

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

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

Scope

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

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

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

Background

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

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

The standard cigarette holder shall cover 9 mm \pm 0.5 mm from the butt end of a cigarette and shall be impermeable to smoke components and to air. The standard cigarette holder shall ensure that the leakage between the cigarette and the cigarette holder is not greater than 0.5 % of the puff volume.

Either the cigarette holder or the smoke trap shall be equipped with a perforated disc (washer) of plain expanded synthetic rubber, closed cell sponge grade, which partly obstructs the butt end of the cigarette. The synthetic rubber shall have a density of 150 kg/m³ to 170 kg/m³, low swell oil resistance and compression-deflection range of 35 kPa to 63 kPa. Four labyrinth seals shall be used; the one closest to the butt end (back seal) shall be reversed. The dimensions of the washer and labyrinth seals are given in figure 1. The washer shall be supported by a structure with a hole in its centre of 4 mm diameter. The assembly shall be constructed so that the cigarette shall be in contact with the washer when the cigarette is inserted to a depth of 9 mm from the sealing annulus of the front labyrinth seal.

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

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

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

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

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

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

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

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

Discussion

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

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

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

Fine-cut tobacco is used at a much higher moisture content than that used for manufactured products and this results in a product which softens more than a manufactured cigarette at the mouth end during smoking.

The tobacco is unevenly distributed through the length of the fine-cut smoking article giving rise to variable density and soft points along the length of the article.

There is often poor packing at the end of the fine-cut smoking article.

Consumers make products with a wide variety of shapes.

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

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

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

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

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

- A. The perforated disc is only relevant to filtered products and is not relevant to hand rolled fine-cut smoking articles. Indeed the presence of the washer may cause distortion of the hand-rolled product which is generally softer than a manufactured cigarette.
- B. The number of seals required to be used may be more than 4 in order to avoid leakage. Alternatively the method of sealing the smoking article should be more applicable to a softer, less even product.
- C. The reason for reversing the fourth seal is to ensure a tight seal between the cigarette and the washer. Since there is no need for the washer there is no need to reverse the fourth seal. Indeed the use of a reversed seal is likely to cause damage to the fine-cut product.
- D. In order to effect a good seal for asymmetric and / or uneven smoking articles, it is probable that the insertion depth of hand rolled smoking articles made from fine-cut tobacco will have to be greater than 9 mm.

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

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

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

Insertion depth / leakage. Either the product could be treated in some way to minimise the possibility of leakage or the insertion depth would have to be increased.

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

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

Experimental evidence

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

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

Diameter from 5.2 mm upwards

Irregular diameter and product shape

Low packing density

Ends devoid of tobacco, or at best at lower packing density than the rest of the smoking article

Able to be used on existing smoking machines, both rotary and linear.

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

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

Their data set is shown Table 1. Note:

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

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

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

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

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

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

They observed no leakage.

They observed no distortion of the smoking articles.

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

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

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

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

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

1. The longer insertion depth is affecting the air dilution

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

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

Conclusions

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

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

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

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

An insertion depth of 15 mm would appear adequate.

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

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

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

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

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

H.F.Dymond and B.J.Camm

ABSTRACT

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

BACKGROUND

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

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

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

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

INTRODUCTION

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

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

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

Fine-cut tobacco is used at a much higher moisture content than that used for manufactured products and this results in a product that softens at the mouth end during smoking more than a manufactured cigarette.

The tobacco is unevenly distributed through the length of the fine-cut smoking article giving rise to variable density and soft points along the length of the article.

There is often poor packing at the end of the fine-cut smoking article.

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

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

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

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

EXPERIMENTAL PRINCIPLE

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

CONCLUSIONS

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

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

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

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

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

Appendix 6. Study 12

Study of a potential Holder for Fine-Cut Smoking Articles

Executive Summary

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

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

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

Two statisticians examined the data independently.

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

Objective

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

Background

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

Protocol

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

Study Design

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

Statistical Methods

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

Results

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

Table 1
Average Results by Laboratory and Holder

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

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

NFDPM

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

Nicotine

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

Puffs

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

Conclusions

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

Appendix 1 – protocol for Study 12

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

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

*) See Annex A2

- Make and smoke the cigarettes according to the protocol used for Co-operative Study 9 (See Annex A1).
- There will be 2 samples (2 types of holders, 1 butt length and 1 type of paper).
- For each sample smoke 5 x 20 cigarettes.
- Results should be sent to:

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

Before the 27th of September

Annex A1

1. PRODUCT SPECIFICATION

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

This will be: $(75 \pm 5) \% \text{ RH}$.

2. SMOKING ARTICLE MAKING

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

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

$(22 \pm 2) ^\circ\text{C}$ and $(75 \pm 5) \% \text{ RH}$.

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

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

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

2.5 300 Tubes will be supplied to each participating laboratory.

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

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

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

2.9 All FCSAs are restored at the conditioning level, $22 ^\circ\text{C}$ and $75 \% \text{ RH}$, for 3 days.

2.10 After re-conditioning weight-select out of each lot by mean weight $\pm 20 \text{ mg}$. (See 3.5).

3. OPERATIONAL DETAILS

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

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

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

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

3.2.2 Determine the moisture content after conditioning.

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

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

3.5 Select each lot of FCSAs for weight as follows:

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

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

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

Do not use these smoking articles for smoking.

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

3.6.3 Report all results and the method used.

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

(20 ± 2) °C

(60 ± 5) % RH

- 3.8 Set up the smoking machine to comply with ISO 3308 : 1991. Pay particular attention to air flow control.
- 3.9 Smoke according to the procedures laid down in ISO 4387:1991.
 - 3.9.1 Bring the conditioned FCSAs into the smoking room in sealed containers, open the containers just long enough to remove those FCSAs required for immediate smoking and re-seal the container until the next set of FCSAs is required.
- 3.9 Analyse the extract for nicotine using method ISO 10315:1991.
- 3.10 Analyse the extract for water using either method ISO 10362/1:1993 or method 10362/2:1994.

Appendix 7. Study 14

Study of a FCSA Holder

Executive Summary

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

The following were studied.

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

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

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

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

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

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

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

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

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

CORESTA Fine-Cut Tobacco Task Force

Objective

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

Background

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

Protocol

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

Study Design

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

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

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

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

Statistical Methods

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

Analysis by Statistician 1

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

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

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

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

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

Table 1:
“repeatability v” & “Reproducibility V” for NFPDM and Nicotine
Nine Laboratories Included

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

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

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

Analysis by Statistician 2

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Conclusions

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

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

Recommendation

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

Appendix 1

PARTICIPANTS:

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

Details Study No 14 :

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

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

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

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

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

1. PRODUCT SPECIFICATION

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

1.1.1 This will be:

(75 ± 5) % RH

2. SMOKING ARTICLE MAKING

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

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

(22 ± 2) °C and (75 ± 5) % RH.

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

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

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

2.5 500 Tubes will be supplied to each participating laboratory.

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

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

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

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

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

3. OPERATIONAL DETAILS

- 3.1 Select at random 1 pouch out of the bundle. Determine the moisture content preferably by using the Karl Fischer method according to ISO 6488 : 1992 . If an alternative method is used, please specify.
- 3.2 Open the pouches and condition at (22 ± 2) °C and (75 ± 5) % RH (Tobacco before making and FCSAs). This to be achieved by using a conditioning cabinet or as follows:
Mix a quantity (generally about 1 kg) of sodium chloride, NaCl, (of at least GPR quality) with water to form a slurry. Ensure that there is always an excess of free salt. Pour the slurry into a desiccator. Place the desiccator in a conditioning cabinet and allow the air to circulate over the slurry.
- 3.2.1 For those laboratories not able to comply with one of these procedures they should still attempt to control the temperature and RH and report the method used.
- 3.2.2 Determine the moisture content after conditioning.
- 3.3 All test products should be made "en bloc" in the participating laboratories.
- 3.4 Recondition the FCSA's at 22 °C and 75 % RH, for 3 days.
- 3.5 Select each lot of FCSAs for weight as follows:
Determine the weight of 100 FCSAs for each product, calculate the mean and select to ± 20 mg on individual pieces.
- 3.6 Prior to smoking perform the following tests on each batch of products.
- 3.6.1 Take 10 pieces of each sample and determine the Draw Resistance according to with ISO 6565 : 1983.
Do not use these smoking articles for smoking.
- 3.6.2 Take sufficient FCSAs to provide a value for moisture content prior to smoking.
Preferably determine the water content using the Karl Fischer method according to ISO/DIS 6488 : 1994.

Repeat this test for each smoking run if possible. Otherwise analyse a sample of the Masterblend conditioned and stored under the same conditions as the FCSAs.
- 3.6.3 Report all results and the method used.
- 3.7 Maintain the conditions of the smoking room to comply with ISO 3402 : 1991 i.e.
 (20 ± 2) °C
 (60 ± 5) % RH
- 3.8 Set up the smoking machine to comply with ISO 3308 : 1991. Pay particular attention to air flow control.
- 3.9 Smoke according to the procedures laid down in ISO 4387 : 1991.
- 3.9.1 Bring the conditioned FCSAs into the smoking room in sealed containers, open the containers just long enough to remove those FCSAs required for immediate smoking and reseal the container until the next set of FCSAs is required.
- 3.9 Analyse the extract for nicotine using method ISO 10315 : 1991.
- 3.10 Analyse the extract for water using either method ISO 10362/1 : 1993 or method 10362/2 : 1994.
- 3.11 Please follow the smoking plans as mentioned in Appendix A1