

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 it 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 issue with 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 it 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 than 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 component	Error term	Expected Mean Square (using unrestricted model)
1 LAB	0.3557	2	(2) + 5.0000(1)
2 Error	0.1301	(2)	

r = 1.010 R = 1.952

Table 1.3

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
-----+-----+-----+-----
                                (-----*-----)
                                (-----*-----)
                                (-----*-----)
                                (-----*-----)
                                (-----*-----)
-----+-----+-----+-----
                                -0.10    -0.00    0.10

POOLED STDEV = 0.1829

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

                                -----+-----+-----+-----
                                (-*-)
                                (-*-)
                                (-*-)
                                (-*-)
                                (-*-)
                                -----+-----+-----
                                -0.60    -0.00    0.60

POOLED STDEV =    1.1158

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   -0.04651
    0.10392   0.06167
 4  0.01892   -0.02333   -0.03091
    0.12710   0.08485   0.07727
 5  0.02033   -0.02192   -0.02950   -0.05268
    0.12851   0.08626   0.07868   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

                                -----+-----+-----+-----
                                (---*---)
                                (---*---)
                                (---*---)
                                (---*---)
                                -----+-----+-----+-----
                                -0.030      0.000      0.030

POOLED STDEV = 0.07313

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 4.4

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

Normalised Plot of sd(NFDPM)

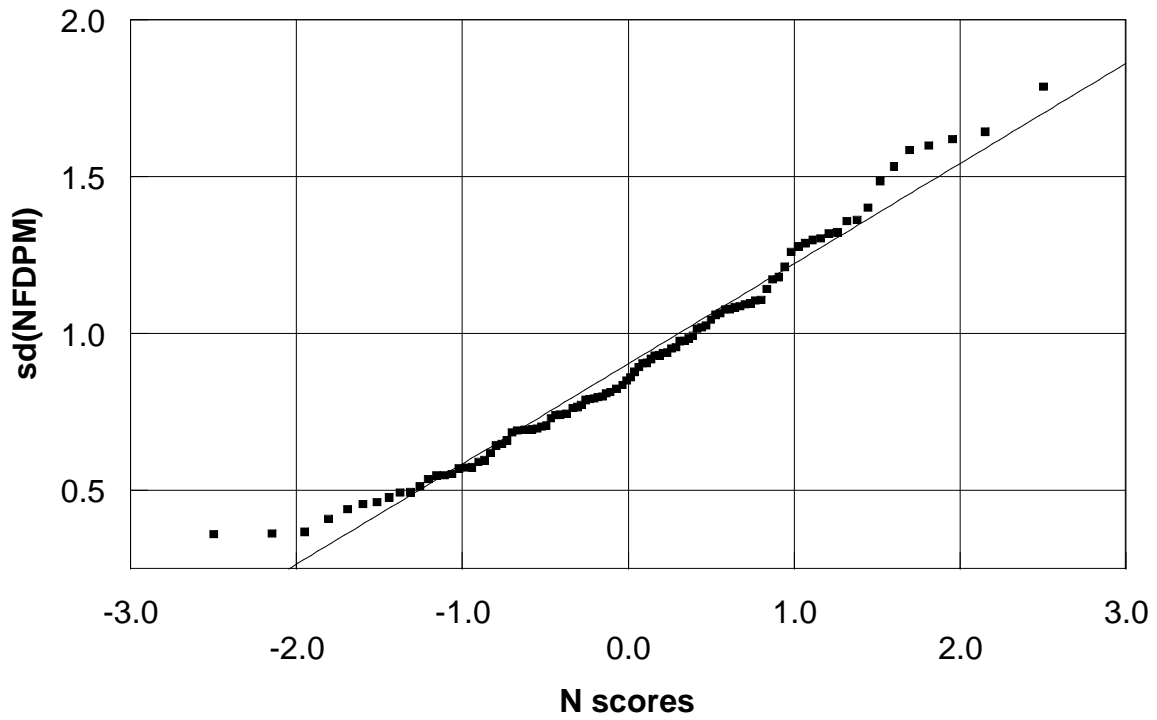


Figure 2.02

Normalised Plot of transformed sd(NFDPM)

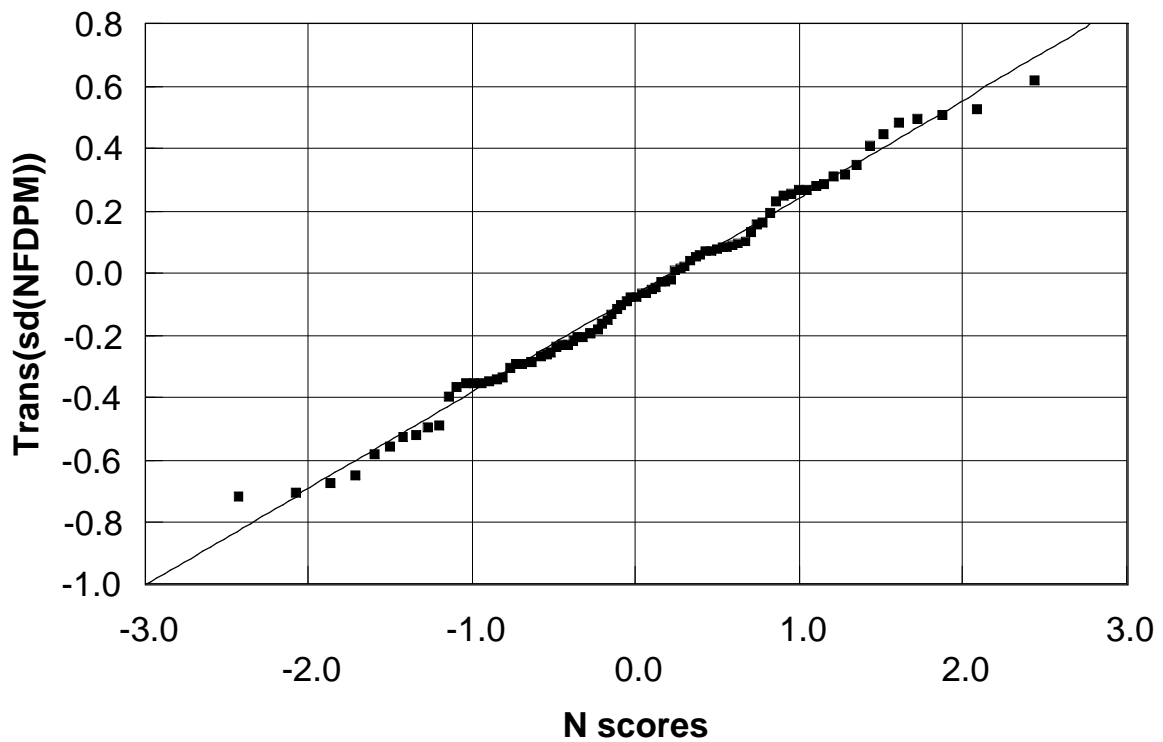


Figure 2.1

Mean Residual (Mean NFDPM)
with 95% confidence intervals

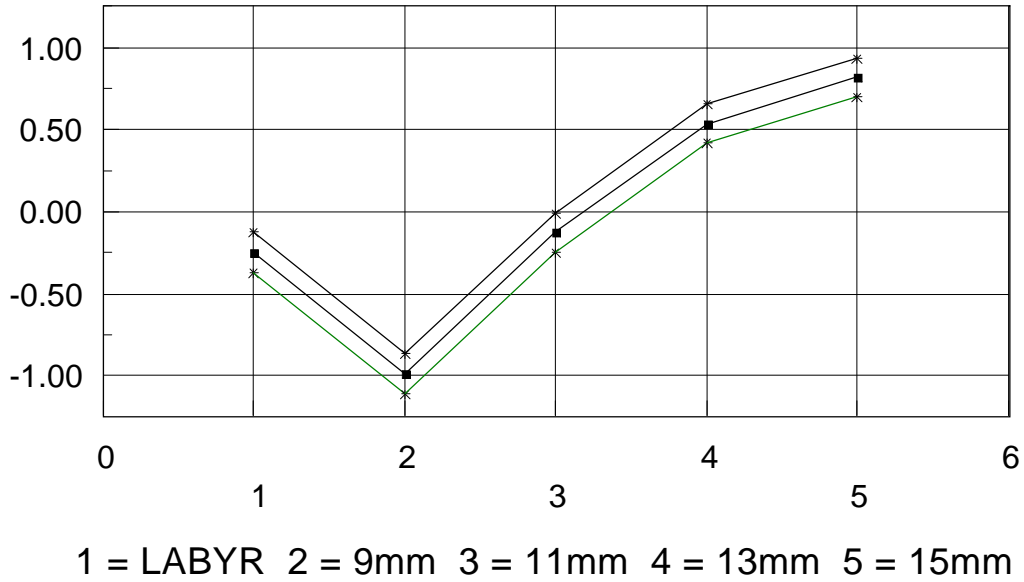


Figure 2.2

Mean Residual Transformed sd(NFDPM)
with 95% confidence intervals

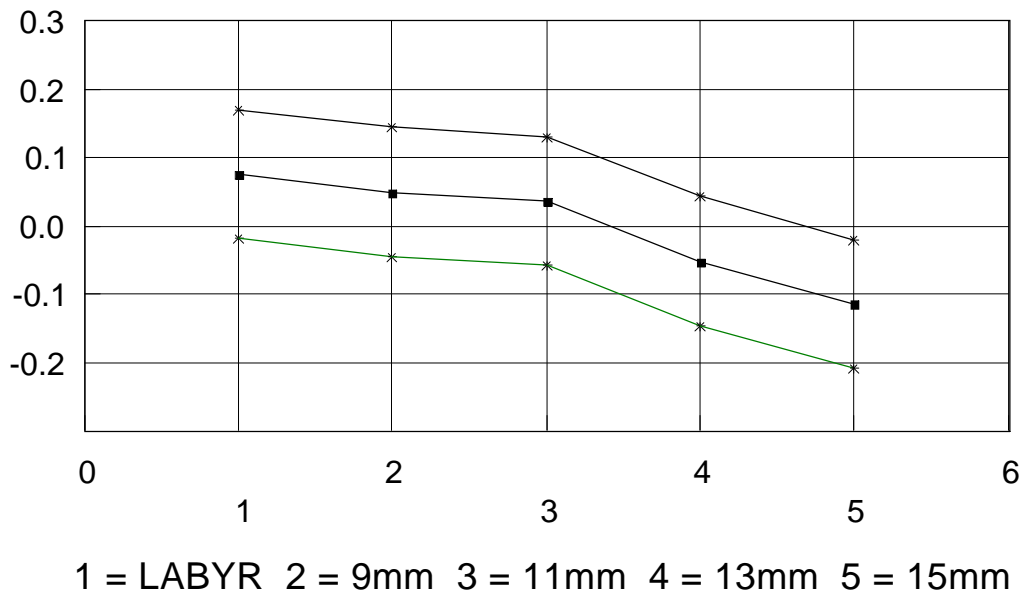
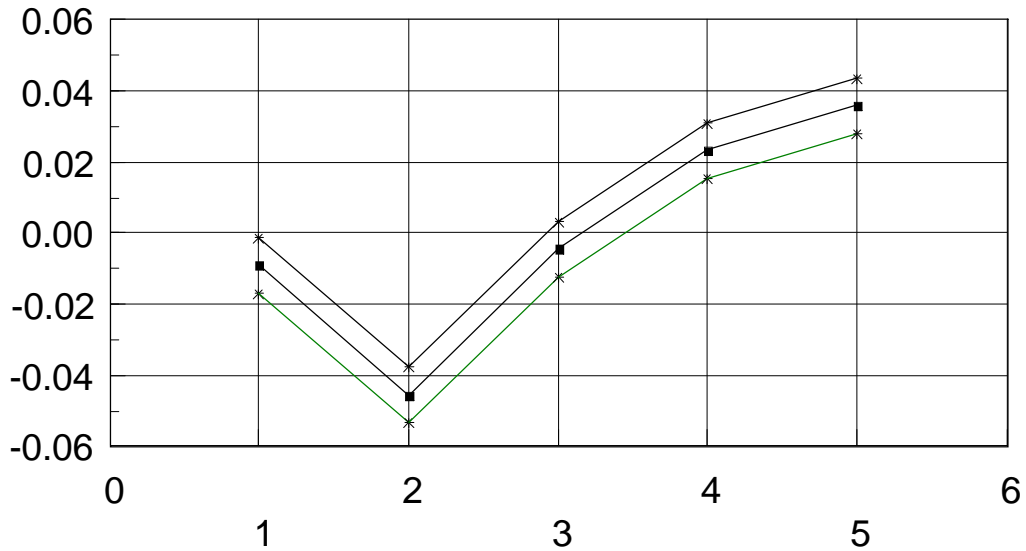


Figure 3.1

Mean Residual (Mean Nicotine)

with 95% confidence intervals

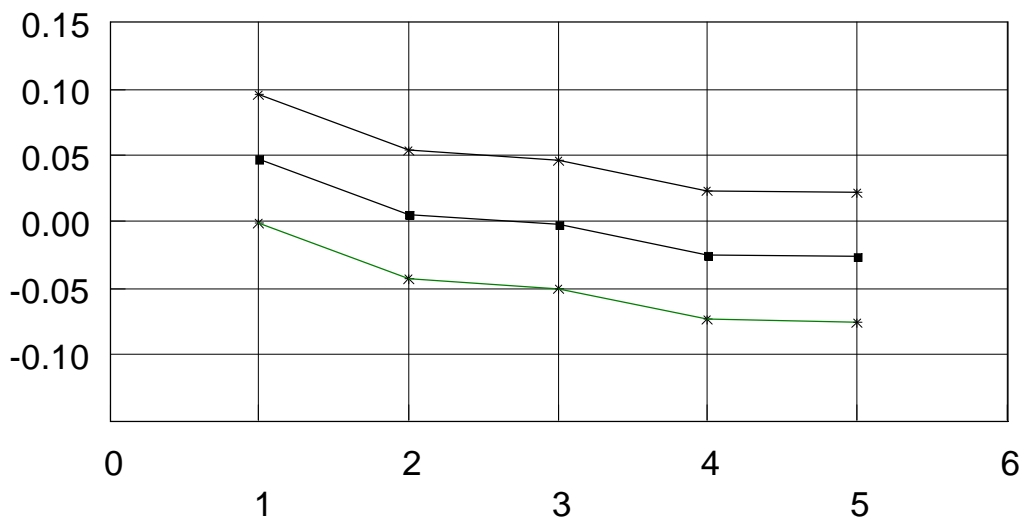


1 = LABYR 2 = 9mm 3 = 11mm 4 = 13mm 5 = 15m

Figure 3.2

Mean Residual Transformed sd(Nicotine)

with 95% confidence intervals



1 = LABYR 2 = 9mm 3 = 11mm 4 = 13mm 5 = 15mm

Figure 4.0

Normalised Plot of sd(Puff Number)

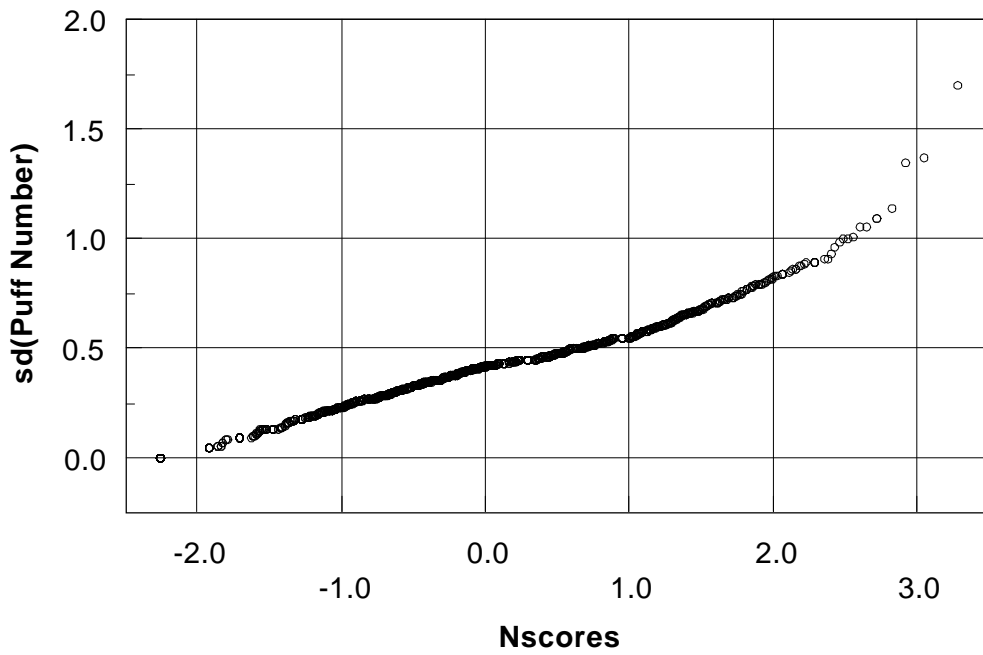


Figure 4.1

Mean Residual (Mean Puff Number)
with 95% confidence intervals

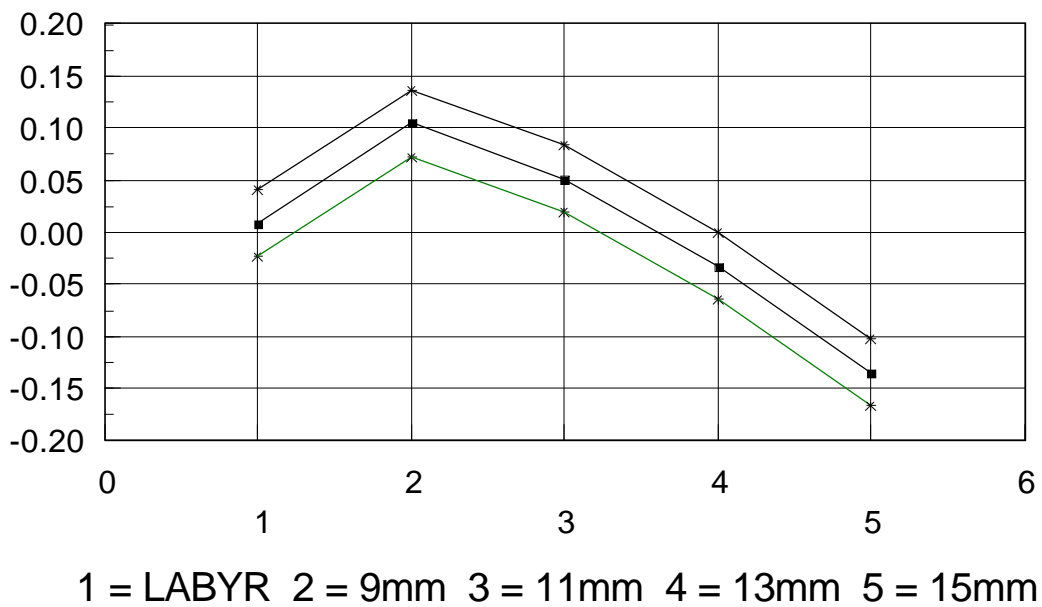
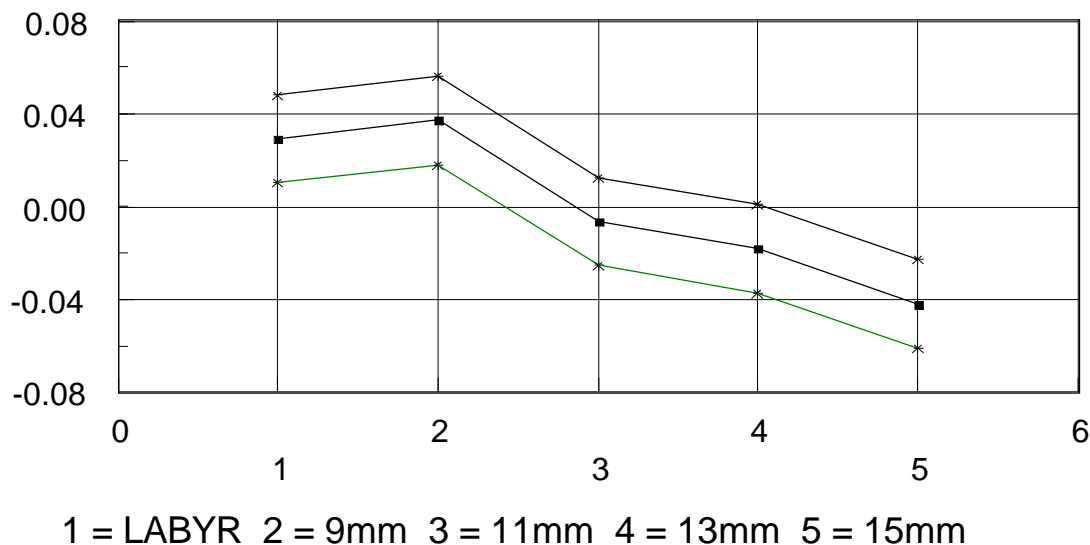


Figure 4.2

Mean Residual sd(Puff Number)

with 95% confidence intervals



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 mgThe 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-channel 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 $22\pm 3^{\circ}\text{C}$, $(60\pm 5)\% \text{RH}$]

- 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 1 st FCSA	##.#	F6.1	
(9)	FCSA2	Puff count for 1 st FCSA	##.#	F6.1	
(10)	FCSA3	Puff count for 2 nd FCSA	##.##	F6.1	
(11)	FCSA4	Puff count for 3 rd FCSA	##.##	F6.1	
(12)	FCSA5	Puff count for 4 th 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]
 - IBM Compatible 3.5 inches Double Density] (1.44 Mb)
- 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