures they should still attempt to control the temperature and RH and report the method used.

3.2.2 Determine the moisture content after conditioning.

3.3 All test products should be made “en bloc” in the participating laboratories.

3.4 Recondition the FCSAs at 22 °C and 75 % RH, for 3 days.

3.5 Select each lot of FCSAs for weight as follows:

Determine the weight of 100 FCSAs for each product, calculate the mean and select to ± 20 mg on individual pieces.

3.6 Prior to smoking perform the following tests on each batch of products.

3.6.1 Take 10 pieces of each sample and determine the Draw Resistance according to ISO 6565: 1983.

Do not use these smoking articles for smoking.

3.6.2 Take sufficient FCSAs to provide a value for moisture content prior to smoking. Preferably determine the water content using the Karl Fischer method according to ISO/DIS 6488:1994 rev. Repeat this test for each smoking run if possible. Otherwise analyse a sample of the Dutch Blend conditioned and stored under the same conditions as the FCSAs.

3.6.3 Report all results and the method used.

3.7 Maintain the conditions of the smoking room to comply with ISO 3402: 1991 i.e.

(20 ± 2) °C
(60 ± 5) % RH

3.8 Set up the smoking machine to comply with ISO 3308 : 1991. Pay particular attention to air flow control.

3.9 Smoke according to the procedures laid down in ISO 4387:1991.

3.9.1 Bring the conditioned FCSAs into the smoking room in sealed containers, open the containers just long enough to remove those FCSAs required for immediate smoking and re-seal the container until the next set of FCSAs is required.

3.9 Analyse the extract for nicotine using method ISO 10315:1991.

3.10 Analyse the extract for water using either method ISO 10362/1:1993 or method 10362/2:1994.

Appendix 7. Study 14

Study of a FCSA Holder

Executive Summary

The objective of this study was to evaluate the holder developed by one supplier as a replacement for the Labyrinth Seal holder, currently being used. Study 12 had been inconclusive as the variability between laboratories eclipsed any holder effect.

The following were studied.
1. Existing Labyrinth Seal holder, 9 mm depth of insertion, 23mm butt length.
2. Existing Labyrinth Seal holder, 9 mm depth of insertion, 27mm butt length.
3. New Sleeve holder, 5.5mm latex sleeve, 16mm depth of insertion, 27mm butt length.

Eleven laboratories were recruited to conduct smoking tests, each providing smoking data based on 100 FCSAs.

A second related study was conducted using a plain manufactured cigarette smoked under standard ISO conditions. Thirteen laboratories took part in this part of the study, including all eleven laboratories that participated in the first part.

Two statisticians examined the data independently. Both Statisticians separately examined Study 14 and Study 14-B, but after a discussion within the Task Force it was agreed that this report should be based on a combined analysis of the two parts.

For reasons described in the body of this report, estimates of within and between laboratory variation throughout this report are denoted v and V, to distinguish them from the formally determined r and R.

The values of v and V from this study when compared to r and R to be expected from manufactured cigarettes given in the CORESTA bulletin show that the within laboratory performance is at an acceptable level. However, it is the mean values between laboratories that give cause for concern.

Feedback from laboratory operators suggests a strong preference for a sleeve holder as opposed to a labyrinth seal holder.

Unfortunately, the important question of depth of insertion and/or butt length cannot be defined from this study.

It is recommended that a new study, with clearly defined objectives and measures of success, should be undertaken to provide more robust data. As many laboratories as possible should participate. Ideally this should be in the range of 12 - 16, split evenly between both types of smoking machine. Once the protocol has been agreed a meeting should be called with laboratory representatives to ensure that this protocol is fully understood and appreciated, and to underline the importance of following the protocol explicitly.
CORESTA Fine-Cut Tobacco Task Force

Objective

To evaluate the Sleeve holder as a replacement for the Labyrinth Seal holder, currently being used.

Background

Study 12, which had the same objective as this study was inconclusive as the variability between laboratories eclipsed any holder effect. Nevertheless, it was established that the existing Labyrinth Seal holder used to smoke manufactured cigarettes was inappropriate for fine-cut smoking articles due to the damage caused to the FCSAs during insertion.

Protocol

A copy of the Protocol as distributed to participants is attached as Appendix 1.

Study Design

As a result of Study 12 being inconclusive the design was modified. The following were studied in Study 14.

1. Existing Labyrinth Seal holder, 9 mm depth of insertion, 23 mm butt length.
2. Existing Labyrinth Seal holder, 9 mm depth of insertion, 27 mm butt length.
3. New Sleeve holder, 5.5 mm latex sleeve, 16 mm depth of insertion, 27 mm butt length.

FCSAs were made using Dutch blend of fine-cut tobacco with a German style paper, to a specification of 7.2mm diameter, 70mm length, 750mg tobacco weight conditioned at 75%RH. Eleven laboratories were recruited to conduct smoking tests, each providing smoking data based on 100 FCSAs.

A second related study (14B) was conducted using a plain manufactured cigarette smoked under standard ISO conditions, i.e. conditioning to 60% RH, standard Labyrinth Seal holder, 9 mm depth of insertion and 23 mm butt length. Thirteen laboratories took part in this part of the study, including all eleven laboratories that participated in the first part.

Statistical Methods

Two statisticians examined the data independently. Both statisticians separately examined Study 14 and Study 14-B, but after a discussion within the Task Force it was agreed that this report should be based on a combined analysis of the two parts.

Analysis by Statistician 1

Only those laboratories which participated in both studies are included. One laboratory which failed to abide by the protocol for Study 14B is excluded. A Principle Components Analysis on the NFDPM mean results for each of the 4 variants from each of the remaining 10 laboratories was run. Standardised scores from the first principle component were used to decide on outlier behaviour of the laboratories. If a laboratory was excluded, then the process was repeated. This was permitted up to a maximum of three times.

As a result of the first run one laboratory was clearly defined as an outlier behaviour (standardised score = - 3.21) and is therefore excluded. The PCA was rerun with 9 laboratories.

At the second run, a second laboratory was 'borderline' (standardised score = - 2.95) in terms of outlier behaviour. In view of the borderline nature, it was decided to examine what effect a decision to include/exclude this laboratory has on subsequent runs and overall conclusions. If this laboratory is retained in the analysis, estimates of repeatability are calculated on the basis of 9 laboratories (comprising 5 linear and 4 rotary smoking machines). If this laboratory is removed as an outlier and the PCA is re-run for the final time, a further laboratory is removed as an outlier (standardised score = -3.70). Thus the options are either to base the analysis of variability on 9 laboratories with just 7 laboratories. (8 laboratories are not an option and calculations based on this have not been included.)

The following tables for repeatability and reproducibility clearly show the degree of fragility in these results. On what is a very borderline decision (to include or exclude a laboratory at the second stage) very different conclusions would be reached. Based on 9 laboratories the Sleeve holder (with a 27 mm butt length) produces data with the lowest between laboratory variation. Based on 7 laboratories that conclusion is not supported: there is some evidence of lower variability using the ISO holder with a 27 mm butt length. However, conclusions based on 7 laboratories are unsatisfactory for several additional reasons and decisions based on such fragile results are unreliable.

(1) A minimum of eight laboratories are required by ISO 5725 for calculation of r & R.
(2) The final split of 5 linear and 2 rotary laboratories is unrepresentative.
(3) A total of 4 laboratories rejected out of a pool of 11 is excessive

Table 1:
"repeatability v" & "Reproducibility V" for NFDPM and Nicotine
Nine Laboratories Included

	NFPDM		Nicotine	
Sample	v	V	v	V
Labyrinth Seal Holder / 23 mm Butt length	1.7	5.2	0.13	0.40
Labyrinth Seal Holder / 27 mm Butt length	1.6	5.1	0.14	0.37
Sleeve Holder / 27 mm Butt length	1.7	2.5	0.14	0.29
Manufactured Plain cigarette / Labyrinth Seal Holder	1.1	4.0	0.10	0.24

Table 2:
"repeatability v" & "Reproducibility V" for NFDPM and Nicotine
Seven Laboratories Included

	NFDPM		Nicotine	
Sample	v	V	v	V
Labyrinth Seal Holder / 23mm Butt length	1.7	2.9	0.12	0.40
Labyrinth Seal Holder / 27mm Butt length	1.5	1.7	0.15	0.29
Sleeve Holder / 27mm Butt length	1.6	2.5	0.14	0.33
Manufactured Plain cigarette / Labyrinth Seal Holder	1.1	2.6	0.08	0.14

Analysis by Statistician 2

The first step in the analysis was to examine the individual data for outliers.
For each holder a one-way analysis of variance between laboratories was used to calculate the standardised residual for each data point. Any standardised residual equal to or greater than 2.0 was noted. The standardised residuals were also plotted against their normalised scores. Using this procedure 1, 5, 7 and 4 data points were deemed to be outliers for NFDPM, nicotine, water and puffs respectively.

For most of the subsequent analyses these data points were deleted; for some of the analyses the outliers were replaced by the cell means to preserve the balanced structure of the data set. The analyses where cell means have been used are annotated and the degrees of freedom adjusted to take account of this.

Taken overall, Grubbs tests suggest that the results from just one laboratory are outliers in this data set and its results were not included in any of the analyses.

For each sample one-way analyses of variance were carried out for both NFDPM and Nicotine to estimate the between-laboratory and the within-laboratory components of variance. These estimates were then used to calculate v and V.

The values of v and V derived from these analyses are shown in tables 3 and 4.

The objective of this experiment requires that we determine whether or not the use of a greater insertion depth, or the use of the Sleeve holder, would reduce the variability previously observed when using the Labyrinth Seal holder (23 mm insertion) for smoking FCSAs, and to compare the data from these holders when smoking FCSAs with that obtained from the smoking of a commercial cigarette.

If leakage previously observed is effecting the variability of the data, and if the alternative designs of holder have reduced leakage, then it would be expected that the within-laboratory variability should be correspondingly reduced.

Table 3.
Table of v (“repeatability” within laboratories)

	NFDPM	NIC
Labyrinth Seal/23 mm	1.62	0.125
Labyrinth Seal/27 mm	1.46	0.111
Sleeve/27 mm	1.62	0.135
Cigarette	1.18	0.077

[Note: The values of r to be expected from the smoking of a 12.5/1.00 commercial cigarette are 1.05 for NFDPM and 0.088 for nicotine. See CORESTA bulletin 1991-3].

The values of v shown in Table 3 suggest however that the within-laboratory variability has not been reduced by the use of the alternative designs of holder. There is some indication that the Labyrinth Seal/27 mm holder (v=1.46 for NFDPM, 0.111 for nicotine) gives slightly less variable results than the Labyrinth Seal/23 mm holder (v=1.62/0.125) or the Sleeve/27 mm holder (v=1.62/0.135). The effect, if it is real, is very small.

As might be expected, the values of v from the smoking of a commercial cigarette are lower than any of the corresponding values of v from the smoking of FCSAs. For those laboratories that used the LINEAR smoking machines the values of v for both NFDPM (1.02) and Nicotine (0.090) are very close to those that might be expected, based upon the data given in CORESTA Bulletin 1991-3. [The expected value of r for NFDPM=1.05; the expected value of r for nicotine =0.088]. The results from the laboratories that used the ROTARY smoking machines are rather higher than expectation for NFDPM (1.32) but lower than expectation for Nicotine (0.058).

The values of V shown in Table 4 indicate that the use of the alternative designs of holder does have a marked effect on the variability between laboratories. The values of V using the Labyrinth Seal/23 mm holder (V=5.02 for NFDPM and V=0.392 for nicotine are higher than those obtained when using the Labyrinth Seal/27 mm holder (4.61 for NFDPM, 0.332 for nicotine) and these in turn are higher than those obtained when using the Sleeve/27 mm holder (2.37 for NFDPM, 0.279 for nicotine).

Table 4. Table of V (“Reproducibility” within laboratories)

	NFDPM	NIC
Labyrinth Seal/23 mm	5.02	0.392
Labyrinth Seal/27 mm	4.61	0.332
Sleeve/27 mm	2.37	0.279
Cigarette	4.62	0.294

Inconsistent changes in yields of both NFDPM and Nicotine, as the laboratories participating in this study change from one holder to another, are reflected in the analyses of variance. This clearly indicates that the data obtained in this study contains large inconsistencies. Given the presence of interaction terms LABS x HOLDERS in all these analyses is highly significant whereas the MACHINES x HOLDERS interaction term is not significant. The fact that the LABS x HOLDERS interaction term is statistically significant (confidence level >99.9% in all analyses) shows quite clearly that the data contains large inconsistencies. Given these inconsistencies in the data it is impossible to come to any firm decision about the relative merits of the holders being used.

The values of v derived from the smoking of a commercially manufactured cigarette (v=1.18 for NFDPM and v=0.077 for Nicotine) are comparable with those that would have been expected from the information published in CORESTA Bulletin 1991-3. That bulletin shows that, in a study of 30 laboratories using the CORESTA Standard Methods governing the smoking of cigarettes, the values of r to be expected from the smoking of a cigarette delivering 12.5 mg NFDPM and 1.0 mg nicotine are r=1.05 for NFDPM and r=0.088 for nicotine.

The values of V derived from the smoking of a commercially manufactured cigarette (R=4.62 for NFDPM and R=0.294 for Nicotine) are larger than would have been expected from the information published in the CORESTA Bulletin which showed the values of R to be expected from the smoking of a cigarette delivering 12.5mg NFDPM and 1.0mg nicotine are R=1.69 for NFDPM and R=0.149 for nicotine.

The values of V derived from the data obtained from the smoking of a commercially manufactured cigarette in those laboratories that used LINEAR machines (R=1.99 for NFDPM, R=0.144 for nicotine) are comparable to the values predicted by the CORESTA study. The values of V derived from the data obtained from the smoking of a commercially manufactured cigarette in those laboratories that used ROTARY machines (V=4.02 for NFDPM, V=0.415 for nicotine) are however considerably higher than the values predicted by the CORESTA study.

[Note: The values of R to be expected from the smoking of a 12.5/1.00 commercial cigarette are 1.69 for NFDPM and 0.149 for nicotine. See CORESTA bulletin 1991-3]

Conclusions

The values of v and V from this study when compared to those to be expected from the CORESTA bulletin show that the within laboratory performance is at an acceptable level. However, it is the mean values between laboratories that give cause for concern.

Feedback from laboratory operators suggests a strong preference for a sleeve holder as opposed to a labyrinth seal holder. The important question of depth of insertion and/or butt length cannot be defined from this study.

Recommendation

After the laboratory representatives have made their recommendations, and such have been implemented, a study, with clearly defined objectives and measures of success, should be undertaken to provide more robust data. As many laboratories as possible should participate. Ideally this should be in the range of 12 - 16, split evenly between both types of smoking machine. Once the protocol has been agreed a meeting should be called with laboratory representatives to ensure that this protocol is fully understood and appreciated, and to underline the importance of following the protocol explicitly.

Appendix 1

PARTICIPANTS:

BAT Benelux
Douwe Egberts Van Nelle
Filtrona
Gallaher
ITL
Pöschl
SEITA
Swedish Match
Theodorus Niemeyer
Tiedemanns

Details Study No 14 :

Tobacco	: Dutch Blend
Tobacco-weight	: 750 mg
Diameter	: 7.2 mm
Paper	: German-style
Making device	: By DEVN, modified Premier-device for diameter 7.2 mm.
Butt length	: 23 mm and 27 mm

Holder	Insert depth (mm)	Butt length (mm)	Code
Labyrinth Seal (4 seals No 3 (6.5 to 7.44 mm) + washer)	9	23	A
Labyrinth Seal (4 seals No 3 (6.5 to 7.44 mm) + washer)	9	27	B
Sleeve holder: 99512 (14 mm) with sleeve (5.5 mm) Front seal 99518 (7.5 mm internal diameter) Rear seal 99518 (7.5 mm internal diameter)	15	27	C

- Make and smoke the cigarettes according to the protocol.
- There will be 3 samples (2 types of holders, 2 butt lengths and 1 type of paper).
- Smoke of each sample 5 x 20 cigarettes.
- Results should, before the 26th of January 1997, be send to:

Douwe Egberts Van Nelle Tob. Int.
Dept. Tabak/I.M.R./Research
Att.: A. de Vries
P.O. Box 3
8500 AA JOURE
Netherlands

Fax: ..31 - (0)513 - 488833

1. <u>PRODUCT SPECIFICATION</u>

1.1 One level of relative humidity (RH) will be used in this study.

1.1.1 This will be:

(75 ± 5) % RH

2. <u>SMOKING ARTICLE MAKING</u>

2.1 The tobacco will be a brand produced in Holland taken from a single batch production, already conditioned packed and sealed dispatched by the manufacturer (1 bundle, 10 packs each).

2.2 Before making, the tobacco will be conditioned at one moisture level. For this the tobacco must be unpacked and conditioned for 4 days at:

(22 ± 2) °C and (75 ± 5) % RH.

Keep the tobacco at this conditioning level until just before making the FCSA's. When removing the tobacco from conditioning for making the FCSAs, keep the tobacco, as much as possible, in a good sealed container to avoid moisture loss during making.

2.3 The test products shall be made from pre-formed tubes in order to minimise the total variability and to increase the sensitivity. The tubes will be provided and sent together with the tobacco. For tubing, a tubing device will also be sent to you.

2.4 Participating laboratories will be required to make their own FCSAs using tobacco, tubes and device supplied.

2.5 500 Tubes will be supplied to each participating laboratory.

2.6 From the conditioned tobacco for each holder at least 150 FCSAs should be made.

2.7 Weigh the amount of tobacco required (750 mg) for each FCSA. Carefully introduce this tobacco into the tubing device, spread the tobacco evenly over the total length of the chamber and press it carefully in place. Transfer the tobacco into the tube until it presses against the end of the tube. Remove the product from the tubing machine. Carefully cut off any tobacco hanging out of the ends. <u>Mark</u> <u>the end fixed to the tubing device as butt-end</u>.

2.8 Store the FCSAs in good sealed containers to avoid the FCSAs drying out.

2.9 All FCSAs are restored at the conditioning level, 22 °C and 75 % RH, for 3 days.

2.10 After re-conditioning weight-select out of each lot by mean weight ± 20 mg (See 3.5).

3. OPERATIONAL DETAILS

3.1 Select at random 1 pouch out of the bundle. Determine the moisture content preferably by using the Karl Fischer method according to ISO 6488 : 1992 . If an alternative method is used, please specify.

3.2 Open the pouches and condition at (22 ± 2) °C and (75 ± 5) % RH (Tobacco before making and FCSAs). This to be achieved by using a conditioning cabinet or as follows:

Mix a quantity (generally about 1 kg) of sodium chloride, NaCl, (of at least GPR quality) with water to form a slurry. Ensure that there is always an excess of free salt. Pour the slurry into a desiccator. Place the desiccator in a conditioning cabinet and allow the air to circulate over the slurry.

3.2.1 For those laboratories not able to comply with one of these procedures they should still attempt to control the temperature and RH and report the method used.

3.2.2 Determine the moisture content after conditioning.

3.3 All test products should be made "en bloc" in the participating laboratories.

3.4 Recondition the FCSA's at 22 °C and 75 % RH, for 3 days.

3.5 Select each lot of FCSAs for weight as follows:

Determine the weight of 100 FCSAs for each product, calculate the mean and select to ± 20 mg on individual pieces.

3.6 Prior to smoking perform the following tests on each batch of products.

3.6.1 Take 10 pieces of each sample and determine the Draw Resistance according to with ISO 6565 : 1983.

Do not use these smoking articles for smoking.

3.6.2 Take sufficient FCSAs to provide a value for moisture content prior to smoking. Preferably determine the water content using the Karl Fischer method according to ISO/DIS 6488 : 1994.

Repeat this test for each smoking run if possible. Otherwise analyse a sample of the Masterblend conditioned and stored under the same conditions as the FCSAs.

3.6.3 Report all results and the method used.

3.7 Maintain the conditions of the smoking room to comply with ISO 3402 : 1991 i.e.

(20 ± 2) °C

(60 ± 5) % RH

3.8 Set up the smoking machine to comply with ISO 3308 : 1991. Pay particular attention to air flow control.

3.9 Smoke according to the procedures laid down in ISO 4387 : 1991.

3.9.1 Bring the conditioned FCSAs into the smoking room in sealed containers, open the containers just long enough to remove those FCSAs required for immediate smoking and reseal the container until the next set of FCSAs is required.

3.9 Analyse the extract for nicotine using method ISO 10315 : 1991.

3.10 Analyse the extract for water using either method ISO 10362/1 : 1993 or method 10362/2 : 1994.

3.11 Please follow the smoking plans as mentioned in Appendix A1

Appendix 8. Study 15

Study of FCSA Holder Type and Insertion Depth

Executive Summary

The objective of this study was to determine the appropriate holder design and insertion depth required to smoke fine-cut smoking articles for the determination of NFDPM and nicotine in smoke. The results were evaluated primarily on the basis of within-laboratory variability.

The fine-cut smoking articles were all made in one location. The following were studied.

1. Existing Labyrinth Seal holder, 9 mm depth of insertion, 29 mm butt length.
2. Sleeve holder, 6.0 mm latex sleeve, 9 mm depth of insertion, 29 mm butt length.
3. Sleeve holder, 6.0 mm latex sleeve, 11 mm depth of insertion, 29 mm butt length.
4. Sleeve holder, 6.0 mm latex sleeve, 13 mm depth of insertion, 29 mm butt length.
5. Sleeve holder, 6.0 mm latex sleeve, 15 mm depth of insertion, 29 mm butt length.

FCSAs were made using a blend of fine-cut tobacco previously used (Dutch blend) with a German style paper, to a specification of 7.2 mm diameter, 70 mm length, 750 mg tobacco weight, conditioned at 75%RH. Eighteen laboratories were recruited to conduct smoking tests, each providing smoking data based on 100 FCSAs for each holder configuration. The method of smoking using a rotary smoking machine was modified to increase the sensitivity to variability. A butt length of 29 mm was used for all smoking of the fine-cut smoking articles to allow valid comparisons between holder configurations.

Since suspected leakage at the holder was one of the reasons for the design of the Sleeve holder, analysis of the puff numbers of individual fine-cut smoking articles was considered necessary in order to provide important insight into this aspect of the design of the holder.

One laboratory (number 7) was completely eliminated from the analysis because it was unable to follow the experimental protocol. The statisticians agreed the statistical approach to be applied. Details are given in the text. As a result of the outlier treatment, one laboratory (number 5) was removed.

Analysis of the data from the test piece indicates that the participating laboratories, after outlier removal, performed well during the course of this study. As the result of the basic statistical analysis (validation of data, outlier treatment), the values of within-laboratory variability were obtained and are shown below.

Values of v (based on 5 products per pad; laboratories 5 and 7 omitted)

	NFDPM	Nicotine
Labyrinth Seal 9 mm	3.0	0.21
Sleeve 9 mm	3.1	0.20
Sleeve 11 mm	2.9	0.18
Sleeve 13 mm	2.9	0.19
Sleeve 15 mm	2.5	0.18
Test piece	1.7	0.17

- There is a significant reduction in the variability of the NFDPM data as the insertion depth is increased ($p<0.01$).
- There is no significant reduction in the variability of the nicotine data as the insertion depth is increased ($p>0.05$).
- There is a highly significant reduction in the variability of the puff number data as the insertion depth is increased ($p<0.001$).

The prime focus of the analysis of this study was the variability within laboratories. It is however of interest to consider the mean values and variability between laboratories, as far as it is possible, considering the experimental design.

- There is a highly significant increase in mean NFDPM (by 1.8 mg; $p<0.001$).
- There is a highly significant increase in mean nicotine (by 0.08 mg; $p<0.001$).
- There is a highly significant decrease in mean puff number (by 0.24; $p<0.001$).

The values for NFDPM and nicotine are shown below.

Values of V (based on 5 products per pad; Laboratories 5 and 7 omitted)

	NFDPM	Nicotine
Labyrinth Seal 9 mm	5.0	0.32
Sleeve 9 mm	3.7	0.26
Sleeve 11 mm	3.7	0.25
Sleeve 13 mm	3.5	0.24
Sleeve 15 mm	3.4	0.24
Test piece	2.3	0.21

It can be seen that when considering both NFDPM and nicotine results, the value of V (an indicator of between laboratory variability) is at a maximum when the Labyrinth Seal holder is being used, whereas all versions of the Sleeve holder give lower values of V.

A further study, examining the effect of making FCSAs in the laboratory where the smoke analysis is performed, and making and smoking 5.2 mm diameter products, is recommended.

This would represent “near-normal” practice of the analysis of FCSAs.

Objective

The objective of this study was:

> To determine the appropriate holder design and insertion depth required to smoke fine-cut smoking articles for the determination of NFDPM and nicotine in smoke.

It was stated that the results would be evaluated primarily on the basis of within-laboratory variability. It was not the intention of this study to estimate the between-laboratory variability, and in particular, the data from this study would not form the basis of the calculation of r and R. This was not possible since the fine-cut smoking articles were made at one location and in the final procedure they will have to be produced by the laboratory that conducts the analysis.

Background

The holders used for the analysis of manufactured cigarettes are difficult to use with fine-cut smoking articles. There is also some evidence that they may be unsuitable for fine-cut smoking articles and give rise to greater variability of data.

Two previous studies (Study 10 and Study 14) had been performed in an attempt to demonstrate the advantages of a specially designed holder. Neither of these two studies proved completely satisfactory. A statistical evaluation of Study 10 was totally inconclusive. A statistical evaluation of Study 14 showed some evidence that the special holder gave improved data but it was not possible to draw definitive conclusions. It was recommended that a new study be performed with a larger number of participating laboratories, a more stringent study design and an improved statistical analysis.

This study was designed with these points in mind, looking particularly at the within-laboratory variability. In order to do this most efficiently, and in order to ensure the maximum sensitivity of the data with respect to the objective, the fine-cut smoking articles were all made at one location and the method of smoking using a rotary smoking machine was modified. For these two reasons, it is not possible to determine the final between-laboratory variability (R) and the objective stated above makes this clear. Nevertheless, it is possible to compute a value for the between-laboratory variability for this study. As the FCSAs were all made at one location this value is close to the true effect of the analytical method alone (i.e. omitting the effect of making). It is thus a good measure of the holder effect and is not unduly influenced by making variables. For this reason, the value must be expected to be better than the final value of R.

Protocol

A copy of the Protocol as distributed to participants is attached as Annex 1.

Study Design

As a result of Study 14 being inconclusive, the design was modified. The following were studied.

1. Existing Labyrinth Seal holder, 9 mm depth of insertion, 29mm butt length.
2. Sleeve holder, 6.0 mm latex sleeve, 9 mm depth of insertion, 29mm butt length.
3. Sleeve holder, 6.0 mm latex sleeve, 11 mm depth of insertion, 29mm butt length.
4. Sleeve holder, 6.0 mm latex sleeve, 13 mm depth of insertion, 29mm butt length.
5. Sleeve holder, 6.0 mm latex sleeve, 15 mm depth of insertion, 29mm butt length.

FCSAs were made using a blend of fine-cut tobacco previously used (Dutch blend) with a German style paper, to a specification of 7.2mm diameter, 70mm length, 750mg tobacco weight, conditioned at 75%RH. All the FCSAs were made at a single location and distributed to participating laboratories. Eighteen laboratories were recruited to conduct smoking tests, each providing smoking data based on 100 FCSAs for each holder configuration.

The CORESTA test piece was included to monitor the performance of the participating laboratories.

Statistical Methods

Outlier Treatment

For each laboratory, a one-way analysis of variance between and within holders was used as a basis for detecting outlying data points for both NFDPM and nicotine. Any data point whose standardised residual was > 3.0 was considered as a possible outlier. These datapoints were then referred to the originating laboratories to allow the data to be checked. Any points whose standardised residual remained > 3.0 after checking were deemed to be outliers and were removed from the analysis.

All the data from the laboratory coded as 7 were eliminated from the analysis since this laboratory was unable to follow the experimental protocol.

Table 0.1 shows a complete list of all the outliers removed from the data. The data (excluding Laboratory 7) contains 2040 records. There were 16 outliers for both NFDPM and nicotine, giving an error rate of 0.78%.

Test piece data

The test piece CM2, smoked through the Labyrinth Seal 9mm holder, was included in this study as a measure of the performance of the participating laboratories. Table 1.1 shows the results of a between and within laboratories analysis of variance of the NFDPM data. Table 1.2 shows a similar analysis of the nicotine data. From these tables it is clear that there is no outlying laboratory for this study.

Also shown in these tables are calculations of r and R. These values of r and R must be treated with caution. This study was not set up to estimate r and R. Furthermore, it must be remembered that the values of NFDPM and nicotine recorded in the data are based upon smoking 5 products through each filter pad, rather than 20, as is usually the case. In order to estimate v and V, the data were therefore pooled over four results to simulate the smoking of 20 products through each filter.

Nonetheless these values of v and V for NFDPM and nicotine indicate that the participating laboratories performed well during the course of this study. The values of r for NFDPM (1.010) and R for nicotine (0.161) are in fact somewhat less than the value to be expected from the information given in CORESTA Report 91/1. The values of R for NFDPM (1.952) and r for nicotine (0.161) are only slightly higher than the expected values.

These findings are summarised in Table 1.3, where the notation v and V has been used in place of r and R to emphasise the fact that this study was not set up to estimate r and R. The results are shown both before and after the pooling to simulate the smoking of 20 products per filter pad.

Variance stabilising transformation.

The measures of interest in this study are the standard deviations of the analytes. The various configurations of the holder were designed with the view to eliminating leakage, and it is believed that the elimination of leakage will lead to lower standard deviations.

The statistical techniques of analysis of variance rely upon the assumption that the data (or more strictly, the residuals after fitting main effects and interactions) are Normally distributed. It is a well-known fact that standard deviations are not Normally distributed.

A Box and Cox transformation y = (x^c)-1/c was chosen, with the parameter c = 0.25, where x is either observed NFDPM or observed nicotine.

The effect of this transformation is demonstrated (using the NFDPM data) in Diagrams 2.01 and 2.02. If data are Normally distributed, then a plot of the data versus the Normalised score of the data should show a straight line.

Diagram 2.01 shows a plot of SD(NFDPM) versus its Normalised score, and it can be seen that both extremes of the data are above the expected straight line, demonstrating curvature in the plot.

Diagram 2.02 shows a plot of transformed (SD (NFDPM)) versus its Normalised score, and now it can be seen that the plot is virtually straight, with no systematic deviations from expectation at either end of the line.

The same transformation was used for both NFDPM and nicotine.

4.0 The analysis of NFDPM

In this study the differences in smoke yields between laboratories and between smoking machines, are of no intrinsic interest.

These effects were therefore removed from the data and the residuals were then examined for any holder effects.

Having removed the effects of laboratories and smoking machines, a one-way analysis of variance (between and within holders) of the residuals showed a significant effect of the holders ($p<0.02$, 98%).

In terms of variability of NFDPM the holders were ranked in the order

Labyrinth Seal 9mm

Sleeve 9mm

Sleeve 11mm

Sleeve 13mm

Sleeve 15mm.

The Labyrinth Seal holder gave results with the largest variability and the Sleeve 15mm holder gave results with the lowest variability.

Pairwise comparisons of the holders showed that the differences between

Labyrinth Seal and Sleeve 13mm

Labyrinth Seal and Sleeve 15mm

Sleeve 9mm and Sleeve 15mm

Sleeve 11mm and Sleeve 15mm

were all significant (95% or greater). These results are shown in Table 2.1.

The differences between the various configurations of the Sleeve holder were also examined using regression analysis (omitting the data from the Labyrinth Seal 9mm holder). The results are shown in Table 2.2 where is can be seen that there is a significant reduction in the variability of the data as the insertion depth is increased (p=0.007, 99.3%). This trend is shown in Diagram 2.1. The point representing the Labyrinth Seal 9mm holder is included in this plot for completeness.

Although the prime measure of interest in this study is the variability of the data, the means of the data were also analysed following the same procedure as that used for the transformed standard deviations. The results are shown in Tables 2.3 and 2.4.

Table 2.3 shows that, in terms of mean NFDPM, the holders were ranked in the order

Sleeve 9mm

Labyrinth Seal 9mm

Sleeve 11mm

Sleeve 13mm

Sleeve 15mm.

The Sleeve 9mm holder gave the lowest mean and the Sleeve 15mm gave the highest mean.

Pairwise comparisons of the holders showed that the differences between

Labyrinth Seal and Sleeve 13mm

Labyrinth Seal and Sleeve 15mm

Sleeve 9mm and Sleeve 11mm

Sleeve 9mm and Sleeve 13mm

Sleeve 9mm and Sleeve 15mm

Sleeve 11mm and Sleeve 13mm

Sleeve 11mm and Sleeve 15mm

Sleeve 13mm and Sleeve 15mm

were all significant (95% or greater).

Table 2.4 shows the results of the regression analysis of the data from the Sleeve holders, and it can be seen that there is a highly significant increase in mean NFDPM as insertion depth is increased ($p<0.001$, >99.9%).

This trend is shown in Diagram 2.2. The point representing the Labyrinth Seal 9mm holder is included in this plot for completeness.

Analysis of nicotine

The analysis of the nicotine data followed the same procedure as that used for the NFDPM data. Having removed the effects of laboratories and smoking machines, a one-way analysis of variance (between and within holders) of the residuals showed no overall significant effect of the holders (p=0.151, 84.9%). In terms of variability of nicotine, the holders were ranked in the order

Labyrinth Seal 9mm

Sleeve 9mm

Sleeve 11mm

Sleeve 13mm

Sleeve 15mm.

The Labyrinth Seal holder gave results with the largest variability and the Sleeve 15mm holder gave results with the lowest variability. Pairwise comparisons of the holders showed that the differences between

Labyrinth Seal and Sleeve 13mm

Labyrinth Seal and Sleeve 15mm

were significant (95% or greater).

It must be remembered however that, taken overall, there was no significant holder effect on the variability of nicotine. These results are shown in Table 3.1

The differences between the various configurations of the Sleeve holder were also examined using regression analysis (omitting the data from the Labyrinth Seal 9mm holder). The results are shown in Table 3.2 where it can be seen that there is no significant reduction in the variability of the data as the insertion depth is increased (p=0.255, 74.5%). The data are shown in Diagram 3.1. The point representing the Labyrinth Seal 9mm holder is included in this plot for completeness.

As with NFDPM, the means of the data were also analysed following the same procedure. The results are shown in Tables 3.3 and 3.4.

Table 3.3 shows that, in terms of mean nicotine, the holders were ranked in the order

Sleeve 9mm

Labyrinth Seal 9mm

Sleeve 11mm

Sleeve 13mm

Sleeve 15mm.

The Sleeve 9mm holder gave the lowest mean and the Sleeve 15mm gave the highest mean.

Pairwise comparisons of the holders showed that the differences between

Labyrinth Seal and Sleeve 13mm

Labyrinth Seal and Sleeve 15mm

Sleeve 9mm and Sleeve 11mm

Sleeve 9mm and Sleeve 13mm

Sleeve 9mm and Sleeve 15mm

Sleeve 11mm and Sleeve 13mm

Sleeve 11mm and Sleeve 15mm

Sleeve 13mm and Sleeve 15mm

were all significant (95% or greater).

Table 3.4 shows the results of the regression analysis of the data from the Sleeve holders, and it can be seen that there is a highly significant increase in mean nicotine as insertion depth is increased ($p<0.001$, >99.9%).

This trend is shown in Diagram 3.2. The point representing the Labyrinth Seal 9mm holder is included in this plot for completeness.

Analysis of puff numbers.

In this study the individual puff number of every FCSA smoked was recorded. It is usual to record the mean puff number of those FCSAs smoked through a single filter-pad. By recording the individual puff numbers, the power of the study to detect any effect of the holder configurations was substantially improved.

There was however an issuewith the data. Diagram 4.0 shows a plot of the standard deviations of individual puff numbers versus their Normalised scores. It can be seen that although the lower half of the graph adheres reasonably well to the expected straight line, there is a marked deviation from linearity at the upper end of the data.

No variance stabilising transformation could be found to alleviate this problem.

The upper 10 data points were examined with a view to deciding whether or not they could be omitted from the analysis. Table 4.0 shows that 5 of these 10 points all came from the Labyrinth Seal holder, and it was therefore decided to retain these points in the analysis.

Although the distribution of the standard deviations of puff numbers gives cause for concern, the techniques of the analysis of variance are reasonably robust against non-Normality, especially if there is no indication of bi-modality in the distribution of the data. It was therefore decided to continue with the analysis. (This point will be referred to again later.)

The analytical procedure used was the same as that for NFDPM and nicotine. Having removed the effects of laboratories and smoking machines, a one-way analysis of variance (between and within holders) of the residuals showed a very significant effect of the holders ($p=0.000$, >99.9%). In terms of the standard deviations of individual puff numbers the holders were ranked in the order

Sleeve 9mm

Labyrinth Seal 9mm

Sleeve 11mm

Sleeve 13mm

Sleeve 15mm

The Sleeve 9mm gave results with the largest variability and the Sleeve 15mm holder gave results with the lowest variability.

Pairwise comparisons of the holders showed that the differences between

Labyrinth Seal and Sleeve 11mm

Labyrinth Seal and Sleeve 13mm

Labyrinth Seal and Sleeve 15mm

Sleeve 9mm and Sleeve 11mm

Sleeve 9mm and Sleeve 13mm

Sleeve 9mm and Sleeve 15mm

Sleeve 11mm and Sleeve 15mm

were significant (95% or greater). These results are shown in Table 4.1.

The differences between the various configurations of the Sleeve holder were also examined using regression analysis (omitting the data from the Labyrinth Seal 9mm holder). The results are shown in Table 4.2 where is can be seen that there is a highly significant reduction in the variability of the data as the insertion depth is increased ($p<0.001$, 99.9%). This trend is shown in Diagram 4.1. The point representing the Labyrinth Seal 9mm holder is included in this plot for completeness.

The fact that this analysis shows very highly significant effects of holder configuration on the standard deviation of individual puff numbers minimises any concerns about the non-Normality of the distribution of the data. Had the analysis produced marginally significant results then there would have been doubts about their reliability. The fact that the significance levels are so high removes these doubts.

The means of the individual puff numbers were also analysed following the same procedure. The results are shown in Tables 4.3 and 4.4.

Table 4.3 shows that, in terms of mean puff number, the holders were ranked in the order

Sleeve 9mm

Labyrinth Seal 9mm

Sleeve 11mm

Sleeve 13mm

Sleeve 15mm.

The Sleeve 9mm holder giving the highest mean puff number and the Sleeve 15mm giving the lowest mean puff number.

Pairwise comparisons of the holders showed that the differences between

Labyrinth Seal and Sleeve 13mm

Labyrinth Seal and Sleeve 15mm

Sleeve 9mm and Sleeve 11mm

Sleeve 9mm and Sleeve 13mm

Sleeve 9mm and Sleeve 15mm

Sleeve 11mm and Sleeve 13mm

Sleeve 11mm and Sleeve 15mm

Sleeve 13mm and Sleeve 15mm

were all significant (95% or greater).

Table 4.4 shows the results of the regression analysis of the mean puff numbers from the Sleeve holders, and it can be seen that there is a highly significant decrease in mean puff number as insertion depth is increased ($p<0.001$, >99.9%).

This trend is shown in Diagram 4.2. The point representing the Labyrinth Seal 9mm holder is included in this plot for completeness.

Variability between laboratories

The variability within laboratories is the prime focus of the analyses of this study. It is however of interest to consider the variability between laboratories. To that end, values of v and V (calculated in the same way as r and R) have been estimated and are shown in Tables 5.1 and 5.2.

It will be noted that the Laboratory coded as 5 (as well as laboratory 7) has been omitted from these calculations. This is because the mean level of the data from Laboratory 5, especially the determinations of nicotine, are much higher that expected, and these high means would have an undue influence on the calculations of v and V.

Included in Tables 5.1 and 5.2 are the values of v and V that might have been expected on the basis of the data given in CORESTA Report 91/1. These CORESTA values are included for guidance only; it must be remembered that the CORESTA data is based upon the smoking of 20 products through each filter pad, whereas in this study only 5 products were smoked through each filter.

From Table 5.1 it can be seen that when considering the NFDPM results the value of V (an indicator of between laboratory variability) is at a maximum when the Labyrinth Seal 9mm holder is being used, whereas all versions of the Sleeve holder give lower, and very similar values, of V.

From Table 5.2 it can be seen that when considering the nicotine results the value of V is at a maximum when the Labyrinth Seal holder is being used, whereas all versions of the Sleeve holder give lower, and very similar, values of V, but the difference between the Labyrinth Seal and Sleeve holders is less marked than is the case when considering the NFDPM data.

Conclusions

This study has demonstrated that the laboratories that took part in this study, after outlier removal, produced very sound data. The data also demonstrate that the Sleeve holder reduces variability as insertion depth increases.

Recommendation

A further study is recommended. Study 15 involved the analysis of FCSAs made at single location. It also only involved articles made at 7.2 mm diameter.

A further study should be conducted in which 5.2 mm diameter products are investigated. The study design should require those laboratories that are analysing the FCSAs also to make them. In this way, the data will be closer to that which would be obtained from a definitive method and estimates of both within and between laboratory variation could then be made.

Table 0.1

CRYO STUDY 15 PART A

SUMMARY OF OUTLIERS

LAB	M/C	RUN	CHN	HLD	NFDPM	NFDPM(e)	V	NIC.	NIC.(e)	V
1	2	87	*	1	9.03	13.07	-3.50	0.82	0.995	-3.31
2	2	10	*	5				0.74	1.032	-3.13
+2	2	8	*	4	14.16					
3	1	4	9	1	6.35	10.89	-4.18	0.55	0.862	-4.38
4	1	3	9	2				1.08	0.850	3.29
5	1	3	6	4	2.84	12.28	-5.63	0.37	1.143	-5.51
5	1	5	11	4	5.51	12.28	-4.04	0.59	1.143	-3.94
5	1	5	15	4	5.22	12.28	-4.22	0.61	1.143	-3.80
+5	1	5	7	4				0.97		
6	1	2	15	2	10.04	13.06	-3.02			
6	1	5	5	2	16.68	13.06	4.25	1.28	1.060	3.23
7	2	14	*	5	10.00	3.77	3.25			
7	2	16	*	2				0.07	0.606	-3.33
8	2	101	*	2	13.18	10.95	3.45			
9	1	3	18	3	16.38	12.73	3.65			
9	1	4	19	1				0.85	1.028	-3.20
10	1	2	20	1	10.22	12.73	-3.22			
11	1	4	5	5	3.80	14.49	-6.54	0.19	1.102	-6.94
+11	1	2	12	4	8.45			0.76		
14	1	5	15	4	19.46	15.03	3.24			
16	2	78	*	1				0.48	0.755	-4.04
16	2	109	*	1				0.55	0.755	-3.01
18	2	24	*	1	12.63	14.40	-3.16			
18	2	26	*	6				1.37	1.52	-3.01

The observations marked + were deleted at the second scan

8 NFDPM/NICOTINE pairs 8 NFDPM 8 NICOTINE

Break-down

HOLDER	NFDPM	NICOTINE
Labyrinth Seal 9mm	4	5
SLEEVE 9mm	3	3
SLEEVE 11mm	1	0
SLEEVE 13mm	6	5
SLEEVE 15mm	2	2
Test Piece	0	1
TOTAL	16 (0.78%)	16 (0.78%)

TOTAL NUMBER OF RECORDS = 2040

Table 1.1

```
ANALYSIS OF VARIANCE OF Test Piece DATA
 MEANS OF 20 FCSAs
 NFDPM
 ANALYSIS OF VARIANCE ON NFDPM
 SOURCE     DF        SS        MS        F        p
 LAB        16    30.535     1.908    14.67    0.000
 ERROR      68     8.849     0.130
 TOTAL      84    39.384
                                    INDIVIDUAL 95 PCT CI'S FOR MEAN
                                    BASED ON POOLED STDEV
  LEVEL      N      MEAN     STDEV  ---+---------+---------+---------+---
      5      5    14.509     0.166  (---*---)
     17      5    14.585     0.211   (---*---)
      9      5    14.748     0.271     (---*---)
     15      5    14.981     0.409        (---*---)
     13      5    15.005     0.309         (---*---)
      8      5    15.026     0.226         (---*---)
     16      5    15.267     0.748            (---*---)
     10      5    15.329     0.262             (---*---)
     18      5    15.409     0.187              (---*---)
      1      5    15.451     0.515              (---*---)
      2      5    15.585     0.368                (---*---)
     12      5    15.791     0.442                  (---*---)
      3      5    15.829     0.344                   (---*---)
     11      5    15.935     0.303                    (---*---)
      6      5    16.149     0.145                       (---*---)
     14      5    16.507     0.301                           (---*---)
      4      5    16.552     0.415                            (---*---)
                                    ---+---------+---------+---------+---
 POOLED STDEV =    0.361            14.40     15.20     16.00     16.80

 Analysis of Variance for NFDPM
 Source      DF         SS         MS       F      P
 LAB         16    30.5349     1.9084   14.67  0.000
 Error       68     8.8490     0.1301
 Total       84    39.3838

 Source      Variance Error Expected Mean Square
            component  term (using unrestricted model)
  1 LAB        0.3557   2   (2) +     5.0000(1)
  2 Error      0.1301       (2)

               r = 1.010      R = 1.952
```

Table 1.2

```
ANALYSIS OF VARIANCE OF Test Piece DATA
MEANS OF 20 FCSAs
NICOTINE
ANALYSIS OF VARIANCE ON nicotine
SOURCE      DF        SS        MS         F        p
LAB         16   0.14819   0.00926     5.05    0.000
ERROR       68   0.12482   0.00184
TOTAL       84   0.27301
                                    INDIVIDUAL 95 PCT CI'S FOR MEAN
                                    BASED ON POOLED STDEV
  LEVEL      N      MEAN     STDEV  -----+---------+---------+---------+-
     10      5    1.4735    0.0695  (----*-----)
      9      5    1.5038    0.0302      (-----*----)
      8      5    1.5135    0.0186        (----*-----)
      2      5    1.5145    0.0989        (----*-----)
     18      5    1.5262    0.0447          (----*----)
     13      5    1.5270    0.0498          (----*-----)
     16      5    1.5320    0.0327          (-----*----)
     12      5    1.5425    0.0443            (----*-----)
      6      5    1.5430    0.0206            (----*-----)
     11      5    1.5435    0.0298            (----*-----)
     14      5    1.5445    0.0301            (-----*----)
      5      5    1.5540    0.0285              (----*----)
     15      5    1.5569    0.0338              (----*-----)
      3      5    1.5610    0.0311               (----*----)
     17      5    1.6125    0.0112                      (----*-----)
      4      5    1.6294    0.0460                        (-----*----)
      1      5    1.6345    0.0211                         (----*-----)
                                     -----+---------+---------+---------+-
 POOLED STDEV =   0.0428               1.470     1.540     1.610     1.680

 Analysis of Variance for nicotine

 Source      DF         SS         MS       F      P
 LAB         16    0.148194   0.009262    5.05  0.000
 Error       68    0.124816   0.001836
 Total       84    0.273010

 Source      Variance Error Expected Mean Square
            component  term (using unrestricted model)
 1 LAB       0.00149   2   (2) +     5.0000(1)
 2 Error     0.00184       (2)
             r = 0.120     R = 0.161
```

Table 1.3

CRYO STUDY 15 PART A - TABLE OF v AND V - LABORATORY 7 OMITTED

NICOTINE

	v	(v calc)	V	(V calc)
Test Piece	0.173	(0.116)	0.210	(0.215)

NFDPM

	v	(v calc)	V	(V calc)
Test Piece	1.705	(1.147)	2.337	(1.801)

TABLE OF v AND V BASED ON THE MEANS OF 20 FCSAs - LABORATORY 7 OMITTED

NICOTINE

	v	(v calc)	V	(V calc)
Test Piece	0.120	(0.116)	0.161	(0.215)

NFDPM

	v	(v calc)	V	(V calc)
Test Piece	1.010	(1.147)	1.952	(1.801)

In the above tables the values of (v calc) and (V calc) are the values of v and V calculated from the data given in CORESTA Report 91/1.

Table 2.1

```
ANALYSIS OF SD(NFDPM) (transformed)
REMOVAL OF LABORATORY AND MACHINE EFFECTS
NOTE LAB 7 OMITTED  HOLDER 6 (Test Piece) OMITTED
MODEL MACHINES LABORATORIES WITHIN MACHINES
Analysis of Variance for SDTAR T
Source     Model DF   Reduced DF     Seq SS
M/C              1            1     0.34563
LAB(M/C)        32           15+    5.11924
Error           51           68     2.57205
Total           84           84     8.03691

RESIDUALS STORED IN 'RES-SDTAR'

ANALYSIS OF VARIANCE ON 'RES-SDTAR'
SOURCE     DF        SS        MS        F        p
HOLDER      4     0.4317    0.1079     3.23    0.018
ERROR      64     2.1403    0.0334
TOTAL      68     2.5720

                                       INDIVIDUAL 95 PCT CI'S FOR MEAN
                                       BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ----------+---------+---------+------
     1     17    0.0774    0.1698                     (-------*-------)
     2     17    0.0504    0.2272                  (-------*-------)
     3     17    0.0366    0.1235               (-------*-------)
     4     17   -0.0507    0.1557        (-------*-------)
     5     17   -0.1138    0.2173  (-------*-------)
                                    ----------+---------+---------+------
POOLED STDEV =   0.1829                    -0.10     -0.00      0.10

Fisher's pairwise comparisons
Individual error rate = 0.0500 (SINGLE-SIDED)
Intervals for (column level mean) - (row level mean)
                 1         2         3         4
         2   -0.0776
              0.1316
         3   -0.0638   -0.0908
              0.1454    0.1184
         4    0.0235   -0.0035   -0.0173
              0.2327    0.2057    0.1919
         5    0.0866    0.0596    0.0458   -0.0415
              0.2958    0.2688    0.2550    0.1677
```

Table 2.2

```
REGRESSION ANALYSIS OF INSERTION DEPTH - Labyrinth Seal 9mm HOLDER OMITTED
RESIDUAL SD(NFDPM) transformed
The regression equation is
RES NFDPM = 0.329 - 0.0290 INS

Predictor       Coef       Stdev    t-ratio        p
Constant      0.3286      0.1252       2.62    0.011
INS        -0.028999    0.010261      -2.83    0.007

Analysis of Variance

SOURCE       DF          SS          MS         F        p
Regression    1     0.28592     0.28592      7.99    0.007
Error        50     1.78979     0.03580
Total        51     2.07570
```

Table 2.3

ANALYSIS OF MEAN NFDPM

REMOVAL OF LABORATORY AND MACHINE EFFECTS

LABORATORY 7 OMITTED. HOLDER 6 (Test Piece) OMITTED.

MODEL:- MACHINES LABORATORIES WITHIN MACHINES

Analysis of Variance for NFDPM

Source	Model DF	Reduced DF	Seq SS
M/C	1	1	461.914
LAB(M/C)	32	15+	631.824
Error	1646	1663	2745.420
Total	1679	1679	3839.158

RESIDUALS STORED AS 'RES-MTAR'

ANALYSIS OF VARIANCE ON RES-MTAR

SOURCE	DF	SS	MS	F	p
HLD	4	679.93	169.98	136.53	0.000
ERROR	1659	2065.49	1.25		
TOTAL	1663	2745.42			

```
                                                INDIVIDUAL 95 PCT CI'S FOR MEAN
                                                BASED ON POOLED STDEV
HOLDER   N      MEAN      STDEV     ---------+---------+---------+------
1       334    -0.248     1.366                (-*-)
2       336    -0.989     1.145      (-*-)
3       338    -0.127     1.044                  (-*-)
4       333     0.539     0.992                             (-*-)
5       339     0.821     0.959                                   (-*-)
                                    ---------+---------+---------+------
POOLED STDEV =    1.1158                  -0.60     -0.00      0.60
```

Fisher's pairwise comparisons

Individual error rate = 0.0500 (SINGLE-SIDED)

Intervals for (column level mean) - (row level mean)

	1	2	3	4
2	0.599			
	0.883			
3	-0.267	-1.004		
	0.021	-0.720		
4	-0.929	-1.670	-0.808	
	-0.645	-1.386	-0.524	
5	-1.211	-1.952	-1.090	-0.424
	-0.927	-1.668	-0.806	-0.140

Table 2.4

REGRESSION ANALYSIS OF INSERTION DEPTH - Labyrinth Seal 9mm HOLDER OMITTED

RESIDUAL MEAN NFDPM

The regression equation is
RES-MTAR = - 2.87 + 0.251 INS DEP

Predictor	Coef	Stdev	t-ratio	p
Constant	-2.8678	0.1382	-20.75	0.000
INS DEP	0.25143	0.01187	21.18	0.000

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	579.03	579.03	444.21	0.000
Error	1662	2166.39	1.30		
Total	1663	2745.42			

Table 3.1

ANALYSIS OF SD(NICOTINE) (transformed)

REMOVAL OF LABORATORY AND MACHINE EFFECTS

NOTE LAB 7 OMITTED HOLDER 6 (Test Piece) OMITTED

MODEL MACHINES LABORATORIES WITHIN MACHINES

Analysis of Variance for SDTNA T

Source	Model DF	Reduced DF	Seq SS
M/C	1	1	0.358813
LAB(M/C)	32	15+	0.770344
Error	51	68	0.633710
Total	84	84	1.762867

RESIDUALS STORED AS 'RES-SDTNA'

ANALYSIS OF VARIANCE ON 'RES-NICOTINE'

SOURCE	DF	SS	MS	F	p
HOLDER	4	0.06227	0.01557	1.74	0.151
ERROR	64	0.57144	0.00893		
TOTAL	68	0.63371			

```
                                                  INDIVIDUAL 95 PCT CI'S FOR MEAN
                                                  BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ----+---------+---------+---------+-
     1     17   0.04790   0.09387               (--------*-------)
     2     17   0.00565   0.09576       (-------*-------)
     3     17  -0.00193   0.08145     (--------*-------)
     4     17  -0.02511   0.08584  (-------*-------)
     5     17  -0.02652   0.11251  (-------*-------)
                                   ----+---------+---------+---------+-
POOLED STDEV =  0.09449             -0.050    -0.000     0.050    0.100
```

Fisher's pairwise comparisons

Individual error rate = 0.0500 (SINGLE-SIDED)

Intervals for (column level mean) - (row level mean)

	1	2	3	4
2	-0.01184 0.09634			
3	-0.00426 0.10392	-0.04651 0.06167		
4	0.01892 0.12710	-0.02333 0.08485	-0.03091 0.07727	
5	0.02033 0.12851	-0.02192 0.08626	-0.02950 0.07868	-0.05268 0.05550

Table 3.2

REGRESSION ANALYSIS OF INSERTION DEPTH - Labyrinth Seal 9mm HOLDER OMITTED

RESIDUAL SD(NICOTINE) transformed

The regression equation is
RES NICOTINE = 0.0598 - 0.00598 INS

Predictor	Coef	Stdev	t-ratio	p
Constant	0.05984	0.06350	0.94	0.351
INS	-0.005984	0.005202	-1.15	0.255

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	0.012176	0.012176	1.32	0.255
Error	50	0.459988	0.009200		
Total	51	0.472164			

Table 3.3

```
ANALYSIS OF MEAN NICOTINE
REMOVAL OF LABORATORY AND MACHINE EFFECTS
LABORATORY 7 OMITTED.  HOLDER 6 (Test Piece) OMITTED.
MODEL:-  MACHINES   LABORATORIES WITHIN MACHINES

Analysis of Variance for NICOTINE
Source    Model DF   Reduced DF       Seq SS
M/C              1            1      1.98469
LAB(M/C)        32          15+      9.47011
Error         1648         1665     10.22761
Total         1681         1681     21.68241

RESIDUALS STORED AS 'RES-Mnic.'
ANALYSIS OF VARIANCE ON RES-Mnic.
SOURCE     DF        SS        MS        F       p
HLD         4   1.34499   0.33625    62.88   0.000
ERROR    1661   8.88262   0.00534
TOTAL    1665  10.22761
                                             INDIVIDUAL 95 PCT CI'S FOR MEAN
                                             BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV      --------+---------+---------+-------
     1    333  -0.00900   0.08677                   (--*--)
     2    338  -0.04542   0.07157     (--*-)
     3    339  -0.00448   0.06736                    (--*-)
     4    334   0.02323   0.06566                           (--*-)
     5    338   0.03582   0.07077                                  (--*--)
                                       --------+---------+---------+-------
POOLED STDEV =  0.07313                     -0.030     0.000     0.030

Fisher's pairwise comparisons
Individual error rate = 0.0500 (SINGLE-SIDED)
Intervals for (column level mean) - (row level mean)
                 1         2         3         4
         2   0.02714
             0.04570
         3  -0.01380   -0.05022
             0.00476   -0.03166
         4  -0.04151   -0.07793   -0.03699
            -0.02295   -0.05937   -0.01843
         5  -0.05410   -0.09052   -0.04958   -0.02187
            -0.03554   -0.07196   -0.03102   -0.00331
```

Table 3.4

```
REGRESSION ANALYSIS OF INSERTION DEPTH - Labyrinth Seal 9mm HOLDER OMITTED
RESIDUAL MEAN NICOTINE

The regression equation is
RES-MNic. = - 0.125 + 0.0110 INS DEP

Predictor       Coef       Stdev    t-ratio       p
Constant   -0.125364    0.008963     -13.99    0.000
INS DEP    0.0109938   0.0007701      14.28    0.000

Analysis of Variance
SOURCE       DF          SS          MS         F        p
Regression    1      1.1065      1.1065    201.86    0.000
Error      1664      9.1211      0.0055
Total      1665     10.2276
```

Table 4.0

```
ANALYSIS OF SD(PUFF NUMBER)
INCIDENCE TABLE OF 10 HIGHEST SD's

HOLDER 1 (Labyrinth Seal 9mm) 5
HOLDER 2 (SLEEVE 9mm)         1
HOLDER 3 (SLEEVE 11mm)        1
HOLDER 4 (SLEEVE 13mm)        1
HOLDER 5 (SLEEVE 15mm)        2
```

Table 4.1

```
ANALYSIS OF SD(PUFF NUMBER)
REMOVAL OF MACHINE AND LABORATORY EFFECTS
NOTE LAB 7 AND HOLDER 6 (Test Piece) OMITTED

MODEL:- MACHINES LABORATORIES WITHIN MACHINES

Analysis of Variance for SD PUFFS
Source    Model DF   Reduced DF     Seq SS
M/C              1            1    0.46798
LAB(M/C)        32          15+    3.11616
Error         1649         1666   53.82596
Total         1682         1682   57.41010

RESIDUALS STORED AS 'RES-SDP'

ANALYSIS OF VARIANCE ON 'RES-SDP'
SOURCE     DF        SS        MS        F        p
HLD         4    1.4530    0.3633    11.53    0.000
ERROR    1662   52.3729    0.0315
TOTAL    1666   53.8260
                                                 INDIVIDUAL 95 PCT CI'S FOR MEAN
                                                 BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV      --------+---------+---------+-------
     1    334    0.0292    0.1930                          (----*-----)
     2    336    0.0371    0.1796                              (-----*----)
     3    340   -0.0063    0.1803                  (----*-----)
     4    339   -0.0181    0.1624            (-----*----)
     5    334   -0.0417    0.1664     (----*----)
                                       --------+---------+---------+-------
POOLED STDEV =   0.17752                    -0.035     0.000     0.035

Fisher's pairwise comparisons
Individual error rate = 0.0500
Intervals for (column level mean) - (row level mean)
                 1         2         3         4
         2   -0.0348
              0.0190
         3    0.0086    0.0165
              0.0624    0.0703
         4    0.0204    0.0283   -0.0151
              0.0742    0.0821    0.0387
         5    0.0440    0.0519    0.0085   -0.0033
              0.0978    0.1057    0.0621    0.0505
```

Table 4.2

REGRESSION ANALYSIS OF INSERTION DEPTH - Labyrinth Seal 9mm HOLDER OMITTED

RESIDUAL SD(PUFF NUMBER)

The regression equation is
RES SDP = 0.142 - 0.0124 INS DEP

Predictor	Coef	Stdev	t-ratio	p
Constant	0.14167	0.02568	5.52	0.000
INS DEP	-0.012413	0.002105	-5.90	0.000

Analysis of Variance

SOURCE	DF	SS	MS	F	p	
Regression	1	1.0338	1.0338	34.79	0.000	
Error	1347	40.0279	0.0297	Total 1348		41.0617

Table 4.3

ANALYSIS OF MEAN PUFF NUMBER

REMOVAL OF MACHINE AND LABORATORY EFFECTS

LABORATORY 7 OMITTED.   HOLDER 6 OMITTED.

MODEL:- MACHINES   LABORATORIES WITHIN MACHINES.

Analysis of Variance for MEANPUFF

Source	Model DF	Reduced DF	Seq SS
M/C	1	1	32.3329
LAB(M/C)	32	15+	189.4675
Error	1651	1668	160.4592
Total	1684	1684	382.2596

RESIDUALS STORED AS 'MEAN-RES'

ANALYSIS OF VARIANCE ON MEAN-RES

SOURCE	DF	SS	MS	F	p	
HLD	4	11.0340	2.7585	30.72	0.000	
ERROR	1664	149.4252	0.0898	TOTAL	1668	160.4592

```
                                                INDIVIDUAL 95 PCT CI'S FOR MEAN
                                                BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV      -------+---------+---------+--------
     1    335    0.0089    0.3371                     (--*--)
     2    336    0.1051    0.2963                             (---*--)
     3    340    0.0517    0.2866                       (--*--)
     4    339   -0.0323    0.2777              (--*--)
     5    335   -0.1342    0.2904   (---*--)
                                           -------+---------+---------+--------
POOLED STDEV =   0.2997                        -0.10     -0.00      0.10
```

Fisher's pairwise comparisons

Individual error rate = 0.0500 (SINGLE-SIDED)

Intervals for (column level mean) - (row level mean)

	1	2	3	4
2	-0.1342			
	-0.0582			
3	0.0712	0.0154		
	-0.0048	0.0914		
4	0.0032	0.0994	0.0460	
	0.0791	0.1754	0.1220	
5	0.1051	0.2013	0.1479	0.0639
	0.1811	0.2773	0.2239	0.1399

Table 4.4

REGRESSIONS ANALYSIS OF INSERTION DEPTH - Labyrinth Seal 9mm HOLDER OMITTED
RESIDUAL MEAN PUFF NUMBER

The regression equation is
MEAN-RES = 0.479 - 0.0401 INS DEP

Predictor	Coef	Stdev	t-ratio	p
Constant	0.47877	0.04285	11.17	0.000
INS DEP	-0.040092	0.003512	-11.42	0.000

Analysis of Variance

SOURCE	DF	SS	MS	F	p
Regression	1	10.799	10.799	128.79	0.000
Error	1332	111.684	0.084		
Total	1333	122.482			

Table 5.1

CRYO STUDY 15 PART A
TABLE OF v AND V
LABORATORIES 5 AND 7 OMITTED
NFDPM

	v	(v calc)	V	(V calc)
Labyrinth Seal 9mm	2.990	(1.049)	4.990	(1.693)
SLEEVE 9mm	3.095	(1.021)	3.714	(1.661)
SLEEVE 11mm	2.908	(1.055)	3.741	(1.701)
SLEEVE 13mm	2.646	(1.078)	3.568	(1.727)
SLEEVE 15mm	2.512	(1.089)	3.376	(1.739)
Test Piece	1.705	(1.147)	2.337	(1.801)

In the above table the values of (v calc) and (V calc)are the values of v and V calculated from the data given in CORESTA Report 91/1.

Table 5.2

CRYO STUDY 15 PART A
TABLE OF v AND V
LABORATORIES 5 AND 7 OMITTED
NICOTINE

	v	v calc)	V	(V calc)
Labyrinth Seal 9mm	0.207	(0.087)	0.322	(0.148)
SLEEVE 9mm	0.195	(0.085)	0.263	(0.143)
SLEEVE 11mm	0.184	(0.088)	0.250	(0.148)
SLEEVE 13mm	0.182	(0.089)	0.245	(0.152)
SLEEVE 15mm	0.182	(0.090)	0.235	(0.153)
Test Piece	0.173	(0.116)	0.210	(0.215)

Figure 2.01

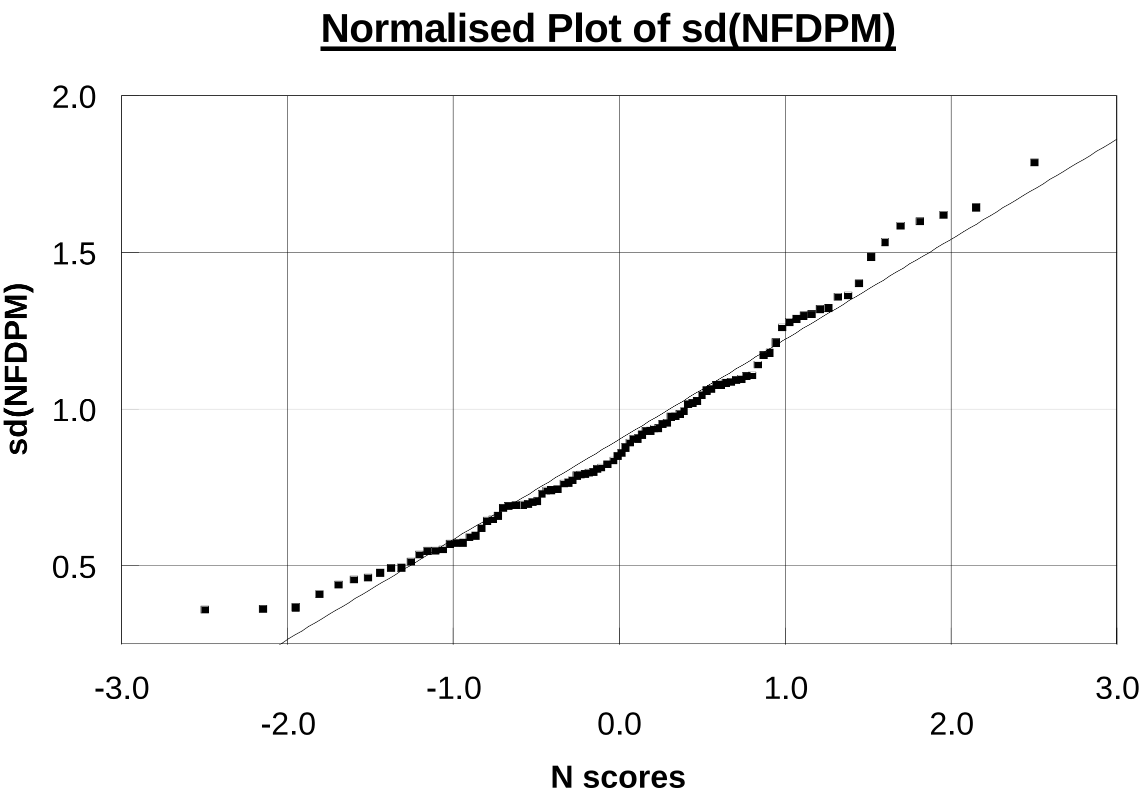


Figure 2.02

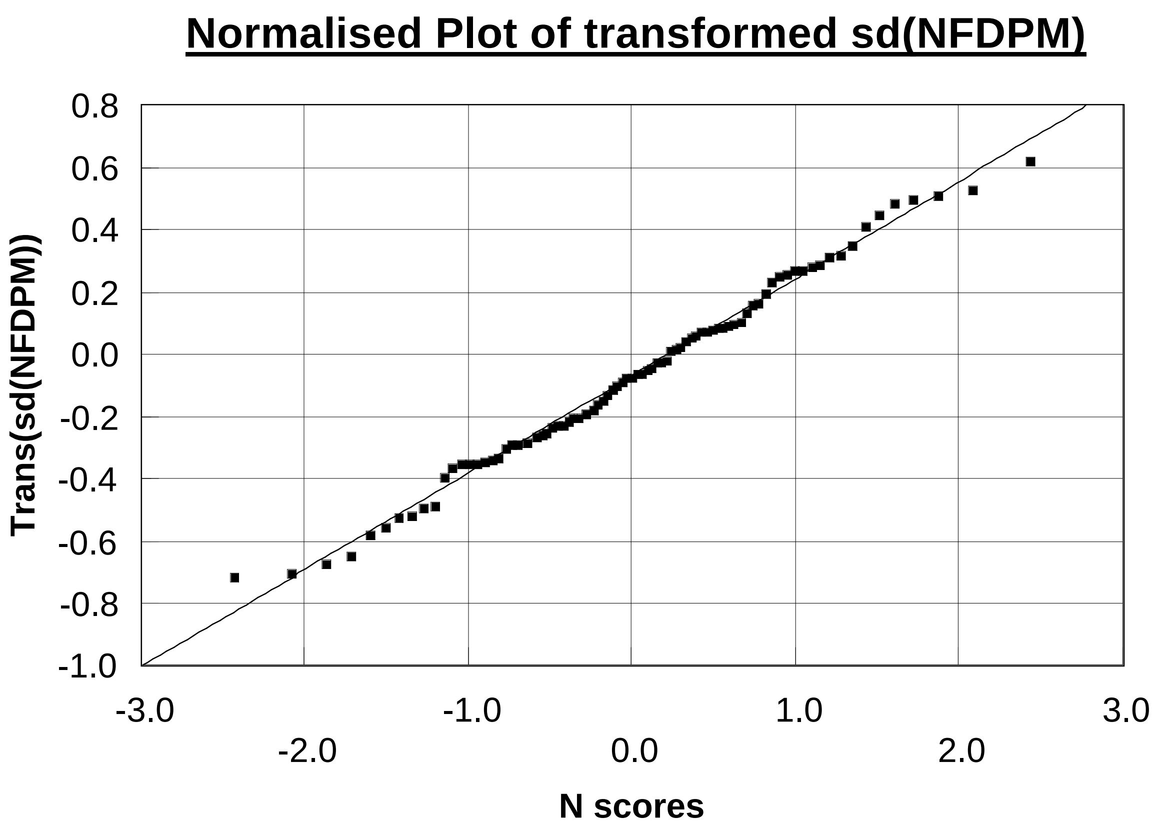


Figure 2.1

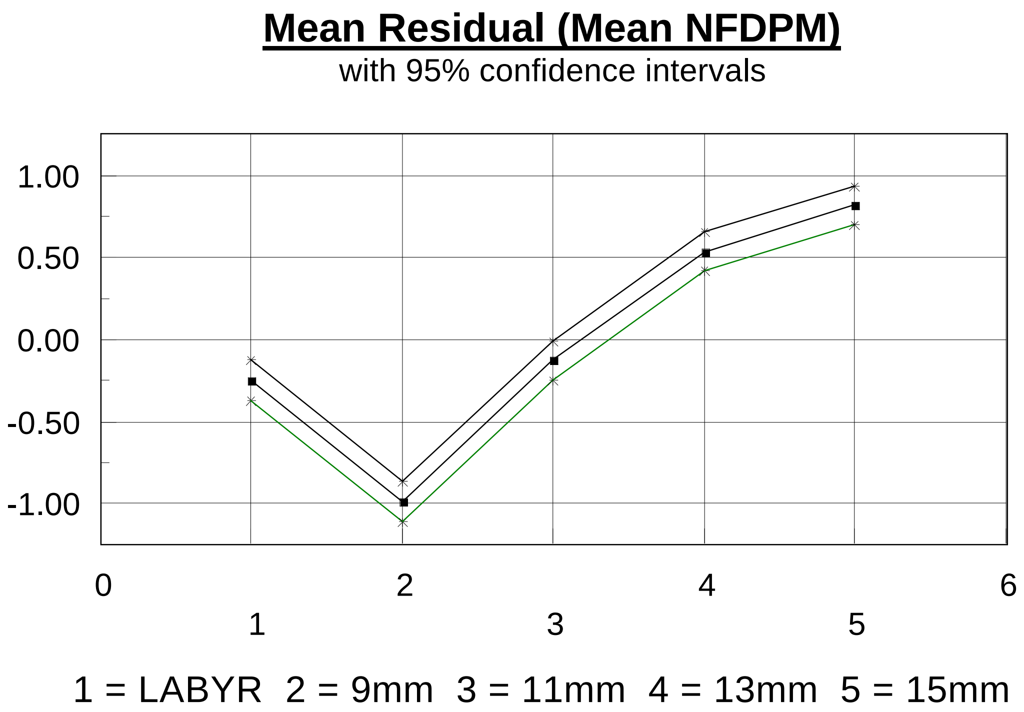


Figure 2.2

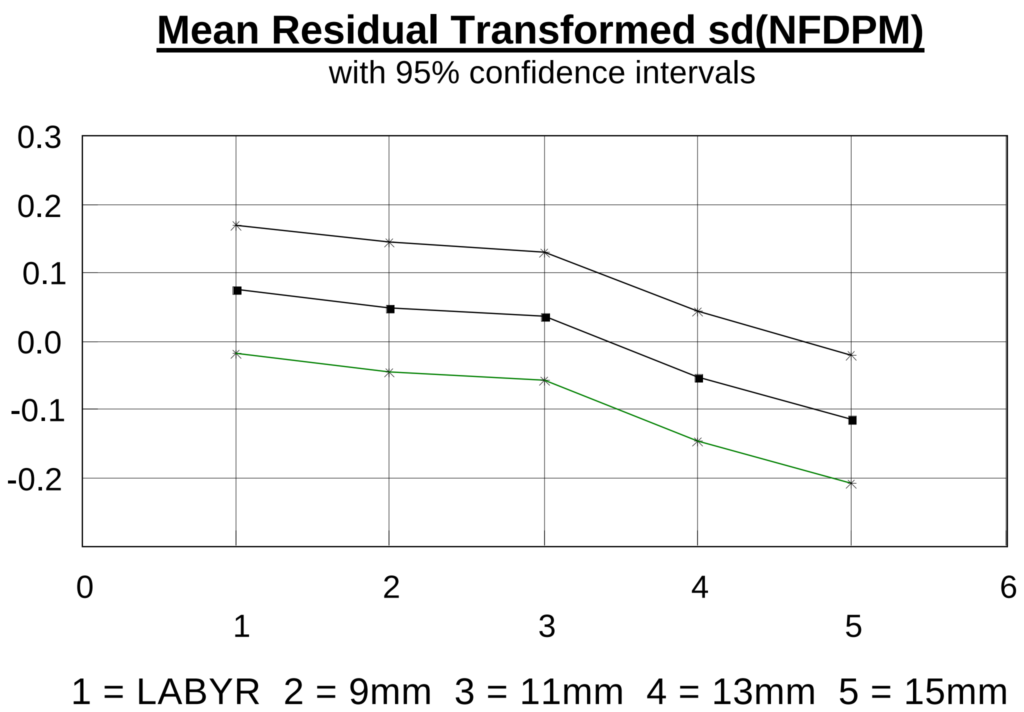


Figure 3.1

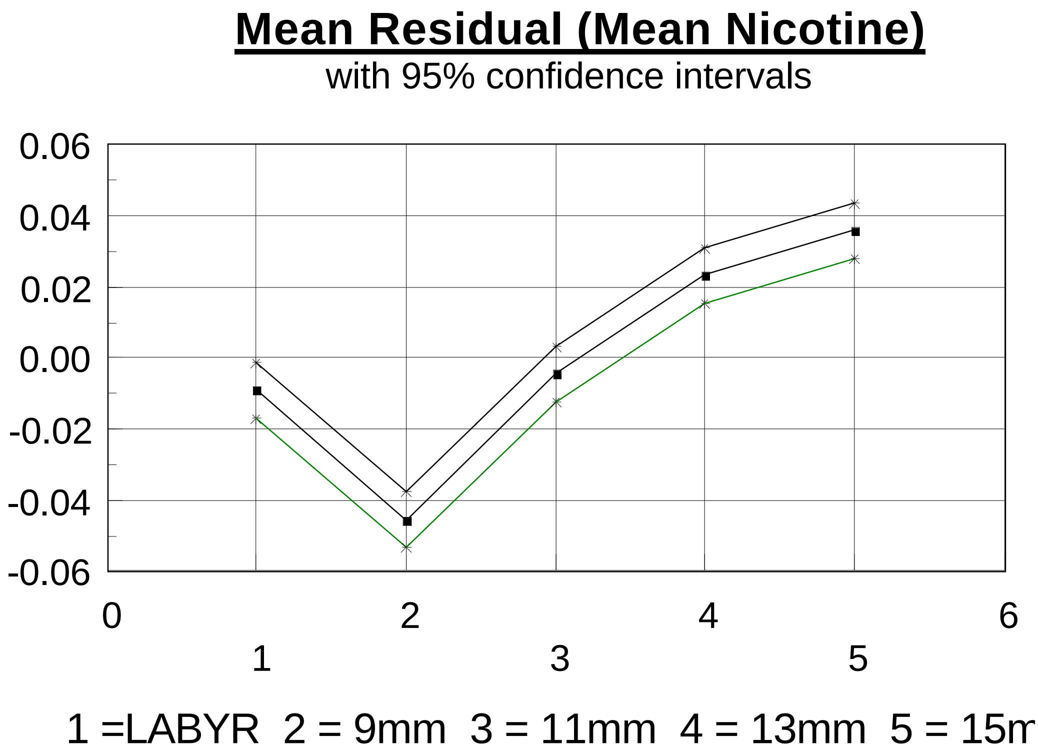


Figure 3.2

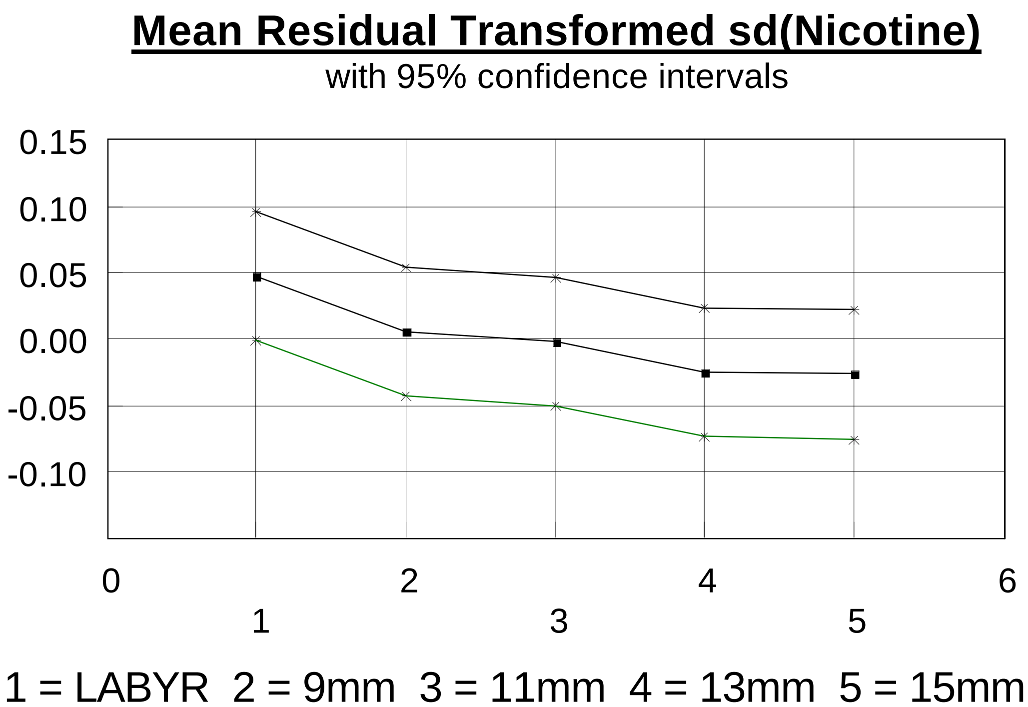


Figure 4.0

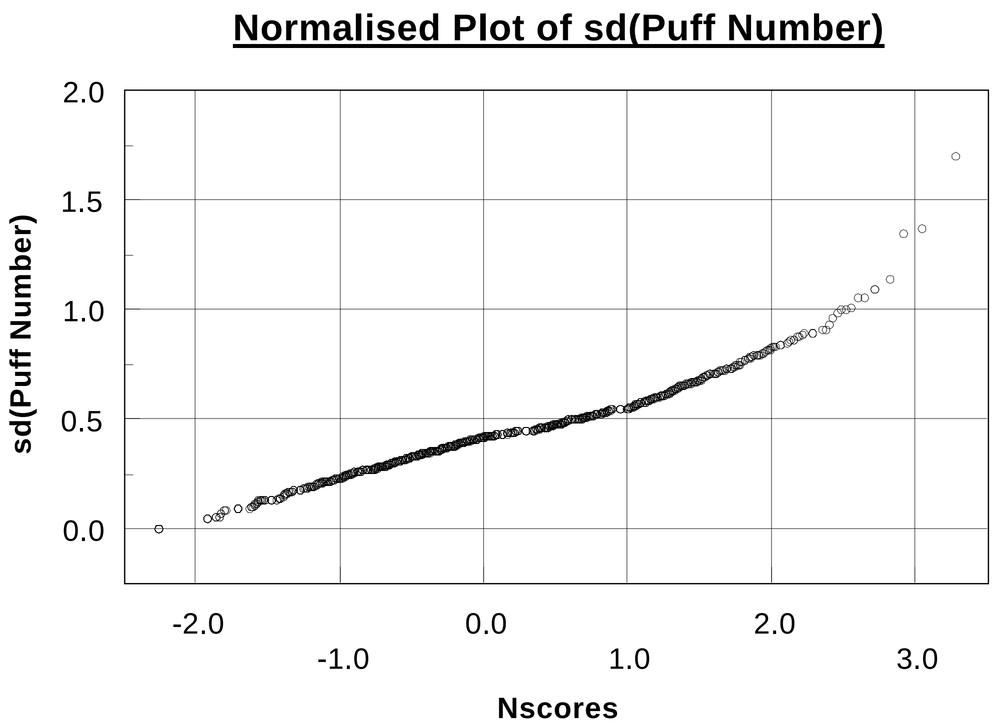


Figure 4.1

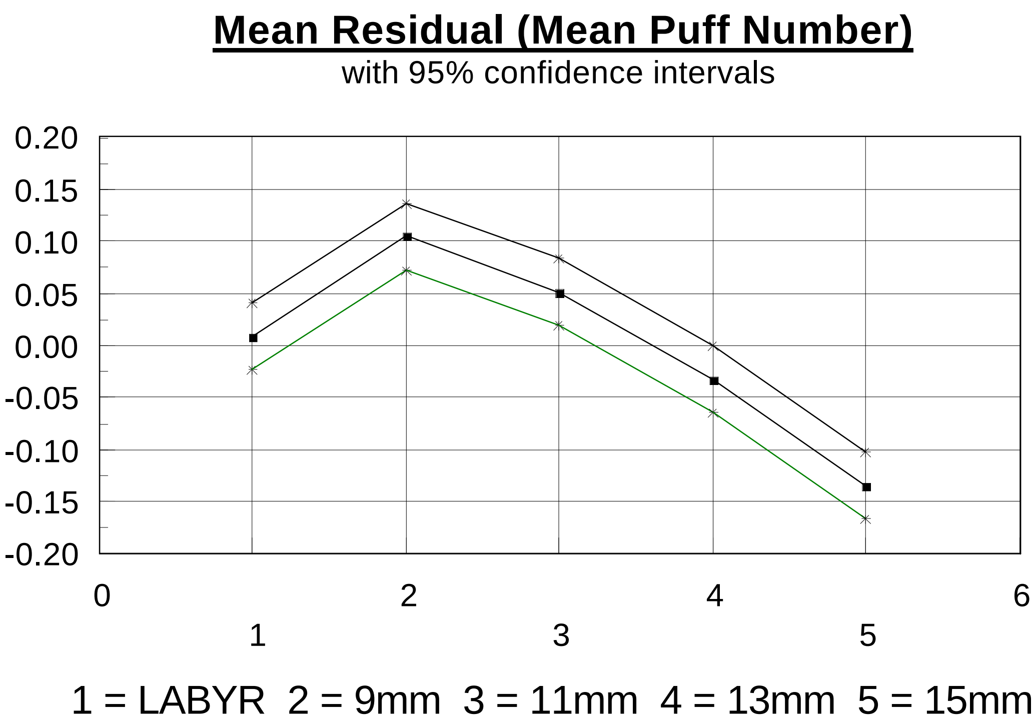
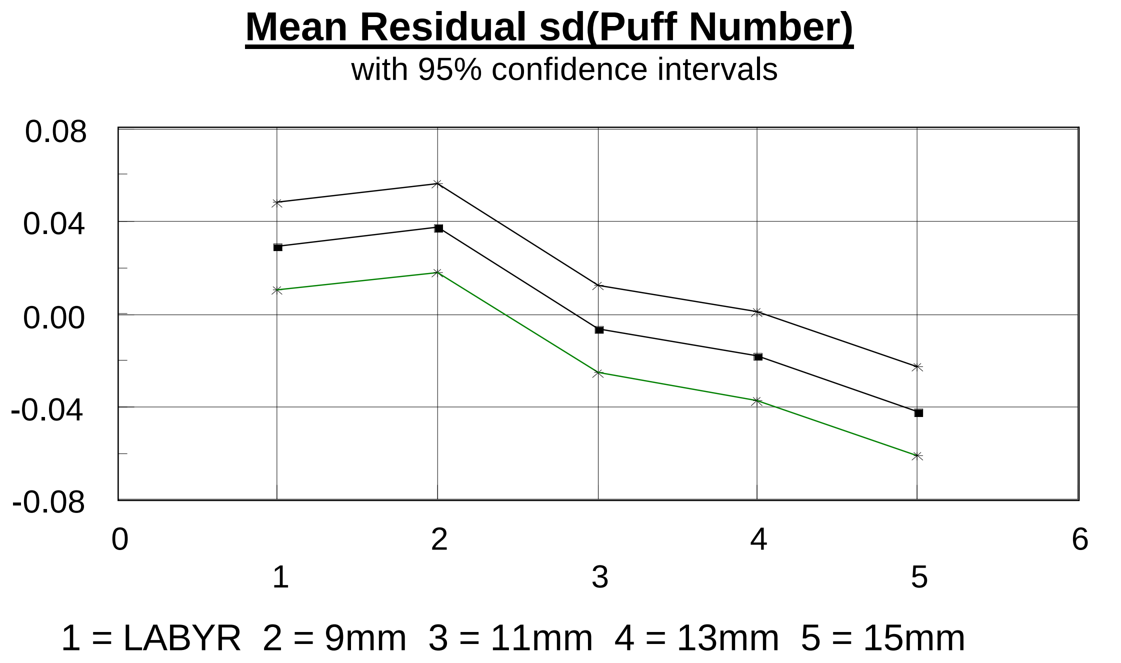


Figure 4.2



Annex 1

EXPERIMENTAL PROTOCOL

0.0 **Introduction**

The holders used for the analysis of manufactured cigarettes are extremely difficult to use with fine-cut smoking articles. There is also some evidence that they may be unsuitable for fine-cut smoking articles and give rise to greater variability of data. Two previous studies (Study 10 and Study 14) were performed in an attempt to demonstrate the advantages of a specially designed holder. Neither of these two studies proved completely satisfactory. A statistical evaluation of Study 10 was totally inconclusive. A statistical evaluation of Study 14 showed some evidence that the special holder gave improved data but it was not possible to draw definitive conclusions. It was recommended that a new study be performed with a larger number of participating laboratories, a more strict experimental design and an improved statistical analysis. This study has been designed with these points in mind, looking particularly at the within-laboratory variability.

1.0 Objectives of the Study

1.0.1 To determine the appropriate holder design and insertion depth required to smoke fine-cut smoking articles for the determination of NFDPM and nicotine in smoke.

1.0.2 The results will be evaluated primarily on the basis of within-laboratory variability. It is not the intention of this study to estimate the between-laboratory variability. In particular, the data from this study will not form the basis of the calculation of r and R.

2.0 Holders

2.0.1 The details of the design and construction of the two holders to be used in this study will be discussed at a meeting of laboratory experts.

2.0.2 The Labyrinth Seal holder is to be used according to ISO Standard Method 3308:1991. For this reason only one insertion depth (9mm) can be used with this holder.

2.0.3 The Sleeve holder will be supplied as four separate designs in such a way that various insertion depths can be used.

2.0.4 Using the Sleeve holder, the fine-cut smoking articles will be smoked using insertion depths of 9 mm, 11 mm, 13 mm, and 15 mm.

2.0.5 A butt length of 29mm will be used for all smoking of the fine-cut smoking articles.

2.0.6 A Test Piece (CM2) will be smoked using the standard Labyrinth Seal holder.

2.0.7 A butt length of 30mm will also be used for the smoking of the Test Piece in line with the protocol given for the use of the Test Piece.

3.0 Fine-cut Smoking Articles

3.0.1 The fine-cut smoking articles to be used in this study will be manufactured, with the modified Canadian tubing device, at one location and distributed to the participating laboratories.

3.0.2 The specification of the fine-cut smoking articles to be used is as follows:-

Length 70 mm

Diameter 7.2 mm

Tobacco weight 750 mg

The tobacco to be used is Drum Master Blend. The moisture content of a sample pouch from each bundle will be determined and if satisfactory the tobacco will be used for making as supplied by the manufacturer. (see also 6.2)

The paper will be of German manufacture and of a type typically used in Germany.

3.0.3 Fine-cut smoking articles will be made in batches such that one laboratory receives the sequential production of one operator. One day's production will be placed in 3 tins. This will give a total of 15 tins per laboratory. The 15 tins will be sealed as a single package.

3.0.5 As excess handling of FCSAs causes damage to them, they will not be strictly randomised but each laboratory will be asked to group tins 1,6 and 11; 2,7 and 12; etc and to take 100 FCSAs from these groupings. Each group of products will be used with one holder type.

3.0.6 On receipt in the laboratory, the fine-cut smoking articles will be conditioned according to CORESTA Recommended Method 42, as they are required.

4.0 Smoking Machines

4.0.1 Two types of smoking machine will be used in this study.

4.0.2 20-channel linear smoking machines smoking 5 articles through each channel on to a 44 mm filter pad.

4.0.3 20-channel rotary smoking machines specially equipped with a trap designed to allow the smoking of 5 articles on to a 44 mm filter pad. (Available from Borgwaldt GmbH part Number 80205340 with part number 80205480.)

4.0.4 Since the evaluation of the data from this study is to be based upon the within-laboratory variability, the 20-channnel rotary smoking machines have been adapted in this way so as to make the variability of the data from the two types of smoking machine directly comparable.

4.0.5 The equipment required to establish standardised ambient airflow conditions according to CORESTA Recommended Method 25 will be used.

5.0 Variables

5.1 Target variables

5.1.1 The variables to be analysed in order to estimate the within-laboratory variability are Nicotine Free Dry Particulate Matter (NFDPM) and nicotine. Ancillary measurements will be Puff number, Total Particulate Matter (TPM), and Water.

5.2 Variables to be reported

5.2.1 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 method.

5.2.2 The ancillary variables are

type of smoking machine used

laboratory temperature during smoking

relative humidity (RH%) in the laboratory during smoking

atmospheric pressure in the laboratory during smoking

date of test

ambient air flow

weight of 5 articles before smoking.

[Note that CORESTA Recommended Method No. 42 specifies that the Test Atmosphere shall be 22±3°C, (60±5)%RH]

5.2.3 The variables to be reported for statistical analysis are

puff number per FCSA or Test Piece

mean TPM per FCSA or Test Piece

mean nicotine yield per FCSA or Test Piece

mean water yield per FCSA or Test Piece

mean NFDPM yield per FCSA or Test Piece

5.3 Dimensions and rounding of test results

5.3.1 Ancillary variables

Laboratory temperature degrees Celsius. ##.#

Laboratory humidity percent RH. ##.#

Laboratory pressure kPa. ###.#

Weight of 5 FCSAs mg. ###.#

5.3.2 Analytical variables

Puff Number ##.#

[Note - This value is required as the individual puff count as well as the mean puff number for the five FCSAs or Test Pieces - see Appendix 3]

TPM mg per FCSA or Test Piece	##.##
Nicotine mg per FCSA or Test Piece	#.##
Water mg per FCSA or Test Piece	#.##
NFDPM mg per FCSA or Test Piece	##.##

[Note - These values are those obtained by dividing the value obtained from one glass fibre filter pad by 5]

[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.]

5.3.3 Note that the puff number for each individual fine-cut smoking article is to be reported.

5.3.3.1 Record and report all attempts to relight.

5.3.4 If leakage at the holder occurs for some individual fine-cut smoking articles, then this may be reflected in an increased puff number.

5.3.5 Since suspected leakage at the holder is one of the reasons for the design of the Sleeve holder, analysis of the puff numbers of individual fine-cut smoking articles may provide important insights into this aspect of the design of the holder.

5.3.6 Note that not all of the recorded variables are required on disc but all should be included on the hard copy data sheets that accompany the disc.

5.4 Internal documentation of tests

5.4.1 Each laboratory will document the method used following (where appropriate) the requirements of ISO Standard 4387:1991. The relevant requirements of Section 8 of ISO 4387:1991 are shown as Appendix 2 to this protocol. (Note that the requirements concerning the number of digits to be reported and other details have been modified to reflect the fact that this study is concerned with fine-cut smoking articles and not manufactured cigarettes as well as to meet the objectives of this study.)

5.4.2 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.

5.5 Exchange of data

5.5.1 All the data arising from this study will be made available to all the laboratories participating in the study on request after confirmation of the validity of the data.

5.5.2 To facilitate the exchange of data, all laboratories will be asked to send their results to H. Dymond for collation prior to the distribution of the data.

5.5.3 The format and medium to be used for the reporting of results are shown in Appendix 3 to this protocol.

6.0 Design of the Study

6.1 Laboratories are required to unpack the fine-cut smoking articles before conditioning

6.2 The fine-cut smoking articles shall be conditioned for a minimum of 72 hours at 22°C and 75% RH. They shall not be left in the conditioning atmosphere for longer than 10 days.

6.2.1 Conditioning will be achieved according to CORESTA Recommended Method Number 42.

6.2.3 The Test Piece (CM2) will be conditioned according to CORESTA Recommended Method Number 21.

6.3 Numbers of fine-cut smoking articles and Test Pieces

6.3.1 The study will use the fine-cut smoking article specified in 3.0 above.

6.3.2 The study will also include the CORESTA Test Piece CM2.

6.3.3 The CM2 Test Piece will only be smoked in the standard Labyrinth Seal holder.

6.3.4 Each participating laboratory will be required to smoke 100 fine-cut smoking articles through each holder. (i.e. 20 runs on a rotary machine or 20 channels on a linear machine.)

6.3.5 Each participating laboratory will be required to smoke 100 Test Pieces through the standard Labyrinth Seal holder using standard procedures according to ISO 4387;1991.

6.4 Smoking plans

6.4.1 The smoking plans show one fine-cut smoking article being smoked through 5 holder configurations shown as A, B, C, D and E and one Test Piece shown as M in the smoking plans.

6.4.2 For each holder configuration, 20 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.

6.4.3 For this study, one test result is defined as the mean yield obtained from smoking 5 fine-cut smoking articles, or 5 CM2 Test Pieces, onto one glass fibre filter pad.

6.5 Smoking methods

6.5.1 The smoking procedures shall be as given in ISO 4387:1991 including clearing puffs.

7.0 General Remarks

7.0.1 Each participating laboratory will be asked to use only one type of smoking machine.

7.0.2 It is hoped that at least 16 laboratories will be recruited to take part in this study.

7.0.3 It would be advantageous if equal numbers of participating laboratories used the 20-channel linear smoking machine and the rotary smoking machine.

7.0.4 Each participating laboratory will use only one operator and one machine to smoke the products throughout the course of the study.

7.0.5 The sealed glass fibre filter holder assembly, without the Sleeve holder or the labyrinth seal holder, will be weighed both before and after smoking. In the case of the Labyrinth Seal holder, if the perforated disc/washer is part of the trap it will be left in place during weighing but if it is part of the holder it will not.

7.0.6 In the case of the Labyrinth Seal holder, a new perforated disc/washer will be used for each smoke run.

7.0.7 The filter holder will be dry-wiped to ensure that any tar deposited in the holder is included in the nicotine/water extraction.

7.0.8 The CM2 Test Piece will be smoked according to the CORESTA Recommended Method Number 23. A new slotted washer will be used for each smoke run of the Test Piece.

7.0.9 No further preparation of the fine-cut smoking articles is required apart from conditioning before smoking. In particular, neither weight nor pressure drop selection procedures are to be used.

7.0.10 Please note: The statements given in 5.3.3 is intentional and although somewhat unusual this information may assist in the interpretation of the data from this study

APPENDIX 1

INTERNAL DOCUMENTATION OF TEST

BASED UPON THE PROVISIONS OF ISO 4387:1991

Note: The following is an adaptation from ISO 4387:1991. It indicates the records that should be kept in-house. It is not a statement of what is to be reported. For this information see Appendix 3.

8. Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The test report shall include all details required for complete identification of the sample. Where appropriate, record the information in 8.1 to 8.4.

[Note the following is not a complete reproduction of sections 8.1, 8.2 and 8.3 but highlights the important points.]

8.1 Characteristic data about the FCSA or Test Piece

a) product name;

b) total length;

c) length of filter;

d) length of overwrap.

8.3 Description of test

Date of test.

Type of smoking machine used.

Type of smoke trap used.

Total number of FCSAs or Test Pieces smoked in the entire determination on that product type.

Number of FCSAs or Test Pieces smoked into each trap.

Butt length.

Room temperature (°C) during smoke operation.

Relative humidity (%) during smoke operation.

Atmospheric pressure (kPa) during smoke operation.

8.4 Test results

The expression of the laboratory data depends on the purpose for which the data are required, and the level of laboratory precision. Tolerances shall be calculated and expressed on the basis of the laboratory data before any rounding has taken place.

Average length of the FCSA or Test Piece to the nearest 0.1 mm.

Average length of the filter to the nearest 0.1 mm.

Average length of the overwrap to the nearest 0.1 mm.

Butt length to which the products were smoked to the nearest 0.1 mm.

Average length of tobacco portion smoked to the nearest 0.1 mm.

Average diameter of the products (mm).

Average draw resistance of the conditioned FCSAs or Test Pieces. [Note: There are a few spare FCSAs supplied to each laboratory. These may be used to determine a value for draw resistance.]

Average mass (milligrams per FCSA or Test Piece) of the conditioned FCSA or Test Piece selected for the smoking operation.

Water content (% weight/weight) of the conditioned FCSA or Test Piece (see ISO 6488). [Note: There are a few spare FCSAs supplied to each laboratory. These may be used to determine a value for water content.]

Ambient air flow to the nearest cm per sec.

Average number of puffs per FCSA or Test Piece for each channel to the nearest 0.01 puff. [Note: For this study you are also required to record the individual puff count]

Total particulate matter (milligrams per FCSA or Test Piece) for each channel to the nearest 0.01 mg and the average per FCSA or Test Piece to the nearest 0.01 mg.

Water delivery for each channel to the nearest 0.01 mg and the average per FCSA or Test Piece to the nearest 0.01 mg.

Dry particulate matter (milligrams per FCSA or Test Piece) for each channel to the nearest 0.01 mg and the average per FCSA or Test Piece to the nearest 0.01 mg.

Nicotine delivery for each channel to the nearest 0.001 mg and the average per FCSA or Test Piece to the nearest 0.001 mg.

Nicotine free dry particulate matter (milligrams per FCSA or Test Piece) for each channel to the nearest 0.01 mg and the average per FCSA or Test Piece to the nearest 0.01 mg.

APPENDIX 2

Data Support for the 15th. Study.

This Appendix gives information concerning the collection of data for the 15th. Study. Given is:-

a list of the variables used and the order in which they must be written on the data file. An example is also given.

a list of codes assigned to each variable. This includes the code for each laboratory. Please use the codes given to simplify the transfer of data into a file suitable for statistical analysis.

details of the characteristics of the floppy disc to be used for data transmission.

If your data is transmitted using a floppy disc then the disc MUST be accompanied by a printout of the file on the disc.

A set of data forms is also attached to be used if your data cannot be transmitted via a floppy disc.

The Means and SDs shown on these data forms are requested solely for the purpose of checking the (manual) entry of the data into the computer. They will not be used for any other purpose. Each data form will be checked as it is entered by comparing the Means and SDs of the data on the computer with those given on the data forms.

If you have any problems please contact Mr. H. Dymond.

Enclosed :-

ANNEX 1:	Order of variables on the smoking data file and the format of the data file.
ANNEX 2:	Order of variables on the puff number data file and the format of the data file.
ANNEX 3:	Characteristics of the floppy discs to be used.
ANNEX 4:	List of participating laboratories, and their associated laboratory codes.

ANNEX 1 ORDER OF VARIABLES ON THE SMOKING DATA FILE

Each row of the data file must contain 12 variables in the following order:-

```
EXP   LAB  DATE  OPER  RNUM   CHAN  HOLDER  PUFF   TPM    WATER    NIC.    NFDPM
(1)   (2)  (3)   (4)    (5)   (6)    (7)     (8)   (9)     (10)   (11)     (12)
```

```
EXAMPLE :
 (1)    (2)     (3)    (4)   (5,6,7)   (8)    (9)    (10)   (11)      (12)
ST15   PMGE   071097   ABCD   1 1 A    9.10   3.79   2.34   0.121     1.33
ST15   PMGE   071097   ABCD   1 2 M    8.90   5.07   2.67   0.252     2.15
```

```
FORMAT(A4,A7,I7,A5,2I3,A2,3F6.2,F6.3,F6.2)
All variables to be right-justified within the field.
```

EXPLANATION OF VARIABLE CODINGS

#	Name	Definition	Value	Length	Comment
(1)	EXP	Experiment code	ST15	A4	
(2)	LAB	Laboratory name	?????	A7	See ANNEX 3
(3)	DATE	Smoking date	DDMMYY	I7	
(4)	OPER	Operator code	?????	A5	Defined by you
(5)	RNUM	Smoking run number	1....6 1..120	I3	Linear machines Rotary machines
(6)	CHAN	Channel number	1...20 *	I3	Linear machines Rotary machines
(7)	HOLDER	Holder code	A,B,C, D,E,M	A2	
(8)	PUFF	Puff number	##.#	F6.1	This is the Mean of the 5 smoked in one trap
(9)	TPM	Total particulate matter	##.##	F6.2	
(10)	WATER	Smoke water	##.##	F6.2	
(11)	NICOTINE	Smoke nicotine	#.###	F6.3	
(12)	NFDPM	Nicotine free dry particulate matter	##.##	F6.2	

The holder configurations used in this study are coded as follows:-

A. Labyrinth Seal 9mm insertion
B. SLEEVE 9mm insertion
C. SLEEVE 11mm insertion
D. SLEEVE 13mm insertion
E. SLEEVE 15mm insertion
M. Labyrinth Seal 9mm insertion CM2 test piece

ANNEX 2 ORDER OF VARIABLES ON THE PUFF NUMBER DATA FILE

Each row of the data file must contain 12 variables in the following order:-

```
EXP   LAB   DATE   OPER   RNUM  CHAN  HOLDER  FCSA1  FCSA2  FCSA3  FCSA4  FCSA5
(1)   (2)   (3)     (4)    (5)   (6)   (7)     (8)    (9)    (10)   (11)  (12)
```

EXAMPLES:

```
(1)    (2)    (3)   (4)    (5,6,7)  (8)  (9)  (10)  (11)  (12)
ST15  PMGE  071097  ABCD   1  1 A    9    8     9     7     5
ST15  PMGE  071097  ABCD   1  2 M    7    7     6     7     6
```

FORMAT(A4,A7,I7,A5,2I3,A2,6I6)

All variables to be right-justified within the field.

EXPLANATION OF VARIABLE CODINGS

#	Name	Definition	Value	Length	Comment
(1)	EXP	Experiment code	ST15	A4	
(2)	LAB	Laboratory name	?????	A7	See ANNEX 3
(3)	DATE	Smoking date	DDMMYY	I7	
(4)	OPER	Operator code	?????	A5	Defined by you
(5)	RNUM	Smoking run number	1....6 1..120	I3	Linear machines Rotary machines
(6)	CHAN	Channel number	1...20 *		Linear machines Rotary machines
(7)	HOLDER	Holder code	A,B,C, D,E,M	A2	
(8)	FCSA1	Puff count for 1st FCSA	##.#	F6.1	
(9)	FCSA2	Puff count for 1st FCSA	##.#	F6.1	
(10)	FCSA3	Puff count for 2nd FCSA	#.##	F6.1	
(11)	FCSA4	Puff count for 3rd FCSA	#.##	F6.1	
(12)	FCSA5	Puff count for 4th FCSA	#.##	F6.1	

REMARKS

1. Please adhere strictly to the formats given above; it is much easier to read the data into computer files if all participating laboratories use the same format.

2. Write the sequence of variables as the first record on the file e.g.

EXP LAB DATE OPER RNUM CHAN HOLDER CIG PUFF TPM WATER NIC. NFDPM
EXP LAB DATE OPER RNUM CHAN HOLDER CIG FCSA1 FCSA2 FCSA3 FCSA4 FCSA5

so that any accidental permutation of the variables can be detected.

3. The format as given occupies 61 characters and so can easily be written as a single line on most terminals.

4. As noted above, missing data are to be coded with an asterisk (*).

5. When completed the floppy disc, together with a hard copy, should be sent to Mr. H. Dymond at

   45 Monarch Way, West End, Hampshire, SO30 3JQ, U.K.

   Telephone - 44-1703 476587 Fax - 44-1703 470707

   The disc MUST be accompanied by a listing of the contents of the file(s) on the disc.

ANNEX 3 CHARACTERISTICS OF FLOPPY DISCS TO BE USED

The following diskettes can be read :-
IBM Compatible 3.5 inches Single Density]
] (1.44 Mb)
IBM Compatible 3.5 inches Double Density]
The file type MUST be plain ASCII.

If you have difficulty in meeting these requirements would you please refer to your own computing departments for assistance or contact Mr. H. Dymond to investigate alternative possibilities.

ANNEX 4 LABORATORY CODES AND EXPECTED USE OF SMOKING MACHINE

LABORATORY	CODE	PROBABLE MACHINE
PM-Europe	PME	ROTARY
BAT-Benelux	BATBE	ROTARY
SEITA	SEITA	LINEAR
Tiedemanns	TIEDE	LINEAR
House of Prince	HOP	LINEAR
Swedish Match	SWM	LINEAR
BAT-Germany	BATGE	ROTARY
Reemtsma	REEMT	ROTARY
RJR-Germany	RJRGE	LINEAR
BAT-Southampton	BATUK	LINEAR
Gallaher	GALLA	LINEAR
Imperial Tob. Co.	IMPTO	LINEAR
Rothmans U.K.	ROTHM	LINEAR
Filtrona, Jarrow	FILTR	LINEAR
Royal Theodorus Niemeyer	NIEM	ROTARY
Alois Poschl	POSCH	ROTARY
Douwe Egberts Van Nelle	DOUWE	ROTARY
Rothmans The Netherlands	TURMAC	ROTARY

APPENDIX 3

SMOKING PLAN FOR 20-PORT MACHINES

Holder codes:

A. Labyrinth Seal 9mm insertion
B. SLEEVE 9mm insertion
C. SLEEVE 11mm insertion
D. SLEEVE 13mm insertion
E. SLEEVE 15mm insertion
M. Labyrinth Seal 9mm insertion CM2 Test Piece

Channel no:	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Run no: 1	D	A	B	C	D	E	M	B	D	A	M	E	C	A	M	E	C	B	D	C
2	C	D	E	M	A	B	C	M	E	C	B	D	A	C	B	D	A	M	E	A
3	B	M	A	B	C	D	E	C	B	D	A	M	E	D	A	M	E	C	B	E
4	E	B	C	D	E	M	A	D	A	M	E	C	B	M	E	C	B	D	A	D
5	M	E	M	A	B	C	D	E	C	B	D	A	M	B	D	A	M	E	C	M
6	A	C	D	E	M	A	B	A	M	E	C	B	D	E	C	B	D	A	M	B

APPENDIX 4

SMOKING PLAN FOR ROTARY MACHINES

Holder codes:

A. Labyrinth Seal 9mm insertion
B. SLEEVE 9mm insertion
C. SLEEVE 11mm insertion
D. SLEEVE 13mm insertion
E. SLEEVE 15mm insertion
M. Labyrinth Seal 9mm insertion CM2 Test Piece

Run number	Holder code	Run number	Holder code	Run number	Holder code	Run number	Holder code
1	D	31	A	61	A	91	C
2	E	32	M	62	E	92	B
3	A	33	D	63	C	93	D
4	C	34	E	64	M	94	M
5	B	35	A	65	E	95	C
6	M	36	B	66	C	96	D
7	B	37	C	67	B	97	E
8	D	38	M	68	D	98	A
9	A	39	B	69	A	99	B
10	E	40	D	70	M	100	M
11	C	41	C	71	E	101	B
12	M	42	D	72	A	102	M
13	A	43	E	73	B	103	C
14	E	44	M	74	C	104	B
15	C	45	E	75	D	105	D
16	B	46	C	76	M	106	A
17	D	47	B	77	D	107	E
18	M	48	D	78	A	108	M
19	A	49	A	79	E	109	A
20	B	50	M	80	C	110	B
21	C	51	D	81	A	111	C
22	B	52	A	82	M	112	D
23	D	53	E	83	E	113	E
24	A	54	C	84	C	114	M
25	E	55	B	85	B	115	C
26	M	56	M	86	D	116	D
27	E	57	B	87	A	117	B
28	C	58	C	88	M	118	A
29	B	59	D	89	A	119	E
30	D	60	E	90	E	120	M

Appendix 9. Study 16

Study of Holder Type and Insertion Depth using FCSAs at two different diameters

Objectives

- To determine the between-laboratory and within-laboratory variability of fine-cut smoking articles (FCSAs) made under "normal" conditions and at two diameters.
- To evaluate the effect of insertion depth on the smoke yields and between-laboratory and within-laboratory variability of 5.2 mm and 7.2 mm diameter FCSAs.
- To compare the between-laboratory and within-laboratory variability of 5.2 mm diameter FCSAs with the between-laboratory and within-laboratory variability of 7.2 mm diameter FCSAs.
- To evaluate the effect of insertion depth on the smoke yields of 7.2 mm diameter FCSAs when these articles are made under "normal" conditions and to contrast these data with those obtained from Study 15 (centrally made).
- To evaluate the local making of FCSAs, and to determine if there are any problems arising from articles being made locally or during the smoking (especially the 5.2 mm diameter FCSAs).

Limitations

As the analytical parameters of the experimental method are not yet fully specified, it must be noted that, although the statistical analysis of this study will be founded upon the within-laboratory and between-laboratory variability, this study cannot be used to estimate the repeatability (r) or the Reproducibility (R) of the method.

Extreme caution must be used when making any comparisons between the data arising from this study and that from Study 15 for the following reasons:

The fine-cut smoking articles were made by a central laboratory in Study 15 but were made by the participating laboratories in Study 16.

In this study the means are to be based upon the smoking of 20 FCSAs whereas in Study 15 the means were based upon the smoking of 5 FCSAs.

The design of the Sleeve holder for use in this study has been modified from that used in Study 15.

The mix of participating laboratories is different between the two studies.

Background

CORESTA Study 15 was conducted in order to determine the appropriate insertion depth required for the satisfactory smoking of fine-cut smoking articles. In order to do this, the study was conducted with fine-cut smoking articles of 7.2 mm diameter. In that study, the protocol for smoking was modified in order to achieve maximum sensitivity to determine the effect of insertion depth. In order to take these studies further, and to develop a satisfactory analytical method, it was necessary to study articles made at a diameter of 5.2 mm and to study the effect of repeating Study 15 under smoking conditions more akin to those that are likely to apply in a final method.

In particular, the current study was conducted with smoking articles made in the laboratories conducting the study and users of the rotary smoking machine operated the smoking machine in its "normal" mode. Although smoking conditions were more akin to those that are likely to apply in a final method, it was not possible to conduct this study in conditions that would allow r and R to be determined according to the rules of international standardisation.

Protocol

A copy of the Protocol as distributed to participants is attached as Annex 1.

Study design

The following holder configurations were studied: (Identified by letter codes given below).

1 7.2 mm diameter articles

A. Existing Labyrinth Seal holder, 9 mm depth of insertion, 29mm butt length.
C. Sleeve holder, 6.0 mm latex sleeve, 9 mm depth of insertion, 29mm butt length.
D. Sleeve holder, 6.0 mm latex sleeve, 13 mm depth of insertion, 29mm butt length.
E. Sleeve holder, 6.0 mm latex sleeve, 15 mm depth of insertion, 29mm butt length.

2 5.2 mm diameter articles

B. Existing Labyrinth Seal holder, 9 mm depth of insertion, 29mm butt length.
F. Sleeve holder, 6.0 mm latex sleeve, 9 mm depth of insertion, 29mm butt length.
G. Sleeve holder, 6.0 mm latex sleeve, 13 mm depth of insertion, 29mm butt length.
H. Sleeve holder, 6.0 mm latex sleeve, 15 mm depth of insertion, 29mm butt length.

FCSAs were made using a blend of fine-cut tobacco previously used (Dutch blend) with a German style wrapper, to a specification of 70mm length, 400 mg and 750 mg tobacco weight respectively at the two diameters. They were conditioned at 75%RH before smoking. The participating laboratories made the FCSAs. Seventeen laboratories were recruited to conduct smoking tests, each providing smoking data based on 100 FCSAs for each holder configuration.

The CORESTA Test Piece (CM2) was also used with the Labyrinth Seal holder and coded M.

Statistical Methods

Outlier Treatment

1.0.1 Linear machines

The analyses of these data are based upon the values of the analytes obtained by smoking 20 FCSAs through a single filter pad when using rotary machines, or the mean values of the analytes obtained by averaging the data for each holder configuration from four channels when using linear smoking machines (each channel smoking 5 FCSAs through a single filter pad).

Thus the data from the linear machines are available as means of 5 FCSAs, and it was agreed that these data should be scanned for outliers prior to the averaging over four channels.

For each laboratory using linear machines, a one-way analysis of variance between and within runs for each holder was used as a basis for detecting outliers for both NFDPM and nicotine. Any data point whose absolute standardised residual exceeded 2.5 was considered to be a possible outlier. These points were then referred to the originating laboratories to allow the data to be checked. Any points whose absolute standardised residual still exceeded 2.75 after checking were deemed to be outliers and were removed from the data.

For the linear machines there were 9 laboratories each producing 20 sets of data for each holder configuration plus the monitor. Thus, a total of 1620 data sets should have been produced for each analyte. One laboratory returned only 19 data sets. Thus, the actual total available for analysis was 1600. This outlier procedure resulted in the removal of the following number of data points from the data for linear machines:

Puffs	15 out of 1600 = 0.94%
TPM	21 out of 1600 = 1.31%
Water	19 out of 1600 = 1.19%
Nicotine	18 out of 1600 = 1.12%
NFDPM	28 out of 1600 = 1.75%

After the removal of these outliers, the data from the linear machines were averaged over four channels or the number of channels remaining per run, per holder configuration.

1.0.2 All machines

The analytes of interest in the analysis of this study are NFDPM and nicotine. It was therefore decided that the (NFDPM, nicotine) pairs of observations should be scanned for outliers, rather than scanning the data for each analyte separately. A 2-variate analysis of variance of the form

(NFDPM, nicotine) = Laboratory effects + error

for each holder configuration was used. This can be written as

$$\underset{(n \times 2)}{[\text{NFDPM, nicotine}]} = \underset{(n \times 17)}{[\text{Laboratory contrasts}]} * \underset{(17 \times 2)}{[\text{Coefficients}]} + \underset{(n \times 2)}{[\text{Error}]}$$

where [NFDPM, nicotine] is an (n x 2) matrix of observations, [Laboratory contrasts] is an (n x 17) matrix which represents the mean differences between the 17 laboratories which participated in this study, and [Error] is an (n x 2) matrix of residual errors. The coefficients of this model were then calculated in the usual way i.e. by making the determinant of the $[E'E]$ matrix a minimum.

Outliers were then detected by an examination of the plots of the (NFDPM, nicotine) elements of the [E] matrix.

In order to eliminate any subjectivity that might arise during the examination of these plots, a principal component decomposition of the [E] matrix was used, and the resulting principal component scores were normalised. The decomposition has the effect of removing the correlation known to exist between the (NFDPM, nicotine) residuals, and the Normalisation allows the presence of outliers to be detected by reference to a contour line drawn on the plot. For this study, any data point lying outside the 3.25 standard deviation (SD), contour line was deemed to be an outlier.

Whenever an outlier was detected, the corresponding (NFDPM, nicotine) data point was removed from the [NFDPM, nicotine] matrix and the [E] matrix was recalculated. This cycle was repeated until no further points appeared outside the 3.25SD contour line on the plots of residuals.

The process is illustrated by the Figures 1 to 6 which arose from the data for Holder E.

Figure 1 shows the plot of the elements [E], and Figure 2 shows the principal component decomposition of this plot with the 3.25SD contour line. From Figure 2 it can be seen that there are two outliers. The more extreme, on the left of the plot, was removed from the data and the [E] matrix was recalculated. Figures 3 and 4 show the plots after recalculation, and Figure 4 shows that the second outlier is still present. This point was removed from the data and the [E] matrix recalculated again. Figures 5 and 6 show the results, and from these two plots it can now be seen that no further outliers are present. Thus two data points were removed from the data for Holder E.

This procedure resulted in the removal of the following numbers of data points:

Holder A	Labyrinth seal	9mm insertion depth	5.2mm FCSAs	0
Holder B	Labyrinth seal	9mm insertion depth	7.2mm FCSAs	1
Holder C	Sleeve	9mm insertion depth	5.2mm FCSAs	3
Holder D	Sleeve	13mm insertion depth	5.2mm FCSAs	4
Holder E	Sleeve	15mm insertion depth	5.2mm FCSAs	2
Holder F	Sleeve	9mm insertion depth	7.2mm FCSAs	1
Holder G	Sleeve	13mm insertion depth	7.2mm FCSAs	2
Holder H	Sleeve	15mm insertion depth	7.2mm FCSAs	4
Monitor test piece	Labyrinth seal	9mm insertion depth		0

Total 17 out of 761 data points = 2.23%

1.0.3 Outlying laboratories

After the removal of outlying data points as described in 1.0.1 and 1.0.2, the data were examined for the presence of outlying laboratories. For this examination the data from the 5.2mm FCSAs and the 7.2mm FCSAs were treated separately. The correlation matrices arising from data shown in Annexes 1 and 2 were subjected to principal component analysis, and the first three principal component scores were used to compute the distance of each laboratory from the centre of the 3 dimensional space represented by the three principal components.

1.0.4 Outlying laboratories - 5.2mm FCSAs.

Annex 1 shows the results. At the first pass, laboratory 2 was deemed to be an outlier within the data from the 5.2mm FCSAs and was therefore deleted from all subsequent analyses of these data.

A repeat of the principal component analysis after the deletion of the data from laboratory 2 revealed no other outlying laboratory.

1.0.5 Outlying laboratories - 7.2mm FCSAs.

Annex 2 shows the results. At the first pass, laboratory 5 was deemed to be an outlier within the data from the 7.2mm FCSAs and was therefore deleted from all subsequent analyses of these data.

At the second pass (after the deletion of the data from Laboratory 5) Laboratory 2 was deemed to be an outlier within the data from the 7.2mm FCSAs and was therefore deleted from all subsequent analyses of these data.

A third pass of the principal component analysis after the deletion of the data from Laboratories 5 and 2 revealed no other outlying laboratory.

Since only Laboratory 2 was deemed an outlier for the data from the 5.2mm FCSAs, but both laboratories 2 and 5 were deemed to be outliers for the data from the 7.2mm FCSAs, subsequent analyses of the data from this study treated the 5.2mm FCSA and 7.2mm FCSA data sets as if they were independent studies.

Note that as a consequence, different laboratories provided the data sets for the 5.2 mm and 7.2 mm FCSAs. The mean CM2 data used is that from the laboratories retained in the 5.2 mm and 7.2 mm analyses respectively and may not be the same value.

2.0 Data for all further analysis

2.0.1 Tables 1 to 6 show the means, within-laboratory standard deviations, and the pooled within-laboratory standard deviations, after the removal of outliers and the deletion of the outlying laboratories as described in the foregoing sections.

3.0 Analysis of means

Using between-laboratory analyses of variance, the between-laboratory effects were removed from the data in order to simplify the between-holder analysis. Thus the analyses of variance shown in Tables 7 to 12 show adjusted means in the printout.

3.0.1 Analysis of means - 5.2mm FCSAs.

Tables 7 to 9 show the results of analyses of variance for puff number, nicotine, and NFDPM of the 5.2mm FCSAs.

Table 7 shows that there are no statistically significant differences between the puff numbers arising from the use of the various holder configurations when smoking 5.2mm FCSAs. The means are shown plotted in Figure 7.

Table 8 shows that the mean nicotine yield from holder A is significantly lower than that from holders C, D, or E. The mean nicotine yield from holder C is significantly lower than that from holder E, but there is no significant difference between the mean nicotine yields from holders D and E. The means are shown plotted in Figure 8.

Table 9 shows that the mean NFDPM yield from holder A is significantly lower than that from holders C, D, or E. The mean NFDPM yield from holder C is significantly lower than that from holders D or E, but there is no significant difference between the mean NFDPM yields from holders D and E. The means are shown plotted in Figure 9.

3.0.2 Analysis of means - 7.2mm FCSAs.

Tables 10 to 12 show the results of analyses of variance for puff number, nicotine, and NFDPM of the 7.2mm FCSAs.

Table 10 shows that there are no statistically significant differences between the puff numbers arising from the use of the various holder configurations when smoking 7.2mm FCSAs. The means are shown plotted in Figure 10.

Table 11 shows that the mean nicotine yield from holders B and F are not significantly different and the mean nicotine yield from holders G and H are not significantly different. There is however a statistically significant difference between the mean nicotine yield of both holder B and holder F and holders G and H. The means are shown plotted in Figure 11.

Table 12 shows that the mean NFDPM yield from holders B and F are not significantly different, and the mean NFDPM yield from holders G and H are not significantly different. There is however a statistically significant difference between the mean NFDPM yield of both holder B and holder F and holders G and H. The means are shown plotted in Figure 12.

3.0.3 Analysis of means - calculation of v and V.

The analyses of variance that enable the calculation of v and V are shown in Tables 13 to 22. The values of v and V are brought together in Tables 23 and 24. The values of v and V are also shown plotted in Figures 13 to 16.

From Tables 23 and 24, and from Figures 13 to 16, it can be seen that in terms of both v and V the 9 mm Labyrinth Seal holder produces the more variable data when used to smoke either 5.2 mm FCSAs or 7.2 mm FCSAs.

The situation with respect to the Sleeve holders is more complex.

In terms of v for nicotine, the data suggest that using a Sleeve holder with 15 mm insertion depth when smoking either 5.2 or 7.2 mm FCSAs gives rise to more variable data compared to that obtained when using either a 9 mm or a 13 mm insertion depth.

In terms of V for nicotine, when smoking either 5.2 mm or 7.2 mm FCSAs, there is little difference between any of the Sleeve holders. When smoking 7.2 mm FCSAs the data from the Labyrinth Seal holder are more variable than that from any of the Sleeve variants. When smoking 5.2 mm FCSAs, there is little difference between the Labyrinth Seal holder and any of the Sleeve variants.

In terms of v for NFDPM, when smoking 7.2 mm FCSAs the data from the Labyrinth Seal holder are more variable, and there is a slight downward trend as Sleeve holder insertion depth is increased. When smoking 5.2 mm FCSAs, there is little difference between the Labyrinth Seal holder and the Sleeve holder using 9 mm or 15 mm insertion. The data arising from the Sleeve holder using 13 mm insertion might be somewhat less variable.

In terms of V for NFDPM, when smoking 7.2 mm FCSAs, the data from the Labyrinth Seal holder are more variable than that from any of the Sleeve holder variants, and there might be a slight downward trend in variability as insertion depth into the Sleeve holder is increased. When smoking 5.2 mm FCSAs the data from the Labyrinth Seal holder are somewhat more variable than arose from any of the Sleeve holders. There is also an indication that the data from the Sleeve holder using 15 mm insertion depth are more variable than that from the holders using either 9 mm or 13 mm insertion.

4.0 Analysis of standard deviations.

4.0.1 Data

The transformed within-laboratory standard deviations, after the removal of outliers and the deletion of outlying laboratories, are shown in Tables 25 to 28. The transformation used is

$$\text{Transformed SD} = (\text{SD})^{0.25}$$

This transformation is used as a variance stabilising transformation, taking account of the fact that standard deviations are not themselves Normally distributed.

4.0.2 Analyses of variance of transformed standard deviations.

The results of the analyses of variance of the transformed standard deviations are shown in Tables 29 to 34.

From Table 29 it can be seen that there are no significant differences between the transformed standard deviations of puff numbers when smoking 5.2mm FCSAs through any of the holder variants used in this study.

From Table 30 it can be seen that there is a statistically significant difference between the transformed standard deviations of nicotine yields of the data from the A and D holders (Labyrinth Seal, Sleeve using 13mm insertion) when smoking 5.2mm FCSAs. No other comparisons of the holder variants showed a significant difference.

From Table 31 it can be seen that there are no significant differences between the transformed standard deviations of NFDPM yields of any of the holder variants used in this study when smoking 5.2mm FCSAs.

From Table 32 it can be seen that there are no significant differences between the transformed standard deviations of puff numbers when smoking 7.2mm FCSAs through any of the holder variants used in this study.

From Table 33 it can be seen that there are no significant differences between the transformed standard deviations of nicotine yields when smoking 7.2mm FCSAs through any of the holder variants used in this study.

From Table 34 it can be seen that there is a statistically significant difference between the transformed standard deviations of NFDPM yields of the data from the B and G holders (Labyrinth Seal, Sleeve using 13mm insertion) when smoking 7.2mm FCSAs. No other comparisons of the holder variants showed a significant difference.

The overall mean transformed standard deviations are shown plotted in Figures 17 to 19.

5.0 Studies 15 and 16.

For completeness, Figures 20 to 23 show plots of data obtained from Studies 15 and 16. Only the 7.2mm FCSA data from Study 16 was used, since no 5.2mm FCSAs were smoked during the execution of Study 15.

From Figure 20 it can be seen that, in terms of mean nicotine yield, there is close agreement between the results from Studies 15 and 16.

From Figure 21 it can be seen that, in terms of mean NFDPM yield, there is close agreement between the results from Studies 15 and 16, although the agreement is perhaps not quite as close as that shown by the nicotine data.

From Figure 22 it can be seen that in terms of the within-laboratory standard deviation of nicotine yields there is some disparity between Studies 15 and 16. It is to be expected that Study 16 would show greater variability than Study 15, given the fact that for Study 15 the FCSAs were made at one location and could therefore be expected to be more uniform than the FCSAs used in Study 16 that were produced in each individual participating laboratory. This might be one of the factors which might explain, at least in part, the difference in general level between the two lines on the graph, but this does not explain the difference between the performance of the Sleeve holder using 15 mm insertion depth.

From Figure 23 it can be seen that in terms of the within-laboratory standard deviations of NFDPM yields, there is some disparity between Studies 15 and 16. In Study 15 the Labyrinth Seal holder gave results which were somewhat less variable than the Sleeve holder using 9 mm insertion depth, whereas in Study 16 the reverse is the case.

TABLE 1

STUDY 16

5.2 mm FCSAs OUTLIERS REMOVED LABORATORY 2 DELETED

PUFFS - MEAN

	A	C	D	E	M
1	5.0400	5.0800	5.0200	5.4600	8.8600
3	4.9200	4.9875	4.8875	4.7875	9.0500
4	4.9450	4.9483	4.8800	4.8100	8.7850
5	4.5050	4.6300	4.5950	4.6500	9.2500
6	4.5250	4.4083	4.5600	4.5550	8.8950
7	4.7120	4.8640	4.9280	4.8760	9.3560
8	5.0200	5.0600	4.9600	4.8000	9.3000
11	5.3800	5.0000	4.9400	5.1000	9.2600
12	5.0900	5.1133	5.0133	5.0867	9.1450
13	5.2050	5.3750	5.3300	5.4900	9.1700
14	5.0750	5.0500	4.9350	4.9850	9.5317
15	5.5150	5.2950	5.2100	5.2183	9.3750
16	4.6950	4.7350	4.8250	4.8550	9.6350
17	4.8600	5.1600	4.8400	4.8200	8.6200
18	4.8400	4.7400	4.6800	4.6600	8.9400
19	4.5800	4.6500	4.5000	4.4600	9.3000
ALL	4.9317	4.9467	4.8863	4.9126	9.1545

PUFFS - STANDARD DEVIATIONS

	A	C	D	E	M
1	0.1673	0.1304	0.1304	0.7893	0.1517
3	0.1081	0.0595	0.2026	0.1436	0.0935
4	0.2080	0.1246	0.1110	0.1232	0.1330
5	0.0570	0.1408	0.1110	0.1090	0.2236
6	0.2910	0.2686	0.1851	0.1483	0.1972
7	0.2195	0.1408	0.1119	0.0654	0.1350
8	0.1095	0.1949	0.0548	0.1581	0.1225
11	0.2387	0.1414	0.2074	0.2000	0.1949
12	0.3029	0.2467	0.1657	0.2463	0.1614
13	0.2960	0.5022	0.4364	0.4666	0.1736
14	0.2391	0.2084	0.1098	0.1318	0.1588
15	0.2673	0.2869	0.1282	0.2000	0.1541
16	0.2923	0.2661	0.1920	0.1535	0.0652
17	0.1673	0.2881	0.1140	0.1095	0.1304
18	0.1140	0.1673	0.1304	0.1517	0.1342
19	0.2280	0.1291	0.1155	0.2191	0.2121
ALL	0.2200	0.2325	0.1775	0.2770	0.1579

TABLE 2

STUDY 16

5.2mm FCSAs OUTLIERS REMOVED LABORATORY 2 DELETED

Nicotine - MEAN (mg)

	A	C	D	E	M
1	0.5976	0.5922	0.5916	0.6080	1.5600
3	0.5640	0.5975	0.5681	0.5794	1.5705
4	0.5594	0.5696	0.5675	0.5628	1.6146
5	0.5730	0.4745	0.5105	0.5455	1.5460
6	0.5939	0.6115	0.6551	0.6532	1.5341
7	0.6524	0.6204	0.6306	0.6448	1.5442
8	0.5654	0.5930	0.6178	0.5754	1.5706
11	0.4760	0.5700	0.5780	0.5225	1.5160
12	0.6000	0.6365	0.6470	0.6502	1.5440
13	0.5802	0.6455	0.6424	0.6753	1.5906
14	0.5355	0.6180	0.6115	0.6360	1.5815
15	0.5265	0.6305	0.6225	0.6437	1.5634
16	0.7027	0.6998	0.7229	0.7006	1.6038
17	0.5980	0.6340	0.6560	0.6820	1.5680
18	0.5080	0.5628	0.6014	0.5892	1.5978
19	0.5622	0.6330	0.6080	0.5948	1.6960
ALL	0.5747	0.6053	0.6151	0.6181	1.5751

Nicotine - STANDARD DEVIATIONS

	A	C	D	E	M
1	0.0329	0.0353	0.0433	0.0335	0.0306
3	0.0129	0.0167	0.0069	0.0461	0.0199
4	0.0289	0.0416	0.0391	0.0416	0.0354
5	0.0330	0.0476	0.0421	0.0612	0.0624
6	0.0595	0.0440	0.0364	0.0886	0.0493
7	0.0188	0.0323	0.0138	0.0319	0.0224
8	0.0380	0.0229	0.0244	0.0275	0.0205
11	0.0513	0.0524	0.0268	0.0395	0.0288
12	0.0352	0.0147	0.0331	0.0363	0.0221
13	0.0480	0.0223	0.0274	0.0486	0.0419
14	0.0323	0.0239	0.0320	0.0258	0.0175
15	0.0540	0.0187	0.0555	0.0312	0.0387
16	0.0460	0.0497	0.0459	0.0300	0.0358
17	0.0517	0.0167	0.0219	0.0130	0.0646
18	0.0629	0.0335	0.0188	0.0207	0.0229
19	0.0340	0.0300	0.0225	0.0393	0.0534
ALL	0.0422	0.0340	0.0334	0.0420	0.0384

TABLE 3

STUDY 16

5.2mm FCSAs OUTLIERS REMOVED LABORATORY 2 DELETED

NFDPM - MEAN (mg)

	A	C	D	E	M
1	8.084	7.624	7.760	7.934	15.652
3	7.042	7.827	7.706	7.925	15.563
4	7.250	7.071	7.492	7.400	15.199
5	7.235	6.295	6.695	7.300	15.695
6	8.011	8.368	8.894	8.938	15.769
7	8.648	8.090	8.376	8.220	15.846
8	7.284	7.302	7.758	7.260	15.260
11	5.238	6.748	7.174	6.483	15.616
12	8.061	8.396	8.718	8.784	15.840
13	7.534	7.852	8.225	8.658	15.767
14	6.718	8.039	8.251	8.558	16.087
15	6.338	8.235	8.248	8.530	16.077
16	9.075	8.476	9.049	8.759	16.357
17	6.832	7.274	7.706	8.060	15.136
18	6.372	6.994	7.554	7.514	15.514
19	7.710	7.782	7.713	8.066	15.698
ALL	7.339	7.644	7.964	8.045	15.692

NFDPM - STANDARD DEVIATIONS

	A	C	D	E	M
1	0.228	0.198	0.479	0.236	0.325
3	0.213	0.300	0.243	0.410	0.225
4	0.463	0.681	0.506	0.260	0.341
5	0.169	0.574	0.632	0.714	0.401
6	0.635	0.657	0.536	0.960	0.408
7	0.318	0.171	0.401	0.481	0.184
8	0.417	0.317	0.139	0.341	0.246
11	0.502	0.324	0.416	0.480	0.425
12	0.677	0.441	0.150	0.587	0.334
13	0.647	0.816	0.347	0.600	0.229
14	0.489	0.358	0.387	0.374	0.443
15	0.552	0.215	0.530	0.158	0.258
16	0.563	0.740	0.350	0.539	0.262
17	0.713	0.210	0.188	0.409	0.407
18	0.530	0.280	0.259	0.287	0.282
19	0.472	0.693	0.407	0.440	0.431
ALL	0.501	0.484	0.401	0.495	0.336

TABLE 4

STUDY 16

7.2mm FCSAs OUTLIERS REMOVED LABORATORIES 2 and 5 DELETED

PUFFS - MEAN

LAB	B	F	G	H	M
1	6.5200	6.7400	6.4600	6.6200	8.8600
3	5.9550	6.3700	6.2217	6.2850	9.0500
4	6.0400	6.1500	5.9500	5.9550	8.7850
6	6.1375	5.8750	6.1950	6.2550	8.8950
7	6.5100	6.6320	6.5480	6.6500	9.3560
8	6.2400	6.5600	6.3200	6.3000	9.3000
11	6.2600	6.7200	6.8000	6.4000	9.2600
12	6.6150	6.3400	6.3800	6.3967	9.1450
13	6.8617	6.9900	6.9300	6.7150	9.1700
14	6.6150	6.1550	6.1450	6.0800	9.5317
15	7.0700	6.5750	6.6550	6.4783	9.3750
16	5.7250	5.8687	5.6479	5.8313	9.6350
17	6.5400	6.4400	6.6200	6.6250	8.6200
18	6.9000	6.8400	6.5750	7.0000	8.9400
19	6.3200	6.7600	6.5000	6.5000	9.3000
ALL	6.4340	6.4758	6.4043	6.4110	9.1482

PUFFS - STANDARD DEVIATIONS

LAB	B	F	G	H	M
1	0.3493	0.2608	0.1342	0.1483	0.1517
3	0.1972	0.1823	0.2281	0.0627	0.0935
4	0.2815	0.3206	0.2455	0.3262	0.1330
6	0.4225	0.2675	0.2654	0.3370	0.1972
7	0.1933	0.2809	0.3672	0.2582	0.1350
8	0.1140	0.2510	0.2775	0.2739	0.1225
11	0.3286	0.1095	0.2345	0.2000	0.1949
12	0.4152	0.2854	0.2321	0.2406	0.1614
13	0.4004	0.4103	0.3680	0.4557	0.1736
14	0.4022	0.1328	0.2672	0.1217	0.1588
15	0.0622	0.0968	0.1653	0.2025	0.1541
16	0.1242	0.1344	0.0980	0.1807	0.0652
17	0.2881	0.1949	0.2049	0.1500	0.1304
18	0.2739	0.2608	0.1708	0.2915	0.1342
19	0.2168	0.1517	0.2000	0.1000	0.2121
ALL	0.2933	0.2399	0.2445	0.2471	0.1525

TABLE 5

STUDY 16

7.2mm FCSAs OUTLIERS REMOVED LABORATORIES 2 and 5 DELETED

Nicotine - MEAN (mg)

LAB	B	F	G	H	M
1	1.0854	1.0252	1.1052	1.1036	1.5600
3	1.2035	1.0592	1.1645	1.1650	1.5705
4	1.0789	1.0040	1.0281	1.0535	1.6146
6	1.0036	1.0196	1.1181	1.1120	1.5341
7	1.2068	1.0840	1.1544	1.1320	1.5442
8	1.0076	1.0026	1.0492	1.0602	1.5706
11	0.9660	0.9620	1.0640	1.0020	1.5160
12	1.0240	1.0735	1.0530	1.0968	1.5440
13	1.0997	1.1296	1.1366	1.1429	1.5906
14	0.8497	0.9370	0.9760	1.0195	1.5815
15	0.7830	1.0315	1.0780	1.0750	1.5634
16	1.1802	1.2049	1.2096	1.2744	1.6038
17	1.0940	1.0500	1.1880	1.1525	1.5680
18	0.9296	1.0046	1.0720	1.1044	1.5978
19	1.2106	1.0556	1.1382	1.1448	1.6960
ALL	1.0470	1.0407	1.1013	1.1064	1.5770

Nicotine - STANDARD DEVIATIONS

LAB	B	F	G	H	M
1	0.0471	0.0275	0.0412	0.0521	0.0306
3	0.0190	0.0236	0.0411	0.0399	0.0199
4	0.0645	0.0546	0.0603	0.0695	0.0354
6	0.0429	0.0463	0.0763	0.0509	0.0493
7	0.0208	0.0205	0.0399	0.0302	0.0224
8	0.0458	0.0388	0.0311	0.0551	0.0205
11	0.0747	0.0259	0.0305	0.0303	0.0288
12	0.0476	0.0468	0.0158	0.0787	0.0221
13	0.0563	0.0676	0.0379	0.0284	0.0419
14	0.0600	0.0286	0.0432	0.0078	0.0175
15	0.0518	0.0141	0.0209	0.0611	0.0387
16	0.0259	0.0309	0.0496	0.0477	0.0358
17	0.0498	0.0534	0.0512	0.0419	0.0646
18	0.0600	0.0658	0.0215	0.0262	0.0229
19	0.0915	0.0627	0.0531	0.0664	0.0534
ALL	0.0544	0.0441	0.0439	0.0495	0.0362

TABLE 6

STUDY 16

7.2mm FCSAs OUTLIERS REMOVED LABORATORIES 2 and 5 DELETED

NFDPM - MEAN (mg)

LAB	B	F	G	H	M
1	13.968	12.834	13.868	14.130	15.652
3	15.671	12.760	14.577	14.403	15.563
4	13.634	12.467	13.015	13.295	15.199
6	13.193	13.703	14.804	14.982	15.769
7	15.484	13.726	14.738	14.078	15.846
8	12.808	12.246	13.028	13.370	15.260
11	11.916	11.456	13.610	13.228	15.616
12	13.356	13.842	13.824	14.716	15.840
13	13.323	13.548	14.181	14.182	15.767
14	10.582	12.286	13.171	13.620	16.087
15	9.265	13.084	14.093	14.090	16.077
16	14.492	14.402	14.815	15.782	16.357
17	12.508	11.748	13.696	13.307	15.136
18	10.916	11.554	13.068	13.226	15.514
19	15.110	12.460	13.982	14.224	15.698
ALL	13.061	12.786	13.897	14.028	15.692

NFDPM - STANDARD DEVIATIONS

LAB	B	F	G	H	M
1	0.556	0.716	0.624	0.687	0.325
3	0.395	0.524	0.614	0.533	0.225
4	0.702	0.265	0.474	0.804	0.341
6	0.650	0.602	0.560	0.399	0.408
7	0.317	0.496	0.447	0.188	0.184
8	0.601	0.564	0.233	0.569	0.246
11	1.297	0.536	0.305	0.361	0.425
12	0.523	0.615	0.549	0.674	0.334
13	0.534	0.940	0.725	0.794	0.229
14	1.135	0.388	0.984	0.343	0.443
15	0.710	0.580	0.633	0.672	0.258
16	0.306	0.791	0.456	0.356	0.262
17	0.541	0.561	0.556	0.628	0.407
18	1.173	0.662	0.095	0.342	0.282
19	0.700	0.754	0.252	0.470	0.431
ALL	0.743	0.617	0.550	0.553	0.331

TABLE 7

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
MONITOR OMITTED
ANALYSIS OF MEAN PUFFS
ANALYSIS OF VARIANCE ON PUFFS
SOURCE     DF        SS        MS         F        p
HLD         3    0.1648    0.0549      1.01    0.389
ERROR     295   16.0902    0.0545
TOTAL     298   16.2550
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
              ADJUSTED             BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ----+---------+---------+---------+--
     A     80    4.9337    0.2289            (---------*---------)
     C     78    4.9437    0.2252              (---------*---------)
     D     78    4.8832    0.1722 (----------*---------)
     E     78    4.9167    0.2736        (---------*---------)
                                   ----+---------+---------+---------+--
POOLED STDEV =   0.2335              4.850     4.900     4.950     5.000

Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)

           A         C         D
     C   -0.0713
          0.0513
     D   -0.0108   -0.0012
          0.1118    0.1222
     E   -0.0443   -0.0347   -0.0952
          0.0783    0.0887    0.0282
```

TABLE 8

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
MONITOR OMITTED
ANALYSIS OF MEAN nicotine
SOURCE     DF        SS        MS         F        p
HLD         3   0.09033   0.03011     16.86    0.000
ERROR     295   0.52671   0.00179
TOTAL     298   0.61704
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
              ADJUSTED             BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  -------+---------+---------+---------
     A     80   0.57517   0.05008 (----*---)
     C     78   0.60539   0.03702                (----*---)
     D     78   0.61520   0.03389                     (----*---)
     E     78   0.61746   0.04181                      (----*---)
                                   -------+---------+---------+--------
POOLED STDEV = 0.04225                  0.580     0.600     0.620

Fisher's pairwise comparisons
Individual error rate = 0.0500
Intervals for (column level mean) - (row level mean)
         A        C        D
     C -0.04131
       -0.01913
     D -0.05112 -0.02097
       -0.02894  0.00135
     E -0.05338 -0.02323 -0.01342
       -0.03120 -0.00091  0.00890
```

TABLE 9

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
MONITOR OMITTED
ANALYSIS OF MEAN NFDPM
SOURCE     DF        SS        MS        F        p
HLD         3    23.833     7.944    24.79    0.000
ERROR     295    94.529     0.320
TOTAL     298   118.362
                                   INDIVIDUAL 95 PCT CI’S FOR MEAN
              ADJUSTED             BASED ON POOLED STDEV
HOLDER      N       MEAN     STDEV  ----------+---------+---------+------
     A     80     7.3446    0.7121   (---*---)
     C     78     7.6487    0.5026             (---*---)
     D     78     7.9682    0.4286                        (---*---)
     E     78     8.0314    0.5206                          (---*---)
                                    ----------+---------+---------+-----
POOLED STDEV =   0.3204                      7.50      7.80      8.10

Fisher’s pairwise comparisons
Individual error rate = 0.0500
Intervals for (column level mean) - (row level mean)
            A         C         D
     C   -0.3882
         -0.2200
     D   -0.7077   -0.4042
         -0.5395   -0.2348
     E   -0.7709   -0.4674   -0.1478
         -0.6027   -0.2980    0.0214
```

TABLE 10

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
MONITOR DATA OMITTED
ANALYSIS OF MEAN PUFFS
ANALYSIS OF VARIANCE ON PUFFS
SOURCE     DF        SS        MS        F        p
HLD         3    0.2054    0.0685     0.88    0.449
ERROR     275   21.3166    0.0775
TOTAL     278   21.5221
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
              ADJUSTED             BASED ON POOLED STDEV
HOLDER      N       MEAN     STDEV  ---+---------+---------+---------+--
     B     73     6.4289    0.3249      (---------*----------)
     F     74     6.4751    0.2581              (---------*----------)
     G     73     6.4092    0.2463   (---------*----------)
     H     73     6.4119    0.2496   (----------*---------)
                                    ---+---------+---------+---------+--
POOLED STDEV =   0.2784             6.360     6.420     6.480     6.540

Fisher's pairwise comparisons
Individual error rate = 0.0500
Intervals for (column level mean) - (row level mean)
            B         F         G
     F   -0.1220
          0.0296
     G   -0.0563   -0.0099
          0.0957    0.1417
     H   -0.0590   -0.0126   -0.0787
          0.0930    0.1390    0.0733
```

TABLE 11

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
MONITOR DATA OMITTED
ANALYSIS OF MEAN nicotine
ANALYSIS OF VARIANCE ON nicotine
SOURCE     DF        SS        MS        F        p
HLD         3   0.26703   0.08901    21.82    0.000
ERROR     275   1.12194   0.00408
TOTAL     278   1.38897
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
               ADJUSTED            BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  --------+---------+---------+-------
     B     73    1.0469    0.0867    (----*----)
     F     74    1.0407    0.0529  (----*----)
     G     73    1.1006    0.0495                      (----*----)
     H     73    1.1070    0.0527                        (----*----)
                                   --------+---------+---------+-------
POOLED STDEV =   0.0639                  1.050     1.080     1.110

Fisher's pairwise comparisons
Individual error rate = 0.0500
Intervals for (column level mean) - (row level mean)
            B         F         G
     F  -0.01119
         0.02359
     G  -0.07114  -0.07729
        -0.03626  -0.04251
     H  -0.07754  -0.08369  -0.02384
        -0.04266  -0.04891   0.01104
```

TABLE 12

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
MONITOR DATA OMITTED
ANALYSIS OF MEAN NFDPM
ANALYSIS OF VARIANCE ON NFDPM
SOURCE     DF        SS        MS        F        p
HLD         3    80.137    26.712    30.01    0.000
ERROR     275   244.760     0.890
TOTAL     278   324.897
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
               ADJUSTED            BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ---------+---------+---------+------
     B     73    13.075     1.372       (----*---)
     F     74    12.790     0.762   (---*---)
     G     73    13.883     0.679                        (----*---)
     H     73    14.024     0.684                           (---*----)
                                   ---------+---------+---------+------
POOLED STDEV =    0.943                  13.00     13.50     14.00

Fisher's pairwise comparisons
Individual error rate = 0.0500
Intervals for (column level mean) - (row level mean)
            B         F         G
     F    0.0282
          0.5418
     G   -1.0656   -1.3498
         -0.5503   -0.8362
     H   -1.2067   -1.4908   -0.3987
         -0.6913   -0.9772    0.1167
```

TABLE 13.

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
CALCULATION OF V and v
HOLDER A
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         15   0.215867   0.014391    8.07  0.000
Error       64   0.114165   0.001784
Total       79   0.330032
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00252   2   (2) +     5.0000(1)
 2 Error     0.00178       (2)
             v = 0.118      V = 0.184
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         15    67.6746     4.5116   17.94  0.000
Error       64    16.0927     0.2514
Total       79    83.7673
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB        0.8520   2   (2) +     5.0000(1)
 2 Error      0.2514       (2)
              v = 1.404     V = 2.941
```

TABLE 14.

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
CALCULATION OF V and v
HOLDER C
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         15   0.179151   0.011943   10.35  0.000
Error       62   0.071555   0.001154
Total       77   0.250706
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00221   2   (2) +     4.8735(1)
 2 Error     0.00115       (2)
             v = 0.095      V = 0.162
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         15    31.0050     2.0670    8.82  0.000
Error       62    14.5248     0.2343
Total       77    45.5298
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB        0.3761   2   (2) +     4.8735(1)
 2 Error      0.2343       (2)
              v = 1.355     V = 2.187
```

TABLE 15.

STUDY 16

5.2mm FCSAs OUTLIERS REMOVED LABORATORY 2 DELETED
CALCULATION OF V and v

HOLDER D

Analysis of Variance for nicotine

Source	DF	SS	MS	F	P
LAB	15	0.170486	0.011366	10.17	0.000
Error	62	0.069280	0.001117		
Total	77	0.239766			

Source	Variance component	Error term	Expected Mean Square (using unrestricted model)
1 LAB	0.00210	2	(2) + 4.8735(1)
2 Error	0.00112		(2)

v = 0.094 V = 0.159

Analysis of Variance for NFDPM

Source	DF	SS	MS	F	P
LAB	15	29.4586	1.9639	12.21	0.000
Error	62	9.9696	0.1608		
Total	77	39.4282			

Source	Variance component	Error term	Expected Mean Square (using unrestricted model)
1 LAB	0.3700	2	(2) + 4.8735(1)
2 Error	0.1608		(2)

v = 1.122 V = 2.040

TABLE 16.

STUDY 16

5.2mm FCSAs OUTLIERS REMOVED LABORATORY 2 DELETED
CALCULATION OF V and v

HOLDER E

Analysis of Variance for nicotine

Source	DF	SS	MS	F	P
LAB	15	0.191259	0.012751	7.22	0.000
Error	62	0.109428	0.001765		
Total	77	0.300686			

Source	Variance component	Error term	Expected Mean Square (using unrestricted model)
1 LAB	0.00225	2	(2) + 4.8735(1)
2 Error	0.00176		(2)

v = 0.117 V = 0.177

Analysis of Variance for NFDPM

Source	DF	SS	MS	F	P
LAB	15	33.0257	2.2017	8.97	0.000
Error	62	15.2181	0.2455		
Total	77	48.2438			

Source	Variance component	Error term	Expected Mean Square (using unrestricted model)
1 LAB	0.4014	2	(2) + 4.8735(1)
2 Error	0.2455		(2)

v = 1.387 V = 2.252

TABLE 17.

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
CALCULATION OF V and v
HOLDER M
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         15   0.130967   0.008731    5.93  0.000
Error       64   0.094170   0.001471
Total       79   0.225137
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00145   2   (2) +     5.0000(1)
 2 Error     0.00147       (2)
             v = 0.107      V = 0.151
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         15     7.9910     0.5327    4.73  0.000
Error       64     7.2043     0.1126
Total       79    15.1954
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.08403   2   (2) +     5.0000(1)
 2 Error     0.11257       (2)
             v = 0.939      V = 1.242
```

TABLE 18

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
CALCULATION OF v and V
HOLDER B
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         14   1.155245   0.082518   27.89  0.000
Error       58   0.171629   0.002959
Total       72   1.326874
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.01635   2   (2) +     4.8650(1)
 2 Error     0.00296       (2)
             v = 0.152      V = 0.389
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         14    233.388     16.671   30.22  0.000
Error       58     31.994      0.552
Total       72    265.382
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB        3.3133   2   (2) +     4.8650(1)
 2 Error      0.5516       (2)
              v = 2.080     V = 5.505
```

TABLE 19

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
CALCULATION OF v and V
HOLDER F
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         14   0.274549   0.019611   10.10  0.000
Error       59   0.114558   0.001942
Total       73   0.389108
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00358   2   (2) +     4.9324(1)
 2 Error     0.00194       (2)
             v = 0.123      V = 0.208
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         14    53.5778     3.8270   10.06  0.000
Error       59    22.4389     0.3803
Total       73    76.0167
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB        0.6988   2   (2) +     4.9324(1)
 2 Error      0.3803       (2)
              v = 1.727     V = 2.909
```

TABLE 20

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
CALCULATION OF v and V
HOLDER G
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         14   0.276713   0.019765   10.26  0.000
Error       58   0.111780   0.001927
Total       72   0.388492
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00367   2   (2) +     4.8650(1)
 2 Error     0.00193       (2)
             v = 0.123      V = 0.210
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         14    27.6594     1.9757    6.53  0.000
Error       58    17.5423     0.3025
Total       72    45.2016
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB        0.3439   2   (2) +     4.8650(1)
 2 Error      0.3025       (2)
              v = 1.54      V = 2.251
```

TABLE 21

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
CALCULATION OF v and V
HOLDER H
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         14   0.278341   0.019881    8.12  0.000
Error       58   0.142061   0.002449
Total       72   0.420402
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00358   2   (2) +     4.8650(1)
 2 Error     0.00245       (2)
             v = 0.139      V = 0.217
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         14    34.5015     2.4644    8.06  0.000
Error       58    17.7367     0.3058
Total       72    52.2383
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB        0.4437   2   (2) +     4.8650(1)
 2 Error      0.3058       (2)
              v = 1.548     V = 2.424
```

TABLE 22

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
CALCULATION OF v and V
HOLDER M
Analysis of Variance for nicotine
Source      DF         SS         MS       F      P
LAB         14   0.126461   0.009033    6.90  0.000
Error       60   0.078575   0.001310
Total       74   0.205036
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.00154   2   (2) +     5.0000(1)
 2 Error     0.00131       (2)
             v = 0.101      V = 0.149
Analysis of Variance for NFDPM
Source      DF         SS         MS       F      P
LAB         14     7.9910     0.5708    5.22  0.000
Error       60     6.5626     0.1094
Total       74    14.5536
Source      Variance Error Expected Mean Square
           component  term (using unrestricted model)
 1 LAB       0.09228   2   (2) +     5.0000(1)
 2 Error     0.10938       (2)
             v = 0.926      V = 1.257
```

TABLE 23.

STUDY 16
TABLE OF v AND V
5.2mm FCSAs
OUTLIERS REMOVED LABORATORY 2 DELETED
nicotine

	ISO 9mm	SLEEVE 9mm	SLEEVE 13mm	SLEEVE 15mm	MONITOR
v	0.118	0.095	0.094	0.117	0.107
V	0.184	0.162	0.159	0.177	0.151

NFDPM

	ISO 9mm	SLEEVE 9mm	SLEEVE 13mm	SLEEVE 15mm	MONITOR
v	1.404	1.355	1.122	1.387	0.939
V	2.941	2.187	2.040	2.252	1.242

TABLE 24.

STUDY 16
TABLE OF v AND V
7.2mm FCSAs
OUTLIERS REMOVED
nicotine

	ISO 9mm	SLEEVE 9mm	SLEEVE 13mm	SLEEVE 15mm	MONITOR
v	0.152	0.123	0.123	0.139	0.101
V	0.389	0.208	0.210	0.217	0.149

NFDPM

	ISO 9mm	SLEEVE 9mm	SLEEVE 13mm	SLEEVE 15mm	MONITOR
v	2.080	1.727	1.540	1.548	0.926
V	5.505	2.909	2.251	2.424	1.257

TABLE 25.

STUDY 16

TABLE OF MEAN TRANSFORMED SDs

OUTLIERS REMOVED OUTLYING LABORATORIES DELETED.

5.2mm FCSAs

PUFFS

LAB	A	C	D	E	M	ALL
1	0.63955	0.60092	0.60092	0.94256	0.62409	0.68161
3	0.57340	0.49389	0.67090	0.61559	0.55297	0.58135
4	0.67533	0.59413	0.57721	0.59245	0.60390	0.60860
5	0.48862	0.61256	0.57721	0.57459	0.68765	0.58812
6	0.73447	0.71991	0.65592	0.62056	0.66639	0.67945
7	0.68448	0.61256	0.57837	0.50570	0.60615	0.59745
8	0.57525	0.66444	0.48383	0.63057	0.59161	0.58914
11	0.69898	0.61321	0.67484	0.66874	0.66444	0.66404
12	0.74186	0.70476	0.63801	0.70448	0.63383	0.68459
13	0.73760	0.84182	0.81278	0.82649	0.64549	0.77283
14	0.69927	0.67565	0.57564	0.60253	0.63127	0.63687
15	0.71903	0.73187	0.59837	0.66874	0.62654	0.66891
16	0.73529	0.71823	0.66195	0.62593	0.50531	0.64934
17	0.63955	0.73263	0.58107	0.57525	0.60092	0.62588
18	0.58107	0.63955	0.60092	0.62409	0.60525	0.61018
19	0.69101	0.59942	0.58297	0.68416	0.67863	0.64724
ALL	0.66342	0.65972	0.61693	0.65390	0.62028	0.64285

Nicotine

LAB	A	C	D	E	M	ALL
1	0.42589	0.43345	0.45617	0.42782	0.41824	0.43232
3	0.33701	0.35948	0.28821	0.46337	0.37559	0.36473
4	0.41231	0.45162	0.44468	0.45162	0.43376	0.43880
5	0.42621	0.46709	0.45297	0.49738	0.49980	0.46869
6	0.49389	0.45800	0.43679	0.54558	0.47121	0.48109
7	0.37029	0.42394	0.34274	0.42262	0.38687	0.38929
8	0.44152	0.38901	0.39523	0.40722	0.37839	0.40227
11	0.47591	0.47845	0.40461	0.44581	0.41195	0.44335
12	0.43315	0.34820	0.42654	0.43649	0.38557	0.40599
13	0.46807	0.38643	0.40685	0.46953	0.45243	0.43666
14	0.42394	0.39319	0.42295	0.40078	0.36371	0.40091
15	0.48206	0.36979	0.48537	0.42028	0.44353	0.44021
16	0.46312	0.47216	0.46286	0.41618	0.43498	0.44986
17	0.47684	0.35948	0.38469	0.33766	0.50415	0.41257
18	0.50080	0.42782	0.37029	0.37931	0.38901	0.41344
19	0.42941	0.41618	0.38730	0.44524	0.48071	0.43177
ALL	0.44128	0.41464	0.41052	0.43543	0.42687	0.42575

TABLE 26.

STUDY 16

TABLE OF MEAN TRANSFORMED SDs

OUTLIERS REMOVED OUTLYING LABORATORIES DELETED.

5.2mm FCSAs

NFDPM

LAB	A	C	D	E	M	ALL
1	0.69101	0.66706	0.83192	0.69699	0.75504	0.72841
3	0.67935	0.74008	0.70210	0.80020	0.68872	0.72209
4	0.82489	0.90842	0.84341	0.71407	0.76417	0.81099
5	0.64117	0.87042	0.89162	0.91923	0.79577	0.82364
6	0.89268	0.90031	0.85564	0.98985	0.79922	0.88754
7	0.75094	0.64306	0.79577	0.83279	0.65494	0.73550
8	0.80359	0.75035	0.61060	0.76417	0.70426	0.72659
11	0.84174	0.75446	0.80311	0.83236	0.80742	0.80782
12	0.90708	0.81491	0.62233	0.87531	0.76022	0.79597
13	0.89686	0.95044	0.76751	0.88011	0.69177	0.83734
14	0.83623	0.77352	0.78873	0.78202	0.81583	0.79927
15	0.86196	0.68094	0.85324	0.63047	0.71270	0.74786
16	0.86622	0.92749	0.76916	0.85684	0.71544	0.82703
17	0.91891	0.67695	0.65848	0.79971	0.79873	0.77055
18	0.85324	0.72743	0.71339	0.73193	0.72872	0.75094
19	0.82887	0.91240	0.79873	0.81445	0.81025	0.83294
ALL	0.81842	0.79364	0.76911	0.80753	0.75020	0.78778

TABLE 27.

STUDY 16

TABLE OF MEAN TRANSFORMED SDs

OUTLIERS REMOVED OUTLYING LABORATORIES DELETED.

7.2mm FCSAs

PUFFS

LAB	B	F	G	H	M	ALL
1	0.76878	0.71462	0.60525	0.62056	0.62409	0.66666
3	0.66639	0.65343	0.69108	0.50040	0.55297	0.61285
4	0.72840	0.75247	0.70390	0.75574	0.60390	0.70888
6	0.80623	0.71917	0.71775	0.76192	0.66639	0.73429
7	0.66307	0.72801	0.77844	0.71284	0.60615	0.69770
8	0.58107	0.70781	0.72580	0.72343	0.59161	0.66594
11	0.75712	0.57525	0.69588	0.66874	0.66444	0.67229
12	0.80272	0.73091	0.69409	0.70036	0.63383	0.71238
13	0.79547	0.80034	0.77886	0.82162	0.64549	0.76836
14	0.79636	0.60367	0.71897	0.59064	0.63127	0.66818
15	0.49940	0.55779	0.63763	0.67082	0.62654	0.59844
16	0.59365	0.60548	0.55951	0.65199	0.50531	0.58319
17	0.73263	0.66444	0.67280	0.62233	0.60092	0.65862
18	0.72343	0.71462	0.64287	0.73478	0.60525	0.68419
19	0.68236	0.62409	0.66874	0.56234	0.67863	0.64323
ALL	0.70647	0.67681	0.68611	0.67323	0.61579	0.67168

Nicotine

LAB	B	F	G	H	M	ALL
1	0.46586	0.40722	0.45053	0.47776	0.41824	0.44392
3	0.37127	0.39195	0.45026	0.44693	0.37559	0.40720
4	0.50395	0.48339	0.49554	0.51345	0.43376	0.48602
6	0.45511	0.46387	0.52557	0.47498	0.47121	0.47815
7	0.37977	0.37839	0.44693	0.41687	0.38687	0.40177
8	0.46261	0.44382	0.41994	0.48449	0.37839	0.43785
11	0.52279	0.40117	0.41790	0.41722	0.41195	0.43421
12	0.46709	0.46512	0.35454	0.52966	0.38557	0.44039
13	0.48711	0.50990	0.44122	0.41052	0.45243	0.46024
14	0.49492	0.41124	0.45590	0.29718	0.36371	0.40459
15	0.47707	0.34459	0.38022	0.49718	0.44353	0.42852
16	0.40117	0.41927	0.47192	0.46734	0.43498	0.43893
17	0.47240	0.48071	0.47568	0.45243	0.50415	0.47707
18	0.49492	0.50647	0.38292	0.40232	0.38901	0.43513
19	0.54999	0.50040	0.48004	0.50762	0.48071	0.50375
ALL	0.46707	0.44050	0.44328	0.45306	0.42201	0.44518

TABLE 28.

STUDY 16

TABLE OF MEAN TRANSFORMED SDs

OUTLIERS REMOVED   OUTLYING LABORATORIES DELETED.

7.2mm FCSAs

NFDPM

LAB	B	F	G	H	M	ALL
1	0.86351	0.91987	0.88878	0.91041	0.75504	0.86753
3	0.79277	0.85081	0.88520	0.85444	0.68872	0.81439
4	0.91534	0.71748	0.82974	0.94692	0.76417	0.83473
6	0.89790	0.88084	0.86506	0.79477	0.79922	0.84756
7	0.75035	0.83921	0.81767	0.65848	0.65494	0.74413
8	0.88048	0.86660	0.69477	0.86852	0.70426	0.80292
11	1.06717	0.85564	0.74315	0.77513	0.80742	0.84970
12	0.85040	0.88556	0.86078	0.90608	0.76022	0.85261
13	0.85484	0.98465	0.92275	0.94396	0.69177	0.87959
14	1.03216	0.78924	0.99598	0.76529	0.81583	0.87970
15	0.91794	0.87268	0.89197	0.90540	0.71270	0.86014
16	0.74376	0.94307	0.82175	0.77244	0.71544	0.79929
17	0.85763	0.86545	0.86351	0.89020	0.79873	0.85510
18	1.04070	0.90202	0.55518	0.76473	0.72872	0.79827
19	0.91469	0.93184	0.70852	0.82799	0.81025	0.83866
ALL	0.89198	0.87366	0.82299	0.83898	0.74716	0.83496

TABLE 29

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
MONITOR DATA OMITTED
ANALYSIS OF TRANSFORMED STANDARD DEVIATIONS
ANALYSIS OF VARIANCE ON TSD PUFF
SOURCE     DF        SS        MS        F        p
HLD         3   0.02199   0.00733     1.05    0.379
ERROR      60   0.42081   0.00701
TOTAL      63   0.44280
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
                                   BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  -------+---------+---------+--------
     A     16   0.66342   0.07448              (----------*---------)
     C     16   0.65972   0.08122             (----------*---------)
     D     16   0.61693   0.07117   (---------*----------)
     E     16   0.65390   0.10414            (---------*----------)
                                   -------+---------+---------+--------
POOLED STDEV =  0.08375                 0.600     0.640     0.680
Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)
            A         C         D
     C  -0.04589
         0.05329
     D  -0.00310  -0.00680
         0.09608   0.09238
     E  -0.04007  -0.04377  -0.08656
         0.05911   0.05541   0.01262
```

TABLE 30

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
MONITOR DATA OMITTED
ANALYSIS OF TRANSFORMED STANDARD DEVIATIONS
ANALYSIS OF VARIANCE ON TSD nicotine
SOURCE     DF        SS        MS        F        p
HLD         3   0.01104   0.00368     1.71    0.174
ERROR      60   0.12883   0.00215
TOTAL      63   0.13987
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
                                   BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ------+---------+---------+---------+
     A     16   0.44128   0.04424               (---------*--------)
     C     16   0.41464   0.04353     (--------*--------)
     D     16   0.41052   0.04977   (--------*--------)
     E     16   0.43543   0.04754             (--------*--------)
                                   ------+---------+---------+---------+
POOLED STDEV =  0.04634                0.400     0.425     0.450     0.475
Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)
           A         C         D
     C  -0.00080
         0.05408
     D   0.00332  -0.02332
         0.05820   0.03156
     E  -0.02159  -0.04823  -0.05235
         0.03329   0.00665   0.00253
```

TABLE 31

```
STUDY 16
5.2mm FCSAs   OUTLIERS REMOVED   LABORATORY 2 DELETED
MONITOR DATA OMITTED
ANALYSIS OF TRANSFORMED STANDARD DEVIATIONS
ANALYSIS OF VARIANCE ON TSD NFDPM
SOURCE     DF        SS        MS        F        p
HLD         3   0.02174   0.00725     0.87    0.462
ERROR      60   0.50003   0.00833
TOTAL      63   0.52177
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
                                   BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ----------+---------+---------+------
     A     16   0.81842   0.08477               (-----------*----------)
     C     16   0.79364   0.10443         (----------*-----------)
     D     16   0.76911   0.08512   (----------*-----------)
     E     16   0.80753   0.08943            (-----------*----------)
                                   ----------+---------+---------+------
POOLED STDEV =  0.09129                    0.760     0.800     0.840
Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)
            A         C         D
     C  -0.02928
         0.07884
     D  -0.00475  -0.02953
         0.10337   0.07859
     E  -0.04317  -0.06795  -0.09248
         0.06495   0.04017   0.01564
```

TABLE 32

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
MONITOR DATA OMITTED
ANALYSIS OF VARIANCE OF TRANSFORMED STANDARD DEVIATIONS
ANALYSIS OF VARIANCE ON TSD PUFF
SOURCE     DF        SS        MS        F        p
HLD         3   0.00999   0.00333     0.55    0.649
ERROR      56   0.33775   0.00603
TOTAL      59   0.34775
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
                                   BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  ----------+---------+---------+-----
     B     15   0.70647   0.09201            (-----------*----------)
     F     15   0.67681   0.07099    (----------*-----------)
     G     15   0.68611   0.05889       (----------*-----------)
     H     15   0.67323   0.08457   (----------*-----------)
                                   ----------+---------+---------+-----
POOLED STDEV =  0.07766                    0.665     0.700     0.735
Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)
            B         F         G
     F  -0.01778
         0.07710
     G  -0.02708  -0.05674
         0.06780   0.03814
     H  -0.01420  -0.04386  -0.03456
         0.08068   0.05102   0.06032
```

TABLE 33

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
MONITOR DATA OMITTED
ANALYSIS OF VARIANCE OF TRANSFORMED STANDARD DEVIATIONS
ANALYSIS OF VARIANCE ON TSD nicotine
SOURCE     DF        SS        MS        F        p
HLD         3   0.00649   0.00216     0.81    0.493
ERROR      56   0.14920   0.00266
TOTAL      59   0.15569
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
                                   BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  -----+---------+---------+---------+-
     B     15   0.46707   0.04981            (----------*----------)
     F     15   0.44050   0.05124   (---------*----------)
     G     15   0.44328   0.04605    (---------*----------)
     H     15   0.45306   0.05857        (---------*----------)
                                   -----+---------+---------+---------+-
POOLED STDEV =  0.05162               0.425     0.450     0.475     0.500
Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)
           B         F         G
     F  -0.00496
         0.05810
     G  -0.00774  -0.03431
         0.05532   0.02875
     H  -0.01752  -0.04409  -0.04131
         0.04554   0.01897   0.02175
```

TABLE 34

```
STUDY 16
7.2mm FCSAs   OUTLIERS REMOVED   LABORATORIES 2 and 5 DELETED
MONITOR DATA OMITTED
ANALYSIS OF VARIANCE OF TRANSFORMED STANDARD DEVIATIONS
ANALYSIS OF VARIANCE ON TSD NFDPM
SOURCE     DF        SS        MS        F        p
HLD         3   0.04474   0.01491     1.86    0.147
ERROR      56   0.44858   0.00801
TOTAL      59   0.49332
                                   INDIVIDUAL 95 PCT CI'S FOR MEAN
                                   BASED ON POOLED STDEV
HOLDER      N      MEAN     STDEV  -----+---------+---------+---------+-
     B     15   0.89198   0.09686               (--------*---------)
     F     15   0.87366   0.06399           (---------*--------)
     G     15   0.82299   0.10869  (---------*--------)
     H     15   0.83898   0.08217      (--------*--------)
                                   -----+---------+---------+---------+-
POOLED STDEV =  0.08950               0.800     0.850     0.900     0.950
Fisher's pairwise comparisons
Individual error rate = 0.0500 single-sided
Intervals for (column level mean) - (row level mean)
            B         F         G
     F  -0.03636
         0.07300
     G   0.01431  -0.00401
         0.12367   0.10535
     H  -0.00168  -0.02000  -0.07067
         0.10768   0.08936   0.03869
```

ANNEX 1.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 5.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

THE DATA:-

LAB	A_{nic}	C_{nic}	D_{nic}	E_{nic}	5MNIC	A NFDPM	C NFDPM	D NFDPM	E NFDPM	5MNFDPM
1	0.598	0.592	0.592	0.608	1.560	8.084	7.624	7.760	7.934	15.652
2	0.685	0.803	0.801	0.767	1.488	7.276	8.700	9.280	9.130	14.020
3	0.564	0.598	0.568	0.579	1.571	7.042	7.827	7.706	7.925	15.563
4	0.559	0.570	0.567	0.563	1.615	7.250	7.071	7.492	7.400	15.199
5	0.573	0.475	0.511	0.545	1.546	7.235	6.295	6.695	7.300	15.695
6	0.594	0.612	0.655	0.653	1.534	8.011	8.368	8.894	8.938	15.769
7	0.652	0.620	0.631	0.645	1.544	8.648	8.090	8.376	8.220	15.846
8	0.565	0.593	0.618	0.575	1.571	7.284	7.302	7.758	7.260	15.260
11	0.476	0.570	0.578	0.522	1.516	5.238	6.748	7.174	6.483	15.616
12	0.600	0.637	0.647	0.650	1.544	8.061	8.396	8.718	8.784	15.840
13	0.580	0.646	0.642	0.675	1.591	7.534	7.852	8.225	8.658	15.767
14	0.535	0.618	0.612	0.636	1.582	6.718	8.039	8.251	8.558	16.087
15	0.526	0.631	0.623	0.644	1.563	6.338	8.235	8.248	8.530	16.077
16	0.703	0.700	0.723	0.701	1.604	9.075	8.476	9.049	8.759	16.357
17	0.598	0.634	0.656	0.682	1.568	6.832	7.274	7.706	8.060	15.136
18	0.508	0.563	0.601	0.589	1.598	6.372	6.994	7.554	7.514	15.514
19	0.562	0.633	0.608	0.595	1.696	7.710	7.782	7.713	8.066	15.698

NOTE THAT THE MONITOR DATA (5MNIC and 5MNFDPM) IS NOT USED

ANNEX 1.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 5.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED.

FIRST PASS

Eigenanalysis of the Correlation Matrix

Eigenvalue	6.1198	1.0303	0.5018	0.2088	0.0793	0.0422
Proportion	0.765	0.129	0.063	0.026	0.010	0.005
Cumulative	0.765	0.894	0.956	0.983	0.992	0.998

Eigenvalue	0.0131	0.0048
Proportion	0.002	0.001
Cumulative	0.999	1.000

Variable	PC1	PC2	PC3
A nicotine	-0.329	0.449	0.471
C nicotine	-0.363	-0.320	0.281
D nicotine	-0.368	-0.262	0.396
E nicotine	-0.381	-0.146	0.154
A NFDPM	-0.246	0.769	-0.077
C NFDPM	-0.369	-0.085	-0.481
D NFDPM	-0.386	-0.092	-0.232
E NFDPM	-0.366	-0.004	-0.478

ROW	LAB	SCORE 1	SCORE 2	SCORE 3
1	1	0.42161	1.09544	-0.02579
2	2	-5.20936	-1.47151	1.22119
3	3	1.09243	0.08588	-0.52210
4	4	2.12079	0.52471	0.22391
5	5	3.91486	1.54308	0.43723
6	6	-1.83694	0.30106	-1.05188
7	7	-1.38342	1.46686	0.08420
8	8	1.38437	0.19142	0.54365
9	11	4.13556	-1.65776	0.63489
10	12	-1.79278	0.33167	-0.81767
11	13	-1.07710	-0.20907	-0.22289
12	14	-0.10983	-0.91233	-0.99089
13	15	-0.22624	-1.44487	-1.01357
14	16	-3.93850	1.18703	0.62454
15	17	-0.13215	-0.53019	1.01692
16	18	2.27586	-0.76952	0.08535
17	19	0.36085	0.26810	-0.22709

ANNEX 1.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 5.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

FIRST PASS

NORMALISED SCORES and DISTANCE

ROW	LAB	NSCORE1	NSCORE2	NSCORE3	DIST
1	1	0.17043	1.07924	-0.03641	1.09322
2	2	-2.10580	-1.44974	1.72395	3.08353
3	3	0.44160	0.08460	-0.73704	0.86337
4	4	0.85729	0.51694	0.31609	1.04981
5	5	1.58252	1.52025	0.61724	2.27958
6	6	-0.74255	0.29661	-1.48494	1.68654
7	7	-0.55922	1.44516	0.11886	1.55414
8	8	0.55961	0.18859	0.76747	0.96837
9	11	1.67173	-1.63323	0.89627	2.50309
10	12	-0.72470	0.32676	-1.15431	1.40157
11	13	-0.43540	-0.20598	-0.31466	0.57533
12	14	-0.04440	-0.89883	-1.39883	1.66331
13	15	-0.09145	-1.42349	-1.43086	2.02041
14	16	-1.59208	1.16946	0.88166	2.16326
15	17	-0.05342	-0.52234	1.43558	1.52859
16	18	0.91998	-0.75814	0.12049	1.19819
17	19	0.14587	0.26414	-0.32058	0.44024

NOTE LAB 2 OUTLYING. OMIT LAB 2

ANNEX 1.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 5.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

LABORATORY 2 OMITTED

SECOND PASS

Eigenanalysis of the Correlation Matrix

16 cases used 1 cases contain missing values

Eigenvalue	6.0229	1.0468	0.4586	0.2847	0.1246	0.0451
Proportion	0.753	0.131	0.057	0.036	0.016	0.006
Cumulative	0.753	0.884	0.941	0.977	0.992	0.998

Eigenvalue	0.0101	0.0070
Proportion	0.001	0.001
Cumulative	0.999	1.000

Variable	PC1	PC2	PC3
A nicotine	-0.305	0.613	0.241
C nicotine	-0.358	-0.294	0.366
D nicotine	-0.364	-0.192	0.538
E nicotine	-0.375	-0.099	0.132
A NFDPM	-0.295	0.648	-0.123
C NFDPM	-0.370	-0.200	-0.408
D NFDPM	-0.385	-0.152	-0.171
E NFDPM	-0.365	-0.091	-0.539

ROW	LAB	SCORE 1	SCORE 2	SCORE 3
1	1	0.15344	1.02480	-0.23069
2	2	*	*	*
3	3	0.93565	-0.00138	-0.64995
4	4	2.07041	0.64540	-0.00598
5	5	4.23878	2.07038	-0.54934
6	6	-2.40935	-0.16235	-0.83495
7	7	-1.89934	1.30791	-0.00389
8	8	1.16005	0.25338	0.67815
9	11	4.24594	-1.34848	0.90906
10	12	-2.38538	-0.11622	-0.57454
11	13	-1.63879	-0.48057	0.06260
12	14	-0.48216	-1.23474	-0.73138
13	15	-0.63945	-1.78695	-0.58760
14	16	-4.92312	0.86636	1.08558
15	17	-0.62120	-0.36994	1.29364
16	18	2.18282	-0.69228	0.21602
17	19	0.01169	0.02467	-0.07674

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 5.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

LABORATORY 2 OMITTED

SECOND PASS

NORMALISED SCORES and DISTANCE

ROW	LAB	NSCORE1	NSCORE2	NSCORE3	DIST
1	1	0.06252	1.00161	-0.34063	1.05980
2	2	*	*	*	*
3	3	0.38125	-0.00135	-0.95972	1.03267
4	4	0.84363	0.63080	-0.00882	1.05343
5	5	1.72718	2.02355	-0.81115	2.78134
6	6	-0.98174	-0.15868	-1.23289	1.58399
7	7	-0.77393	1.27832	-0.00574	1.49436
8	8	0.47269	0.24765	1.00135	1.13467
9	11	1.73010	-1.31798	1.34231	2.55580
10	12	-0.97197	-0.11359	-0.84836	1.29512
11	13	-0.66776	-0.46970	0.09244	0.82163
12	14	-0.19647	-1.20681	-1.07996	1.63135
13	15	-0.26056	-1.74653	-0.86764	1.96750
14	16	-2.00603	0.84676	1.60297	2.70382
15	17	-0.25312	-0.36157	1.91019	1.96051
16	18	0.88943	-0.67662	0.31898	1.16218
17	19	0.00476	0.02412	-0.11332	0.11595

NOTE NO FURTHER OUTLIERS.

ANNEX 2.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

THE DATA:-

LAB	B nic	F nic	G nic	H nic	7MNIC	B NFDPM	F NFDPM	G NFDPM	H NFDPM	7MTAR
1	1.085	1.025	1.105	1.104	1.560	13.968	12.834	13.868	14.130	15.652
2	1.179	1.423	1.356	1.362	1.488	12.242	15.307	15.566	17.450	14.020
3	1.204	1.059	1.164	1.165	1.571	15.671	12.760	14.577	14.403	15.563
4	1.079	1.004	1.028	1.054	1.615	13.634	12.467	13.015	13.295	15.199
5	1.042	0.764	0.694	0.722	1.546	12.585	9.123	8.106	8.840	15.695
6	1.004	1.020	1.118	1.112	1.534	13.193	13.703	14.804	14.982	15.769
7	1.207	1.084	1.154	1.132	1.544	15.484	13.726	14.738	14.078	15.846
8	1.008	1.003	1.049	1.060	1.571	12.808	12.246	13.028	13.370	15.260
11	0.966	0.962	1.064	1.002	1.516	11.916	11.456	13.610	13.228	15.616
12	1.024	1.074	1.053	1.097	1.544	13.356	13.842	13.824	14.716	15.840
13	1.100	1.130	1.137	1.143	1.591	13.323	13.548	14.181	14.182	15.767
14	0.850	0.937	0.976	1.020	1.582	10.582	12.286	13.171	13.620	16.087
15	0.783	1.031	1.078	1.075	1.563	9.265	13.084	14.093	14.090	16.077
16	1.180	1.205	1.210	1.274	1.604	14.492	14.402	14.815	15.782	16.357
17	1.094	1.050	1.188	1.153	1.568	12.508	11.748	13.696	13.307	15.136
18	0.930	1.005	1.072	1.104	1.598	10.916	11.554	13.068	13.226	15.514
19	1.211	1.056	1.138	1.145	1.696	15.110	12.460	13.982	14.224	15.698

NOTE THAT THE MONITOR DATA (7MNIC and 7MNFDPM) IS NOT USED

ANNEX 2.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

FIRST PASS

Eigenanalysis of the Correlation Matrix

Eigenvalue	5.7739	1.6476	0.2994	0.2184	0.0326	0.0202
Proportion	0.722	0.206	0.037	0.027	0.004	0.003
Cumulative	0.722	0.928	0.965	0.992	0.996	0.999

Eigenvalue	0.0047	0.0031
Proportion	0.001	0.000
Cumulative	1.000	1.000

Variable	PC1	PC2	PC3
B nicotine	-0.211	0.654	0.342
F nicotine	-0.391	-0.063	0.546
G nicotine	-0.400	-0.056	0.155
H nicotine	-0.407	-0.061	0.184
B NFDPM	-0.147	0.704	-0.426
F NFDPM	-0.384	-0.116	-0.285
G NFDPM	-0.385	-0.147	-0.491
H NFDPM	-0.399	-0.172	-0.152

ROW	LAB	SCORE 1	SCORE 2	SCORE 3
1	1	-0.22887	0.50710	-0.33840
2	2	-4.83269	-0.82114	1.30886
3	3	-1.25451	1.66116	-0.36722
4	4	0.76134	0.59627	-0.10951
5	5	6.79710	1.56478	0.79597
6	6	-0.73862	-0.48664	-0.90782
7	7	-1.41358	1.54786	-0.48634
8	8	0.91232	-0.11409	-0.03806
9	11	1.42604	-0.62730	-0.16747
10	12	-0.44420	-0.20005	-0.47168
11	13	-1.00550	0.14465	0.13365
12	14	1.80140	-1.80567	-0.40659
13	15	0.71852	-3.00231	-0.16761
14	16	-2.85283	0.62182	0.13231
15	17	-0.05604	0.07807	0.65952
16	18	1.20904	-1.25407	0.45408
17	19	-0.79891	1.58956	-0.02367

ANNEX 2.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

FIRST PASS

NORMALISED SCORES and DISTANCE

ROW	LAB	NSCORE1	NSCORE2	NSCORE3	DIST
1	1	-0.09525	0.39506	-0.61840	0.73998
2	2	-2.01120	-0.63972	2.39184	3.18983
3	3	-0.52208	1.29415	-0.67107	1.54846
4	4	0.31684	0.46454	-0.20012	0.59685
5	5	2.82871	1.21907	1.45457	3.40640
6	6	-0.30739	-0.37913	-1.65898	1.72929
7	7	-0.58828	1.20588	-0.88876	1.60938
8	8	0.37967	-0.08888	-0.06955	0.39609
9	11	0.59347	-0.48871	-0.30605	0.82747
10	12	-0.18486	-0.15585	-0.86196	0.89523
11	13	-0.41845	0.11269	0.24423	0.49744
12	14	0.74968	-1.40674	-0.74301	1.75869
13	15	0.29902	-2.33900	-0.30629	2.37785
14	16	-1.18725	0.48444	0.24178	1.30487
15	17	-0.02332	0.06082	1.20522	1.20698
16	18	0.50316	-0.97700	0.82980	1.37705
17	19	-0.33248	1.23837	-0.04326	1.28296

NOTE LABORATORY 5 OUTLYING. OMIT LABORATORY 5

ANNEX 2.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

LABORATORY 5 OMITTED

SECOND PASS

Eigenanalysis of the Correlation Matrix

16 cases used 1 cases contain missing values

Eigenvalue	5.6989	1.4559	0.4910	0.2354	0.0667	0.0400
Proportion	0.712	0.182	0.061	0.029	0.008	0.005
Cumulative	0.712	0.894	0.956	0.985	0.993	0.998

Eigenvalue	0.0078	0.0043
Proportion	0.001	0.001
Cumulative	0.999	1.000

Variable	PC1	PC2	PC3
B nicotine	-0.295	0.576	0.152
F nicotine	-0.391	-0.186	0.226
G nicotine	-0.386	-0.020	0.488
H nicotine	-0.397	-0.065	0.343
B NFDPM	-0.187	0.720	-0.295
F NFDPM	-0.358	-0.201	-0.600
G NFDPM	-0.378	-0.077	-0.268
H NFDPM	-0.383	-0.255	-0.227

ROW	LAB	SCORE 1	SCORE 2	SCORE 3
1	1	0.28278	0.66075	-0.20969
2	2	-6.17385	-1.90974	0.66184
3	3	-1.23750	1.63975	0.00745
4	4	1.80279	0.93258	-0.13771
5	5	*	*	*
6	6	-0.58027	-0.47055	-1.08206
7	7	-1.41381	1.44135	-0.62365
8	8	1.97714	0.29768	0.15820
9	11	2.51789	-0.02106	0.30161
10	12	0.06096	-0.24122	-1.06864
11	13	-0.76512	0.08107	-0.08074
12	14	3.12206	-1.23076	-0.47105
13	15	1.50533	-2.62733	-0.24548
14	16	-3.38503	0.08493	-0.16139
15	17	0.48956	0.42696	1.57266
16	18	2.31370	-0.69395	1.08136
17	19	-0.51662	1.62952	0.29728

ANNEX 2.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

LABORATORY 5 OMITTED

SECOND PASS

NORMALISED SCORES and DISTANCE

ROW	LAB	NSCORE1	NSCORE2	NSCORE3	DIST
1	1	0.11845	0.54761	-0.29926	0.63519
2	2	-2.58620	-1.58272	0.94456	3.17579
3	3	-0.51838	1.35896	0.01064	1.45451
4	4	0.75518	0.77289	-0.19654	1.09831
5	5	*	*	*	*
6	6	-0.24307	-0.38997	-1.54428	1.61120
7	7	-0.59224	1.19453	-0.89005	1.60308
8	8	0.82822	0.24671	0.22578	0.89319
9	11	1.05473	-0.01745	0.43045	1.13932
10	12	0.02553	-0.19992	-1.52513	1.53838
11	13	-0.32051	0.06719	-0.11523	0.34716
12	14	1.30782	-1.02000	-0.67226	1.78962
13	15	0.63058	-2.17743	-0.35034	2.29381
14	16	-1.41797	0.07039	-0.23033	1.43828
15	17	0.20508	0.35384	2.24445	2.28140
16	18	0.96920	-0.57512	1.54328	1.91097
17	19	-0.21641	1.35049	0.42426	1.43201

NOTE LABORATORY 2 OUTLYING. OMIT LABORATORY 2

ANNEX 2.

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

LABORATORIES 5 and 2 OMITTED

THIRD PASS

Eigenanalysis of the Correlation Matrix

15 cases used 2 cases contain missing values

Eigenvalue	5.4702	1.2941	0.6506	0.3846	0.1484	0.0334
Proportion	0.684	0.162	0.081	0.048	0.019	0.004
Cumulative	0.684	0.846	0.927	0.975	0.993	0.998

Eigenvalue	0.0142	0.0045
Proportion	0.002	0.001
Cumulative	0.999	1.000

Variable	PC1	PC2	PC3
B nicotine	-0.326	0.540	0.229
F nicotine	-0.387	-0.089	-0.247
G nicotine	-0.364	0.206	-0.515
H nicotine	-0.388	0.040	-0.408
B NFDPM	-0.315	0.463	0.521
F NFDPM	-0.325	-0.464	0.384
G NFDPM	-0.364	-0.219	0.032
H NFDPM	-0.351	-0.422	0.188

ROW	LAB	SCORE 1	SCORE 2	SCORE 3
1	1	-0.15929	0.35532	0.42809
2	2	*	*	*
3	3	-2.14758	1.10162	0.21316
4	4	1.78058	0.95164	0.91446
5	5	*	*	*
6	6	-1.15777	-1.38253	0.56558
7	7	-2.30622	0.61337	0.69495
8	8	1.98884	0.58629	0.27393
9	11	2.76967	0.54734	-0.00675
10	12	-0.46944	-1.13494	1.00285
11	13	-1.56856	-0.27025	-0.28172
12	14	3.59428	-1.01808	0.53415
13	15	1.49588	-2.39064	-0.96470
14	16	-5.02846	-0.99950	-0.67818
15	17	0.05164	1.37588	-1.71251
16	18	2.40784	0.30731	-1.19453
17	19	-1.25143	1.35716	0.21122

STUDY 16

PRINCIPAL COMPONENT ANALYSIS OF 7.2mm FCSAs

nicotine and NFDPM combined using the CORRELATION matrix

MONITOR DATA OMITTED

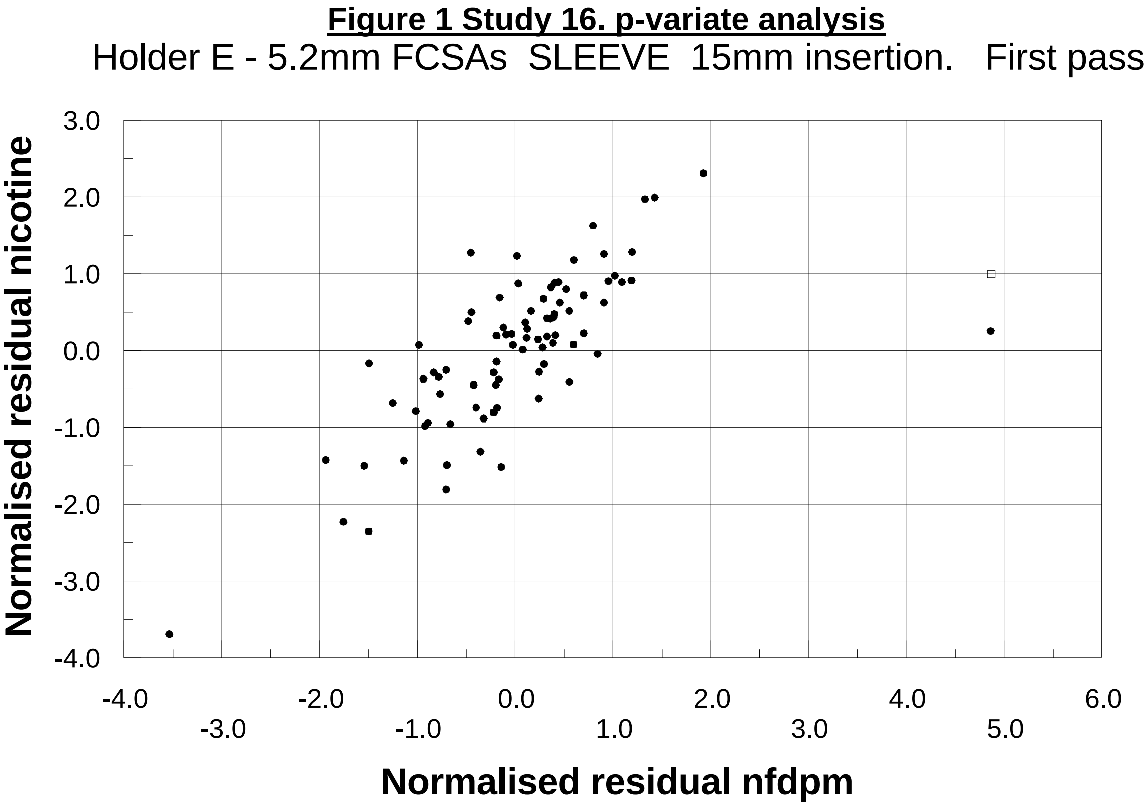
LABORATORIES 5 and 2 OMITTED

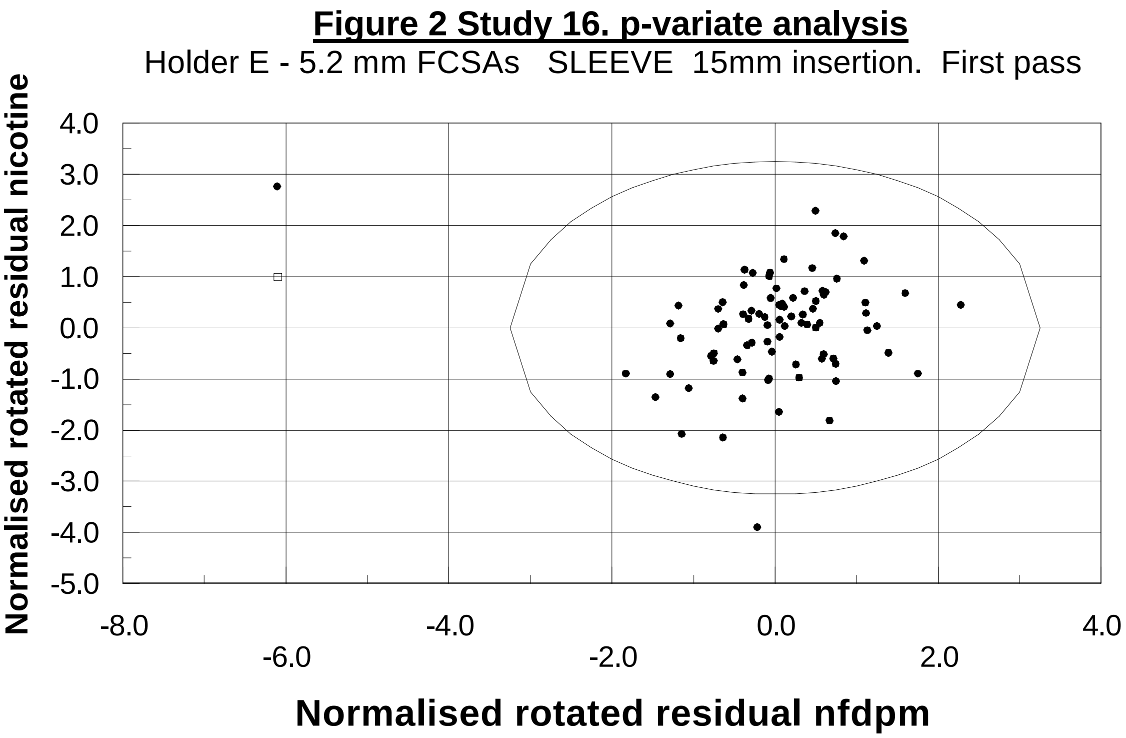
THIRD PASS

NORMALISED SCORES and DISTANCE

ROW	LAB	NSCORE1	NSCORE2	NSCORE3	DIST
1	1	-0.06811	0.31235	0.53075	0.61959
2	2	*	*	*	*
3	3	-0.91822	0.96838	0.26427	1.36042
4	4	0.76131	0.83654	1.13374	1.60149
5	5	*	*	*	*
6	6	-0.49502	-1.21531	0.70120	1.48785
7	7	-0.98605	0.53918	0.86160	1.41611
8	8	0.85035	0.51538	0.33962	1.05074
9	11	1.18421	0.48114	-0.00837	1.27824
10	12	-0.20071	-0.99768	1.24334	1.60671
11	13	-0.67066	-0.23757	-0.34928	0.79260
12	14	1.53678	-0.89495	0.66224	1.89768
13	15	0.63958	-2.10150	-1.19604	2.50117
14	16	-2.14997	-0.87861	-0.84080	2.47008
15	17	0.02208	1.20947	-2.12317	2.44359
16	18	1.02950	0.27014	-1.48098	1.82378
17	19	-0.53506	1.19302	0.26187	1.33348

NOTE NO FURTHER OUTLYING LABORATORIES





The dotted line is the 3.25 sd contour line

Figure 3 Study 16. p-variate analysis

Holder E - 5.2mm FCSAs SLEEVE 15mm insertion. Second pass

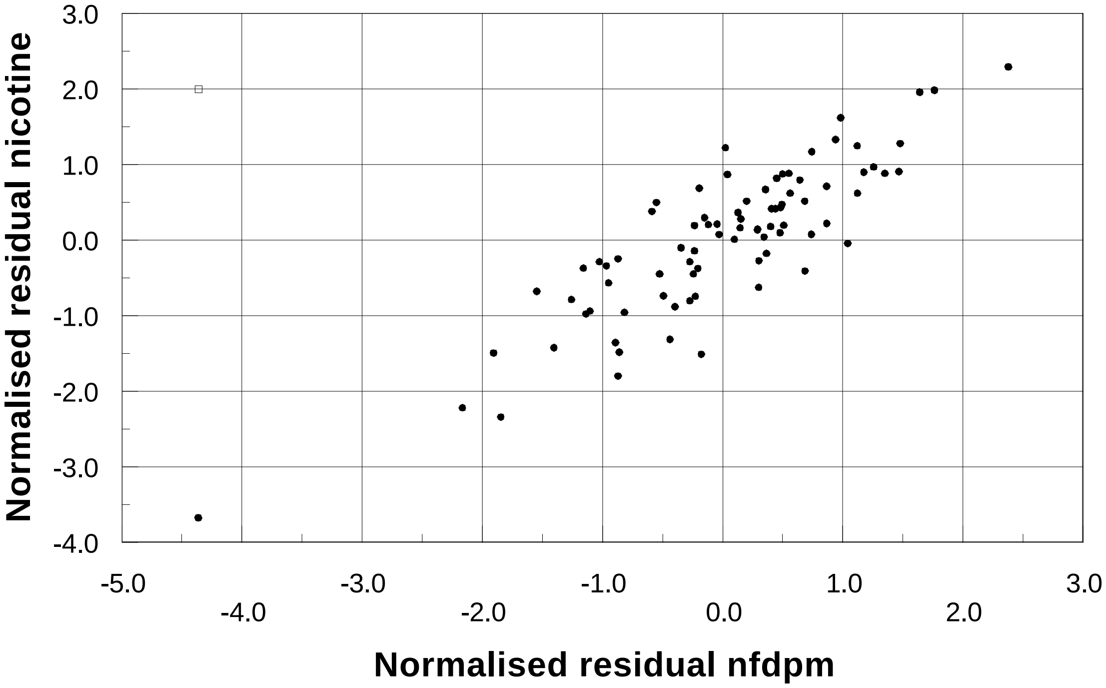
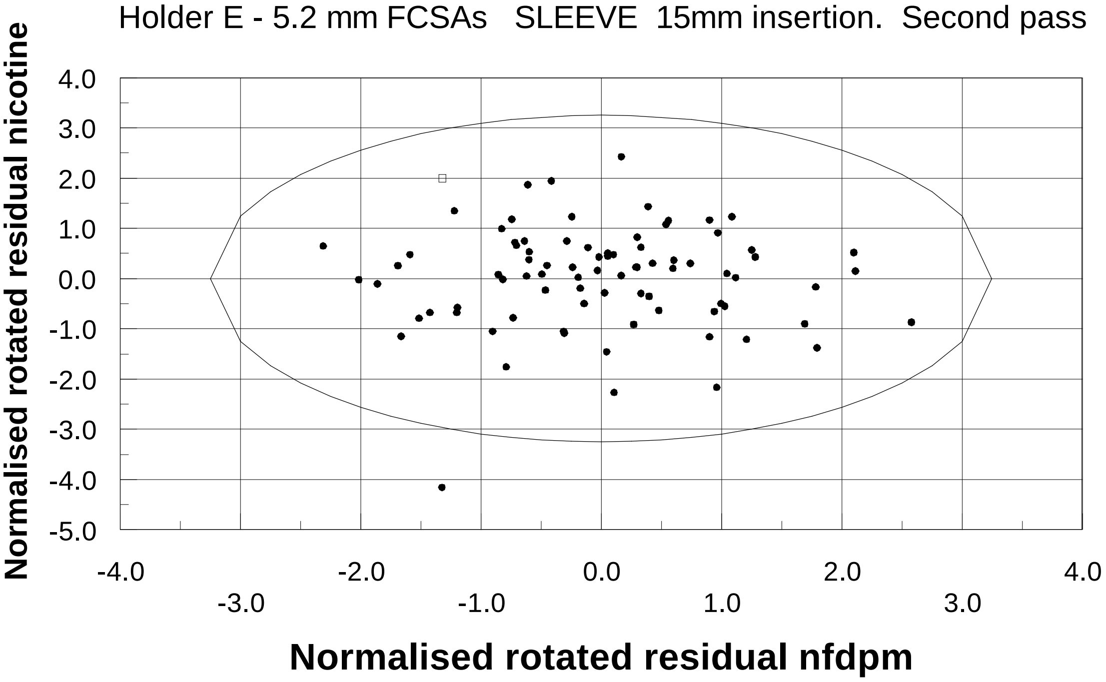


Figure 4 Study 16. p-variate analysis

Holder E - 5.2 mm FCSAs SLEEVE 15mm insertion. Second pass



The dotted line is the 3.25 sd contour line

Figure 5 Study 16. p-variate analysis

Holder E - 5.2mm FCSAs SLEEVE 15mm insertion. Third pass

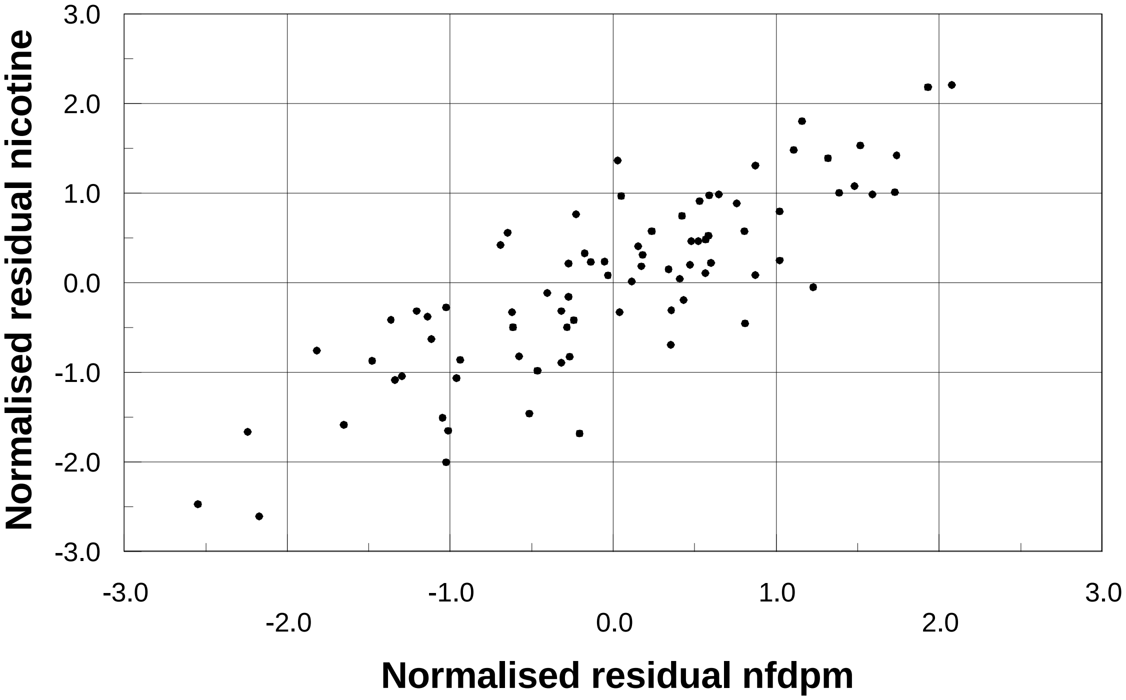
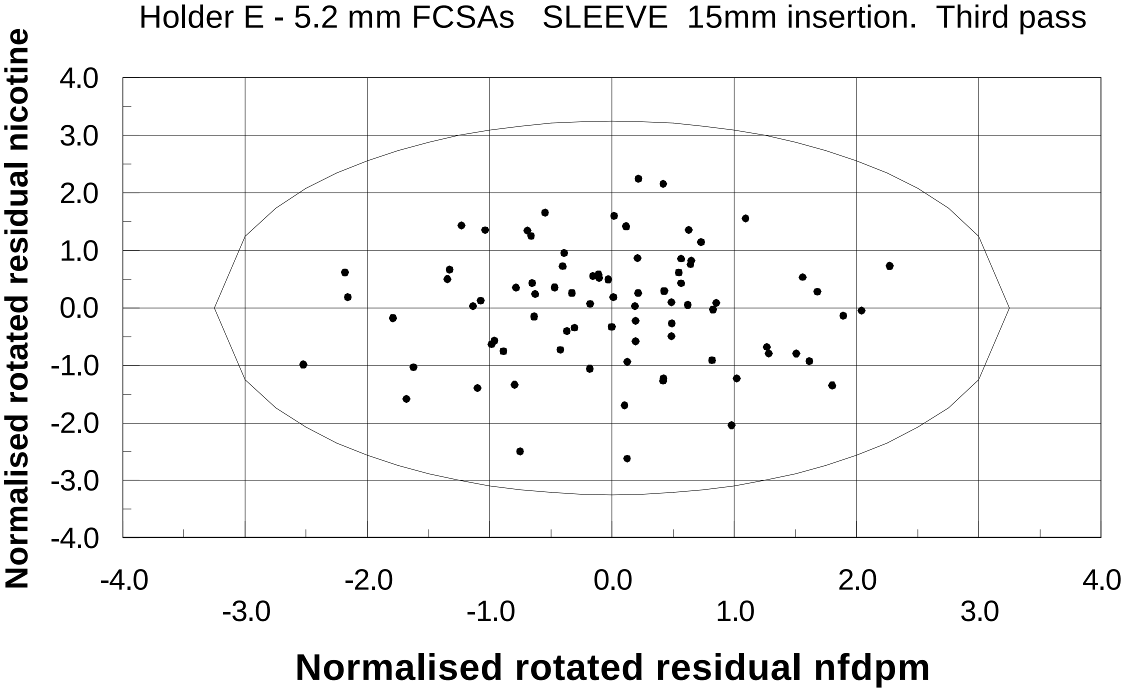


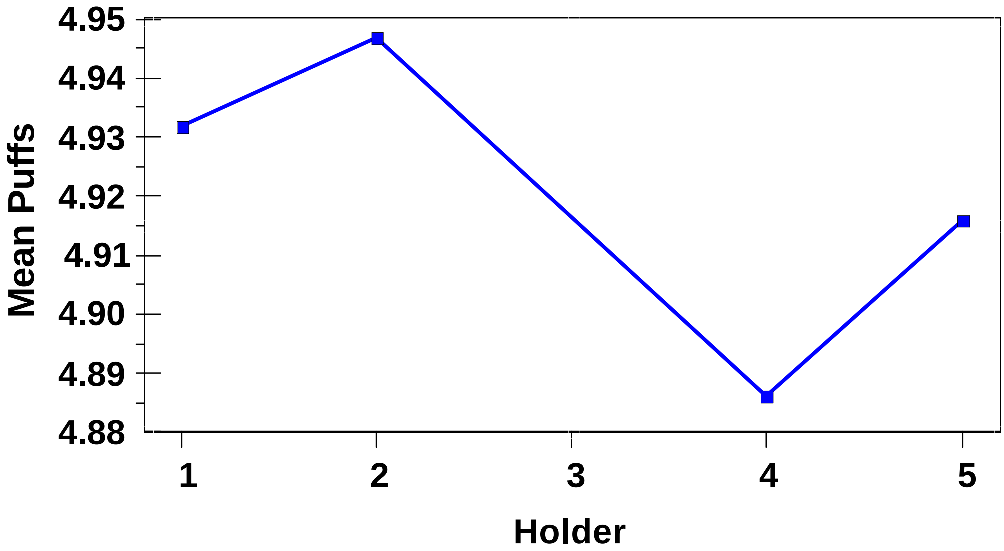
Figure 6 Study 16. p-variate analysis

Holder E - 5.2 mm FCSAs SLEEVE 15mm insertion. Third pass



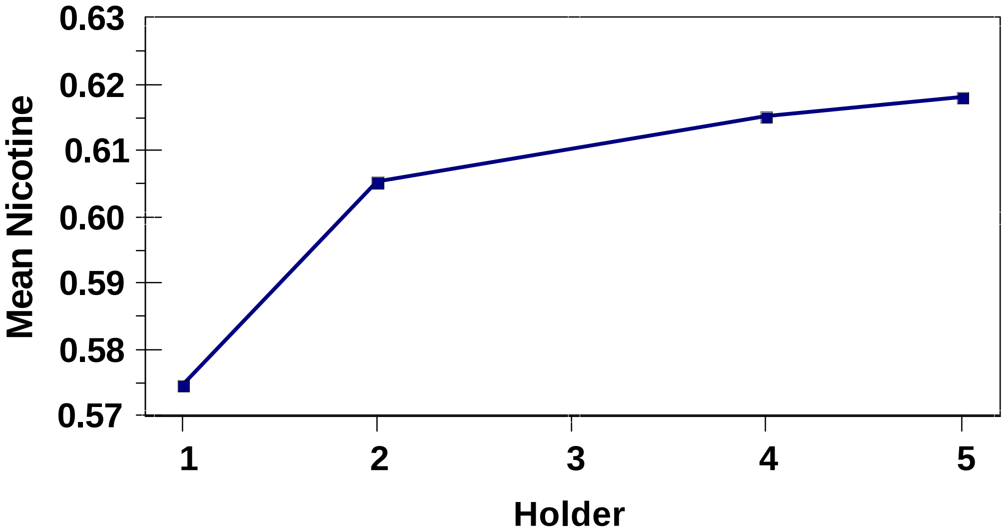
The dotted line is the 3.25 sd contour line

Figure 7 Study 16. Mean Puff number. 5.2mm FCSAs
Outliers removed. Laboratory 2 deleted



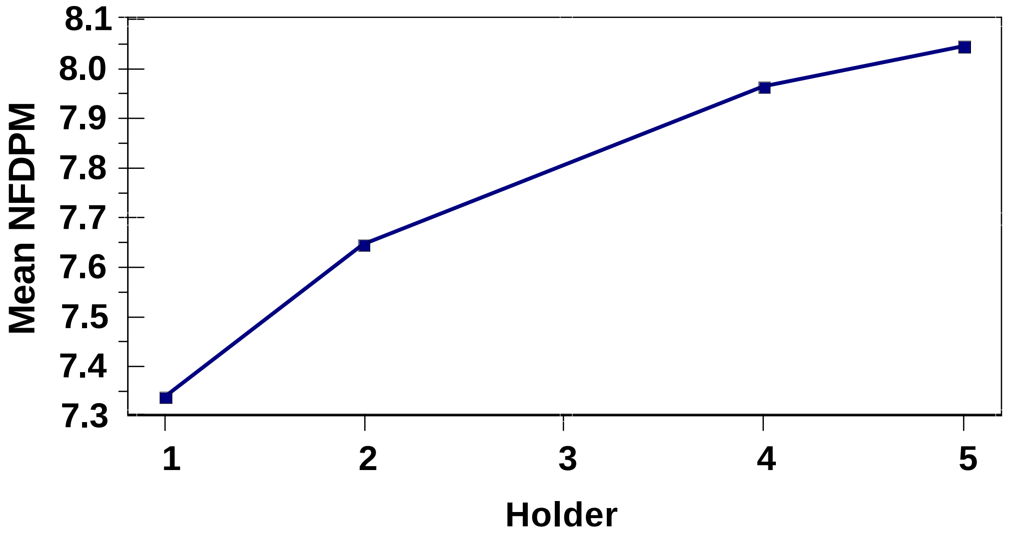
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = CM 2

Figure 8 Study 16. Mean Nicotine. 5.2mm FCSAs
Outliers removed. Laboratory 2 deleted



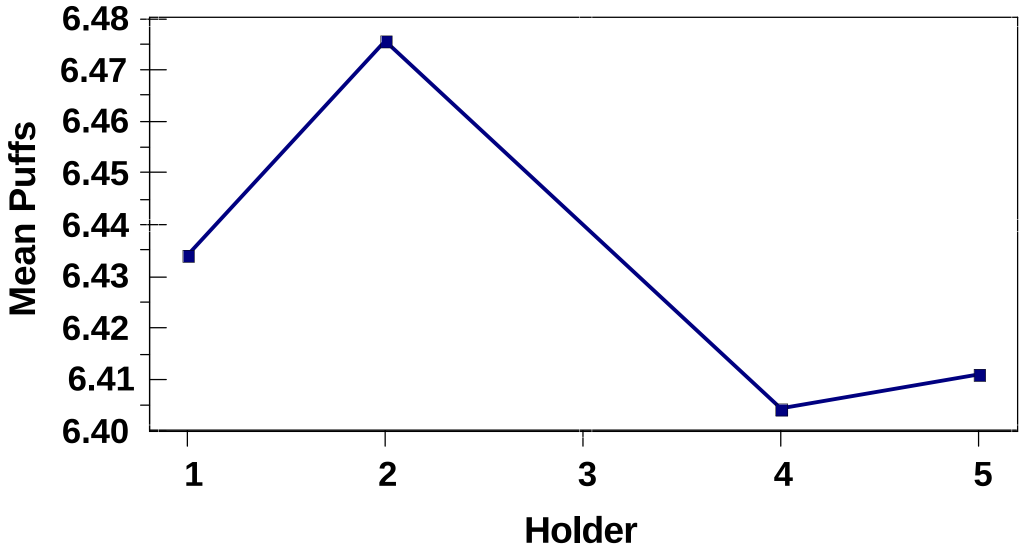
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15

Figure 9. Study 16. Mean NFDPM. 5.2mm FCSAs
Outliers removed. Laboratory 2 deleted



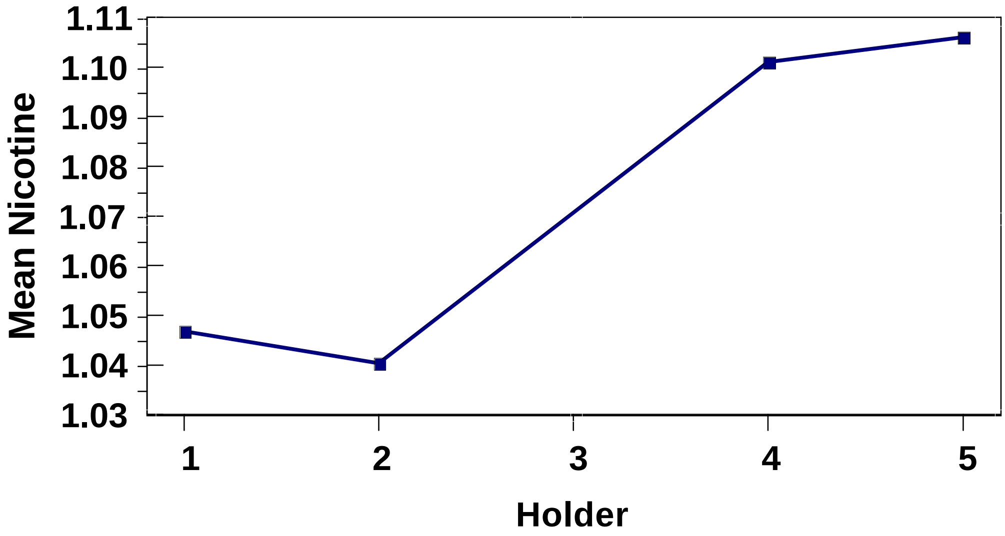
1 = LABYR 9mm 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 1

Figure 10. Study 16. Mean Puff number. 7.2mm FCSAs
Outliers removed. Laboratories 2 and 5 deleted.



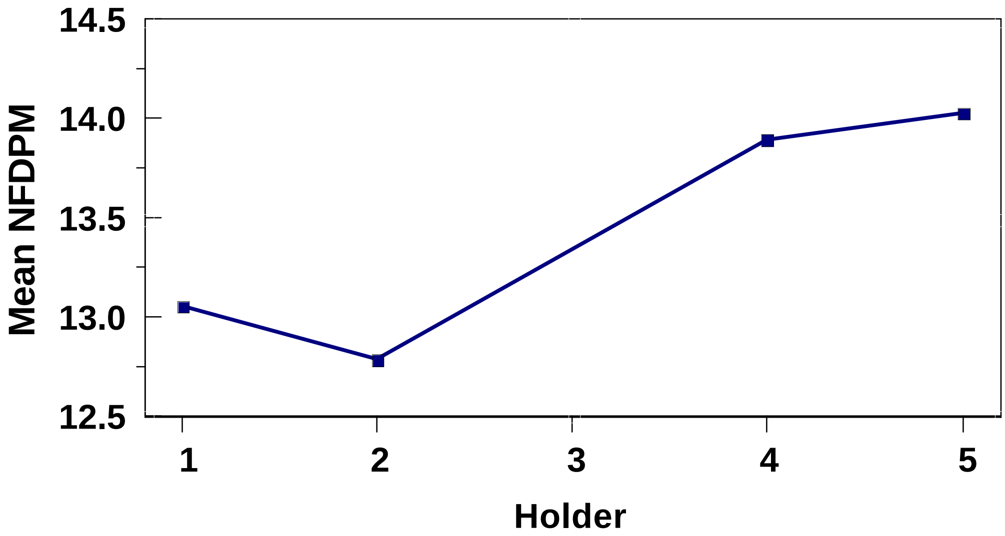
1 = LABYR 9mm 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 1

Figure 11 Study 16. Mean Nicotine. 7.2mm FCSAs
Outliers removed. Laboratories 2 and 5 deleted.



1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15m

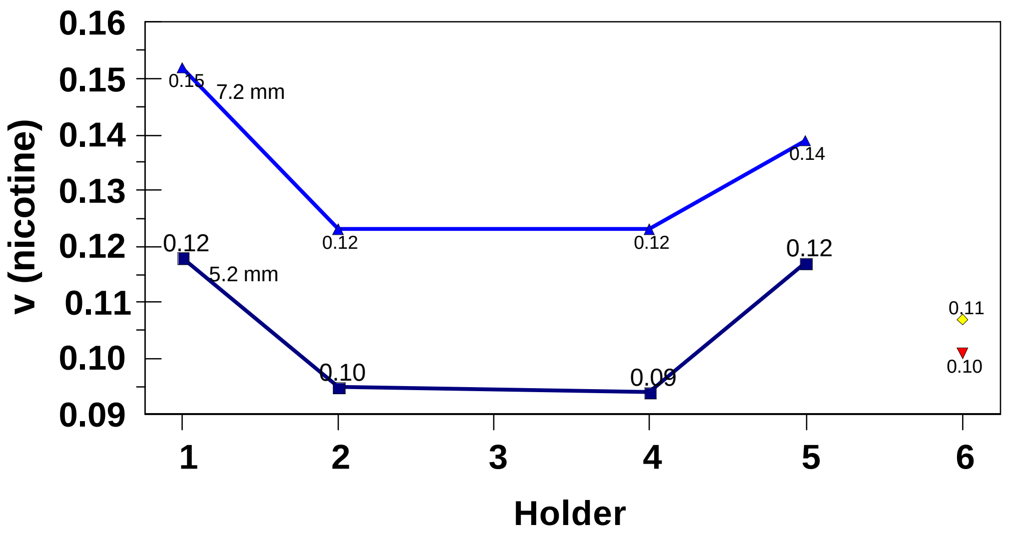
Figure 12 Study 16. Mean NFDPM. 7.2mm FCSAs
Outliers removed. Laboratories 2 and 5 deleted.



1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15m

Figure 13 Study 16. Plot of v for Nicotine

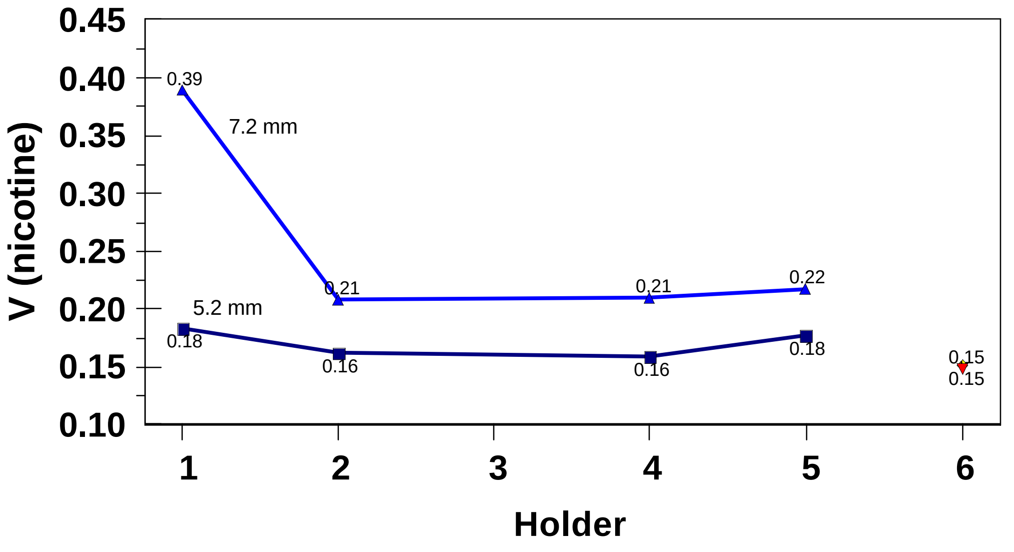
Outliers removed. Outlying laboratories omitted.



1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 =

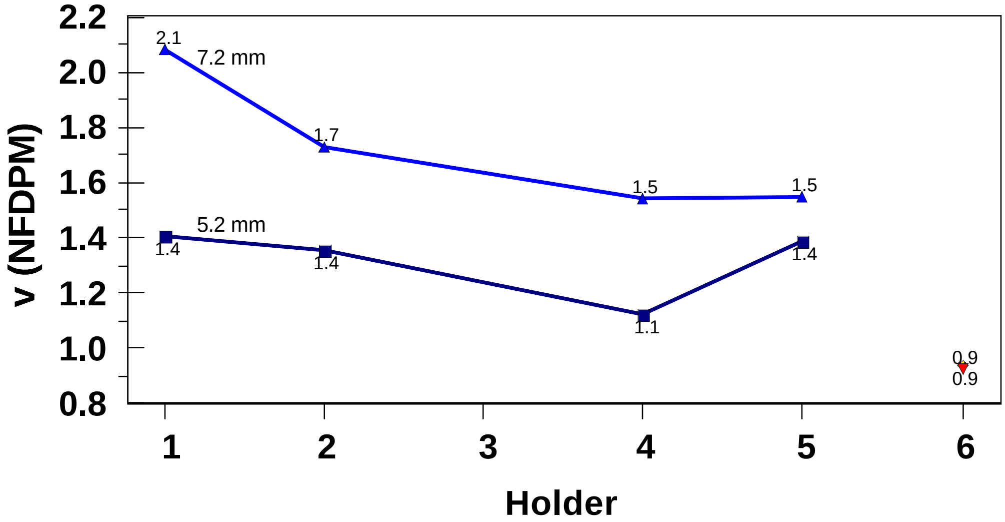
Figure 14 Study 16. Plot of V for Nicotine

Outliers removed. Outlying laboratories omitted.



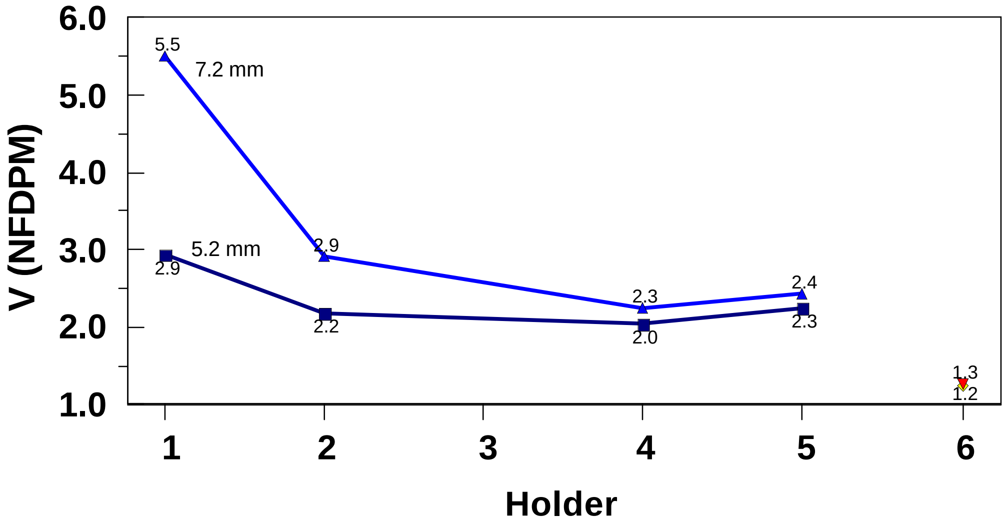
1 =LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CN

Figure 15 Study 16. Plot of v for NFDPM
Outliers removed. Outlying laboratories omitted.



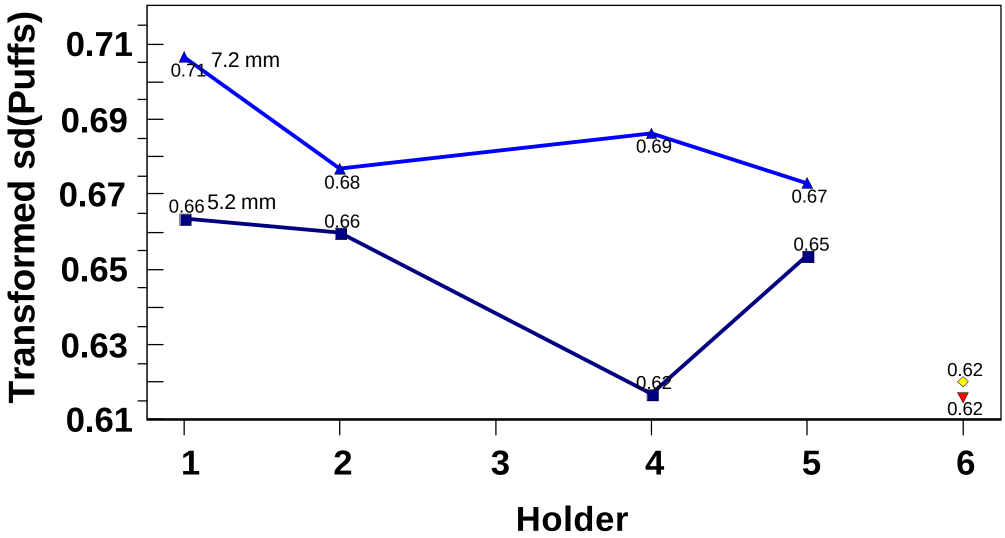
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CM

Figure 16 Study 16. Plot of V for NFDPM
Outliers removed. Outlying laboratories omitted.



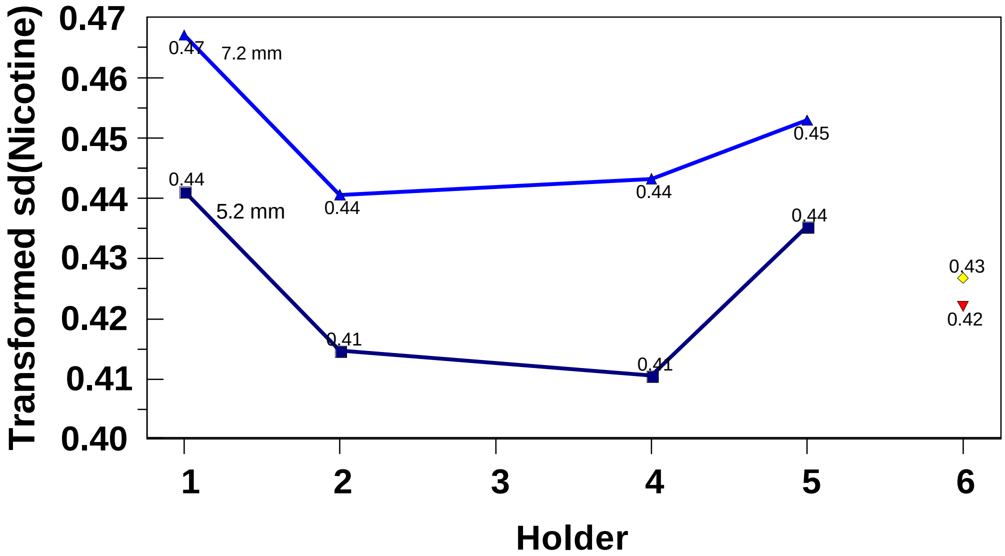
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CM

Figure 17 Study 16. Transformed within-laboratory SDs. Puff number
Outliers removed. Outlying laboratories omitted.



1 =LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CI

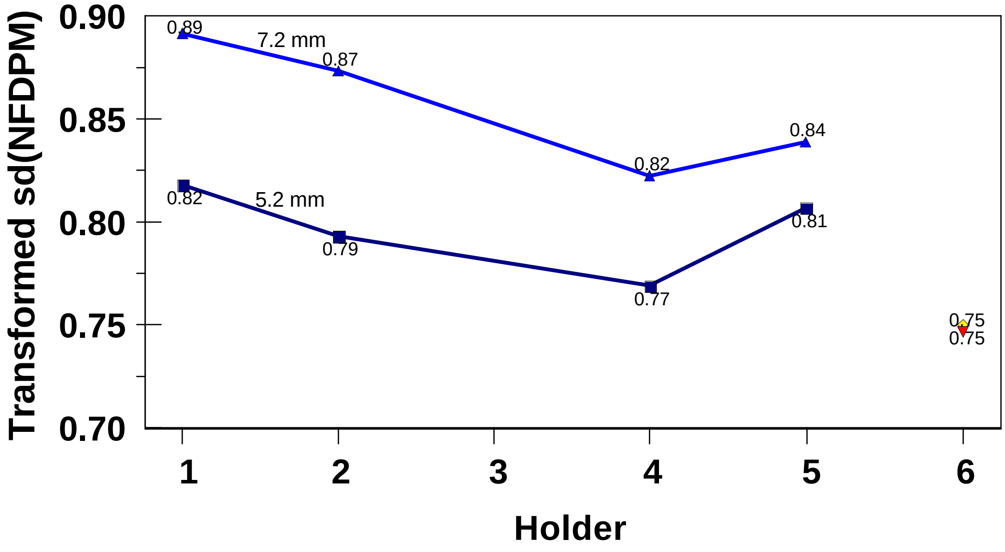
Figure 18 Study 16. Transformed within-laboratory SDs. Nicotine
Outliers removed. Outlying laboratories omitted.



1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CI

Figure 19 Study 16. Transformed within-laboratory SDs

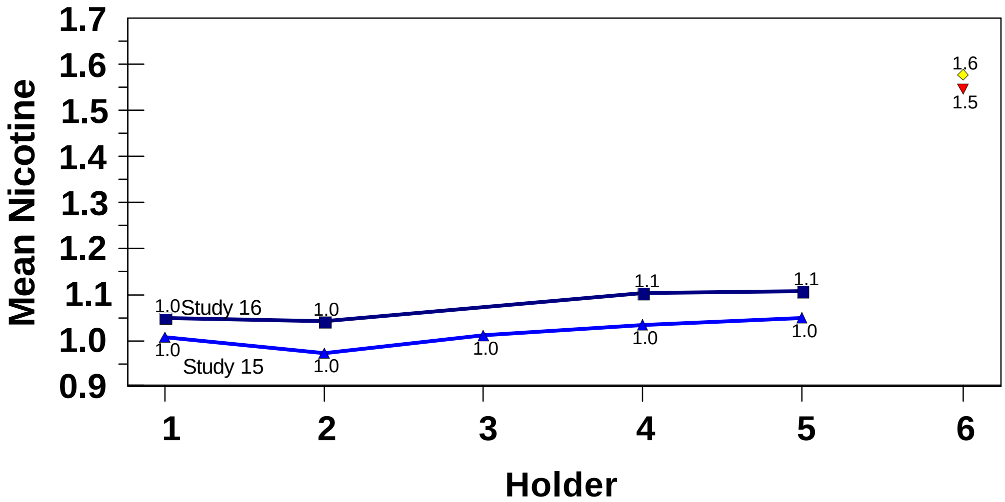
Outliers removed. Outlying laboratories omitted.



1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CI

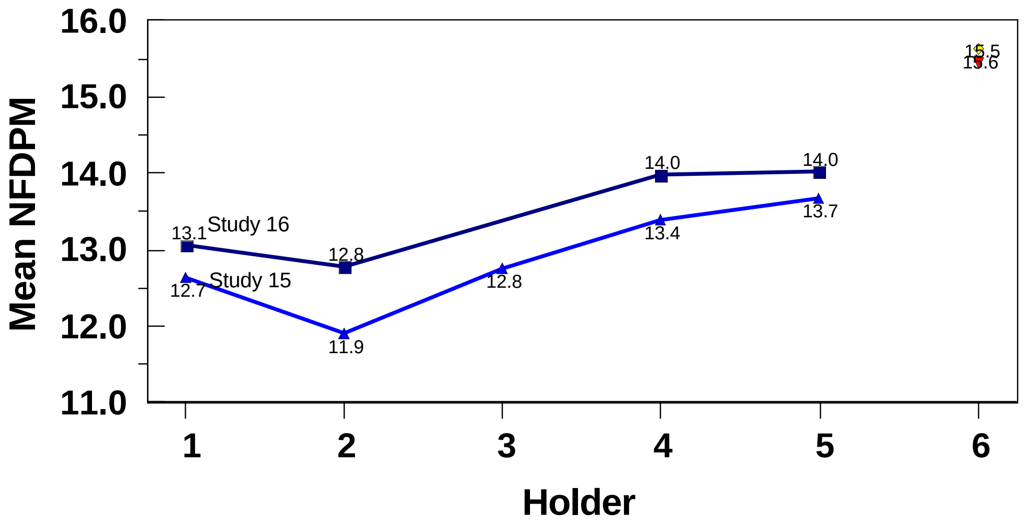
Figure 20 Studies 15 and 16. Mean Nicotine

7.2mm FCSAs Outliers removed. Laboratories 2 and 5 deleted.



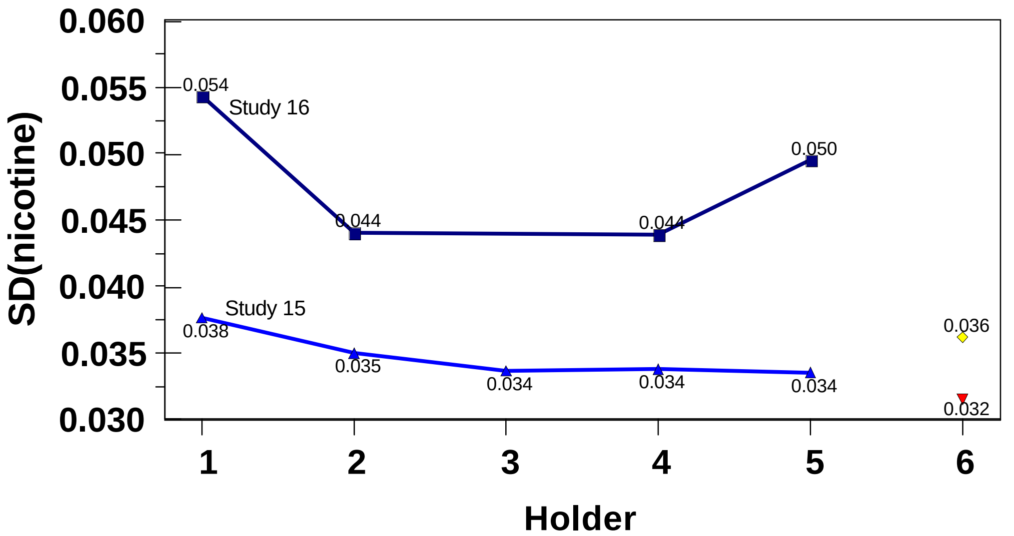
1 = LABYR 2 = SLEEVE 9mm 3 = 11mm SLEEVE 4 = SLEEVE 13mm 5 = SLEEV
6 = CM2

Figure 21 Studies 15 and 16. Mean NFDPM
7.2mm FCSAs Outliers removed. Laboratories 2 and 5 deleted.



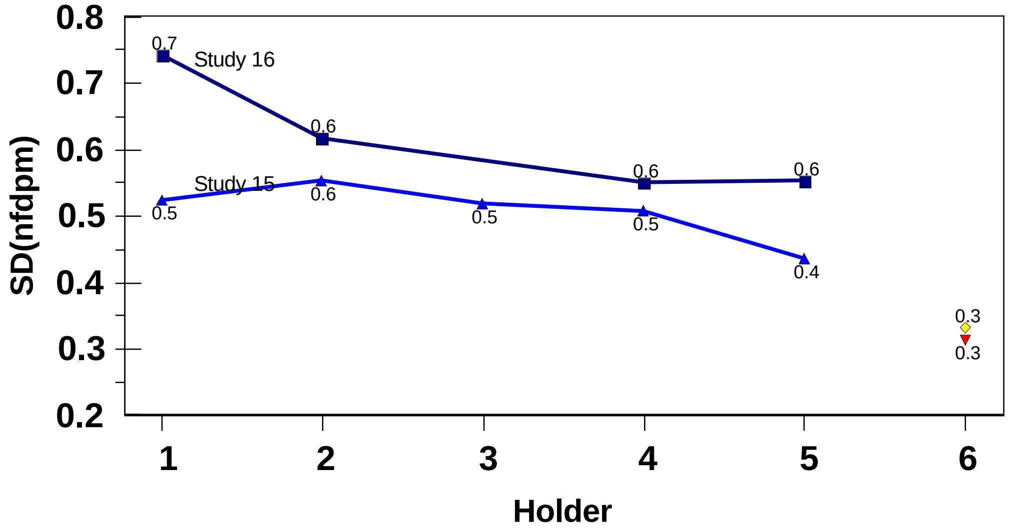
1 = LABYR 2 = SLEEVE 9mm 3 = 11mm SLEEVE 4 = SLEEVE 13mm 5 = SLEE
6 = CM2

Figure 22 Studies 15 and 16. Within-laboratory standard deviation (nicotine)
7.2mm FCSAs Outliers removed. Laboratories 2 and 5 deleted.



1 = LABYR 2 = SLEEVE 9mm 3 = 11mm SLEEVE 4 = SLEEVE 13mm 5 = SLEE'
6 = CM2

Figure 23 Studies 15 and 16. Within-laboratory standard deviation (nfdpm)
7.2mm FCSAs Outliers removed. Laboratories 2 and 5 deleted.



1 = LABYR 2 = SLEEVE 9mm 3 = 11mm SLEEVE 4 = SLEEVE 13mm 5 = SLEEVI
6 = CM2

Annex 1

EXPERIMENTAL PROTOCOL

0.0 INTRODUCTION

CORESTA Study 15 was conducted in order to determine the appropriate insertion depth required for the satisfactory smoking of fine-cut smoking articles. In order to do this, the study was conducted with fine-cut smoking articles of 7.2 mm diameter. In that study, the protocol of smoking was modified in order to achieve maximum sensitivity to determine the effect of insertion depth. In order to take these studies further, and to develop a satisfactory analytical method, it is necessary to study articles made at a diameter of 5.2 mm and to study the effect of repeating Study 15 under smoking conditions more akin to those that are likely to apply in a final method. In particular the current study will be conducted with smoking articles made in the laboratories conducting the study and users of the rotary smoking machine will operate the smoking machine in its “normal” mode. Although smoking conditions will be more akin to those that are likely to apply in a final method, it is not possible to conduct this study in conditions that would allow r and R to be determined.
The definitions of the terms used in this protocol, are given in Appendix 1.

1.0 OBJECTIVES OF THE STUDY

1.0.1 To determine the between-laboratory and within-laboratory variability of fine-cut smoking articles made under “normal” conditions and at two diameters.

1.0.2 To evaluate the effect of insertion depth on the smoke yields and between-laboratory and within-laboratory variability of 5.2 mm diameter FCSAs.

1.0.3 To compare the between-laboratory and within-laboratory variability of 5.2 mm diameter FCSAs with the between-laboratory and within-laboratory variability of 7.2 mm diameter FCSAs.

1.0.4 To evaluate the effect of insertion depth on the smoke yields of 7.2 mm diameter FCSAs when these articles are made under “normal” conditions and to contrast these data with those obtained from Study 15.

1.0.5 To evaluate the local making of FCSAs, and to determine if there are any problems arising from articles being made locally or during the smoking (especially the 5.2 mm diameter FCSAs).

1.1 Limitations

1.1.1 As the analytical parameters of the experimental method are not yet fully specified it must be noted that, although the statistical analysis of this study will be founded upon the between-laboratory and within-laboratory variability, this study cannot be used to estimate the Reproducibility (R) or the Repeatability (r) of the method.

1.1.2 Extreme caution must be used when making any comparisons between the data arising from this study and that from Study 15 for the following reasons:

The fine-cut smoking articles were made by a central laboratory in Study 15 but will be made by the participating laboratories in Study 16.

In this study the means are to be based upon the smoking of 20 FCSAs whereas in Study 15 the means were based upon the smoking of 5 FCSAs.

The design of the Sleeve holder for use in this study has been modified from that used in Study 15.

The mix of participating laboratories is different for the two studies.

2.0 HOLDERS

2.0.1 The design and construction of the holders to be used in this study are similar to those used in Study 15.

2.0.2 The ISO holder is to be used according to ISO Standard Methods 3308 and 4387. For this reason only one insertion depth (9 mm) can be used with this holder. Improvements have been made to ensure that the front seal does not bulge. An aluminium cap is incorporated and the front of this will be used as the point of reference for depth of insertion.

2.0.3 The Sleeve holder is designed in such a way that various insertion depths can be used.

2.0.4 Using the Sleeve holder, the fine-cut smoking articles will be smoked using insertion depths of 9 mm, 13 mm, and 15 mm.

2.0.5 A butt length of 29 mm will be used for all smokings of the FCSAs.

2.0.6 A monitor test piece will be smoked using the standard ISO holder.

2.0.7 A butt length of 30 mm will also be used for the smoking of the monitor test piece.

2.0.8 Insert the marked end of the FCSA (see making) into the holder.

2.0.9 There is concern that the smoulder rate of the fine-cut smoking articles will be such that damage may occur to the holder Sleeves. It is an important part of this study that smoking is performed as close as possible to “normal” procedures. For this reason users of the rotary machine will allow the FCSAs to smoulder until the machine reaches its normal position for ejection. Users of the linear machine should cut or extinguish the smouldering cone as they would normally. In most cases this means that the cutting/extinguishing process will commence when the first FCSA reaches its butt mark.

2.0.9 Estimate and report the observed length of tobacco burned after the butt mark. It is most important that we are aware of the maximum length likely to be required for the convenient smoking of these articles. This is best done by observing the length remaining after extinction and subtracting this from 29 mm.

3.0 FINE CUT SMOKING ARTICLES

3.0.1 The fine-cut smoking articles to be used in this study will be manufactured by each participating laboratory.

3.0.2 The devices to be used to make the FCSAs must be the devices manufactured by EFKA Werke. The making of FCSAs is described in Appendix 2.

3.0.3 The specifications of the two fine-cut smoking articles to be used in this study are as follows:-

Length	70mm	Length	70mm
Diameter	5.2mm	Diameter	7.2mm
Tobacco weight	400mg	Tobacco weight	750 mg

The tobacco to be used is Drum Master Blend.
The cigarette paper will be Type A (see Appendix 1), of German manufacture.

3.0.4 The tobacco will be used direct from the pouches provided (without prior conditioning) when the FCSAs are made.

3.0.5 The FCSAs may be check weighed at the time of making.

3.0.6 There will be no subsequent weight or pressure drop selection procedures.

4.0 SMOKING MACHINES

4.0.1 Two types of smoking machines will be used in this study viz.

4.0.2 20-channel linear smoking machines smoking 5 articles through each channel on to a 44 mm filter pad.

4.0.3 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 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.

5.0 VARIABLES

5.1 Target variables

5.1.1 The variables to be analysed in order to estimate the between-laboratory and within-laboratory variability are Nicotine Free Dry Particulate Matter (NFDPM) and Nicotine. Other analytical measurements will be Puff number, Total Particulate Matter (TPM), and Water.

5.2 Variables to be reported

5.2.1 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.

5.2.2 The ancillary variables are

- type of smoking machine used
- laboratory temperature during smoking
- relative humidity (RH%) in the laboratory during smoking
- atmospheric pressure in the laboratory during smoking
- ambient air flow

Note that ISO Standard Method No. 3402 specifies that the Test Atmosphere shall be:

- 22 (+/- 2) degrees C
- 60 (+/-5) % RH
- 96 (+/- 10) kPa .

5.2.3 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

5.3 Dimensions and rounding of test results

5.3.1 Ancillary variables

- laboratory temperature	degrees Celsius.	##
- laboratory humidity	percent RH.	##
- laboratory pressure	kPa.	###

5.3.2 Analytical variables

- Puff number	Number	##.#
- TPM	mg per cigarette	##.##
- Nicotine	mg per cigarette	#.###
- Water	mg per cigarette	##.##
- NFDPM	mg per cigarette	##.##

[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.]

5.4 Internal documentation of tests

5.4.1 Each laboratory will document the method used following (where appropriate) the recommendations of ISO Standard 4387.

5.4.2 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.

6.0 EXCHANGE OF DATA

6.0.1 The validated data from this study will be made available to all the laboratories participating in the study.

6.0.2 To facilitate the exchange of data, all laboratories will be asked to send their results to Mr. H. Dymond for collation prior to the distribution of the data.

6.0.3 The format and medium to be used for the reporting of results are shown in Appendix 3 to this protocol.

7.0 DESIGN OF THE STUDY

7.1 Numbers of fine-cut smoking articles

7.1.1 The study will use the two fine-cut smoking articles specified in 3.0 above.

7.1.2 The study will also use the CORESTA monitor test piece CM2 as a monitor.

7.1.3 The monitor test piece will only be smoked in the standard ISO holder.

7.1.4 Each participating laboratory will be required to smoke 100 FCSAs of each diameter through each holder configuration.

7.1.5 Each participating laboratory will be required to smoke 100 monitor test pieces through the standard ISO holder.

7.2 Smoking plans

7.2.1 The smoking plans in Appendices 5 and 6 show two FCSAs (5.2mm and 7.2mm diameter) being smoked through four holder configurations. The combinations are coded as A, B, C, D, E, F, G, H, and one monitor test piece coded as M.

7.2.2 For each holder 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.

7.2.3 For this study, one test result is defined as the mean yield obtained from smoking 20 FCSAs or 20 monitor test pieces.

8.0 GENERAL REMARKS

8.0.1 Each participating laboratory will be asked to use only one smoking machine.

8.0.2 It is hoped that at least 16 laboratories will be recruited to take part in this study.

8.0.3 It would be advantageous if equal numbers of participating laboratories used the 20-channel linear smoking machine and the rotary smoking machine.

8.0.4 If possible, each participating laboratory will use only one operator when operating the smoking machine.

8.0.5 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.

8.0.6 The sealed glass fibre filter holder assembly, without the Sleeve holder or the labyrinth seal holder, will be weighed both before and after smoking.

8.0.7 The filter holder will be dry-wiped to ensure that any tar deposited in the holder is included in the nicotine/water extraction.

8.0.8 A new slotted washer will be used for each smoke run when using the ISO holder and care must be taken to use the correct size.

8.0.9 Using the ISO holder, the 5.2 mm FCSAs will be smoked with yellow labyrinth seals.

8.0.10 Using the ISO holder, the 7.2 mm FCSAs will be smoked with green labyrinth seals.

E. B. Wilkes / H. F. Dymond
14th. February, 1999

APPENDIX 1

DEFINITION OF TERMS

Fine-cut smoking article (FCSA). An article, suitable for smoking, produced by combining fine-cut tobacco with a wrapper. In this study it will be the article made using the EFKA werke making device and combining Drum Masterblend tobacco with the tubes supplied by EFKA and distributed by Albert De Vries.

"Normal" conditions. In the previous study FCSAs were made by one laboratory. In this study FCSAs are to be made within the participating laboratory (locally made articles) using the equipment and methods described in Appendix 3, i.e. under "normal" conditions.

Maximum Free Burn Zone. The difference in the length of tobacco between the insertion depth and the butt mark.

Required Free Burn Length. The length of tobacco burned after the butt mark has been reached before the burning coal is extinguished. You will be asked to note the longest length burnt for both article diameters.

EFKA A Company in Germany supplying wrappers for fine-cut tobacco and associated equipment**.** They will supply the tubes and the making devices to be used in this study.

Paper Type A. A wrapper for fine-cut tobacco classified as type A according to Draft F of the CORESTA Recommended Method No. 44. The particular paper to be used in this study will be similar to the best selling paper in Germany.

Validated data. Data with outlying data points removed. The procedure for identifying outliers will be given in the report of the statistical analysis of the data.

APPENDIX 2

THE MAKING OF FINE- CUT SMOKING ARTICLES USING THE EFKA WERKE MAKING DEVICE.

A2.1 On receipt of the tobacco from Albert De Vries, keep it in the sealed pouches in an appropriate room, preferably at 22 C and 75 % RH.

A2.2 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, preferably at 22 C and 75 % RH, until required. Do not open more than one pouch at a time.

A2.2.1 If possible use a balance in an air conditioned room (preferably at 22 C and 75 % RH)

A2.2.2 Using one airtight container at a time, weigh sufficient tobacco to end up with 750 +/- 10 mg of the Drum Masterblend tobacco for the 7.2 mm diameter fine-cut smoking articles or 400 +/- 10 mg tobacco for the 5.2 mm diameter fine-cut smoking articles.

NOTE: A2.2.2 is written in this way as different laboratories have different methods for making the fine-cut smoking articles. 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 net tobacco weight i.e. allowing for paper weight.

A2.2.3 Separate the tobacco to avoid lumps as each amount is weighed.

A2.2.4 Keep the weighed tobacco in good condition until required for making.

A2.3 Attach a pre-made tube onto the making device and mark the end attached.

A2.3.1 Carefully separate the tobacco from one weighing and spread it evenly onto the making device.

A2.3.2 Using a spatula, carefully introduce the tobacco into the receiving chamber of the making device.

A2.3.3 Partially close the chamber using the lever of the device.

A2.3.4 Reopen the chamber and scrape any remaining strands of tobacco into the chamber.

A2.3.5 Gently close the container completely again using the spatula to ensure that the tobacco is evenly spread and completely in the chamber.

A2.3.6 Using the thumb to hold the tube in place, gently and smoothly rotate the lever to push the tobacco into the tube.

A2.3.7 Open the chamber and ensure that no more than a few short strands of tobacco remain.

A2.3.7 Remove the fine-cut smoking article from the making device and inspect it for visual defects, e.g. bad shape, uneven filling, etc. Reject poorly made articles.

A2.3.8 Push in any tobacco strands hanging from the ends of the fine-cut smoking article. Do not cut them off.

A2.3.9 Weigh the individual fine-cut smoking article and reject any that are outside the limit ± 20 mg.

A2.4 Mark the insertion depth and butt mark

A2.5 Repeat the process to make sufficient FCSAs to produce 110 satisfactory ones for each holder variant and at each diameter. I.e. make at least 440 satisfactory smoking articles at each diameter.

A2.5.1 Weigh 22 fine-cut smoking articles and ensure that the mean weight is within ± 10 mg.

A2.6 Place the 22 finished articles carefully into a tin or plastic box capable of holding 22 FCSAs.

A2.7 Number the tins/boxes sequentially, starting at one, seal the tin and keep it in store preferably at 22 C and 75 % RH until required for conditioning prior to smoking.

A2.8 Tins 1, 5, 9, 13 and 17 will be used with the Labyrinth Seal holder; tins 2, 6, 10, 14 and 18 will be used with the 9 mm Sleeve holder; tins 3, 7, 11, 15 and 19 will be used with the 13 mm Sleeve holder; tins 4, 8, 12, 16 and 20 will be used with the 15 mm Sleeve holder.

A2.9 Repeat for the other diameter.

A2.10 Prior to smoking, open the tins of a batch to be used with the appropriate holder such that the articles can be conditioned for at least 3, but not more than 10, days at 22 C and 75 % RH.

APPENDIX 3

DATA SUPPORT FOR THE 16TH. CRYO STUDY.

This Appendix gives information concerning the collection of data for the 16th. CRYO study.

Given is :-

- a list of the variables used and the order in which they must be written on the data file. An example is also given.
- a list of codes assigned to each variable. This includes the code for each laboratory. Please use the codes given to simplify the transfer of data into a file suitable for statistical analysis.
- details of the characteristics of the floppy disc to be used for data transmission.

If your data is transmitted using a floppy disc then the disc MUST be accompanied by a printout of the file on the disc.

A set of data forms is also attached to be used if your data cannot be transmitted via a floppy disc.

The Means and SD's shown on these data forms are requested for the purpose of checking the (manual) entry of the data into the computer. Each data form will be checked as it is entered by comparing the Means and SD's of the data on the computer with those given on the data forms.

If you have any problems please contact Mr. H. Dymond.

Enclosed :-

ANNEX 1 : Order of variables on the smoking data file, and the format of the data file.

ANNEX 2 : Characteristics of the floppy discs to be used.

ANNEX 3 : List of participating laboratories, and their associated laboratory codes.

ANNEX 1 ORDER OF VARIABLES ON THE SMOKING DATA FILE

Each row of the data file must contain 12 variables in the following order:-

```
EXP   LAB  DATE  OPER  RNUM   CHAN  HOLDER  PUFF   TPM    WATER    NIC.    NFDPM
(1)   (2)  (3)   (4)    (5)   (6)    (7)     (8)   (9)     (10)   (11)     (12)

EXAMPLE :
 (1)    (2)     (3)    (4)   (5,6,7)   (8)    (9)    (10)   (11)     (12)
ST16   PMGE   070399   ABCD   1 1 A    9.1    3.79   2.34   0.121    1.33
ST16   PMGE   070399   ABCD   1 2 M    8.9    5.07   2.67   0.252    2.15

FORMAT(A4, A7, I7, A5, 2I3, A2, F5.1, 2F6.2, F6.3, F6.2)
All variables to be right-justified within the field.
```

EXPLANATION OF VARIABLE CODINGS

#	Name	Definition	Value	Length	Comment
(1)	EXP	Experiment code	ST16	A4	
(2)	LAB	Laboratory name	?????	A7	See ANNEX 3
(3)	DATE	Smoking date	DDMMYY	I7	
(4)	OPER	Operator code	?????	A5	Defined by you
(5)	RNUM	Smoking run number	1...10 1.. 45	I3	Linear machines Rotary machines
(6)	CHAN	Channel number	1...20 *	I3	Linear machines Rotary machines
(7)	HOLDER	Holder code	A,B,C,D,E F,G,H,M	A2	
(8)	PUFF	Puff count	##.#	F6.1	
(9)	TPM	Total particulate matter	##.##	F6.2	
(10)	WATER	Smoke water	##.##	F6.2	
(11)	NICOTINE	Smoke nicotine	#.###	F6.3	
(12)	NFDPM	Nicotine free dry particulate matter	##.##	F6.2	

Missing data are to be coded as *

The holder configurations used in this study are coded as follows:-

A. 5.2mm diameter FCSA ISO 9mm insertion
B. 7.2mm diameter FCSA ISO 9mm insertion
C. 5.2mm diameter FCSA SLEEVE 9mm insertion
D. 5.2mm diameter FCSA SLEEVE 13mm insertion
E. 5.2mm diameter FCSA SLEEVE 15mm insertion
F. 7.2mm diameter FCSA SLEEVE 9mm insertion
G. 7.2mm diameter FCSA SLEEVE 13mm insertion
H. 7.2mm diameter FCSA SLEEVE 15mm insertion
M. CM? Monitor test piece ISO 9mm insertion
* Not used (linear machines only; may be used for in-house monitor)

REMARKS

1. Please adhere strictly to the formats given above; it is much easier to read the data into computer files if all participating laboratories use the same format.

2. Write the sequence of variables as the first record on the file e.g.

EXP LAB DATE OPER RNUM PORT CIG PUFF TPM WATER TNA NFDPM

so that any accidental permutation of the variables can be detected.

3. The format as given occupies 60 characters and so can easily be written as a single line on most terminals.

4. As noted above, missing data are to be coded with an asterisk (*).

5. If you are using Excel or another spread sheet package in your laboratory please do not send the disc in this format. Instead go to “File”, on the drop down menu choose “Save As”, in the dialogue box “Save as type” choose and highlight “Formatted Text (Space delimited)”. Ensure that the file name is correct and that the “Save in” box shows 3½ Floppy (A), click Save. This will correctly format your disk and save considerable time.

6. As the file is to be in plain ASCII, it is not possible to issue a pre formatted disk, as discussed at the laboratory experts meeting. However the above instruction will ensure that the disk is easy to read.

7. When completed the floppy disc should be sent to

Mr. H. Dymond
45 Monarch Way
West End, Hampshire
SO3 3JQ
U.K.

Telephone 01703 476587 Fax 01703 470707 e-mail h.dymond@btinternet.com

The disc MUST be accompanied by a listing of the contents of the file(s) on the disc.

ANNEX 2 CHARACTERISTICS OF FLOPPY DISCS TO BE USED

The following diskettes can be read :-
IBM Compatible 3.5 inches Single Density (1.44 Mb)
IBM Compatible 3.5 inches Double Density (1.44 Mb)
CD-ROM

The file type MUST be plain ASCII.

If you have difficulty in meeting these requirements would you please refer to your own computing departments for assistance or contact Mr. H. Dymond to investigate alternative possibilities.

ANNEX 3 LABORATORY CODES

LABORATORY	CODE
PM-Europe	PME
BAT-Benelux	BATBE
SEITA	SEITA
Tiedemanns	TIEDE
House of Prince	HOP
Swedish Match	SWED
Rothmans (Europe)	ROTHE
BAT-Germany	BATGE
Poschl	POSC
BAT-Southampton	BATUK
Gallaher	GALLA
Imperial Tob. Co.	IMPTO
Rothmans U.K.	ROTUK
Filtrona, Jarrow	FILTR
Imperial Tobacco (Netherlands)	DOUWE
Royal Theodorus Niemeyer	NIEM
LTR Saint-Girons	LTRSTG

APPENDIX 5

SMOKING PLAN FOR 20-CHANNEL MACHINES

Holder codes:

A. 5.2mm diameter FCSA ISO 9mm insertion
B. 7.2mm diameter FCSA ISO 9mm insertion
C. 5.2mm diameter FCSA SLEEVE 9mm insertion
D. 5.2mm diameter FCSA SLEEVE 13mm insertion
E. 5.2mm diameter FCSA SLEEVE 15mm insertion
F. 7.2mm diameter FCSA SLEEVE 9mm insertion
G. 7.2mm diameter FCSA SLEEVE 13mm insertion
H. 7.2mm diameter FCSA SLEEVE 15mm insertion
M. CM2 Monitor test piece ISO 9mm insertion
* Not used (may be used for in-house monitor)

Channel no.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Run no. 1	E	M	E	C	A	D	E	C	D	C	D	A	E	*	A	D	*	M	C	A
2	B	H	G	H	*	G	M	F	B	F	B	H	*	B	F	G	H	F	M	G
3	G	B	H	F	H	M	H	B	G	M	G	F	G	F	*	F	B	H	B	*
4	D	C	*	A	E	A	C	D	M	A	E	*	A	D	C	M	E	C	D	E
5	*	E	A	*	C	E	A	M	E	D	M	C	D	C	D	A	C	A	E	D
6	M	F	B	M	G	F	B	*	F	H	*	G	H	G	H	B	G	B	F	H
7	H	G	M	B	F	B	G	H	*	B	F	M	B	H	G	*	F	G	H	F
8	C	A	D	E	D	*	D	A	C	*	C	E	C	E	M	E	A	D	A	M
9	A	D	C	D	M	C	*	E	A	E	A	D	M	A	E	C	D	E	*	C
10	F	*	F	G	B	H	F	G	H	G	H	B	F	M	B	H	M	*	G	B

Note that each consecutive pairs of runs are to be carried out as closely together as possible i.e. Run 2 will follow Run 1 as quickly as possible on the same day.

APPENDIX 6

SMOKING PLAN FOR ROTARY MACHINES

Holder codes:

A. 5.2mm diameter FCSA ISO 9mm insertion
B. 7.2mm diameter FCSA ISO 9mm insertion
C. 5.2mm diameter FCSA SLEEVE 9mm insertion
D. 5.2mm diameter FCSA SLEEVE 13mm insertion
E. 5.2mm diameter FCSA SLEEVE 15mm insertion
F. 7.2mm diameter FCSA SLEEVE 9mm insertion
G. 7.2mm diameter FCSA SLEEVE 13mm insertion
H. 7.2mm diameter FCSA SLEEVE 15mm insertion
M. CM? Monitor test piece ISO 9mm insertion

Run number	Holder code	Run number	Holder code	Run number	Holder code
1	A	16	F	31	C
2	E	17	D	32	M
3	H	18	C	33	G
4	D	19	G	34	E
5	M	20	M	35	A
6	G	21	D	36	H
7	F	22	H	37	E
8	B	23	F	38	A
9	C	24	C	39	B
10	A	25	E	40	M
11	G	26	B	41	D
12	B	27	A	42	G
13	H	28	F	43	C
14	E	29	B	44	F
15	M	30	D	45	H

NOTE A full set of reply forms was sent to all participants.

Appendix 10. Study 13

Study of paper parameters

Executive Summary

The Objective of this study was to evaluate a range of fine cut smoking articles made from different wrappers for yield of NFDPM and nicotine and to determine whether one or more test procedures, such as combustibility or air permeability, can be used in a simple manner, to classify papers into two categories.

An improved protocol was devised for the application of the Louisville Combustibility Test (LCT), and a large number of laboratories performed this test and also determined air-permeability by the standard CORESTA method. This study was devised to investigate further, relationships between these paper parameters and smoke yields.

Seven papers were examined and two statisticians examined the data independently using regression analysis.

Regression analysis showed that, based on overall means of the data, neither of the single paper parameters can be used on its own to predict smoke yields but that there is a clear relationship between the smoke data (NFDPM and nicotine) and the paper data combined (Permeability and LCT). This does not mean that the relationship can be used to predict yield. Further analysis showed that, even for the laboratories that participated in this study, the prediction equations perform poorly. It would be expected that if these predictors were to be used by laboratories that did not participate in this study then their performance is likely to be much worse. The poor performance of the prediction equations is only to be expected since, at this time, there is also considerable variation between the laboratories for the determination of smoke yields. This will be a major impediment to the establishment of any predictor between smoke yields and paper measurements.

Since the data from one paper parameter alone is not sufficient to classify paper for constituent yield it must be concluded that the classification should be done using another procedure.

It is clear that a direct method based on the determination of smoke yields must be more reliable than an indirect method.

However, the statistical studies carried out in this report demonstrate that the LCT method has potential for use within the paper converting industry. It is therefore recommended that further work is undertaken to develop the LCT method so that it can be used as a quality control tool within the paper converting industry.

Objective

To evaluate a range of fine cut smoking articles made from different wrappers for yield of NFDPM and nicotine and to determine whether one or more test procedures can be used in a simple manner, to classify papers into two categories.

Background

CORESTA RYO Study 10 indicated that different papers used with fine-cut tobaccos gave rise to differing NFDPM and nicotine values. These differences appeared to be correlated with results from the Louisville Combustibility Test (LCT) suggesting that the LCT might form the basis for a procedure to categorise papers. However, it was noted that the range of papers for Study 10 had only given results at the extremes of the LCT range. As a result, due to this selection of papers, there was uncertainty about the outcome of this study. In addition, the variability of the data from the LCT gave rise to some concern.

In order to conduct this study a defined protocol was devised for the application of the LCT, as well as recruiting a large number of laboratories to carry out air-permeability by the standard CORESTA method. A copy of the protocol used for the LCT test is given in Appendix 1.

This study was devised to investigate further, relationships between paper parameters and smoke yields.

The nature and extent of changes made to the LCT test mean that results of Studies 10 & 13 cannot be combined.

Protocol

A copy of the Protocol as distributed to participants is attached as Appendix 2.

Study Design

The cigarette papers investigated are described below.

1. NL : Typical Dutch paper (also in Study 10)
2. D : Typical German paper (also in Study 10)
3. UK : Typical UK paper (also in Study 10)
4. CND : Typical Canadian paper (also in Study 10)
5. WAT3 : Watten's Paper 3: 20-25 CU - no burn additive
6. WAT4 : Watten's Paper 4: 10 CU and up to 2% burn additive
7. WAT5 : Watten's Paper 5: Mid range base porosity, with additional perforation

FCSAs were made using the Dutch blend of fine-cut tobacco previously used in Study 10. Eight laboratories were recruited to conduct smoking tests. Twelve laboratories were recruited to provide Louisville combustibility tests and ten recruited to provide air permeability at 0.25kPa and 1kPa. In all 16 laboratories were involved in some part of the analysis.

Statistical Methods:

Two statisticians examined the data independently.

Analysis by Statistician 1

To meet the objective given above, a stepwise multiple regression procedure was adopted. The variables inputted into the model were as follows:

(a) Dependent Variables

NFDPM

Nicotine

Puff Number

(b) Independent variables.

LCT	Louisville Combustibility Test data.
log(APlk)	where APIk is Air Permeability measured in CU per second when a pressure of 1 kPa is applied.
$\log\left(\frac{4 \times AP25}{AP1k}\right)$	where AP25 is Air Permeability measured in CU per second when a pressure of 025 kPa is applied.

For each paper the average LCT of the 12 participating laboratories was used, while for log(APlk) and $\log\left(\frac{4 \times AP25}{AP1k}\right)$ the average of eight laboratories who supplied results to the required precision were used.

CORESTA recommended method No. 40 includes a definition of linearity as applied to papers. By constructing $\log\left(\frac{4\times AP25}{AP1k}\right)$ linear papers give values very close to zero. For clarity negative results were classified as zero, while positive values indicate the extent of non-linearity.

(c) Dummy Variables

In order to retain individual laboratory smoking data in the analysis, the following dummy variables were introduced:

FIL - Returns a value 1 if the participating laboratory uses a 20-channel linear smoking machine and 0 otherwise.
LAB2 - Returns a value 1 if Lab 2 supplied the smoke results and 0 otherwise.
LAB4 - Returns a value 1 if Lab 4 supplied the smoke results and 0 otherwise.
LAB6 - Returns a value 1 if Lab 6 supplied the smoke results and 0 otherwise.
LAB8 - Returns a value 1 if Lab 8 supplied the smoke results and 0 otherwise.
LAB11 - Returns a value 1 if Laboratory 11 supplied the smoke results and 0 otherwise.
LAB12 - Returns a value 1 if Laboratory 12 supplied the smoke results and 0 otherwise.

Two laboratories failed to report air-permeability to the required precision (0.5 CU). These laboratories were excluded from the averages of the two variables involving air permeability.

The remainder of the data were screened for outlier behaviour and after running some diagnostic checks, with the exception of one single value for the UK paper under the LCT, no further data were removed.

This analysis resulted in regressions for NFDPM, nicotine and puff number, fitted on 56 observations, which are presented below:

NFDPM = 11.47 + 0.0887*LCT + 4.113*FIL - 0.868*log(AP1K)+ 3.875*LAB6 + 1.623*LAB8 - 1.017*LAB11
$R^2 = 0.87$

Nicotine = 0.979 + 0.0058*LCT + 0.342*FIL - 0.0512*log(AP1K) + 0.342*LAB6 + 0.20*LAB8 - 0.129*LAB12
$R^2 = 0.86$

Puffs = 9.46 - 0.79*log(AP1K) + 5.61*log(4*AP25/AP1K) - 0.47*LAB2- 0.21*LAB6 - 0.52*LAB8 - 0.25*LAB12
$R^2 = 0.89$

The equations generated above can be used to ‘predict’ the outcome for NFDPM, nicotine and puff number for a combination of paper parameters. The predictions for NFDPM and nicotine are shown in the following table.

Table 1.
Predicted Values for NFDPM and Nicotine by LCT and Air Permeability

LCT	Air Permeability (CU)	NFDPM (mg)	Nicotine (mg)
40	5	15.7	1.29
40	60	13.6	1.16
60	5	17.5	1.41
60	60	15.3	1.28
80	5	19.3	1.52
80	60	17.1	1.39

This analysis suggests that data from the LCT may be an important factor in the prediction of NFDPM and nicotine yields for the papers used in this study. However, the above table also shows that data from an air permeability test (in particular air permeability measured at 1 kPascal) are also required.

It will be noted that $\log\left(\frac{4 \times AP25}{AP1k}\right)$ did not appear in the regression equations for NFDPM and nicotine, but did for puffs. This indicates that for the papers used in this study, non-linearity is only a factor with respect to puff number.

The need for a second paper parameter to predict constituent yield is demonstrated as follows (considering NFDPM only):

Given the ability of a system based on two parameters to predict NFDPM or nicotine, how much worse do the predictions become if one parameter, e.g. air permeability, is ignored. This is answered by running the regression for NFDPM with data on air permeability omitted. The average residual (i.e. the difference between what a model predicts and what an individual laboratory determines) is calculated for each paper in both situations. If residual values for any paper increase consistently in one direction then there is evidence of lack of fit which would be overcome by retaining the additional term. The model for NFDPM thus obtained is:

NFDPM = 6.54 + 0.124*LCT + 4.113*FIL + 3.875*LAB6 + 1.623*LAB8 - 1.017*LAB11
$R^2 = 0.80$

In terms of variation, loss of information on air permeability of the paper has increased the unexplained variation by 50% ($1-R^2$ rises from 0.13 to 0.20). What is more relevant is the effect on individual papers as measured by the average residual across laboratories. See Table 2.

Table 2.
Average residuals for NFDPM under 2 models

	Average Residual Under Regression	
Paper	With Air Permeability	Without Air Permeability
WAT3	0.20	0.54
WAT4	-0.33	0.62
WAT5	-0.58	-1.66
D	0.24	0.35
NL	0.52	-0.04
UK	0.19	0.05
CND	-0.24	0.14

WAT5 is poorly specified under the regression which excludes air permeability. It is noteworthy that all eight laboratories obtained a negative residual for this paper (ranging from -0.21 to -3.66). This demonstrates that, for these data, air permeability must be included.

Having established that more than one parameter is required it is now important to consider the reliability of the individual data.

The data given above are based on composite values from all participating laboratories. If a single laboratory was to classify papers on the basis of results of these tests, it would be necessary for the data to be repeatable within a laboratory, but more importantly, reproducible across laboratories.

The following tables illustrate the values for r and R for the LCT and for Air Permeability determined at 1 kPa.

Table 3.
Repeatability and Reproducibility values for the LCT test

Paper Type	Mean (sec)	r	R
Wat 3	56.9	7.0	20.9
Wat 4	49.7	7.6	18.5
Wat 5	53.4	6.0	13.6
D	45.0	7.0	11.7
NL	52.4	8.2	12.9
UK	76.7	12.7	21.4
CND	69.7	10.1	21.6

1 Outlier value removed from the UK paper.

Table 4.
Repeatability and Reproducibility values for the Air Permeability test

Paper Type	Mean (CU)	r	R
Wat 3	19.2	2.4	3.4
Wat 4	12.7	1.3	5.6
Wat 5	112.5	15.0	21.5
D	40.3	6.4	7.9
NL	64.2	12.2	16.8
UK	15.1	9.8	11.3
CND	10.8	3.1	4.0

Analysis by Statistician 2

The first step in the analysis was to examine the individual data for outliers.

OUTLIERS - AIR PERMEABILITY DATA MEASURED AT 1 kPa

The data from laboratory 13 presented problems. Each data point was reported to the nearest 5 units, whereas, for nearly all of the other laboratories, each data point was reported to the nearest 0.1 unit. (The only exception was laboratory 11 which reported it's data to the nearest 1 unit.) The air permeability data from laboratory 13 were therefore omitted from the analysis.

For each paper, a one-way analysis of variance between laboratories was used to calculate the standardised residual for each data point. The standardised residuals were also plotted against their normalised scores. Any data point with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure, eleven data points were deemed to be outliers and deleted from all subsequent analysis.

Having deleted the outlying data points, plots of laboratory means for paper WAT4 tended to indicate that laboratory 2 was an outlier and the Grubbs test confirmed this.

No other laboratory was indicated as a possible outlier by these plots.

Having deleted the outlying data points, and the data for paper WAT4 from laboratory 2, the means for each paper from each laboratory were calculated, as shown in Table 5.

Table 5
Mean permeability (CU) for each paper measured at 1 kPa

LAB	WAT3-1 Mean	WAT4-1 Mean	WAT5-1 Mean	D-1 Mean	NL-1 Mean	UK-1 Mean	CND-1 Mean
2	20.54	—	119.14	42.85	66.12	17.43	12.42
5	19.55	12.97	112.00	40.54	62.95	19.09	11.11
9	18.21	11.28	105.78	38.56	61.32	12.20	9.95
10	18.10	11.00	113.40	39.70	68.00	13.40	10.20
11	19.80	12.29	107.40	41.35	61.42	14.88	10.56
14	19.14	12.39	107.40	38.67	63.39	13.13	12.47
15	20.11	13.26	112.58	41.97	64.29	14.33	11.82
17	18.96	12.35	121.79	39.79	72.44	12.34	10.24
18	19.80	12.52	103.30	31.30	53.30	14.54	11.18
19	18.45	11.65	105.65	37.30	57.75	14.25	9.90
ALL	19.27	12.19	110.71	39.20	62.93	14.58	10.98

<u>OUTLIERS - AIR PERMEABILITY DATA MEASURED AT 0.25 kPa</u>

For each paper, the ratio of the air permeability measurements at 1 kPa and 0.25 kPa was calculated (after the removal of any outliers in the 1 kPa data). A one-way analysis of variance between laboratories was used to calculate the standardised residual for each ratio. The standardised residuals were also plotted against their normalised scores. Any ratio with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure 18 ratios were deemed to be outliers.

These data points for air permeability measured at 0.25 kPa were deleted from all subsequent analyses.

Plots of laboratory means for paper WAT5 tended to indicate that the mean ratio from laboratories 17 and 18 were outliers and the Grubbs test confirmed this. The measurements taken at 0.25 kPa for this paper by these two laboratories were therefore rejected.

Plots of laboratory means for paper D tended to indicate that the mean ratio from laboratory 18 was an outliers and the Grubbs test confirmed this. The measurements taken at 0.25 kPa for this paper by this laboratory was therefore rejected.

No other laboratory was indicated as a possible outlier by these plots.

Having deleted the outlying data as above, the mean ratio of the measurements taken at 1 kPa and 0.25 kPa for each paper were calculated. Results are shown in Table 6.

Table 6
Mean air permeability ratios

Lab.	WAT3-R Mean	WAT4-R Mean	WAT5-R Mean	D-R Mean	NL-R Mean	UK-R Mean	CND-R Mean
2	4.351	4.785	2.915	3.987	2.773	2.977	3.555
5	3.957	4.262	2.934	3.958	2.878	2.848	3.902
9	3.901	3.973	2.884	3.885	2.782	2.978	3.795
10	4.525	5.500	2.879	4.182	2.895	3.400	4.204
11	4.074	4.210	2.935	4.016	2.882	3.031	3.927
14	—	—	2.896	4.068	2.988	—	—
15	3.901	4.068	2.999	4.044	2.909	3.204	3.859
17	4.116	4.324	—	4.054	2.979	3.475	4.117
18	4.061	—	—	—	—	3.054	—
19	3.969	3.900	2.927	3.927	2.881	2.936	3.914
ALL	4.0970	4.3635	2.9207	4.0134	2.8776	3.0676	3.9053
	Linear	Linear	Non-Linear	Linear	Non-Linear	Non-Linear	Linear

In table 6, after consideration of the individual laboratory data, each paper is graded as being LINEAR or NON-L(inear).

OUTLIERS - LOUISVILLE COMBUSTION TEST

For each paper, a one-way analysis of variance between laboratories was used to calculate the standardised residual for each data point. The standardised residuals were also plotted against their normalised scores. Any ratio with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure, 17 data points were deemed to be outliers and were deleted from all subsequent analyses.

Plots of laboratory means for paper WAT3 tended to indicate that laboratories 9 and 13 were outliers and the Grubbs test confirmed this.

No other laboratory was indicated as a possible outlier by these plots.

Having deleted the outlying data, means for each paper from each laboratory were calculated, as shown in Table 7.

Table 7
Mean LCT data for each paper (sec)

Lab	WAT3 Mean	WAT4 Mean	WAT5 Mean	D Mean	NL Mean	UK Mean	CND Mean
2	59.22	52.03	53.25	45.58	52.24	81.85	68.59
5	62.78	52.68	54.66	48.54	54.09	76.45	70.54
6	62.68	57.82	60.40	44.39	57.22	76.66	71.21
7	55.40	48.50	53.60	42.00	50.70	78.30	69.80
9	—	34.22	48.57	48.77	47.10	70.51	84.55
10	57.67	51.22	56.00	44.20	53.50	88.00	72.33
11	55.80	47.80	52.67	41.80	48.20	74.60	61.20
13	—	43.96	44.09	38.84	47.72	63.80	56.54
14	58.54	51.72	53.08	45.64	56.72	82.27	71.00
15	64.50	55.90	59.20	51.50	58.70	84.00	76.40
18	60.30	52.80	55.50	44.80	54.10	76.50	70.00
19	58.60	48.80	51.40	44.30	50.10	73.56	66.70
ALL	59.57	50.04	53.52	45.03	52.33	77.16	69.87

OUTLIERS - SMOKE DATA

A two-way analysis of variance was used to calculate the standardised residual for each data point for NFDPM, nicotine and puffs. The standardised residuals were also plotted against their normalised scores. Any data point with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure 2, 5 and 7 data points were deemed to be outliers for NFDPM, nicotine and puffs respectively.

These data were deleted from all subsequent analyses and the means of the smoke data for each paper from each laboratory were calculated, as shown in Table 8.

Table 8
A. Means for PUFFS by paper and laboratory

LAB	CND	D	NL	UK	WAT3	WAT4	WAT5
2	7.78	7.04	7.96	8.86	7.32	7.48	7.16
5	7.26	6.34	7.70	8.86	6.94	7.00	7.14
6	7.42	6.94	7.72	9.00	6.98	7.66	7.56
7	7.28	6.30	7.46	8.38	6.68	6.82	6.78
9	7.44	6.54	7.96	8.82	6.80	7.20	7.28
10	7.85	6.72	8.18	9.18	6.96	7.54	7.38
11	7.06	6.34	7.44	8.63	6.58	6.66	6.62
13	7.27	6.73	7.78	8.69	6.74	6.75	7.35
ALL	7.409	6.619	7.766	8.805	6.876	7.139	7.159

B. Means for NFDPM by Paper and Laboratory

LAB	CND	D	NL	UK	WAT3	WAT4	WAT5
2	14.54	13.25	13.44	16.01	14.77	13.24	12.19
5	19.91	16.12	17.03	19.84	18.26	18.77	16.37
6	15.66	12.26	14.28	15.76	13.78	11.50	9.50
7	15.71	13.55	13.91	16.29	14.20	13.15	10.78
9	18.56	16.10	16.04	20.22	18.56	17.28	15.64
10	18.38	14.68	14.70	20.40	16.90	16.94	14.96
11	16.42	15.05	14.36	16.40	15.36	15.75	13.95
13	19.70	15.85	17.08	20.98	17.99	16.69	14.77
ALL	17.33	14.61	15.10	18.24	16.23	15.42	13.45

C. Means for Nicotine by Paper and Laboratory

LAB	CND	D	NL	UK	WAT3	WAT4	WAT5
2	1.226	1.122	1.150	1.272	1.252	1.170	1.108
5	1.458	1.184	1.304	1.548	1.354	1.378	1.318
6	1.286	1.054	1.168	1.270	1.186	1.004	0.878
7	1.182	1.112	1.134	1.246	1.176	1.088	0.912
9	1.520	1.344	1.362	1.664	1.524	1.462	1.378
10	1.660	1.310	1.380	1.766	1.498	1.514	1.420
11	1.474	1.309	1.278	1.358	1.365	1.389	1.264
13	1.508	1.273	1.324	1.553	1.344	1.296	1.184
ALL	1.413	1.212	1.262	1.452	1.337	1.288	1.183

REGRESSION ANALYSIS

Tables 5 to 8 provided the data for the regression analysis of the smoke data. The overall means of NFDPM and nicotine were regressed against the overall means of PERM, LCT, and against the indicator variable TYPE. The indicator variable TYPE was set equal to 1 for LINEAR papers and 2 for NON-L(inear) papers. The results of the regressions, based on seven data points follow.

For NFDPM the equation is

NFDPM = 11.6 - 0.0300 PERM + 0.594 TYPE + 0.0772 LCT (R^2 = 98%)

The analysis also shows that TYPE in non-significant; using PERM and LCT only as predictors the regression equation is

NFDPM = 11.2 - 0.0226 PERM + 0.0935 LCT (R^2 = 97%)

For smoke nicotine the regression equation is

Nicotine = 0.987 - 0.00120 PERM + 0.00628 LCT (R^2 = 98%)

These regressions show that, based on overall means of the data, neither of the single paper parameters can be used on its own but that there is a clear relationship between the smoke data (NFDPM and nicotine) and the paper data combined (PERM and LCT).

It is tempting to believe that the regression equations derived from these data could be used as general prediction equations for smoke yield if the values of PERM and LCT were known for any paper. This is most unlikely to be the case.

Tables 9 and 10 show the results of using these regressions to predict the NFDPM and nicotine data from the paper measurements for the laboratories that participated in this study.

Table 9 shows that, for laboratory 2, the predicted value is always higher than the observed value, whereas for laboratory 5 the reverse is the case. Laboratory 9 shows wide variations in the ERROR, from -5.3 to 3.1. The NFDPM values from laboratories 10 and 11 come somewhat closer to prediction.

Table 9
Prediction of NFDPM (mg)

LAB	PAPER	PERM	LCT	NFDPM Observed	NFDPM Predicted	ERROR (Obs-Pred)
2	CND	12.425	68.590	14.358	17.329	-2.971
2	D	42.848	45.580	13.250	14.490	-1.240
2	NL	66.117	52.240	13.444	14.586	-1.142
2	UK	17.427	81.850	16.026	18.456	-2.430
2	WAT3	20.545	59.220	14.772	16.270	-1.498
2	WAT4	*	52.030	13.238	*	*
2	WAT5	119.140	53.250	12.194	13.480	-1.286
5	CND	11.111	70.541	19.906	17.541	2.365
5	D	40.540	48.544	16.116	14.819	1.297
5	NL	62.950	54.088	17.032	14.830	2.202
5	UK	19.090	76.446	19.836	17.913	1.923
5	WAT3	19.550	62.875	18.262	16.634	1.628
5	WAT4	12.970	52.685	18.770	15.830	2.940
5	WAT5	112.000	54.657	16.370	13.773	2.597
9	CND	9.950	84.550	18.560	18.877	-0.317
9	D	38.560	48.770	16.100	14.885	1.215
9	NL	61.320	47.100	16.040	14.214	1.826
9	UK	12.200	70.510	12.220	17.514	-5.294
9	WAT3	18.210	*	18.560	*	*
9	WAT4	11.280	34.225	17.280	14.143	3.137
9	WAT5	105.780	48.567	15.640	13.344	2.295
10	CND	10.200	72.333	18.375	17.730	0.645
10	D	39.700	44.200	14.680	14.432	0.248
10	NL	68.000	53.500	14.700	14.661	0.039
10	UK	13.400	88.000	20.400	19.122	1.278
10	WAT3	18.100	57.667	16.900	16.180	0.720
10	WAT4	11.000	51.222	16.940	15.738	1.202
10	WAT5	113.400	56.000	14.960	13.867	1.093
11	CND	10.560	61.200	16.422	16.681	-0.259
11	D	41.350	41.800	15.054	14.170	0.883
11	NL	61.425	48.200	14.358	14.314	0.044
11	UK	14.878	74.600	16.400	17.836	-1.436
11	WAT3	19.800	55.800	15.362	15.967	-0.605
11	WAT4	12.290	47.800	15.746	15.389	0.357
11	WAT5	107.400	52.667	13.952	13.691	0.261

Table 10 shows that, for laboratory 2, the predicted value is always less than the observed value, whereas for laboratories 9 and 10 the reverse is the case. For laboratory 5 only 2 values of ERROR are negative, and for laboratory 11 only 1 value of ERROR is negative.

Table 10
Prediction of Nicotine (mg)

LAB	PAPER	PERM	LCT	Nicotine Observed	Nicotine Predicted	ERROR (Obs-Pred)
2	CND	12.425	68.590	1.226	1.403	-0.177
2	D	42.848	45.580	1.122	1.222	-0.100
2	NL	66.117	52.240	1.150	1.236	-0.086
2	UK	17.427	81.850	1.272	1.481	-0.209
2	WAT3	20.545	59.220	1.252	1.335	-0.083
2	WAT4	*	52.030	1.170	*	*
2	WAT5	119.140	53.250	1.108	1.179	-0.071
5	CND	11.111	70.541	1.458	1.417	0.041
5	D	40.540	48.544	1.184	1.244	-0.060
5	NL	62.950	54.088	1.304	1.252	0.052
5	UK	19.090	76.446	1.548	1.445	0.103
5	WAT3	19.550	62.875	1.354	1.359	-0.005
5	WAT4	12.970	52.685	1.378	1.303	0.075
5	WAT5	112.000	54.657	1.318	1.196	0.122
9	CND	9.950	84.550	1.520	1.506	0.014
9	D	38.560	48.770	1.344	1.247	0.097
9	NL	61.320	47.100	1.362	1.210	0.152
9	UK	12.200	70.510	1.664	1.416	0.248
9	WAT3	18.210	*	1.524	*	*
9	WAT4	11.280	34.225	1.462	1.189	0.273
9	WAT5	105.780	48.567	1.378	1.166	0.212
10	CND	10.200	72.333	1.660	1.429	0.231
10	D	39.700	44.200	1.310	1.217	0.093
10	NL	68.000	53.500	1.380	1.242	0.138
10	UK	13.400	88.000	1.766	1.524	0.242
10	WAT3	18.100	57.667	1.498	1.328	0.170
10	WAT4	11.000	51.222	1.514	1.296	0.218
10	WAT5	113.400	56.000	1.420	1.203	0.217
11	CND	10.560	61.200	1.474	1.359	0.115
11	D	41.350	41.800	1.309	1.200	0.109
11	NL	61.425	48.200	1.278	1.216	0.062
11	UK	14.878	74.600	1.358	1.438	-0.080
11	WAT3	19.800	55.800	1.365	1.314	0.051
11	WAT4	12.290	47.800	1.389	1.273	0.116
11	WAT5	107.400	52.667	1.264	1.189	0.075

The difference in the sign of ERROR in passing from one laboratory to another, coupled with the magnitude of the ERROR, shows that, even for the laboratories that participated in this study, the prediction equations perform poorly. It would be expected that if these predictors were to be used by laboratories that did not participate in this study then their performance is likely to be much worse.

The poor performance of the prediction equations is only to be expected. Examination of tables to 8 shows that there is considerable variation between the laboratories for both the paper measurements and the determination of smoke yields.

The data was reanalysed excluding the Dutch and Wat 5 papers. These two papers are clearly perforated and it is important to know whether the LCT could be used as a predictor for NFDPM for the non-perforated papers examined in this study.

Conclusions

It is acknowledged that FCSAs are intrinsically more variable than commercially manufactured cigarettes. Nonetheless, the variability of the data reported in this study, especially as reflected in the high between laboratory variation, must be a major impediment to the establishment of any predictor between smoke yields and paper measurements.

Since the data from one test alone is not sufficient to classify paper for constituent yield it must be concluded that the classification should be done using another procedure. It is clear that a direct method based on the determination of smoke yields must be more reliable than an indirect method.

However, it is also apparent that, on the basis of this test, the LCT is a useful predictor of combustibility, and therefore yields, for the type of non-perforated papers examined in this study. A procedure based on the LCT may have value within the paper industry as a tool for testing such papers.

The data shown in Table 3 indicates that the LCT is a basis on which to develop further improvements for the testing of paper combustibility. Further improvements to this test method are likely to provide less variable data.

Appendix 1

Paper Classification (A: LCT & B: Air Permeability)

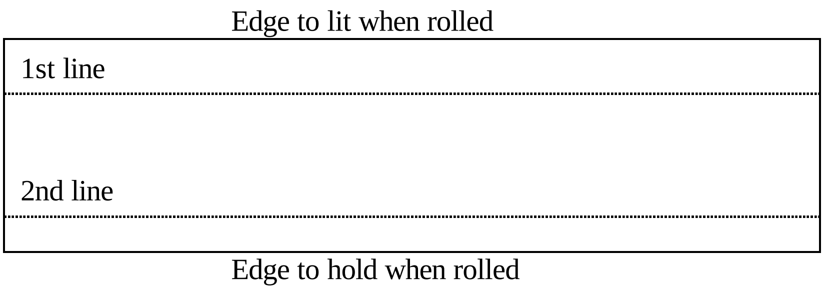
A) The Louisville Combustibility Test

Principle of the test:

Two lines are marked on a strip of paper 15 mm apart. The paper length is cut to 60 mm and rolled around a needle. The needle is removed and the paper clamped at one end. The paper is ignited and the time to smoulder between the two lines is a measure of the combustibility. The determination of the burn-time is conducted within a specially constructed box to prevent the influence of air-movements.

Figure 1

Direction of rolling →



Construction of the box:

The box should be constructed from Plexiglas (if necessary using a wooden or metal frame) and measure approximately: 50 cm wide x 40 cm deep by 30 cm high including any framing (see figure 2).

The front of the box should have a hole cut in it, large enough to allow the operator to insert the strip (diameter approximately 12 cm).

The centre of the hole should be placed about 12 cm from the left hand edge and 12 cm from the top of the box.

The top of the box should have a hole to allow the passage of air (diameter 2.5 cm). The centre of the hole should be placed about 12 cm from the right hand edge and 20 cm from the front of the box.

Within the box, construct a stand to hold the clamped paper strip (see procedure). This should be placed such that the lit end of the paper roll is exactly in the middle of the floor area of the box with the remainder of the roll facing the right hand side (see figure 3).

The box should be placed in a normal fume hood but there should be no air flow in the hood during the time of determination. The fume hood should be switched on to allow exhaust fumes to be removed after the determination.

PROCEDURE:

Take 10 strips of the cigarette-paper, each 60 mm length. The wire side is taken to be inside (is marked on the strips). On the “outside” of each paper-strip, draw a pencil line 5 mm from one edge, draw a second line 15 mm away from the first line. With the pencil-lines on the outside, wind a single strip round a steel needle, 2.5 mm in diameter, to form a small roll (the needles will be supplied). Remove the roll from the needle and hold it tightly using a “fold-back” paper clamp (will be supplied) at the end furthest from the pencil lines.

With the clamp, hold the roll and ignite the cigarette paper (by using non-flame ignition, such as a hot surface or an electric lighter). Still holding the clamp, transfer the ignited paper to the inside of the box, placing it accurately in position on the stand. Ensure the strip is held perfectly horizontally and measure the time taken for it to burn between the pencil marks, using a stop clock. Carry out the determination on each of the 10 strips.

Report the burning time in seconds for each of the 10 strips.

B) Air Permeability

The air permeability will be determined according to CORESTA recommended methods No. 40. In particular section 7.5 will be followed and any non linearity will be determined according to note 1. The air permeability will be measured on at least 10 strips and the individual data reported.

Appendix 2

Protocol for study 13

0. INTRODUCTION

0.1 This second experiment will test the viability of a possible matrix approach for the declaration of tar/nicotine yields of FCSAs.

0.2 This study is set up to investigate the relation between the LCT values of different papers and the tar/nicotine deliveries.

Tubes made of 7 commercially available papers with diameters of 7.2 mm will be filled with 750 mg of fine cut tobacco, which has been conditioned at 75 % RH before making.

The special constructed tubing device used in former studies will be used for making the FCSAs.

The Dutch blend of fine cut tobacco will be the same as used in other studies.

1. OBJECTIVES OF THE STUDY

1.1 To test the viability of that aspect of a possible matrix approach concerned with classification of different papers to the assessment of NFDPM and nicotine in the smoke from FCSAs. This study will be done with 1 diameter and 7 different papers, resulting in 7 samples.

2. STUDY DESIGN

2.1

Tube Diameter (mm)	Tobacco Papers Weight (mg)	Paper
7.2	750	NL, D, CND, UK
		WAT3, WAT4, WAT5

2.2 Before making, the tobacco will be conditioned at 22 °C and 75 % RH. After making the smoking articles will also be conditioned at 22 °C and 75 % RH.

2.3 All laboratories will analyse products made by tubing using the Dutch blend of RYO tobacco in the supplied tubes.

3. PRODUCT SPECIFICATION

3.1 One level of relative humidity (RH) will be used in this study.

3.1.1 This will be:

(75 ± 5) % RH

3.2 One blend of RYO will be used.

3.2.1 This will be:

Dutch blend

3.3 One diameter of tubes will be used.

3.3.1 These will be:

7 Types of tubes of commercially available papers with a diameter of 7.2 mm.

4. SMOKING ARTICLE MAKING

4.1 The tobacco will be a brand produced in Holland taken from a single batch production, already conditioned packed and sealed dispatched by the manufacturer (3 bundles, 10 packs each).

4.2 Before making, the tobacco will be conditioned at one moisture level. For this the tobacco must be unpacked and conditioned for 4 days at:

(22 ± 2) °C and (75 ± 5) % RH.

Keep the tobacco at this conditioning level until just before making the FCSAs. When removing the tobacco from conditioning for making the FCSAs, keep the tobacco, as much as possible, in a good sealed container to avoid moisture loss during making.

4.3 The test products shall be made from pre-formed tubes in order to minimise the total variability and to increase the sensitivity. The tubes will be provided and sent together with the tobacco. For tubing, one tubing device will also be sent to you. These tubing devices are modified to suit the two diameters.

4.4 Participating laboratories will be required to make their own FCSAs using tobacco, tubes and device supplied.

4.5 300 Tubes of each paper quality will be supplied to each participating laboratory.

4.6 From the conditioned tobacco for each paper quality (7 lots) at least 150 FCSAs should be made.

4.7 Weigh the amount of tobacco required (See 2.1) for each FCSA. Carefully introduce this tobacco into the tubing device, spread the tobacco evenly over the total length of the chamber and press it carefully in place. Transfer the tobacco into the tube until it presses against the end of the tube. Remove the product from the tubing machine. Carefully cut off any tobacco hanging out of the ends. Mark the end fixed to the tubing device as butt-end.

4.8 Store the FCSAs in good sealed containers to avoid the FCSAs drying out.

4.9 All FCSAs are restored at the conditioning level, 22 °C and 75 % RH, for 3 days.

4.10 After reconditioning weight-select out of each lot by mean weight ± 20 mg (See 6.4).

5. VARIABLES

5.1 Product NL is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper NL.

Product D is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a
Product CND is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper CND.

Product UK is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper UK.

Product WAT3 is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper WAT3.

Product WAT4 is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper WAT4.

Product WAT5 is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper WAT5.

5.2 The variables to be reported are as follows:

1. NFDPM in mg/FCSA.
2. Smoke Nicotine in mg/FCSA.
3. The total weight of 20 FCSAs prior to one smoking run.
4. Tobacco moisture (%) obtained prior to smoking (See 6.6.2).
5. Puff number.
6. Laboratory conditions during smoking.
7. Date of tests.

5.2.1 These seven variables will be reported as the test report.

5.2.2 An example of a test report is given in Appendix A2.

5.3 Other variables to be reported separately are:

1. Type of smoking machine used.
2. Moisture content of the tobacco before tubing (See 6.2.2).
3. Draw Resistance of the fine cut smoking articles prior to smoking (See 6.5.1).

5.4 It is important to note that for this study one test result is defined as the mean yield obtained from smoking 20 FCSAs in a single run. See smoking plan (Appendix A1) for the different types of smoking machine.

6. OPERATIONAL DETAILS

6.1 Select at random 1 pouch out of every bundle. Determine the moisture content preferably *by using the Karl Fischer method* according to ISO 6488 : 1992 rev.. If an alternative method is used, please specify.

6.2 Open the pouches and condition at (22 ± 2) °C and (75 ± 5) % RH (Tobacco before making and FCSAs). This to be achieved by using a conditioning cabinet or as follows:

Mix a quantity (generally about 1 kg) of sodium chloride, NaCl, (of at least GPR quality) with water to form a slurry. Ensure that there is always an excess of free salt. Pour the slurry into a desiccator. Place the desiccator in a conditioning cabinet and allow the air to circulate over the slurry.

6.2.1 For those laboratories not able to comply with one of these procedures they should still attempt to control the temperature and RH and report the method used.

6.2.2 Determine the moisture content after conditioning.

6.3 All test products should be made "en bloc" in the participating laboratories.

6.4 Recondition the FCSAs at 22 °C and 75 % RH, for 3 days.

6.5 Select each lot of FCSAs for weight as follows:

Determine the weight of 100 FCSAs for each product, calculate the mean and select to ±20 mg on individual pieces.

6.6 Prior to smoking perform the following tests on <u>each</u> batch of products.

6.6.1 Take 10 pieces of each sample and determine the Draw Resistance according to ISO 6565 : 1983.

Do not use these smoking articles for smoking.

6.6.2 Take sufficient FCSAs to provide a value for moisture content prior to smoking. p<u>referably determine the water content using the Karl Fischer</u> <u>method</u> according to ISO/DIS 6488 : 1994 rev.. Repeat this test for each smoking run if possible. Otherwise analyse a sample of the Masterblend conditioned and stored under the same conditions as the FCSAs.

6.6.3 Report all results and the method used.

6.7 Maintain the conditions of the smoking room to comply with ISO 3402:1991 i.e.

(20 ± 2) °C
(60 ± 5) % RH

6.8 Set up the smoking machine to comply with ISO 3308:1991. Pay particular attention to air flow control.

6.9 Use holders which conform to ISO 3308:1991.

6.10 Smoke all products to a butt length of 27 mm for all FCSAs with labyrinth seals according to ISO 3308:1991.

6.11 Smoke according to the procedures laid down in ISO 4387:1991.

6.11.1 Bring the conditioned FCSAs into the smoking room in sealed containers, open the containers just long enough to remove those FCSAs required for immediate smoking and reseal the container until the next set of FCSAs is required.

6.12 Analyse the extract for nicotine using method ISO 10315:1991.

6.13 Analyse the extract for water using either method ISO 10362/1:1993 or method 10362/2:1994.

6.14 Replicate the analyses 5 times (i.e. smoke 100 FCSAs of each sample) according to the smoking plan supplied (See Appendix A1).

7. <u>MISCELLANEOUS REQUIREMENTS</u>

7.1 It is preferable

- to complete the smoking procedure for the samples in 1 week.
- to use the same person to make all the test products.
- to use the same smoking machine throughout.
- to use the same operator for the smoking machine throughout.

7.2 If you are unable to comply with any of the details given in Section 6 report this fact and the alternative used.

8. REPORTING

8.1 Completed result sheets should be returned to:

Mr A. de Vries
Douwe Egberts van Nelle Tobacco Company
Department IMR Tobacco - Research
P.O. Box 3
8500 AA Joure
The Netherlands.
Fax: (0)513 - 488833.

8.2 The planning group will collate and analyse the data and produce full summary for the main task force meeting, held in Amsterdam on the 23rd of October 1996.

9. GENERAL REMARKS

9.1 The data will, as always, be reported anonymously. No reference will be made to a brand name or to individual companies. It will not be possible to determine the source of any individual data set from the report.

9.2 Actual time needed for smoking work should not exceed 2 weeks.

Appendix 11. Study 11

Study of filter tubes packed at various densities

Executive Summary

The objective of this study was to investigate the effect of packing density on smoke yields when fine-cut tobacco is used with different pre-made filter tubes.

Eight laboratories took part. The study examined 2 pre-made filter tubes, one ventilated and one non ventilated, each packed at densities of 220, 260 & 300 mg/cm^3.

Two statisticians examined the data independently.

The data show that with the non ventilated tube, NFDPM increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With the ventilated tube there is no significant density effect. This pattern of data is repeated for smoke nicotine.

From the data it is clear that a tubed product can be tested at two densities and that densities of 220 and 300 mg/cm^3 are appropriate. However with highly ventilated tubes, it is possible that there will be very little difference in yield as a result of the two densities. In these circumstances, it is recommended that a single figure, based on the average, will be more meaningful to the consumer. The TF concluded that a single figure should be quoted if the difference in yields is less than 15%, or where the difference in yield is less than 1 mg NFDPM or less than 0.1 mg Nicotine.

Objective

To investigate the effect of packing density on smoke yields when fine-cut tobacco is used with different pre-made filter tubes.

Protocol

A copy of the Protocol as distributed to the participants is attached as Annex 1.

Study Design

Eight laboratories were recruited. Five smoking runs (i.e. 100 FCSAs) were completed for each of six products. The study comprised a fully factorial 2 (filter tubes) x 3 (densities), with tube A (a non ventilated tube) and tube B (a ventilated tube) being the pre-made filter tubes packed at densities of 220, 260 & 300 mg/cm^3. The eight laboratories were split evenly between users of 20 channel rotary smoking machines and users of 20 channel linear machines.

Statistical Methods

Two statisticians examined the data independently. Statistical analysis was achieved in both cases using stepwise regression and three-way analyses of variance. Type of filter tube, density, and laboratory are main factors. Particular interest was directed (towards filter tube*density) interactions, to see if different pre-made filter tubes responded differently across the range of density. The main difference in approach between the two statisticians was in the treatment of outliers. One statistician took the approach of retaining as much individual data as possible, the other removed any data point with a standardised residual greater than 2.0 and which showed a marked deviation from the expected linear plot of normalised scores.

Analysis of data

Approach by Statistician 1

The stepwise regression analysis showed that for all analytes (NFDPM, Nicotine & Puff Number) most factors and interactions were significant, and that response of yield to density was dependent on the type of filter tube. Residuals and statistical 'measures of influence' were estimated from the models generated to examine if any data points were either bringing excess influence to bear on the conclusions and/or were grossly outlying. Results for the NFDPM model indicated three data points more than +/- 3 standard deviations from their predicted values. In the case of puff number there was just one. Further examination of these cases led to the data in question being retained.

In the case of nicotine, thirteen data points were deemed as 'influential' and six of these had residuals in excess of +/- 3 standard deviations. All of these data came from one laboratory. Consequently, it was decided to remove the data of this laboratory from the analysis of nicotine. Analysis of nicotine over the seven remaining laboratories produced a similar outcome to that for NFDPM and puff number. Two individual data points had residuals outside the range of +/- 3 standard deviations, but were retained on further investigation.

A diagnostic test (using principle components) indicated very strongly the independence of the three analytes considered and thus the data from the one laboratory, deemed an outlier for nicotine, relating to NFDPM and puff number were retained.

Approach by Statistician 2

The outlier approach used by this statistician resulted in the identification of 8 individual data points for puff number, 6 for NFDPM and 10 for nicotine. These data points were deleted from all subsequent analyses.

It is obvious that one laboratory had serious problems with the measurement of smoke nicotine. Of the 30 data points reported, 9 were deemed to be outliers and were not included in the subsequent analysis. Notably, all nicotine data for Tube A at 260 mg/cm^3 were deleted for that laboratory.

Grubbs test was used to determine if any laboratory was outlying within each cell of 2 x 3 design for each analyte. Although one Laboratory was most frequently tested for such behaviour the test failed on all occasions to confirm this. Consequently, no laboratory was removed.

Full analyses of variance for puff number, NFDPM, and nicotine showed that there was no significant difference between the two types of smoking machine used. There are highly significant main effects between Tubes A and B, between packing densities and between laboratories within smoking machines.

These analyses also show that the MACHINE*DENSITY, DENSITY*LABS, TUBES*LABS, and DENSITY*TUBES interaction terms are highly significant. The second order interaction term, DENSITY*TUBE*LABS is also highly significant.

In order to unravel the relationships within the data that give rise to these interactions, analyses of variance for the data from each tube taken separately were carried out.

When Tube A is used, the effect of DENSITY, and the MACHINE*DENSITY and DENSITY*LABS interactions are all highly significant. By contrast, when Tube B is used, the effect of DENSITY, and the MACHINE*DENSITY interaction, are not significant; the DENSITY*LABS interaction is significant.

This contrast in the performance of the two tubes is the main reason for the presence of the highly significant interaction terms involving TUBES reported in the full analyses of variance. Changes in packing density have a much greater effect on the delivery of NFDPM and Nicotine when Tube A is used than when Tube B is used.

Results

What follows is as a result of amalgamating the analyses of both statisticians, and is possible because the two modes of analysis produced virtually identical outcomes. Table 1 provides averages for NFDPM across all laboratories for both filter tubes at each density. This shows that with Tube A, NFDPM increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With Tube B there is no significant density effect.

Table 1: Mean NFDPM (mg/piece) across all laboratories

	Density mg/cm^3		
Filter tube	**220**	**260**	**300**
A	15.2	17.6	18.6
B	10.4	11.1	10.9

Table 2 shows the averages for nicotine in smoke across all laboratories for both filter tubes at each density. This shows that for Tube A, nicotine increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With Tube B there is little evidence of a density effect.

Table 2: Mean Nicotine (mg/piece) across laboratories

	Density mg/cm^3		
Filter tube	**220**	**260**	**300**
A	1.33	1.60	1.73
B	1.10	1.13	1.13

Table 3 shows the averages for puff number across all laboratories for both filter tubes at each density. This shows that for both filter tubes, puff number increases significantly across all densities, in proportion to the amount of tobacco burnt.

Table 3: Mean puff number across laboratories

	Density mg/cm^3		
Filter tube	**220**	**260**	**300**
A	6.9	8.4	9.7
B	8.1	9.8	11.5

Conclusions and Recommendations:

From the above data it is clear that a tubed product can be tested at two densities and that densities of 220 and 300 mg/cm^3 are appropriate. However with highly ventilated tubes, it is possible that there will be very little difference in yield as a result of the two densities. In these circumstances, it is recommended that a single figure, based on the average, will be more meaningful to the consumer. A single figure should be quoted if the difference in yield is less than 15% or where the difference in yield is less than 1 mg NFDPM or less than 0.1 mg Nicotine.

Annex 1 - Protocol

0. INTRODUCTION

0 .1 This experiment will test the viability of an appendix to the RYO matrix for the declaration of tar/nic yields of filter tubes.

0.2 This study is set up to investigate the influence of two different filter tubes at three different densities.

The two pre made tubes are commercially available with a diameter of 8.0 mm and will be filled with different amounts of fine cut tobacco, which has been conditioned at 75 % RH before making.

The tubes have a different design and give different yields.

A commercial available tubing device will be used for making the FCSAs.

The standard Dutch blend of fine cut tobacco will be the same as used in previous studies.

1. OBJECTIVES OF THE STUDY

1.1 To test the viability of an appendix to the RYO matrix concerned with diameters and different densities to the assessment of NFDPM and nicotine in the smoke from FCSAs. This study will be done with 2 different filter tubes and with 3 densities (220; 260; 300 mg/cm^3), resulting in 6 samples.

2. STUDY DESIGN

2.1

Tube (mm)	Density mg/cm^3)	Tobacco Weight (mg)
A	220 - 260 - 300	760 - 900 – 1040
B	220 - 260 - 300	760 - 900 - 1040

2.2 Before making, the tobacco will be conditioned at 22 °C and 75 % RH. After making the smoking articles will also be conditioned at 22 °C and 75 % RH.

2.3 All laboratories will analyse products made by tubing using the Dutch blend of RYO in the supplied filter tubes.

3. PRODUCT SPECIFICATION

3.1 One level of relative humidity (RH) will be used in this study.

3.1.1 This will be: (75 ± 5) % RH

3.2 One blend of RYO will be used.

3.2.1 This will be: Dutch blend

3.3 Two designs of filter tubes will be used.

3.3.1 These will be 2 types of filter tubes, commercially available, with a diameter of 8.0 mm.

A: Design as the market leader in Europe

B: Typical Scandinavian design

4. SMOKING ARTICLE MAKING

4.1 The tobacco will be a brand produced in Holland taken from a single batch production, already conditioned packed and sealed dispatched by the manufacturer (1 bundle, 10 packs each).

4.2 Before making, the tobacco will be conditioned at one moisture level. For this the tobacco must be unpacked and conditioned for 4 days at (see appendix A3):

(22 ± 2) °C and (75 ± 5) % RH.

Keep the tobacco at this conditioning level until just before making the FCSAs. When removing the tobacco from conditioning for making the FCSAs, keep the tobacco, as much as possible, in a good sealed container to avoid moisture loss during making.

4.3 The test products shall be made with pre-formed filter tubes. The filter tubes will be provided and sent together with the tobacco. For tubing, one tubing device will also be sent to you.

4.4 Participating laboratories will be required to make their own FCSAs using tobacco, tubes and device supplied.

4.5 400 filter tubes of each design will be supplied to each participating laboratory.

4.6 From the conditioned tobacco for each filtertube (2 lots) at FCSAs should be made.

4.7 Weigh the amount of tobacco required (See 2.1) for each FCSA. Carefully introduce this tobacco into the tubing device, spread the tobacco evenly over the total length of the chamber and press it carefully in place. Transfer the tobacco into the tube until it presses against the end of the tube. Remove the product from the tubing machine. Carefully cut off any tobacco hanging out of the ends.

4.8 Store the FCSAs in good sealed containers to avoid the FCSAs drying out.

4.9 All FCSAs are restored at the conditioning level, 22 °C and 75 % RH, for 3 days.

4.10 After reconditioning weight-select out of each lot by mean weight ± 20 mg (See 6.4).

5. VARIABLES

5.1 Product 220A is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 760 mg with filtertube A.

Product 220B is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 760 mg with filtertube B.

Product 260A is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 900 mg with filtertube A.

Product 260B is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 900 mg with filtertube B.

Product 300A is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 1040 mg with filtertube A.

Product 300B is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 1040 mg with filtertube B.

5.2 The variables to be reported are as follows:

1. NFDPM in mg/FCSA.
2. Smoke Nicotine in mg/FCSA.
3. The total weight of 20 FCSAs prior to one smoking run.
4. Tobacco moisture (%) obtained prior to smoking (See 6.6.2).
5. Puff number.
6. Laboratory conditions during smoking.
7. Date of tests.

5.2.1 These seven variables will be reported as the test report.

5.2.2 An example of a test report is given in Appendix A2.

5.3 Other variables to be reported separately are:

1. Type of smoking machine used.
2. Moisture content of the tobacco before tubing (See 6.2.2).
3. Draw Resistance of the fine cut smoking articles prior to smoking (See 6.5.1).

5.4 It is important to note that for this study one test result is defined as the mean yield obtained from smoking 20 FCSAs in a single run. See smoking plan (Appendix A1) for the different types of smoking machine.

6. OPERATIONAL DETAILS

6.1 Select at random 1 pouch out of every bundle. Determine the moisture content preferably by using the Karl Fischer method according to ISO 6488 : 1992 rev. If an alternative method is used, please specify.

6.2 Open the pouches and condition at (22 ± 2) °C and (75 ± 5) % RH (Tobacco before making and FCSAs). This to be achieved by using a conditioning cabinet or as follows:

Mix a quantity (generally about 1 kg) of sodium chloride, NaCl, (of at least GPR quality) with water to form a slurry. Ensure that there is always an excess of free salt. Pour the slurry into a desiccator. Place the desiccator in a conditioning cabinet and allow the air to circulate over the slurry (see appendix A3).

6.2.1 For those laboratories not able to comply with one of these procedures they should still attempt to control the temperature and RH and report the method used.

6.2.2 Determine the moisture content after conditioning.

6.3 All test products should be made “en bloc” in the participating laboratories.

6.4 Recondition the FCSAs at 22 °C and 75 % RH, for 3 days.

6.5 Select each lot of FCSAs for weight as follows:

Determine the weight of 100 FCSAs for each product, calculate the mean and select to ± 20 mg on individual pieces.

6.6 Prior to smoking perform the following tests on each batch of products.

6.6.1 Take 10 pieces of each sample and determine the Draw Resistance according to with ISO 6565 : 1983.

Do not use these FCSAs for smoking.

6.6.2 Take sufficient FCSAs to provide a value for moisture content prior to smoking. Preferably determine the water content using the Karl Fischer method according to ISO/DIS 6488 : 1994 rev..

Repeat this test for each smoking run if possible. Otherwise analyse a sample of the Dutch blend conditioned and stored under the same conditions as the FCSAs.

6.6.3 Report all results and the method used.

6.7 Maintain the conditions of the smoking room to comply with ISO 3402 : 1991 i.e.

(20 ± 2) °C
(60 ± 5) % RH

6.8 Set up the smoking machine to comply with ISO 3308 : 1991. Pay particular attention to air flow control.

6.9 Use holders which conform to ISO 3308 : 1991.

6.10 Smoke all products to a butt length of 28 mm for all FCSAs with labyrinth seals according to ISO 3308 : 1991.

6.11 Smoke according to the procedures laid down in ISO 4387 : 1991.

6.11.1 Bring the conditioned FCSAs into the smoking room in sealed containers, open the containers just long enough to remove those FCSAs required for immediate smoking and reseal the container until the next set of FCSAs is required.

6.12 Analyse the extract for nicotine using method ISO 10315 : 1991.

6.13 Analyse the extract for water using either method ISO 10362/1 : 1993 or method 10362/2 : 1994.

6.14 Replicate the analyses 5 times (i.e. smoke 100 FCSAs of each sample) according to the smoking plan supplied (See Appendix A1).

7. MISCELLANEOUS REQUIREMENTS

7.1 It is preferable

- to complete the smoking procedure for the samples in 1 week.
- to use the same person to make all the test products.
- to use the same smoking machine throughout.
- to use the same operator for the smoking machine throughout.

7.2 If you are unable to comply with any of the details given in Section 6 report this fact and the alternative used.

8. REPORTING

8.1 Completed result sheets should be returned a.s.a.p. but not later than by the 20th of September 1996 to:

Mr A. de Vries
Douwe Egberts Van Nelle Tobacco Company
Department IMR Tobacco - Research
P.O. Box 3
8500 AA Joure
The Netherlands.
Fax: (0)513 - 488299.

8.2 The planning group will collate and analyse the data and produce full summary for the main task force meeting, held in Amsterdam on the 23rd of October 1996.

9. GENERAL REMARKS

9.1 The data will, as always, be reported anonymously. No reference will be made to a brand name or to individual companies. It will not be possible to determine the source of any individual data set from the report.

9.2 Actual time needed for smoking work should not exceed 2 weeks.