

## **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

$$(\text{NFDPM, nicotine}) = \text{Laboratory effects} + \text{error}$$

for each holder configuration was used. This can be written as

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

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  
 BASED ON POOLED STDEV

HOLDER	N	ADJUSTED 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

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 0.1118	-0.0012 0.1222	
E	-0.0443 0.0783	-0.0347 0.0887	-0.0952 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  
 BASED ON POOLED STDEV

HOLDER	N	ADJUSTED 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

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 -0.02894	-0.02097 0.00135	
E	-0.05338 -0.03120	-0.02323 -0.00091	-0.01342 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  
 BASED ON POOLED STDEV

HOLDER	N	ADJUSTED 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

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  
 BASED ON POOLED STDEV

HOLDER	N	ADJUSTED 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

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
                                BASED ON POOLED STDEV
HOLDER      N      ADJUSTED 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
                                BASED ON POOLED STDEV
HOLDER      N      ADJUSTED 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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 (using unrestricted model)	Mean Square	
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 (using unrestricted model)	Mean Square	
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 (using unrestricted model)	Mean Square	
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 (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	Square
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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 component	Error term	Expected (using unrestricted model)	Mean Square	
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

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

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 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

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 0.05532	-0.03431 0.02875	
H	-0.01752 0.04554	-0.04409 0.01897	-0.04131 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

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 0.12367	-0.00401 0.10535	
H	-0.00168 0.10768	-0.02000 0.08936	-0.07067 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 <sub>nic</sub>	C <sub>nic</sub>	D <sub>nic</sub>	E <sub>nic</sub>	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

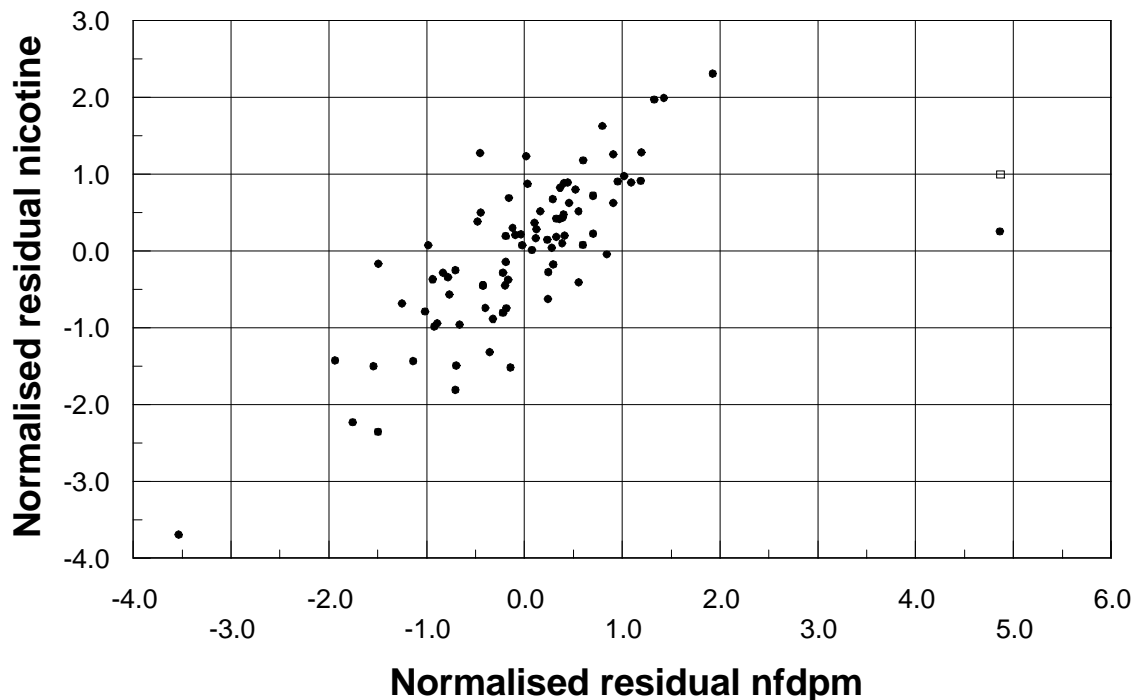
NORMALISED SCORES and DISTANCE

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

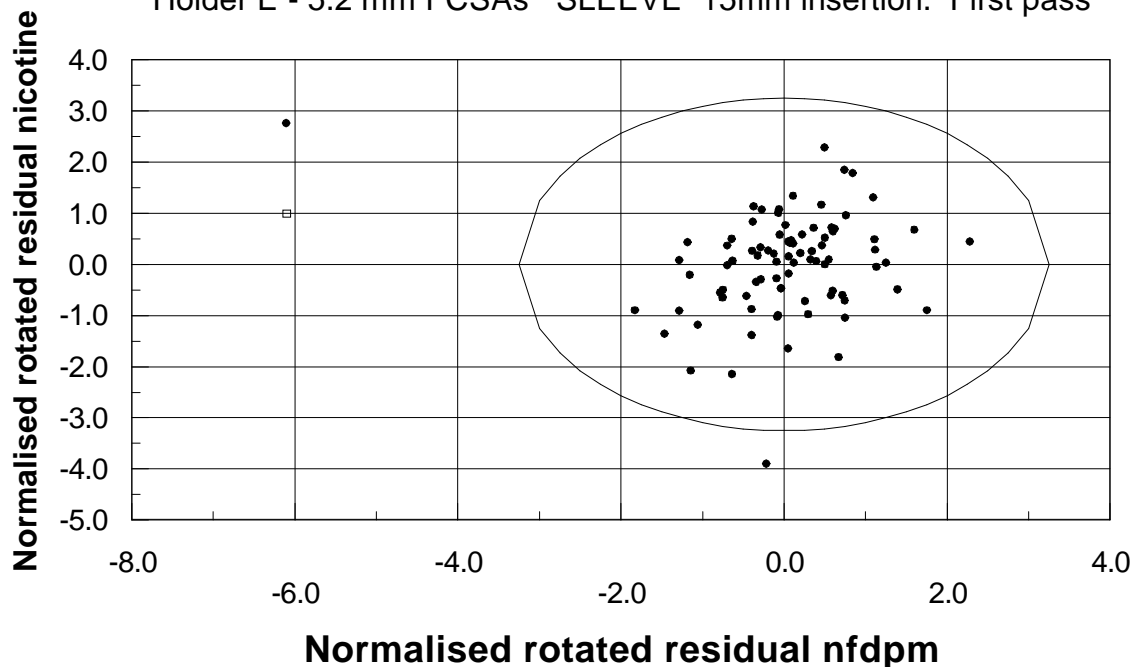
**Figure 1 Study 16. p-variate analysis**

Holder E - 5.2mm FCSAs SLEEVE 15mm insertion. First pass



**Figure 2 Study 16. p-variate analysis**

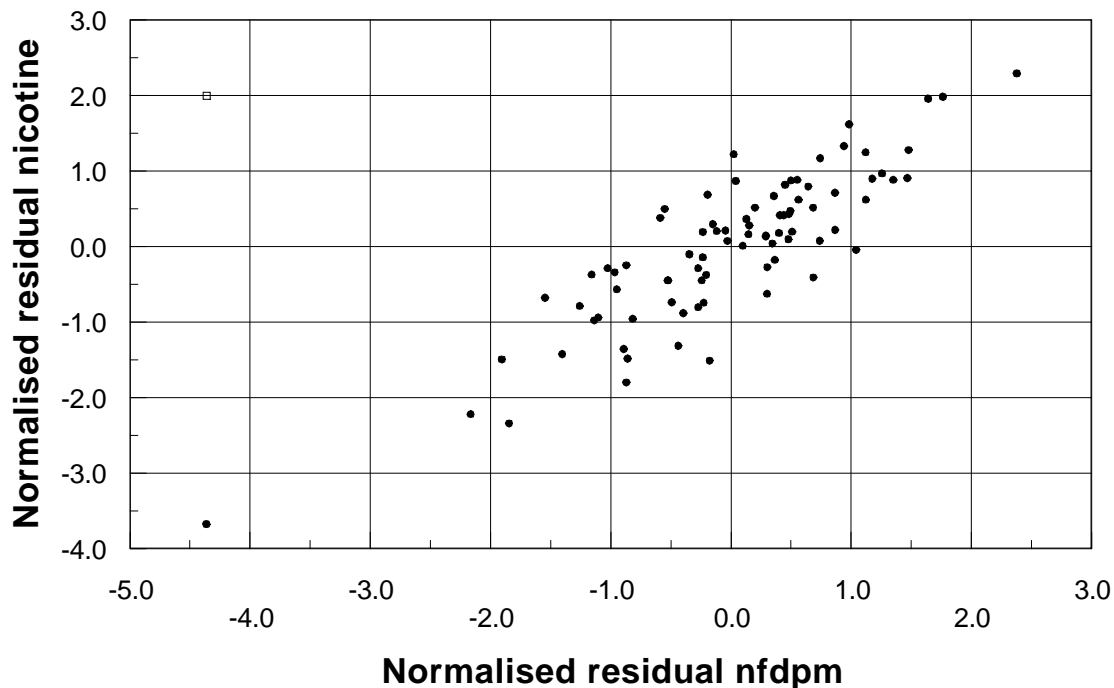
Holder E - 5.2 mm FCSAs SLEEVE 15mm insertion. First pass



The dotted line is the 3.25 sd contour line

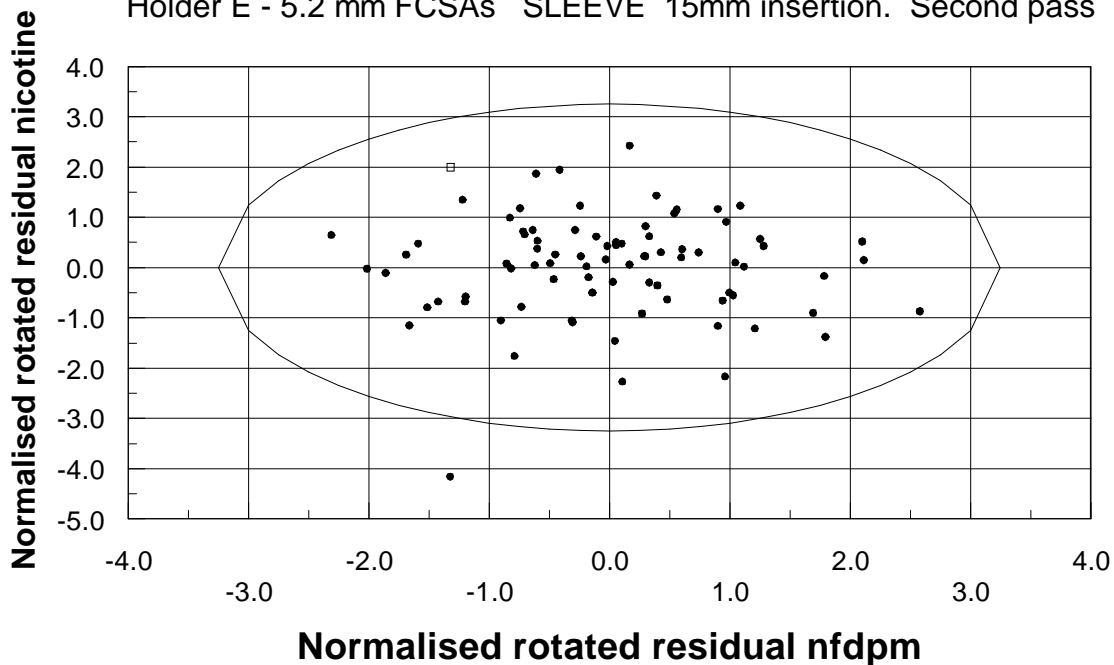
**Figure 3 Study 16. p-variate analysis**

Holder E - 5.2mm FCSAs SLEEVE 15mm insertion. Second pass



**Figure 4 Study 16. p-variate analysis**

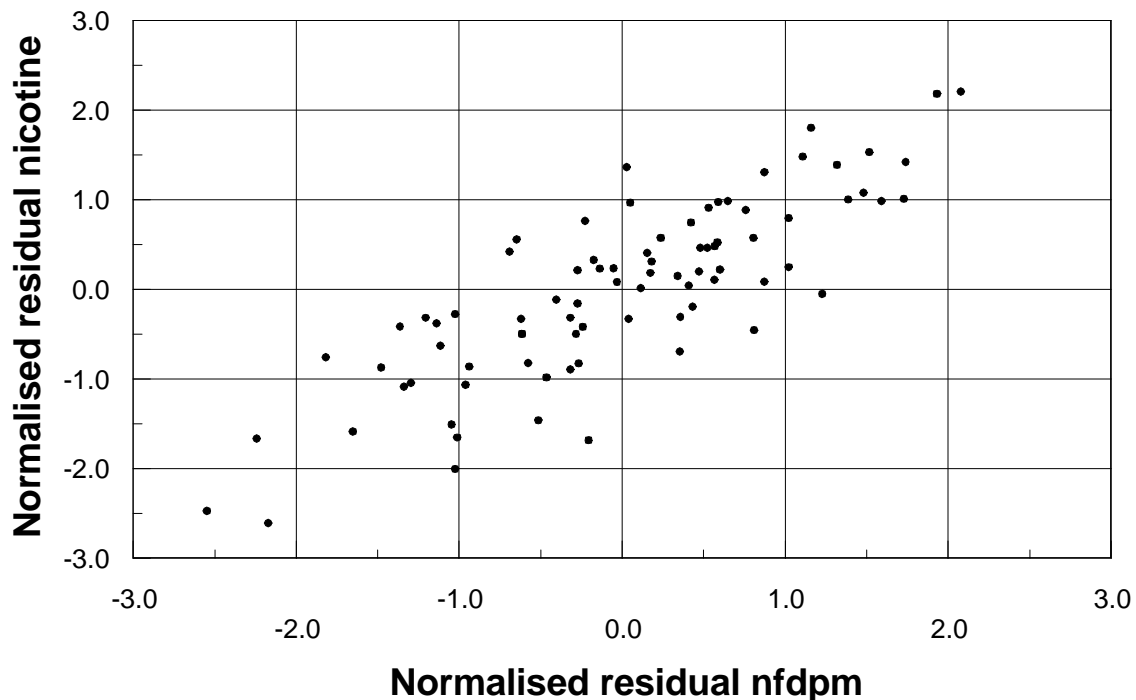
Holder E - 5.2 mm FCSAs SLEEVE 15mm insertion. Second pass



The dotted line is the 3.25 sd contour line

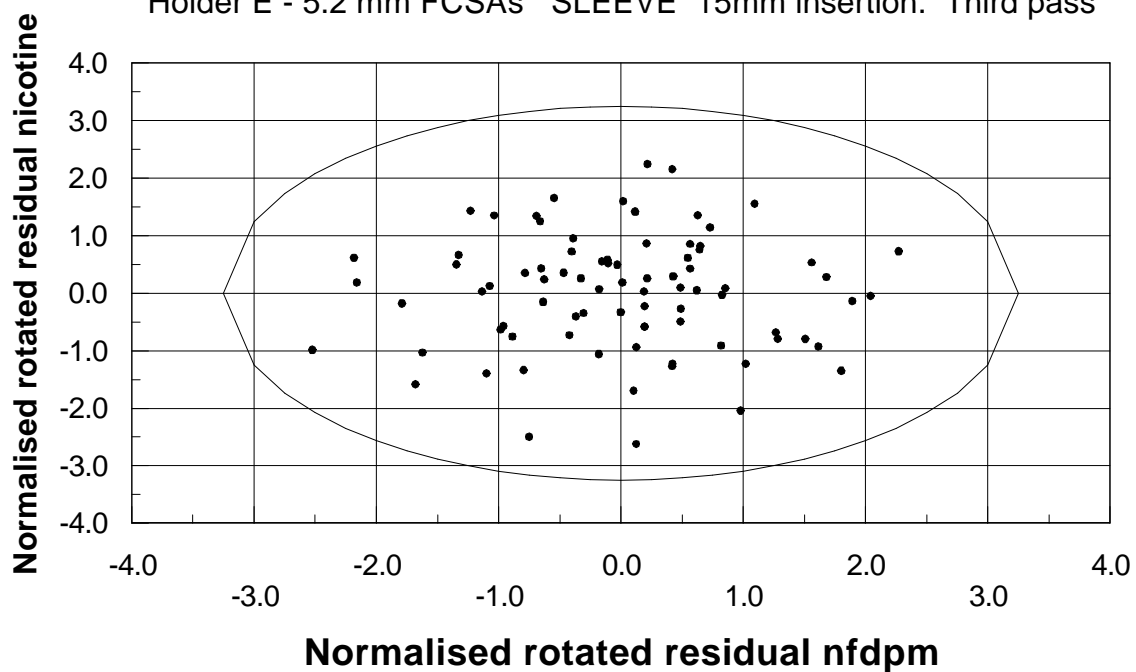
**Figure 5 Study 16. p-variate analysis**

Holder E - 5.2mm FCSAs SLEEVE 15mm insertion. Third pass



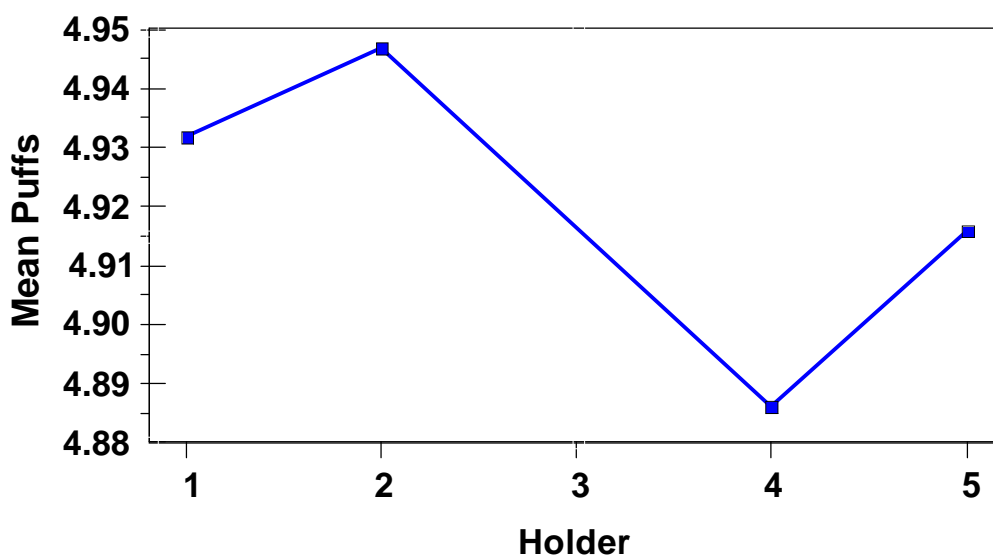
**Figure 6 Study 16. p-variate analysis**

Holder E - 5.2 mm FCSAs SLEEVE 15mm insertion. Third pass



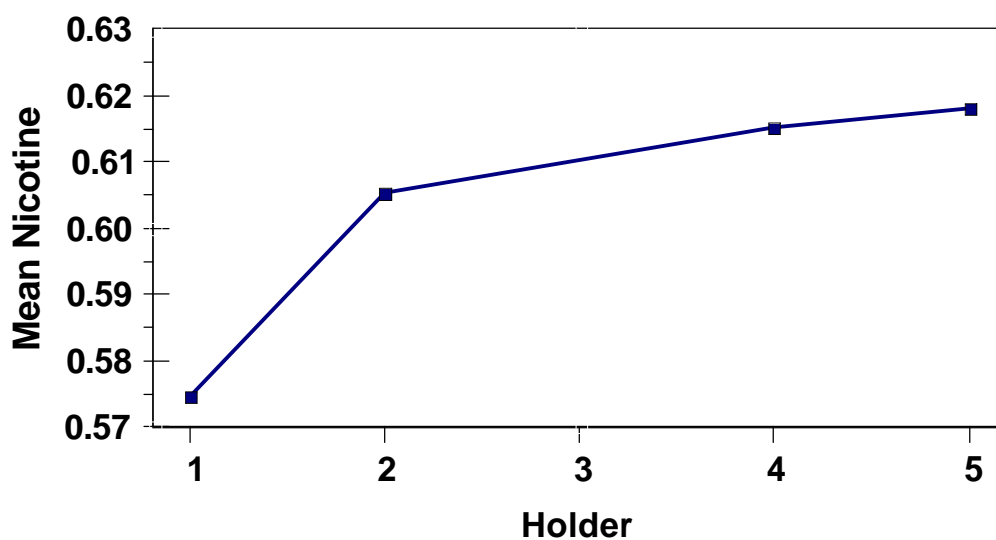
The dotted line is the 3.25 sd contour line

**Figure 7 Study 16. Mean Puff number. 5.2mm FCSAs  
Outliers removed. Laboratory 2 deleted**

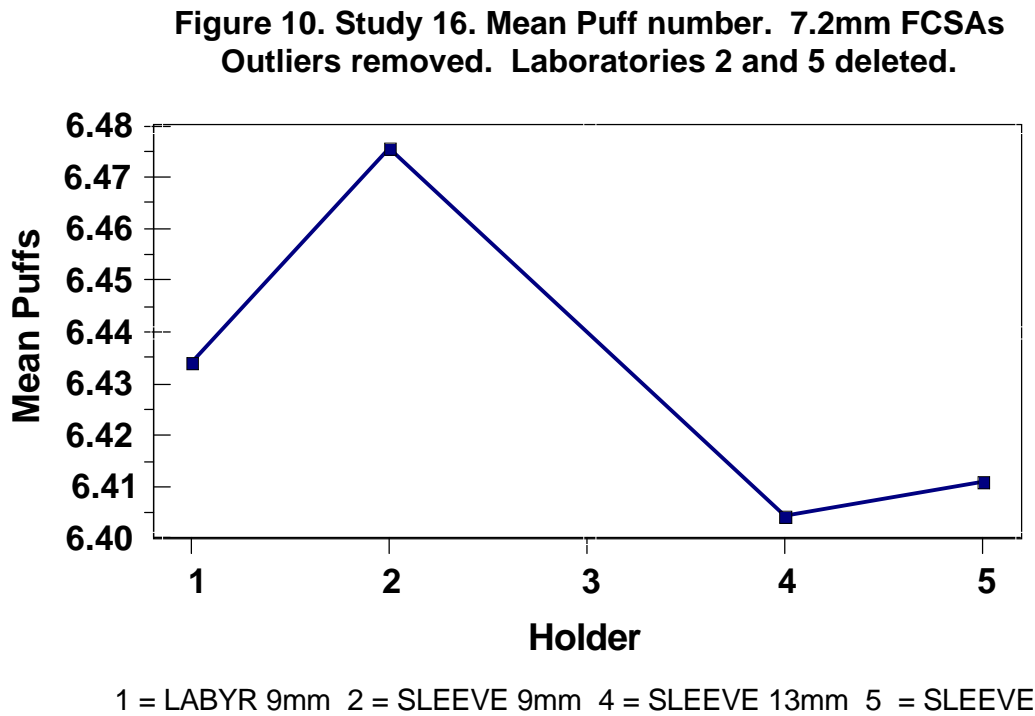
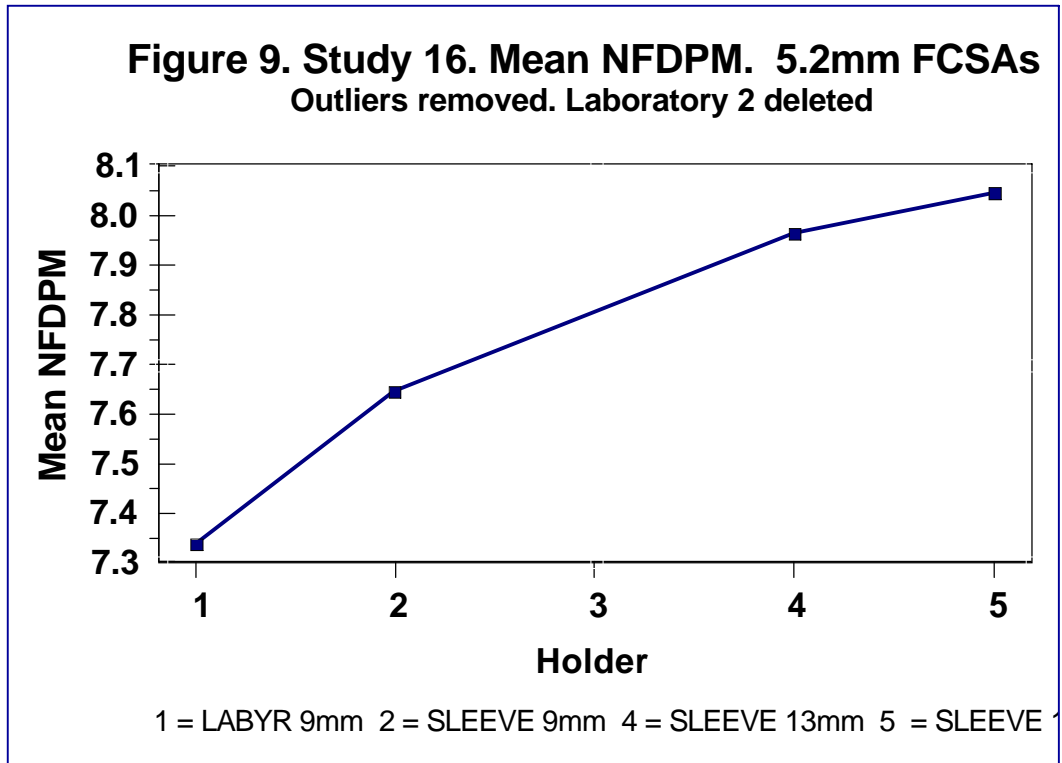


1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = CM 2

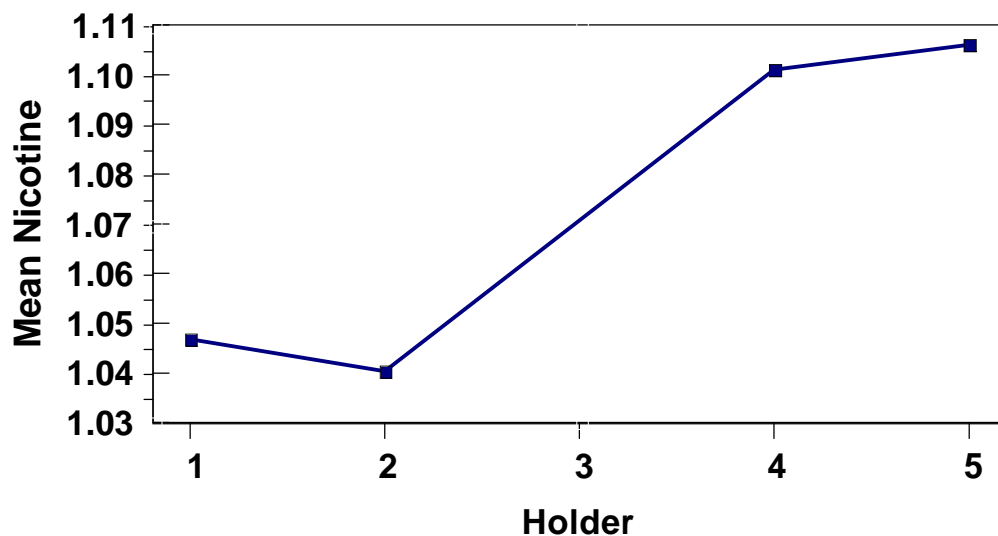
**Figure 8 Study 16. Mean Nicotine. 5.2mm FCSAs  
Outliers removed. Laboratory 2 deleted**



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

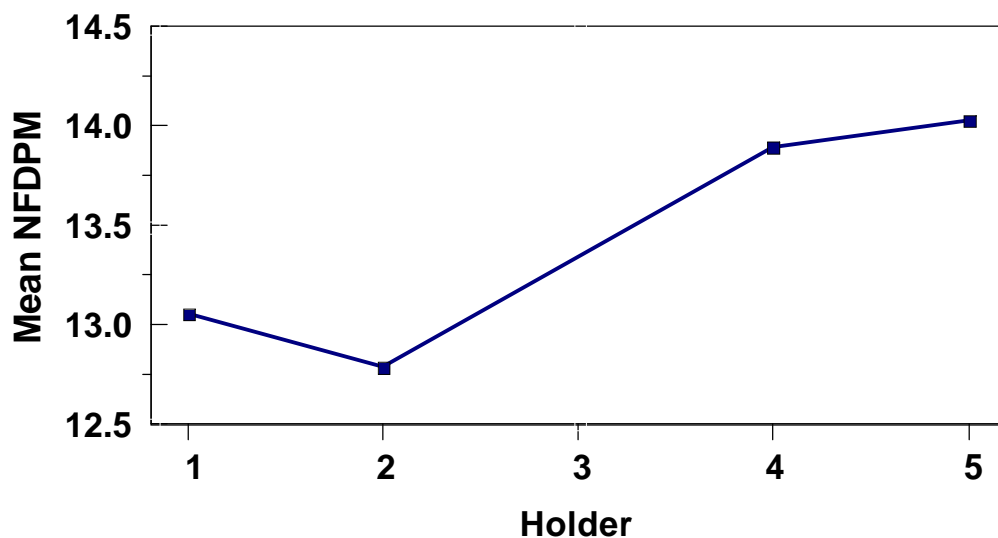


**Figure 11 Study 16. Mean Nicotine. 7.2mm FCSAs**  
Outliers removed. Laboratories 2 and 5 deleted.



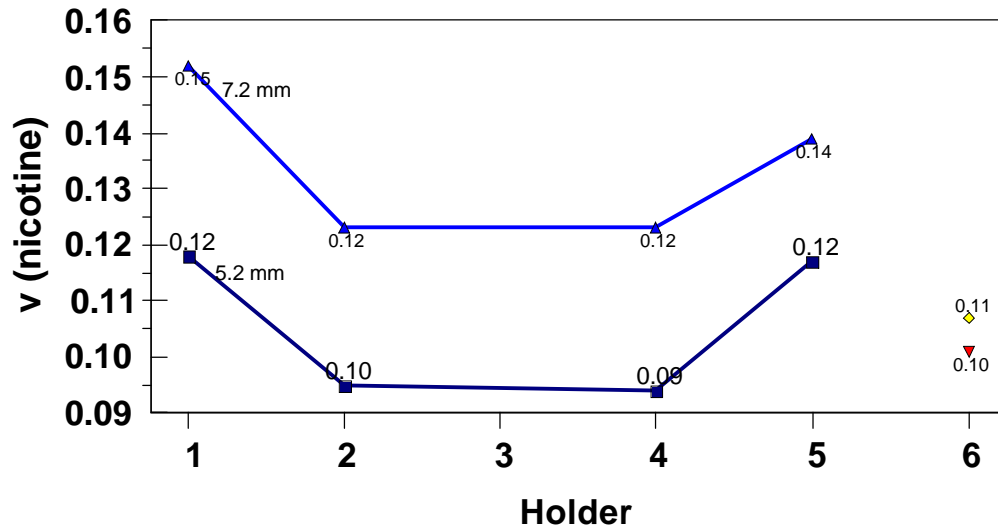
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15r

**Figure 12 Study 16. Mean NFDPM. 7.2mm FCSAs**  
Outliers removed. Laboratories 2 and 5 deleted.



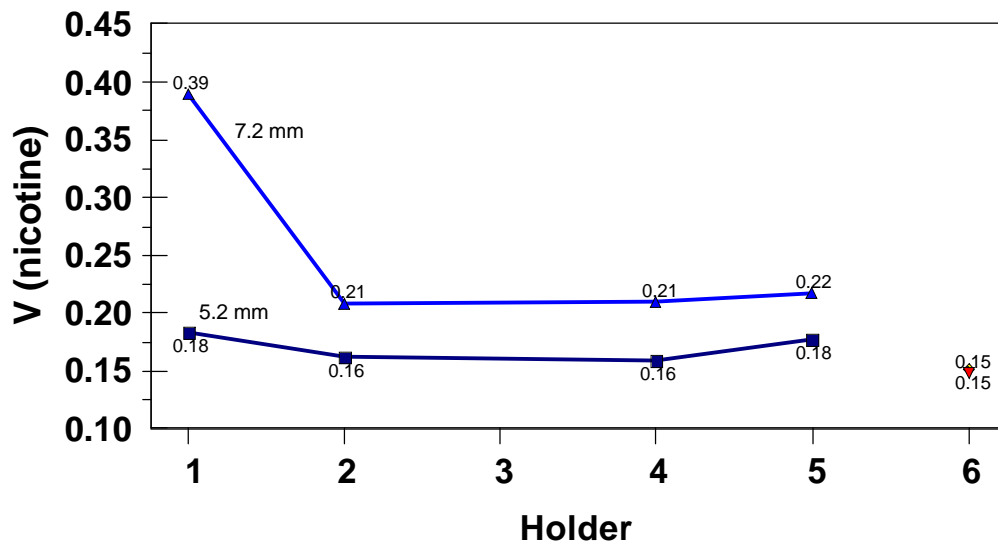
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15r

**Figure 13 Study 16. Plot of v for Nicotine**  
 Outliers removed. Outlying laboratories omitted.



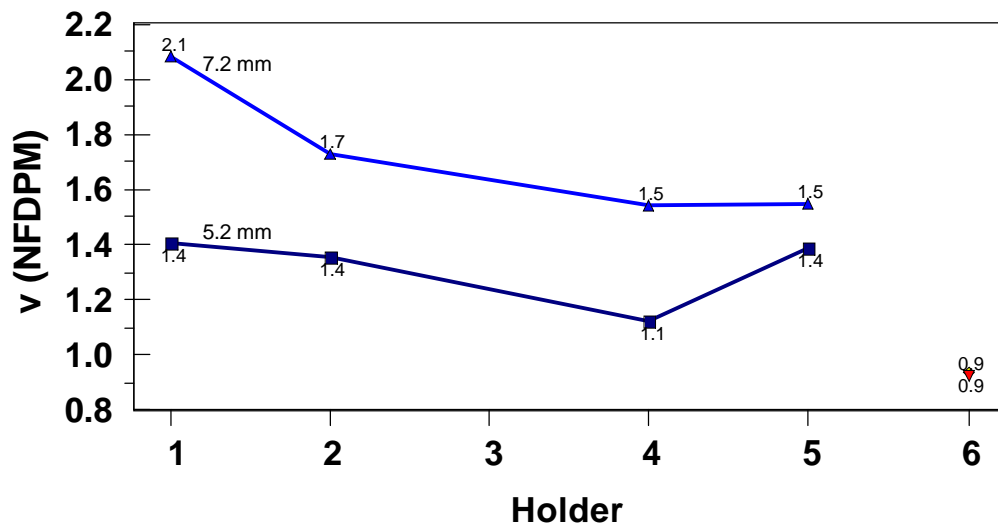
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 =

**Figure 14 Study 16. Plot of V for Nicotine**  
 Outliers removed. Outlying laboratories omitted.



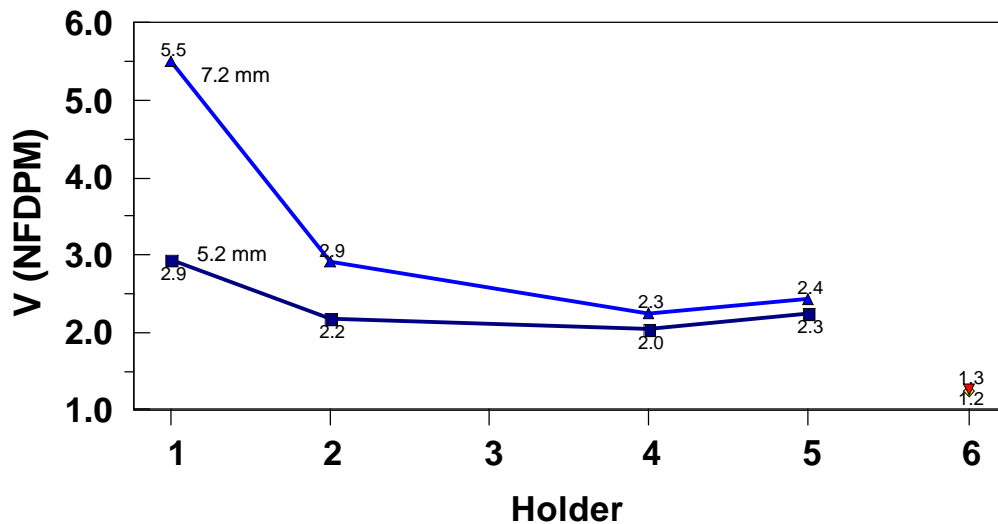
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CA

**Figure 15 Study 16. Plot of v for NFDPM  
Outliers removed. Outlying laboratories omitted.**



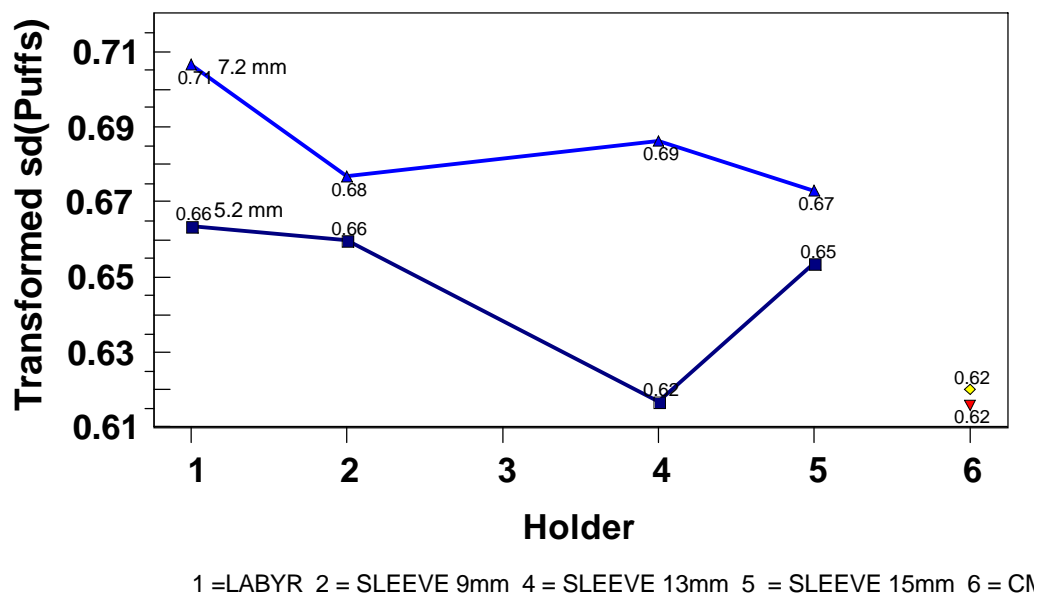
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CM

**Figure 16 Study 16. Plot of V for NFDPM  
Outliers removed. Outlying laboratories omitted.**

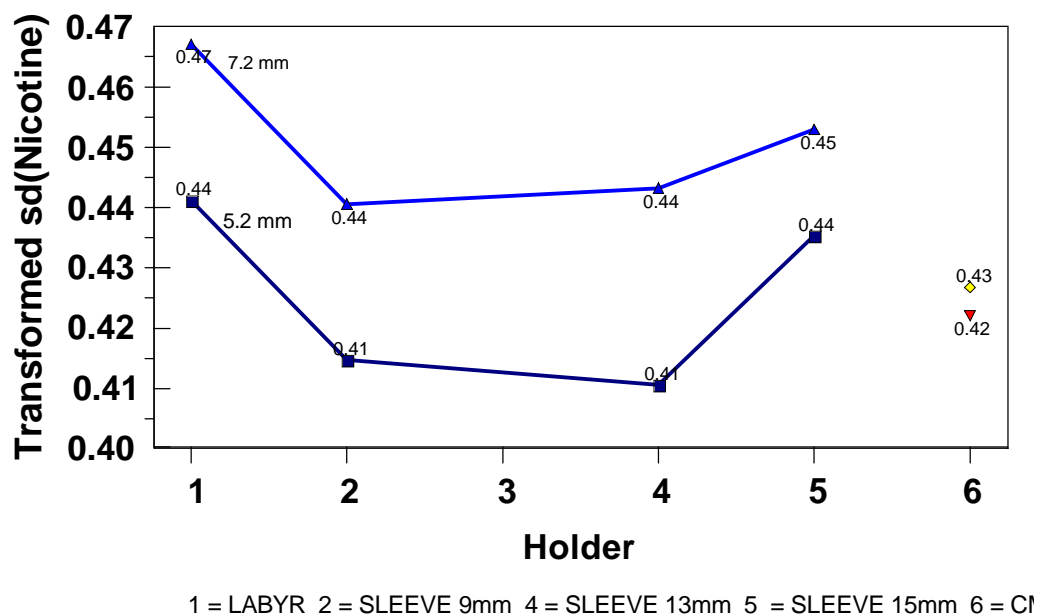


1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CM

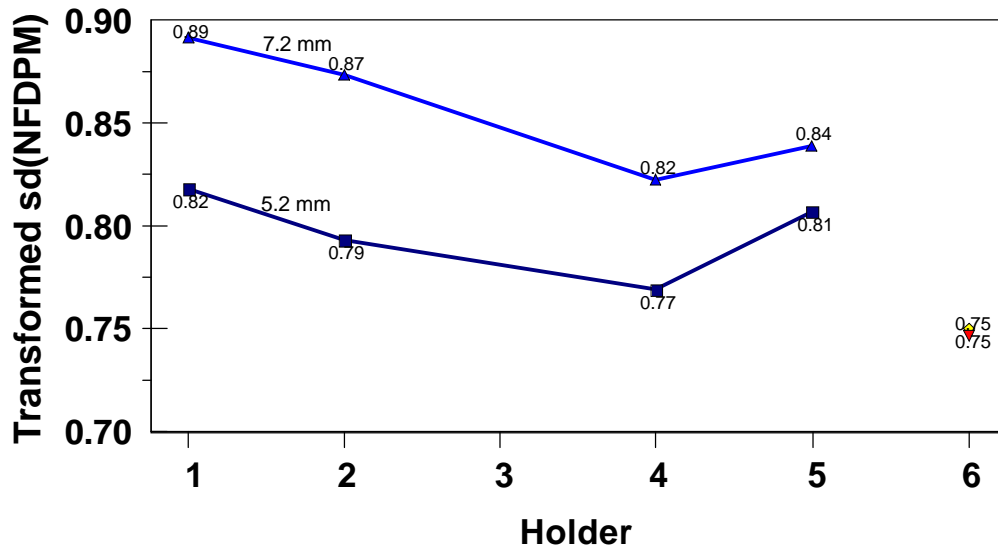
**Figure 17 Study 16. Transformed within-laboratory SDs. Puff number  
Outliers removed. Outlying laboratories omitted.**



**Figure 18 Study 16. Transformed within-laboratory SDs. Nicotine  
Outliers removed. Outlying laboratories omitted.**

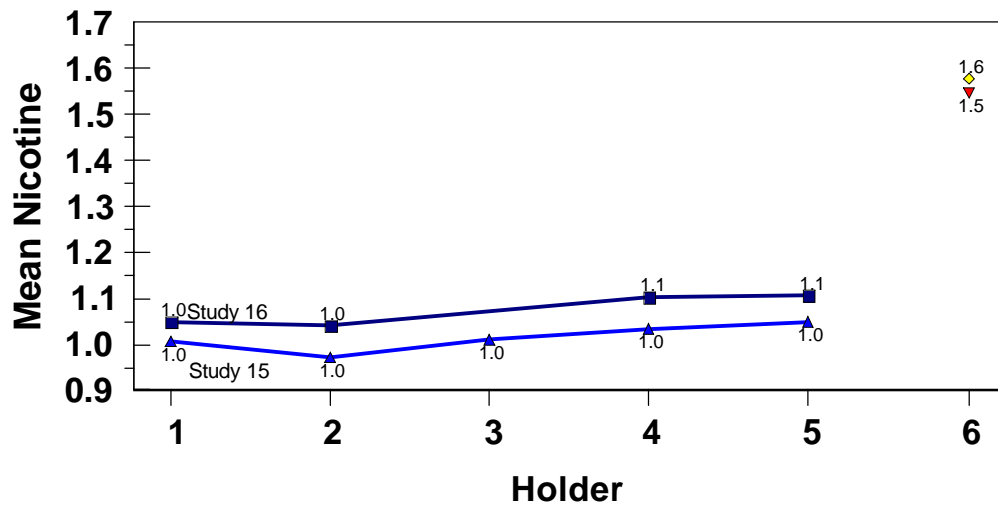


**Figure 19 Study 16. Transformed within-laboratory SDs**  
 Outliers removed. Outlying laboratories omitted.



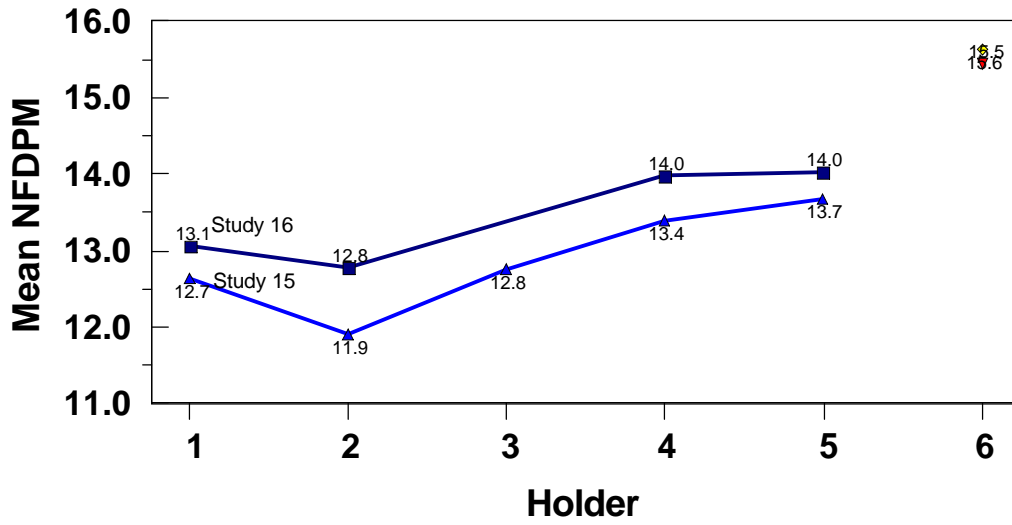
1 = LABYR 2 = SLEEVE 9mm 4 = SLEEVE 13mm 5 = SLEEVE 15mm 6 = CI

**Figure 20 Studies 15 and 16. Mean Nicotine**  
 7.2mm FCSAs Outliers removed. Laboratories 2 and 5 deleted.



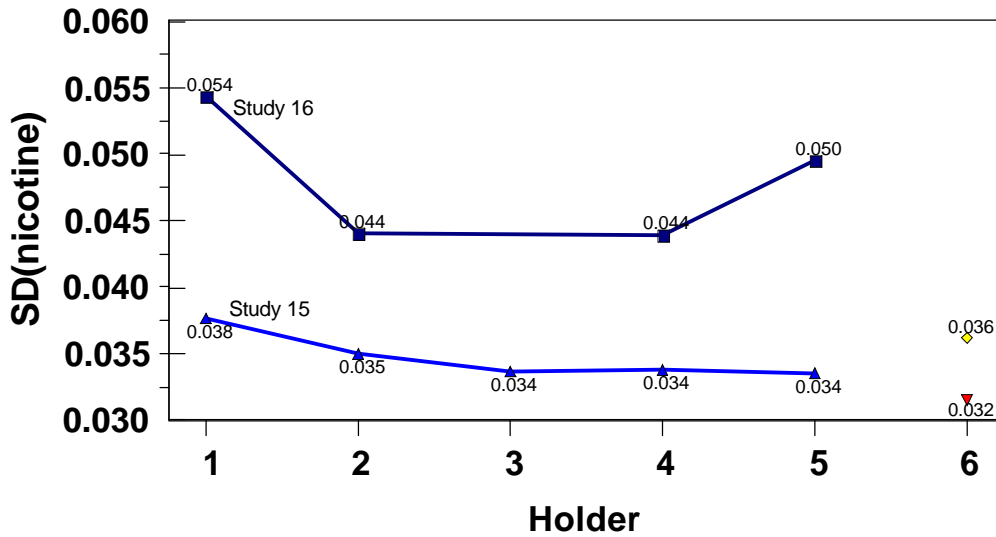
1 = LABYR 2 = SLEEVE 9mm 3 = 11mm SLEEVE 4 = SLEEVE 13mm 5 = SLEEV  
 6 = CM2

**Figure 21 Studies 15 and 16. Mean NFDPM**  
 7.2mm FCSAs Outliers removed. Laboratories 2 and 5 deleted.



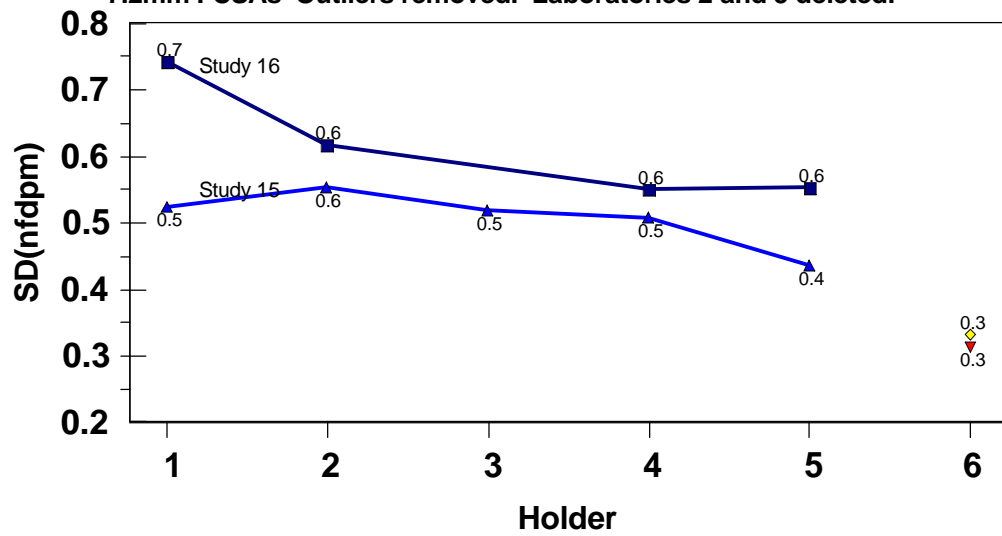
1 = LABYR 2 = SLEEVE 9mm 3 = 11mm SLEEVE 4 = SLEEVE 13mm 5 = SLEEVE 16mm  
 6 = CM2

**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 = SLEEVE 16mm  
 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 = SLEEVE 15mm  
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  $\pm 10$  mg and that individual articles are within  $\pm 20$  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  $\pm 20$  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  $\pm 10$  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.