Appendix 10. Study 13

Study of paper parameters

Executive Summary

The Objective of this study was to evaluate a range of fine cut smoking articles made from different wrappers for yield of NFDPM and nicotine and to determine whether one or more test procedures, such as combustibility or air permeability, can be used in a simple manner, to classify papers into two categories.

An improved protocol was devised for the application of the Louisville Combustibility Test (LCT), and a large number of laboratories performed this test and also determined air-permeability by the standard CORESTA method. This study was devised to investigate further, relationships between these paper parameters and smoke yields.

Seven papers were examined and two statisticians examined the data independently using regression analysis.

Regression analysis showed that, based on overall means of the data, neither of the single paper parameters can be used on its own to predict smoke yields but that there is a clear relationship between the smoke data (NFDPM and nicotine) and the paper data combined (Permeability and LCT). This does not mean that the relationship can be used to predict yield. Further analysis showed that, even for the laboratories that participated in this study, the prediction equations perform poorly. It would be expected that if these predictors were to be used by laboratories that did not participate in this study then their performance is likely to be much worse. The poor performance of the prediction equations is only to be expected since, at this time, there is also considerable variation between the laboratories for the determination of smoke yields. This will be a major impediment to the establishment of any predictor between smoke yields and paper measurements.

Since the data from one paper parameter alone is not sufficient to classify paper for constituent yield it must be concluded that the classification should be done using another procedure.

It is clear that a direct method based on the determination of smoke yields must be more reliable than an indirect method.

However, the statistical studies carried out in this report demonstrate that the LCT method has potential for use within the paper converting industry. It is therefore recommended that further work is undertaken to develop the LCT method so that it can be used as a quality control tool within the paper converting industry.

Objective

To evaluate a range of fine cut smoking articles made from different wrappers for yield of NFDPM and nicotine and to determine whether one or more test procedures can be used in a simple manner, to classify papers into two categories.

Background

CORESTA RYO Study 10 indicated that different papers used with fine-cut tobaccos gave rise to differing NFDPM and nicotine values. These differences appeared to be correlated with results from the Louisville Combustibility Test (LCT) suggesting that the LCT might form the basis for a procedure to categorise papers. However, it was noted that the range of papers for Study 10 had only given results at the extremes of the LCT range. As a result, due to this selection of papers, there was uncertainty about the outcome of this study. In addition, the variability of the data from the LCT gave rise to some concern.

In order to conduct this study a defined protocol was devised for the application of the LCT, as well as recruiting a large number of laboratories to carry out air-permeability by the standard CORESTA method. A copy of the protocol used for the LCT test is given in Appendix 1.

This study was devised to investigate further, relationships between paper parameters and smoke yields.

The nature and extent of changes made to the LCT test mean that results of Studies 10 & 13 cannot be combined.

Protocol

A copy of the Protocol as distributed to participants is attached as Appendix 2.

Study Design

The cigarette papers investigated are described below.

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

FCSAs were made using the Dutch blend of fine-cut tobacco previously used in Study 10. Eight laboratories were recruited to conduct smoking tests. Twelve laboratories were recruited to provide Louisville combustibility tests and ten recruited to provide air permeability at 0.25kPa and 1kPa. In all 16 laboratories were involved in some part of the analysis.

Statistical Methods:

Two statisticians examined the data independently.

Analysis by Statistician 1

To meet the objective given above, a stepwise multiple regression procedure was adopted. The variables inputted into the model were as follows:

(a) Dependent Variables

NFDPM

Nicotine

Puff Number

(b) Independent variables.

LCT	Louisville Combustibility Test data.
log(APlk)	where APIk is Air Permeability measured in CU per second when a pressure of 1 kPa is applied.
$\log\left(\frac{4 \times AP25}{AP1k}\right)$	where AP25 is Air Permeability measured in CU per second when a pressure of 025 kPa is applied.

For each paper the average LCT of the 12 participating laboratories was used, while for log(APlk) and $\log\left(\frac{4 \times AP25}{AP1k}\right)$ the average of eight laboratories who supplied results to the required precision were used.

CORESTA recommended method No. 40 includes a definition of linearity as applied to papers. By constructing $\log\left(\frac{4 \times AP25}{AP1k}\right)$ linear papers give values very close to zero. For clarity negative results were classified as zero, while positive values indicate the extent of non-linearity.

(c) Dummy Variables

In order to retain individual laboratory smoking data in the analysis, the following dummy variables were introduced:

FIL - Returns a value 1 if the participating laboratory uses a 20-channel linear smoking machine and 0 otherwise.
LAB2 - Returns a value 1 if Lab 2 supplied the smoke results and 0 otherwise.
LAB4 - Returns a value 1 if Lab 4 supplied the smoke results and 0 otherwise.
LAB6 - Returns a value 1 if Lab 6 supplied the smoke results and 0 otherwise.
LAB8 - Returns a value 1 if Lab 8 supplied the smoke results and 0 otherwise.
LAB11 - Returns a value 1 if Laboratory 11 supplied the smoke results and 0 otherwise.
LAB12 - Returns a value 1 if Laboratory 12 supplied the smoke results and 0 otherwise.

Two laboratories failed to report air-permeability to the required precision (0.5 CU). These laboratories were excluded from the averages of the two variables involving air permeability.

The remainder of the data were screened for outlier behaviour and after running some diagnostic checks, with the exception of one single value for the UK paper under the LCT, no further data were removed.

This analysis resulted in regressions for NFDPM, nicotine and puff number, fitted on 56 observations, which are presented below:

NFDPM = 11.47 + 0.0887*LCT + 4.113*FIL - 0.868*log(AP1K)+ 3.875*LAB6 + 1.623*LAB8 - 1.017*LAB11
$R^2 = 0.87$

Nicotine = 0.979 + 0.0058*LCT + 0.342*FIL - 0.0512*log(AP1K) + 0.342*LAB6 + 0.20*LAB8 - 0.129*LAB12
$R^2 = 0.86$

Puffs = 9.46 - 0.79*log(AP1K) + 5.61*log(4*AP25/AP1K) - 0.47*LAB2- 0.21*LAB6 - 0.52*LAB8 - 0.25*LAB12
$R^2 = 0.89$

The equations generated above can be used to ‘predict’ the outcome for NFDPM, nicotine and puff number for a combination of paper parameters. The predictions for NFDPM and nicotine are shown in the following table.

Table 1.
Predicted Values for NFDPM and Nicotine by LCT and Air Permeability

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

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

It will be noted that $\log\left(\frac{4\times AP25}{AP1k}\right)$ did not appear in the regression equations for NFDPM and nicotine, but did for puffs. This indicates that for the papers used in this study, non-linearity is only a factor with respect to puff number.

The need for a second paper parameter to predict constituent yield is demonstrated as follows (considering NFDPM only):

Given the ability of a system based on two parameters to predict NFDPM or nicotine, how much worse do the predictions become if one parameter, e.g. air permeability, is ignored. This is answered by running the regression for NFDPM with data on air permeability omitted. The average residual (i.e. the difference between what a model predicts and what an individual laboratory determines) is calculated for each paper in both situations. If residual values for any paper increase consistently in one direction then there is evidence of lack of fit which would be overcome by retaining the additional term. The model for NFDPM thus obtained is:

NFDPM = 6.54 + 0.124*LCT + 4.113*FIL + 3.875*LAB6 + 1.623*LAB8 - 1.017*LAB11
$R^2 = 0.80$

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

Table 2.
Average residuals for NFDPM under 2 models

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

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

Having established that more than one parameter is required it is now important to consider the reliability of the individual data.

The data given above are based on composite values from all participating laboratories. If a single laboratory was to classify papers on the basis of results of these tests, it would be necessary for the data to be repeatable within a laboratory, but more importantly, reproducible across laboratories.

The following tables illustrate the values for r and R for the LCT and for Air Permeability determined at 1 kPa.

Table 3.
Repeatability and Reproducibility values for the LCT test

Paper Type	Mean (sec)	r	R
Wat 3	56.9	7.0	20.9
Wat 4	49.7	7.6	18.5
Wat 5	53.4	6.0	13.6
D	45.0	7.0	11.7
NL	52.4	8.2	12.9
UK	76.7	12.7	21.4
CND	69.7	10.1	21.6

1 Outlier value removed from the UK paper.

Table 4.
Repeatability and Reproducibility values for the Air Permeability test

Paper Type	Mean (CU)	r	R
Wat 3	19.2	2.4	3.4
Wat 4	12.7	1.3	5.6
Wat 5	112.5	15.0	21.5
D	40.3	6.4	7.9
NL	64.2	12.2	16.8
UK	15.1	9.8	11.3
CND	10.8	3.1	4.0

Analysis by Statistician 2

The first step in the analysis was to examine the individual data for outliers.

<u>OUTLIERS - AIR PERMEABILITY DATA MEASURED AT 1 kPa</u>

The data from laboratory 13 presented problems. Each data point was reported to the nearest 5 units, whereas, for nearly all of the other laboratories, each data point was reported to the nearest 0.1 unit. (The only exception was laboratory 11 which reported it's data to the nearest 1 unit.) The air permeability data from laboratory 13 were therefore omitted from the analysis.

For each paper, a one-way analysis of variance between laboratories was used to calculate the standardised residual for each data point. The standardised residuals were also plotted against their normalised scores. Any data point with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure, eleven data points were deemed to be outliers and deleted from all subsequent analysis.

Having deleted the outlying data points, plots of laboratory means for paper WAT4 tended to indicate that laboratory 2 was an outlier and the Grubbs test confirmed this.

No other laboratory was indicated as a possible outlier by these plots.

Having deleted the outlying data points, and the data for paper WAT4 from laboratory 2, the means for each paper from each laboratory were calculated, as shown in Table 5.

Table 5
Mean permeability (CU) for each paper measured at 1 kPa

LAB	WAT3-1 Mean	WAT4-1 Mean	WAT5-1 Mean	D-1 Mean	NL-1 Mean	UK-1 Mean	CND-1 Mean
2	20.54	—	119.14	42.85	66.12	17.43	12.42
5	19.55	12.97	112.00	40.54	62.95	19.09	11.11
9	18.21	11.28	105.78	38.56	61.32	12.20	9.95
10	18.10	11.00	113.40	39.70	68.00	13.40	10.20
11	19.80	12.29	107.40	41.35	61.42	14.88	10.56
14	19.14	12.39	107.40	38.67	63.39	13.13	12.47
15	20.11	13.26	112.58	41.97	64.29	14.33	11.82
17	18.96	12.35	121.79	39.79	72.44	12.34	10.24
18	19.80	12.52	103.30	31.30	53.30	14.54	11.18
19	18.45	11.65	105.65	37.30	57.75	14.25	9.90
ALL	19.27	12.19	110.71	39.20	62.93	14.58	10.98

OUTLIERS - AIR PERMEABILITY DATA MEASURED AT 0.25 kPa

For each paper, the ratio of the air permeability measurements at 1 kPa and 0.25 kPa was calculated (after the removal of any outliers in the 1 kPa data). A one-way analysis of variance between laboratories was used to calculate the standardised residual for each ratio. The standardised residuals were also plotted against their normalised scores. Any ratio with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure 18 ratios were deemed to be outliers.

These data points for air permeability measured at 0.25 kPa were deleted from all subsequent analyses.

Plots of laboratory means for paper WAT5 tended to indicate that the mean ratio from laboratories 17 and 18 were outliers and the Grubbs test confirmed this. The measurements taken at 0.25 kPa for this paper by these two laboratories were therefore rejected.

Plots of laboratory means for paper D tended to indicate that the mean ratio from laboratory 18 was an outliers and the Grubbs test confirmed this. The measurements taken at 0.25 kPa for this paper by this laboratory was therefore rejected.

No other laboratory was indicated as a possible outlier by these plots.

Having deleted the outlying data as above, the mean ratio of the measurements taken at 1 kPa and 0.25 kPa for each paper were calculated. Results are shown in Table 6.

Table 6
Mean air permeability ratios

Lab.	WAT3-R Mean	WAT4-R Mean	WAT5-R Mean	D-R Mean	NL-R Mean	UK-R Mean	CND-R Mean
2	4.351	4.785	2.915	3.987	2.773	2.977	3.555
5	3.957	4.262	2.934	3.958	2.878	2.848	3.902
9	3.901	3.973	2.884	3.885	2.782	2.978	3.795
10	4.525	5.500	2.879	4.182	2.895	3.400	4.204
11	4.074	4.210	2.935	4.016	2.882	3.031	3.927
14	—	—	2.896	4.068	2.988	—	—
15	3.901	4.068	2.999	4.044	2.909	3.204	3.859
17	4.116	4.324	—	4.054	2.979	3.475	4.117
18	4.061	—	—	—	—	3.054	—
19	3.969	3.900	2.927	3.927	2.881	2.936	3.914
ALL	4.0970	4.3635	2.9207	4.0134	2.8776	3.0676	3.9053
	Linear	Linear	Non-Linear	Linear	Non-Linear	Non-Linear	Linear

In table 6, after consideration of the individual laboratory data, each paper is graded as being LINEAR or NON-L(inear).

OUTLIERS - LOUISVILLE COMBUSTION TEST

For each paper, a one-way analysis of variance between laboratories was used to calculate the standardised residual for each data point. The standardised residuals were also plotted against their normalised scores. Any ratio with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure, 17 data points were deemed to be outliers and were deleted from all subsequent analyses.

Plots of laboratory means for paper WAT3 tended to indicate that laboratories 9 and 13 were outliers and the Grubbs test confirmed this.

No other laboratory was indicated as a possible outlier by these plots.

Having deleted the outlying data, means for each paper from each laboratory were calculated, as shown in Table 7.

Table 7
Mean LCT data for each paper (sec)

Lab	WAT3 Mean	WAT4 Mean	WAT5 Mean	D Mean	NL Mean	UK Mean	CND Mean
2	59.22	52.03	53.25	45.58	52.24	81.85	68.59
5	62.78	52.68	54.66	48.54	54.09	76.45	70.54
6	62.68	57.82	60.40	44.39	57.22	76.66	71.21
7	55.40	48.50	53.60	42.00	50.70	78.30	69.80
9	—	34.22	48.57	48.77	47.10	70.51	84.55
10	57.67	51.22	56.00	44.20	53.50	88.00	72.33
11	55.80	47.80	52.67	41.80	48.20	74.60	61.20
13	—	43.96	44.09	38.84	47.72	63.80	56.54
14	58.54	51.72	53.08	45.64	56.72	82.27	71.00
15	64.50	55.90	59.20	51.50	58.70	84.00	76.40
18	60.30	52.80	55.50	44.80	54.10	76.50	70.00
19	58.60	48.80	51.40	44.30	50.10	73.56	66.70
ALL	59.57	50.04	53.52	45.03	52.33	77.16	69.87

OUTLIERS - SMOKE DATA

A two-way analysis of variance was used to calculate the standardised residual for each data point for NFDPM, nicotine and puffs. The standardised residuals were also plotted against their normalised scores. Any data point with a standardised residual greater than 2.0 and which showed a marked deviation from the expected straight line plot of normal scores was deemed to be an outlier. Using this procedure 2, 5 and 7 data points were deemed to be outliers for NFDPM, nicotine and puffs respectively.

These data were deleted from all subsequent analyses and the means of the smoke data for each paper from each laboratory were calculated, as shown in Table 8.

Table 8
A. Means for PUFFS by paper and laboratory

LAB	CND	D	NL	UK	WAT3	WAT4	WAT5
2	7.78	7.04	7.96	8.86	7.32	7.48	7.16
5	7.26	6.34	7.70	8.86	6.94	7.00	7.14
6	7.42	6.94	7.72	9.00	6.98	7.66	7.56
7	7.28	6.30	7.46	8.38	6.68	6.82	6.78
9	7.44	6.54	7.96	8.82	6.80	7.20	7.28
10	7.85	6.72	8.18	9.18	6.96	7.54	7.38
11	7.06	6.34	7.44	8.63	6.58	6.66	6.62
13	7.27	6.73	7.78	8.69	6.74	6.75	7.35
ALL	7.409	6.619	7.766	8.805	6.876	7.139	7.159

B. Means for NFDPM by Paper and Laboratory

LAB	CND	D	NL	UK	WAT3	WAT4	WAT5
2	14.54	13.25	13.44	16.01	14.77	13.24	12.19
5	19.91	16.12	17.03	19.84	18.26	18.77	16.37
6	15.66	12.26	14.28	15.76	13.78	11.50	9.50
7	15.71	13.55	13.91	16.29	14.20	13.15	10.78
9	18.56	16.10	16.04	20.22	18.56	17.28	15.64
10	18.38	14.68	14.70	20.40	16.90	16.94	14.96
11	16.42	15.05	14.36	16.40	15.36	15.75	13.95
13	19.70	15.85	17.08	20.98	17.99	16.69	14.77
ALL	17.33	14.61	15.10	18.24	16.23	15.42	13.45

C. Means for Nicotine by Paper and Laboratory

LAB	CND	D	NL	UK	WAT3	WAT4	WAT5
2	1.226	1.122	1.150	1.272	1.252	1.170	1.108
5	1.458	1.184	1.304	1.548	1.354	1.378	1.318
6	1.286	1.054	1.168	1.270	1.186	1.004	0.878
7	1.182	1.112	1.134	1.246	1.176	1.088	0.912
9	1.520	1.344	1.362	1.664	1.524	1.462	1.378
10	1.660	1.310	1.380	1.766	1.498	1.514	1.420
11	1.474	1.309	1.278	1.358	1.365	1.389	1.264
13	1.508	1.273	1.324	1.553	1.344	1.296	1.184
ALL	1.413	1.212	1.262	1.452	1.337	1.288	1.183

REGRESSION ANALYSIS

Tables 5 to 8 provided the data for the regression analysis of the smoke data. The overall means of NFDPM and nicotine were regressed against the overall means of PERM, LCT, and against the indicator variable TYPE. The indicator variable TYPE was set equal to 1 for LINEAR papers and 2 for NON-L(inear) papers. The results of the regressions, based on seven data points follow.

For NFDPM the equation is

NFDPM = 11.6 - 0.0300 PERM + 0.594 TYPE + 0.0772 LCT (R^2 = 98%)

The analysis also shows that TYPE in non-significant; using PERM and LCT only as predictors the regression equation is

NFDPM = 11.2 - 0.0226 PERM + 0.0935 LCT (R^2 = 97%)

For smoke nicotine the regression equation is

Nicotine = 0.987 - 0.00120 PERM + 0.00628 LCT (R^2 = 98%)

These regressions show that, based on overall means of the data, neither of the single paper parameters can be used on its own but that there is a clear relationship between the smoke data (NFDPM and nicotine) and the paper data combined (PERM and LCT).

It is tempting to believe that the regression equations derived from these data could be used as general prediction equations for smoke yield if the values of PERM and LCT were known for any paper. This is most unlikely to be the case.

Tables 9 and 10 show the results of using these regressions to predict the NFDPM and nicotine data from the paper measurements for the laboratories that participated in this study.

Table 9 shows that, for laboratory 2, the predicted value is always higher than the observed value, whereas for laboratory 5 the reverse is the case. Laboratory 9 shows wide variations in the ERROR, from -5.3 to 3.1. The NFDPM values from laboratories 10 and 11 come somewhat closer to prediction.

Table 9
Prediction of NFDPM (mg)

LAB	PAPER	PERM	LCT	NFDPM Observed	NFDPM Predicted	ERROR (Obs-Pred)
2	CND	12.425	68.590	14.358	17.329	-2.971
2	D	42.848	45.580	13.250	14.490	-1.240
2	NL	66.117	52.240	13.444	14.586	-1.142
2	UK	17.427	81.850	16.026	18.456	-2.430
2	WAT3	20.545	59.220	14.772	16.270	-1.498
2	WAT4	*	52.030	13.238	*	*
2	WAT5	119.140	53.250	12.194	13.480	-1.286
5	CND	11.111	70.541	19.906	17.541	2.365
5	D	40.540	48.544	16.116	14.819	1.297
5	NL	62.950	54.088	17.032	14.830	2.202
5	UK	19.090	76.446	19.836	17.913	1.923
5	WAT3	19.550	62.875	18.262	16.634	1.628
5	WAT4	12.970	52.685	18.770	15.830	2.940
5	WAT5	112.000	54.657	16.370	13.773	2.597
9	CND	9.950	84.550	18.560	18.877	-0.317
9	D	38.560	48.770	16.100	14.885	1.215
9	NL	61.320	47.100	16.040	14.214	1.826
9	UK	12.200	70.510	12.220	17.514	-5.294
9	WAT3	18.210	*	18.560	*	*
9	WAT4	11.280	34.225	17.280	14.143	3.137
9	WAT5	105.780	48.567	15.640	13.344	2.295
10	CND	10.200	72.333	18.375	17.730	0.645
10	D	39.700	44.200	14.680	14.432	0.248
10	NL	68.000	53.500	14.700	14.661	0.039
10	UK	13.400	88.000	20.400	19.122	1.278
10	WAT3	18.100	57.667	16.900	16.180	0.720
10	WAT4	11.000	51.222	16.940	15.738	1.202
10	WAT5	113.400	56.000	14.960	13.867	1.093
11	CND	10.560	61.200	16.422	16.681	-0.259
11	D	41.350	41.800	15.054	14.170	0.883
11	NL	61.425	48.200	14.358	14.314	0.044
11	UK	14.878	74.600	16.400	17.836	-1.436
11	WAT3	19.800	55.800	15.362	15.967	-0.605
11	WAT4	12.290	47.800	15.746	15.389	0.357
11	WAT5	107.400	52.667	13.952	13.691	0.261

Table 10 shows that, for laboratory 2, the predicted value is always less than the observed value, whereas for laboratories 9 and 10 the reverse is the case. For laboratory 5 only 2 values of ERROR are negative, and for laboratory 11 only 1 value of ERROR is negative.

Table 10
Prediction of Nicotine (mg)

LAB	PAPER	PERM	LCT	Nicotine Observed	Nicotine Predicted	ERROR (Obs-Pred)
2	CND	12.425	68.590	1.226	1.403	-0.177
2	D	42.848	45.580	1.122	1.222	-0.100
2	NL	66.117	52.240	1.150	1.236	-0.086
2	UK	17.427	81.850	1.272	1.481	-0.209
2	WAT3	20.545	59.220	1.252	1.335	-0.083
2	WAT4	*	52.030	1.170	*	*
2	WAT5	119.140	53.250	1.108	1.179	-0.071
5	CND	11.111	70.541	1.458	1.417	0.041
5	D	40.540	48.544	1.184	1.244	-0.060
5	NL	62.950	54.088	1.304	1.252	0.052
5	UK	19.090	76.446	1.548	1.445	0.103
5	WAT3	19.550	62.875	1.354	1.359	-0.005
5	WAT4	12.970	52.685	1.378	1.303	0.075
5	WAT5	112.000	54.657	1.318	1.196	0.122
9	CND	9.950	84.550	1.520	1.506	0.014
9	D	38.560	48.770	1.344	1.247	0.097
9	NL	61.320	47.100	1.362	1.210	0.152
9	UK	12.200	70.510	1.664	1.416	0.248
9	WAT3	18.210	*	1.524	*	*
9	WAT4	11.280	34.225	1.462	1.189	0.273
9	WAT5	105.780	48.567	1.378	1.166	0.212
10	CND	10.200	72.333	1.660	1.429	0.231
10	D	39.700	44.200	1.310	1.217	0.093
10	NL	68.000	53.500	1.380	1.242	0.138
10	UK	13.400	88.000	1.766	1.524	0.242
10	WAT3	18.100	57.667	1.498	1.328	0.170
10	WAT4	11.000	51.222	1.514	1.296	0.218
10	WAT5	113.400	56.000	1.420	1.203	0.217
11	CND	10.560	61.200	1.474	1.359	0.115
11	D	41.350	41.800	1.309	1.200	0.109
11	NL	61.425	48.200	1.278	1.216	0.062
11	UK	14.878	74.600	1.358	1.438	-0.080
11	WAT3	19.800	55.800	1.365	1.314	0.051
11	WAT4	12.290	47.800	1.389	1.273	0.116
11	WAT5	107.400	52.667	1.264	1.189	0.075

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

The poor performance of the prediction equations is only to be expected. Examination of tables to 8 shows that there is considerable variation between the laboratories for both the paper measurements and the determination of smoke yields.

The data was reanalysed excluding the Dutch and Wat 5 papers. These two papers are clearly perforated and it is important to know whether the LCT could be used as a predictor for NFDPM for the non-perforated papers examined in this study.

Conclusions

It is acknowledged that FCSAs are intrinsically more variable than commercially manufactured cigarettes. Nonetheless, the variability of the data reported in this study, especially as reflected in the high between laboratory variation, must be a major impediment to the establishment of any predictor between smoke yields and paper measurements.

Since the data from one test alone is not sufficient to classify paper for constituent yield it must be concluded that the classification should be done using another procedure. It is clear that a direct method based on the determination of smoke yields must be more reliable than an indirect method.

However, it is also apparent that, on the basis of this test, the LCT is a useful predictor of combustibility, and therefore yields, for the type of non-perforated papers examined in this study. A procedure based on the LCT may have value within the paper industry as a tool for testing such papers.

The data shown in Table 3 indicates that the LCT is a basis on which to develop further improvements for the testing of paper combustibility. Further improvements to this test method are likely to provide less variable data.

Appendix 1

Paper Classification (A: LCT & B: Air Permeability)

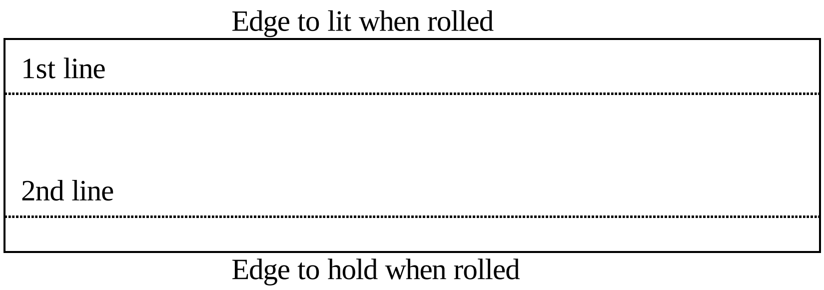
A) The Louisville Combustibility Test

Principle of the test:

Two lines are marked on a strip of paper 15 mm apart. The paper length is cut to 60 mm and rolled around a needle. The needle is removed and the paper clamped at one end. The paper is ignited and the time to smoulder between the two lines is a measure of the combustibility. The determination of the burn-time is conducted within a specially constructed box to prevent the influence of air-movements.

Figure 1

Direction of rolling →



Construction of the box:

The box should be constructed from Plexiglas (if necessary using a wooden or metal frame) and measure approximately: 50 cm wide x 40 cm deep by 30 cm high including any framing (see figure 2).

The front of the box should have a hole cut in it, large enough to allow the operator to insert the strip (diameter approximately 12 cm).

The centre of the hole should be placed about 12 cm from the left hand edge and 12 cm from the top of the box.

The top of the box should have a hole to allow the passage of air (diameter 2.5 cm). The centre of the hole should be placed about 12 cm from the right hand edge and 20 cm from the front of the box.

Within the box, construct a stand to hold the clamped paper strip (see procedure). This should be placed such that the lit end of the paper roll is exactly in the middle of the floor area of the box with the remainder of the roll facing the right hand side (see figure 3).

The box should be placed in a normal fume hood but there should be no air flow in the hood during the time of determination. The fume hood should be switched on to allow exhaust fumes to be removed after the determination.

PROCEDURE:

Take 10 strips of the cigarette-paper, each 60 mm length. The wire side is taken to be inside (is marked on the strips). On the "outside" of each paper-strip, draw a pencil line 5 mm from one edge, draw a second line 15 mm away from the first line. With the pencil-lines on the outside, wind a single strip round a steel needle, 2.5 mm in diameter, to form a small roll (the needles will be supplied). Remove the roll from the needle and hold it tightly using a "fold-back" paper clamp (will be supplied) at the end furthest from the pencil lines.

With the clamp, hold the roll and ignite the cigarette paper (by using non-flame ignition, such as a hot surface or an electric lighter). Still holding the clamp, transfer the ignited paper to the inside of the box, placing it accurately in position on the stand. Ensure the strip is held perfectly horizontally and measure the time taken for it to burn between the pencil marks, using a stop clock. Carry out the determination on each of the 10 strips.

Report the burning time in seconds for each of the 10 strips.

B) Air Permeability

The air permeability will be determined according to CORESTA recommended methods No. 40. In particular section 7.5 will be followed and any non linearity will be determined according to note 1. The air permeability will be measured on at least 10 strips and the individual data reported.

Appendix 2

Protocol for study 13

0. INTRODUCTION

0.1 This second experiment will test the viability of a possible matrix approach for the declaration of tar/nicotine yields of FCSAs.

0.2 This study is set up to investigate the relation between the LCT values of different papers and the tar/nicotine deliveries.

Tubes made of 7 commercially available papers with diameters of 7.2 mm will be filled with 750 mg of fine cut tobacco, which has been conditioned at 75 % RH before making.

The special constructed tubing device used in former studies will be used for making the FCSAs.

The Dutch blend of fine cut tobacco will be the same as used in other studies.

1. OBJECTIVES OF THE STUDY

1.1 To test the viability of that aspect of a possible matrix approach concerned with classification of different papers to the assessment of NFDPM and nicotine in the smoke from FCSAs. This study will be done with 1 diameter and 7 different papers, resulting in 7 samples.

2. STUDY DESIGN

2.1

Tube Diameter (mm)	Tobacco Papers Weight (mg)	Paper
7.2	750	NL, D, CND, UK
		WAT3, WAT4, WAT5

2.2 Before making, the tobacco will be conditioned at 22 °C and 75 % RH. After making the smoking articles will also be conditioned at 22 °C and 75 % RH.

2.3 All laboratories will analyse products made by tubing using the Dutch blend of RYO tobacco in the supplied tubes.

3. PRODUCT SPECIFICATION

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

3.1.1 This will be:

(75 ± 5) % RH

3.2 One blend of RYO will be used.

3.2.1 This will be:

Dutch blend

3.3 One diameter of tubes will be used.

3.3.1 These will be:

7 Types of tubes of commercially available papers with a diameter of 7.2 mm.

4. SMOKING ARTICLE MAKING

4.1 The tobacco will be a brand produced in Holland taken from a single batch production, already conditioned packed and sealed dispatched by the manufacturer (3 bundles, 10 packs each).

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

(22 ± 2) °C and (75 ± 5) % RH.

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

4.3 The test products shall be made from pre-formed tubes in order to minimise the total variability and to increase the sensitivity. The tubes will be provided and sent together with the tobacco. For tubing, one tubing device will also be sent to you. These tubing devices are modified to suit the two diameters.

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

4.5 300 Tubes of each paper quality will be supplied to each participating laboratory.

4.6 From the conditioned tobacco for each paper quality (7 lots) at least 150 FCSAs should be made.

4.7 Weigh the amount of tobacco required (See 2.1) for each FCSA. Carefully introduce this tobacco into the tubing device, spread the tobacco evenly over the total length of the chamber and press it carefully in place. Transfer the tobacco into the tube until it presses against the end of the tube. Remove the product from the tubing machine. Carefully cut off any tobacco hanging out of the ends. Mark the end fixed to the tubing device as butt-end.

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

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

4.10 After reconditioning weight-select out of each lot by mean weight ± 20 mg (See 6.4).

5. VARIABLES

5.1 Product NL is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper NL.

Product D is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a
Product CND is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper CND.

Product UK is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper UK.

Product WAT3 is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper WAT3.

Product WAT4 is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper WAT4.

Product WAT5 is the FCSA conditioned at (75 ± 5) % RH, a diameter of 7.2 mm and a tobacco weight of 750 mg with paper WAT5.

5.2 The variables to be reported are as follows:

1. NFDPM in mg/FCSA.
2. Smoke Nicotine in mg/FCSA.
3. The total weight of 20 FCSAs prior to one smoking run.
4. Tobacco moisture (%) obtained prior to smoking (See 6.6.2).
5. Puff number.
6. Laboratory conditions during smoking.
7. Date of tests.

5.2.1 These seven variables will be reported as the test report.

5.2.2 An example of a test report is given in Appendix A2.

5.3 Other variables to be reported separately are:

1. Type of smoking machine used.
2. Moisture content of the tobacco before tubing (See 6.2.2).
3. Draw Resistance of the fine cut smoking articles prior to smoking (See 6.5.1).

5.4 It is important to note that for this study one test result is defined as the mean yield obtained from smoking 20 FCSAs in a single run. See smoking plan (Appendix A1) for the different types of smoking machine.

6. OPERATIONAL DETAILS

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

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

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

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

6.2.2 Determine the moisture content after conditioning.

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

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

6.5 Select each lot of FCSAs for weight as follows:

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

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

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

Do not use these smoking articles for smoking.

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

6.6.3 Report all results and the method used.

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

(20 ± 2) °C
(60 ± 5) % RH

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

6.9 Use holders which conform to ISO 3308:1991.

6.10 Smoke all products to a butt length of 27 mm for all FCSAs with labyrinth seals according to ISO 3308:1991.

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

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

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

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

6.14 Replicate the analyses 5 times (i.e. smoke 100 FCSAs of each sample) according to the smoking plan supplied (See Appendix A1).

7. MISCELLANEOUS REQUIREMENTS

7.1 It is preferable

- to complete the smoking procedure for the samples in 1 week.
- to use the same person to make all the test products.
- to use the same smoking machine throughout.
- to use the same operator for the smoking machine throughout.

7.2 If you are unable to comply with any of the details given in Section 6 report this fact and the alternative used.

8. REPORTING

8.1 Completed result sheets should be returned to:

Mr A. de Vries
Douwe Egberts van Nelle Tobacco Company
Department IMR Tobacco - Research
P.O. Box 3
8500 AA Joure
The Netherlands.
Fax: (0)513 - 488833.

8.2 The planning group will collate and analyse the data and produce full summary for the main task force meeting, held in Amsterdam on the 23rd of October 1996.

9. GENERAL REMARKS

9.1 The data will, as always, be reported anonymously. No reference will be made to a brand name or to individual companies. It will not be possible to determine the source of any individual data set from the report.

9.2 Actual time needed for smoking work should not exceed 2 weeks.

Appendix 11. Study 11

Study of filter tubes packed at various densities

Executive Summary

The objective of this study was to investigate the effect of packing density on smoke yields when fine-cut tobacco is used with different pre-made filter tubes.

Eight laboratories took part. The study examined 2 pre-made filter tubes, one ventilated and one non ventilated, each packed at densities of 220, 260 & 300 mg/cm^3.

Two statisticians examined the data independently.

The data show that with the non ventilated tube, NFDPM increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With the ventilated tube there is no significant density effect. This pattern of data is repeated for smoke nicotine.

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

Objective

To investigate the effect of packing density on smoke yields when fine-cut tobacco is used with different pre-made filter tubes.

Protocol

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

Study Design

Eight laboratories were recruited. Five smoking runs (i.e. 100 FCSAs) were completed for each of six products. The study comprised a fully factorial 2 (filter tubes) x 3 (densities), with tube A (a non ventilated tube) and tube B (a ventilated tube) being the pre-made filter tubes packed at densities of 220, 260 & 300 mg/cm^3. The eight laboratories were split evenly between users of 20 channel rotary smoking machines and users of 20 channel linear machines.

Statistical Methods

Two statisticians examined the data independently. Statistical analysis was achieved in both cases using stepwise regression and three-way analyses of variance. Type of filter tube, density, and laboratory are main factors. Particular interest was directed (towards filter tube*density) interactions, to see if different pre-made filter tubes responded differently across the range of density. The main difference in approach between the two statisticians was in the treatment of outliers. One statistician took the approach of retaining as much individual data as possible, the other removed any data point with a standardised residual greater than 2.0 and which showed a marked deviation from the expected linear plot of normalised scores.

Analysis of data

Approach by Statistician 1

The stepwise regression analysis showed that for all analytes (NFDPM, Nicotine & Puff Number) most factors and interactions were significant, and that response of yield to density was dependent on the type of filter tube. Residuals and statistical 'measures of influence' were estimated from the models generated to examine if any data points were either bringing excess influence to bear on the conclusions and/or were grossly outlying. Results for the NFDPM model indicated three data points more than +/- 3 standard deviations from their predicted values. In the case of puff number there was just one. Further examination of these cases led to the data in question being retained.

In the case of nicotine, thirteen data points were deemed as 'influential' and six of these had residuals in excess of +/- 3 standard deviations. All of these data came from one laboratory. Consequently, it was decided to remove the data of this laboratory from the analysis of nicotine. Analysis of nicotine over the seven remaining laboratories produced a similar outcome to that for NFDPM and puff number. Two individual data points had residuals outside the range of +/- 3 standard deviations, but were retained on further investigation.

A diagnostic test (using principle components) indicated very strongly the independence of the three analytes considered and thus the data from the one laboratory, deemed an outlier for nicotine, relating to NFDPM and puff number were retained.

Approach by Statistician 2

The outlier approach used by this statistician resulted in the identification of 8 individual data points for puff number, 6 for NFDPM and 10 for nicotine. These data points were deleted from all subsequent analyses.

It is obvious that one laboratory had serious problems with the measurement of smoke nicotine. Of the 30 data points reported, 9 were deemed to be outliers and were not included in the subsequent analysis. Notably, all nicotine data for Tube A at 260 mg/cm^3 were deleted for that laboratory.

Grubbs test was used to determine if any laboratory was outlying within each cell of 2 x 3 design for each analyte. Although one Laboratory was most frequently tested for such behaviour the test failed on all occasions to confirm this. Consequently, no laboratory was removed.

Full analyses of variance for puff number, NFDPM, and nicotine showed that there was no significant difference between the two types of smoking machine used. There are highly significant main effects between Tubes A and B, between packing densities and between laboratories within smoking machines.

These analyses also show that the MACHINE*DENSITY, DENSITY*LABS, TUBES*LABS, and DENSITY*TUBES interaction terms are highly significant. The second order interaction term, DENSITY*TUBE*LABS is also highly significant.

In order to unravel the relationships within the data that give rise to these interactions, analyses of variance for the data from each tube taken separately were carried out.

When Tube A is used, the effect of DENSITY, and the MACHINE*DENSITY and DENSITY*LABS interactions are all highly significant. By contrast, when Tube B is used, the effect of DENSITY, and the MACHINE*DENSITY interaction, are not significant; the DENSITY*LABS interaction is significant.

This contrast in the performance of the two tubes is the main reason for the presence of the highly significant interaction terms involving TUBES reported in the full analyses of variance. Changes in packing density have a much greater effect on the delivery of NFDPM and Nicotine when Tube A is used than when Tube B is used.

Results

What follows is as a result of amalgamating the analyses of both statisticians, and is possible because the two modes of analysis produced virtually identical outcomes. Table 1 provides averages for NFDPM across all laboratories for both filter tubes at each density. This shows that with Tube A, NFDPM increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With Tube B there is no significant density effect.

Table 1: Mean NFDPM (mg/piece) across all laboratories

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

Table 2 shows the averages for nicotine in smoke across all laboratories for both filter tubes at each density. This shows that for Tube A, nicotine increases as the density is increased from 220 to 260 mg/cm^3 and continues to increase significantly, albeit at a reduced rate, between a density of 260 mg/cm^3 and 300 mg/cm^3. With Tube B there is little evidence of a density effect.

Table 2: Mean Nicotine (mg/piece) across laboratories

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

Table 3 shows the averages for puff number across all laboratories for both filter tubes at each density. This shows that for both filter tubes, puff number increases significantly across all densities, in proportion to the amount of tobacco burnt.

Table 3: Mean puff number across laboratories

	Density mg/cm^3		
Filter tube	**220**	**260**	**300**
A	6.9	8.4	9.7
B	8.1	9.8	11.5

Conclusions and Recommendations:

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

Annex 1 - Protocol

0. **INTRODUCTION**

0 .1 This experiment will test the viability of an appendix to the RYO matrix for the declaration of tar/nic yields of filter tubes.

0.2 This study is set up to investigate the influence of two different filter tubes at three different densities.

The two pre made tubes are commercially available with a diameter of 8.0 mm and will be filled with different amounts of fine cut tobacco, which has been conditioned at 75 % RH before making.

The tubes have a different design and give different yields.

A commercial available tubing device will be used for making the FCSAs.

The standard Dutch blend of fine cut tobacco will be the same as used in previous studies.

1. **OBJECTIVES OF THE STUDY**

1.1 To test the viability of an appendix to the RYO matrix concerned with diameters and different densities to the assessment of NFDPM and nicotine in the smoke from FCSAs. This study will be done with 2 different filter tubes and with 3 densities (220; 260; 300 mg/cm^3), resulting in 6 samples.

2. **STUDY DESIGN**

2.1

Tube (mm)	Density mg/cm^3)	Tobacco Weight (mg)
A	220 - 260 - 300	760 - 900 – 1040
B	220 - 260 - 300	760 - 900 - 1040

2.2 Before making, the tobacco will be conditioned at 22 °C and 75 % RH. After making the smoking articles will also be conditioned at 22 °C and 75 % RH.

2.3 All laboratories will analyse products made by tubing using the Dutch blend of RYO in the supplied filter tubes.

3. **PRODUCT SPECIFICATION**

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

3.1.1 This will be: (75 ± 5) % RH

3.2 One blend of RYO will be used.

3.2.1 This will be: Dutch blend

3.3 Two designs of filter tubes will be used.

3.3.1 These will be 2 types of filter tubes, commercially available, with a diameter of 8.0 mm.

A: Design as the market leader in Europe

B: Typical Scandinavian design

4. **SMOKING ARTICLE MAKING**

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

4.2 Before making, the tobacco will be conditioned at one moisture level. For this the tobacco must be unpacked and conditioned for 4 days at (see appendix A3):

(22 ± 2) °C and (75 ± 5) % RH.

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

4.3 The test products shall be made with pre-formed filter tubes. The filter tubes will be provided and sent together with the tobacco. For tubing, one tubing device will also be sent to you.

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

4.5 400 filter tubes of each design will be supplied to each participating laboratory.

4.6 From the conditioned tobacco for each filtertube (2 lots) at FCSAs should be made.

4.7 Weigh the amount of tobacco required (See 2.1) for each FCSA. Carefully introduce this tobacco into the tubing device, spread the tobacco evenly over the total length of the chamber and press it carefully in place. Transfer the tobacco into the tube until it presses against the end of the tube. Remove the product from the tubing machine. Carefully cut off any tobacco hanging out of the ends.

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

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

4.10 After reconditioning weight-select out of each lot by mean weight ± 20 mg (See 6.4).

5. <u>VARIABLES</u>

5.1 Product 220A is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 760 mg with filtertube A.

Product 220B is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 760 mg with filtertube B.

Product 260A is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 900 mg with filtertube A.

Product 260B is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 900 mg with filtertube B.

Product 300A is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 1040 mg with filtertube A.

Product 300B is the FCSA conditioned at (75 ± 5) % RH, with a tobacco weight of 1040 mg with filtertube B.

5.2 The variables to be reported are as follows:

1. NFDPM in mg/FCSA.
2. Smoke Nicotine in mg/FCSA.
3. The total weight of 20 FCSAs prior to one smoking run.
4. Tobacco moisture (%) obtained prior to smoking (See 6.6.2).
5. Puff number.
6. Laboratory conditions during smoking.
7. Date of tests.

5.2.1 These seven variables will be reported as the test report.

5.2.2 An example of a test report is given in Appendix A2.

5.3 Other variables to be reported separately are:

1. Type of smoking machine used.
2. Moisture content of the tobacco before tubing (See 6.2.2).
3. Draw Resistance of the fine cut smoking articles prior to smoking (See 6.5.1).

5.4 It is important to note that for this study one test result is defined as the mean yield obtained from smoking 20 FCSAs in a single run. See smoking plan (Appendix A1) for the different types of smoking machine.

6. OPERATIONAL DETAILS

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

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

Mix a quantity (generally about 1 kg) of sodium chloride, NaCl, (of at least GPR quality) with water to form a slurry. Ensure that there is always an excess of free salt. Pour the slurry into a desiccator. Place the desiccator in a conditioning cabinet and allow the air to circulate over the slurry (see appendix A3).

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

6.2.2 Determine the moisture content after conditioning.

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

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

6.5 Select each lot of FCSAs for weight as follows:

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

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

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

Do not use these FCSAs for smoking.

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

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

6.6.3 Report all results and the method used.

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

(20 ± 2) °C
(60 ± 5) % RH

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

6.9 Use holders which conform to ISO 3308 : 1991.

6.10 Smoke all products to a butt length of 28 mm for all FCSAs with labyrinth seals according to ISO 3308 : 1991.

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

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

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

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

6.14 Replicate the analyses 5 times (i.e. smoke 100 FCSAs of each sample) according to the smoking plan supplied (See Appendix A1).

7. MISCELLANEOUS REQUIREMENTS

7.1 It is preferable

- to complete the smoking procedure for the samples in 1 week.
- to use the same person to make all the test products.
- to use the same smoking machine throughout.
- to use the same operator for the smoking machine throughout.

7.2 If you are unable to comply with any of the details given in Section 6 report this fact and the alternative used.

8. REPORTING

8.1 Completed result sheets should be returned a.s.a.p. but not later than by the 20th of September 1996 to:

Mr A. de Vries
Douwe Egberts Van Nelle Tobacco Company
Department IMR Tobacco - Research
P.O. Box 3
8500 AA Joure
The Netherlands.
Fax: (0)513 - 488299.

8.2 The planning group will collate and analyse the data and produce full summary for the main task force meeting, held in Amsterdam on the 23rd of October 1996.

9. GENERAL REMARKS

9.1 The data will, as always, be reported anonymously. No reference will be made to a brand name or to individual companies. It will not be possible to determine the source of any individual data set from the report.

9.2 Actual time needed for smoking work should not exceed 2 weeks.