



1 Cascade Yarns, Inc.'s Motion for a Preliminary Injunction to Enjoin Defendant Knitting  
2 Fever, Inc.

3 2. Attached as Exhibit 1 is a true and correct copy of an article by F.J.  
4 Wortmann and W. Arns, entitled *Quantitative Fiber Mixture Analysis by Scanning*  
5 *Electron Microscopy: Part I: Blends of Mohair and Cashmere with Sheep's Wool*, from 56  
6 Textile Research Journal 442 (1986).

7 3. Attached as Exhibit 2 is a true and correct copy of a webpage (located at  
8 [http://www.cashmere.org/cm/news\\_article.php?id=36&public=Y](http://www.cashmere.org/cm/news_article.php?id=36&public=Y) maintained by the  
9 Cashmere and Camel Hair Manufacturers Institute and last accessed on July 26, 2010)  
10 reporting the results of trials performed by fiber analysis laboratories to ascertain their  
11 accuracy in detecting the quantity of fine animal hair present in fiber and yarn samples of  
12 known composition.

13 4. Attached as Exhibit 3 is a true and correct copy of an article written by  
14 W.D. Ainsworth and Liqin Zhang, titled *Microscope Analysis of Animal Fibre Blends*  
15 from November 2005.

16 5. Attached as Exhibit 4 is a true and correct copy of an article written by F.J.  
17 Wortmann et al., entitled *Quantitative Fiber Mixture Analysis by Scanning Electron*  
18 *Microscopy: Part VI: Possibilities and Limitations of the Analysis of Binary Specialty*  
19 *Fiber/Wool Blends in View of Test Method IWTO-58*, from 73 Textile Research Journal  
20 782 (2003).

1 I am personally familiar with the foregoing facts and the same are true and correct  
2 to the best of my knowledge, information and belief. I take this verification subject to the  
3 penalties of Title 28 U.S.C. § 1746 relating to unsworn declarations.

4 DATED this 26th day of July, 2010.

5  
6 /s/ Joshua R. Slavitt \_\_\_\_\_

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# **EXHIBIT 1**

# Quantitative Fiber Mixture Analysis by Scanning Electron Microscopy

## Part I: Blends of Mohair and Cashmere with Sheep's Wool

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### ABSTRACT

The possibilities and limitations of analyzing by scanning electron microscopy wool blended with two unmedullated specialty fibers, mohair and cashmere, were studied. The measurement of the height of the cuticle scale at the scale edge was used as an objective criterion for distinguishing between the wool and the specialty fibers. For both blend types and for the experimental procedure used, the mean absolute error of 1% was less than a third of the mean confidence distances and compared well with the accuracy of the chemical methods of analyzing fiber blends. The remaining errors were attributed to purely random variations, so that systematic errors of any kind can largely be excluded.

Labeling textiles to indicate their composition requires analytical means for control, not only for the final product but also for the raw materials and during all stages of processing. Besides the legal aspects of labeling, the price difference of the components for various common fiber blends is a major motivation for developing exact analytical procedures.

Quite common fiber blends for high quality textile goods are blends of sheep's wool, hereafter referred to simply as wool, with other fine animal hair fibers, *e.g.*, mohair, cashmere, camel hair, angora, etc., referred to as specialty fibers [1, 12].

Though substantial progress has been made recently to detect chemically [4, 5, 11, 13] or physically [15], the differences between different keratin fibers (*e.g.*, between wool and specialty fibers), the most important means for identifying and quantifying them has been light microscopy, where the accuracy that can be achieved depends largely on the ability of the operator to identify the different fibers [1].

Though Wildman [16] and others, as recently reviewed by Langley [12], extensively investigated the differences in the appearance of various animal hairs under the light microscope, no reliable objective criterion emerged to identify specific animal hairs [12, 16] because of the similarity of their morphologies [14, 16]. Instead it is a combination of certain features of the fiber image that allows the expert in certain cases to make a good guess as to its identity. For wool/mohair and wool/cashmere mixtures, extensive round trials have shown that analyzing these blends by light microscopy is unreliable, if not impossible [14].

Kusch and Arns [10], during an investigation of the appearance of wool and specialty fibers in the electron

microscope, confirmed the observation made by Dobb *et al.* [6] about the differences in the cuticle scale height at the distal scale edge for wool and mohair, and the usefulness of these differences for distinguishing between these fibers [9].

Kusch and others [10, 11] showed that the cuticle scale height at the scale edge is about 0.7–1  $\mu\text{m}$  for wool and 0.3–0.5  $\mu\text{m}$  for all other specialty fibers they investigated. The largest scale edge height with  $1.0 \pm 0.05 \mu\text{m}$  (95% confidence limits) was for a sample of Argentine wool and the lowest for French wool ( $0.75 \pm 0.02 \mu\text{m}$ ) [10, 11]. Kusch and Stephani [11] also investigated the scale heights for mohair, cashmere, alpaca, llama, camel, and angora rabbit fibers; they found the highest scale edge for Argentine mohair ( $0.49 \pm 0.02 \mu\text{m}$ ) and the lowest for a sample of alpaca ( $0.25 \pm 0.02 \mu\text{m}$ ). The cashmere samples they investigated showed scale heights between  $0.39 \pm 0.02 \mu\text{m}$  and  $0.35 \pm 0.01 \mu\text{m}$ .

The confidence limits of the scale heights are such that, regardless of the processing conditions (*e.g.*, dyeing, bleaching, chlorination) that might obscure the image of the fibers in the light microscope [12], no case has been found so far where the differentiation between wool and specialty fibers was doubtful [10], so long as enough cuticle cells for identification were left on the fiber. The height of the cuticle cell can thus be used as an objective criterion for distinguishing between wool and the specialty fibers, and hence as a basis for a quantitative fiber composition analysis of their mixtures [8, 9, 11].

No objective criteria have been found so far for microscopic identification of specific specialty fibers. The operator still has to resort to his expertise with the vi-

JULY 1986

443

sual/microscopical appearances of different fibers, though for most practical purposes, this limitation is not very severe.

This article is concerned with the possibilities and limitations of analyzing, by scanning electron microscopy (SEM), wool blended with the unmedullated specialty fibers mohair and cashmere, and applying the measured scale edge heights of the cuticle cells as a criterion for fiber identification.

### Experimental

Using the hand microtome as described in IWTO-8-66 [7], we obtained fiber snippets 0.4 mm long, regardless of fiber diameter, from pure wool, cashmere, and mohair tops. Two wool tops were chosen to match approximately the mean diameters of the cashmere and mohair, respectively. By weighing and mixing different amounts of fiber snippets preconditioned at 65% RH and 20°C for at least 24 hours, we obtained mixtures of 5–10 mg total weight containing approximately 10–90% wool. All results refer to the dry weight of the mixtures: we have assumed that the relative moisture loss of the fibers in the vacuum of the microscope does not vary with fiber type, so the composition of the blend is unchanged.

Applying the procedure developed by Kusch and others [9, 11], which is somewhat similar to the procedure proposed for vegetable fibers in ASTM D629-77 [1], the snippets of a fiber mixture were suspended in a 10 mm diameter test tube with 1–2 ml ethyl acetate by stirring with a 0.5 mm diameter stainless steel rod. The snippet suspension was then poured onto a glass plate to give, after evaporation, a spot roughly 10 cm in diameter. Three strips of single sided adhesive tape were pressed in parallel onto the snippets. Seven sample mounts used for the SEM (aluminium, slotted head, ½" diameter) were covered with double sided adhesive tape and pressed on the single sided tape strips in the arrangement as indicated in Figure 1. The single sided tapes were then stripped off the glass plate and the sample holders cut out. The samples (*i.e.*, the fiber snippets adhering to the tape on a sample mount) were sputter-coated with gold.

For the analysis, using a Novascan 30 (Zeiss), the sample was scanned in a manner similar to the procedures described in IWTO-8-66 [7], ASTM D629-77 [1], and ASTM D2130-78 [2]. After each vertical scan, the trace was displaced horizontally by a distance twice the snippet length. All fibers appearing on the monitor with a length that permitted their identification were used for the analysis, thus preventing the repeated measurement of snippets or their subjective selection

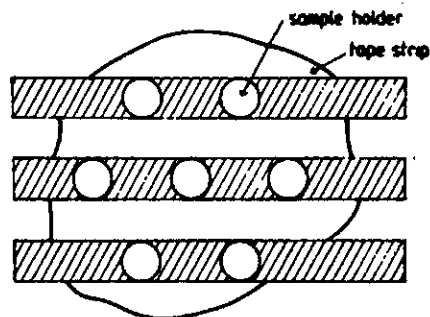


FIGURE 1. Graphical representation of the sample spot on the glass plate with the tape strips, showing the arrangement of the sample holders.

by the operator. Using the instructions of ASTM D629-77 as a guideline for the fiber blend analysis, 150 fibers for each of the seven subsamples (a total of 1050 fibers) were checked for identity. The height of the scale edge was determined on the screen at a suitable spot on the fiber edge at 25,000 magnification. Experience showed that most of the fibers were readily identified by checking whether the cuticle edge height was high ( $\geq 0.6 \mu\text{m}$ ) or low ( $\ll 0.5 \mu\text{m}$ ). For fibers where this distinction was not obvious, two or three cuticle cells were measured to allow safe identification.

For 105 fibers of each component (*e.g.*, 15 fibers of each component per subsample), the diameter was measured on the screen with a vernier at 1000 magnification. No distinction was made between fine undercoat and coarse outercoat cashmere.

Figure 2 shows a mohair fiber and a Buenos Aires wool fiber and a close-up of their surface scale edges at 1000 and 25,000 magnification, respectively. This example is especially relevant, since certain lustrous

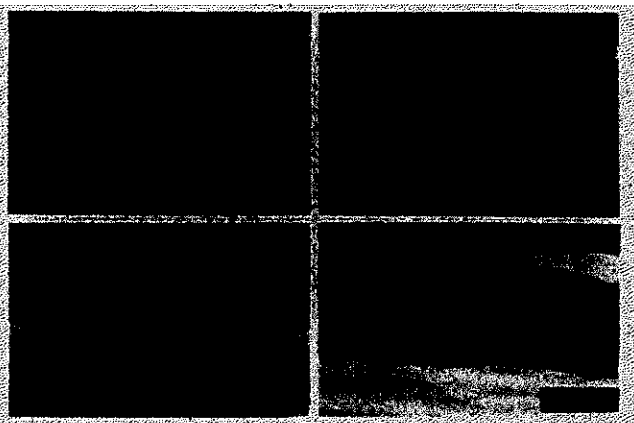


FIGURE 2. SEM image of mohair (left) and wool (right) fibers (originals at 1000X) and close-ups of their surface scale edges at a higher magnification (originals at 25,000X).

Buenos Aires wools are sometimes substituted for mohair, without this being detectable under the light microscope [12].

### Data Evaluation and Results

The analysis yields, for a total number of 1050 fibers, the combined count for the fibers of a given component for the seven subsamples. For the whole sample, the mean diameters and the standard deviations were calculated from the diameter readings of 105 fibers for each component. For a sample of this size, the 95% confidence interval for the mean diameter is about  $\pm 4\%$  [7]. The weight fractions  $w$  of the wool, noted as subscript  $w$ , and the speciality fiber, noted as subscript  $s$ , for a sample were calculated by applying the formula given by Wildman [16]:

$$w_w = [n_w(\bar{d}_w^2 + s_w^2)] / [n_w(\bar{d}_w^2 + s_w^2) + n_s(\bar{d}_s^2 + s_s^2)] \quad (1)$$

and

$$w_s = 1 - w_w \quad (2)$$

where  $n_w$  = number of wool fibers,  $n_s$  = number of speciality fibers,  $\bar{d}_w$  = mean diameter of wool,  $s_w$  = standard deviation for  $\bar{d}_w$ ,  $\bar{d}_s$  = mean diameter of speciality fiber, and  $s_s$  = standard deviation for  $\bar{d}_s$ . All parameters relate to the whole sample.

The application of Equation 1 implies the assumption of circular cross sections for the component fibers and equal and uniform densities. The 95% confidence distance  $q$ , yielding the upper confidence limit ( $w_w + q$ ) and the lower confidence limit ( $w_w - q$ ), is calculated by applying the rules of the Gaussian error progression to Equation 1:

$$q = 1.98$$

$$\times \sqrt{(\partial w_w / \partial n_w)^2 s_n^2 + (\partial w_w / \partial \bar{d}_w)^2 s_w^2 / 105 + (\partial w_w / \partial \bar{d}_s)^2 s_s^2 / 105} \quad (3)$$

$s_n^2$  is the variance of the binomial distribution given by

$$s_n^2 = n_w(1050 - n_w)/1050 \quad (4)$$

The factor 1.98 in Equation 3 is the factor  $t$  of the Student distribution at the 95% probability level for two sided confidence limits and a degree of freedom greater than 100.

Table I (mohair/wool blends) and Table II (cashmere/wool blends) give the weight fractions of wool as weighed  $W$ , as determined by the SEM procedure  $w_w$ , the 95% confidence distance  $q$ , and the error of the analysis  $\Delta = W - w_w$ . Table III gives the mean values

for the confidence distances, the error, and the absolute error for the two blend types as calculated from the data in Tables I and II. Table IV gives the mean di-

TABLE I. The weight fraction of wool in mohair/wool blends as predetermined by weighing  $W$  and as determined by the SEM method  $w_w$ . The values  $q$  are the 95% confidence distances related to  $w_w$  and the  $\Delta = W - w_w$  values are the errors of the analysis.

$W, \%$	$w_w, \%$	$q$	$\Delta$
11.4	10.1	2.1	1.3
19.7	20.0	3.0	-0.3
28.8	29.4	3.8	-0.6
38.4	40.0	4.3	-1.6
50.6	48.9	4.6	1.7
59.7	60.2	4.3	-0.5
70.3	69.2	4.0	1.1
77.7	78.1	3.6	-0.4
88.4	88.9	2.4	-0.5

TABLE II. The weight fraction of wool in cashmere/wool blends as predetermined by weighing  $W$  and as determined by the SEM method  $w_w$ . The  $q$  values are the 95% confidence distances related to  $w$  and the  $\Delta = W - w_w$  values are the errors of the analysis.

$W, \%$	$w_w, \%$	$q$	$\Delta$
9.9	9.2	2.1	0.7
20.0	20.4	3.3	-0.4
29.3	30.6	3.9	-1.3
40.6	39.2	4.6	1.4
46.4	47.1	4.5	-0.7
60.1	59.4	4.6	0.7
69.4	67.4	4.2	2.0
79.9	79.8	3.3	0.1
89.4	88.0	2.7	1.4
93.2	92.5	1.9	0.7

TABLE III. The mean values  $\bar{x}$  and standard deviations  $s$  for the confidence distances  $q$  of the errors  $\Delta$  and the absolute errors  $|\Delta|$  of the analysis for the two sets of blends.

Blend	$q$	$\Delta$	$ \Delta $
Wool/mohair			
$\bar{x}$	3.6	0.0	0.9
$s$	0.9	1.1	0.5
Wool/cashmere			
$\bar{x}$	3.5	0.5	0.9
$s$	1.0	1.0	0.6

TABLE IV. The mean diameters  $\bar{d}$  (vacuum) of the wool, mohair, and cashmere; the standard deviations  $s$  for the two sets of blends; and the coefficients of variation.

Fiber type	$\bar{d}, \mu\text{m}$	$s, \mu\text{m}$	CV, %
Wool 1	35.3	8.3	23.6
Mohair	37.4	10.3	27.5
Wool 2	17.1	3.4	19.7
Cashmere	16.8	5.8	34.3

ameters (vacuum) and the standard deviations for the two wools and the specialty fibers in the two sets of blends, as well as the coefficients of variation.

### Discussion

Figures 3 and 4 are graphical representations of Tables I and II. The solid lines run through the origin with a slope of unity. Table III shows that the mean absolute error of the analysis of the wool weight fraction is the same for mohair/wool and cashmere/wool

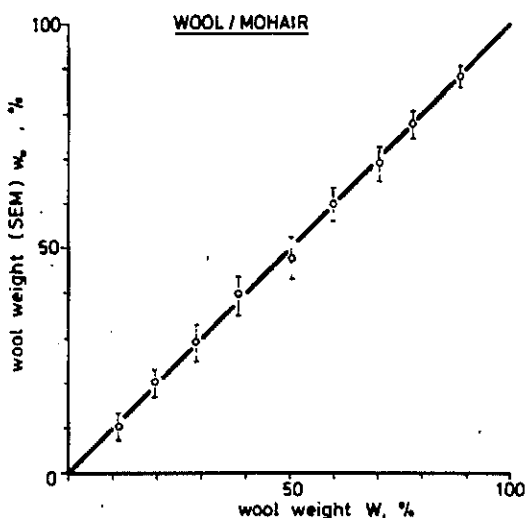


FIGURE 3. Wool weight fraction in wool/mohair blends as determined by the SEM procedure  $w_w$  with the 95% confidence limits, plotted against the predetermined composition  $W$  of the samples.

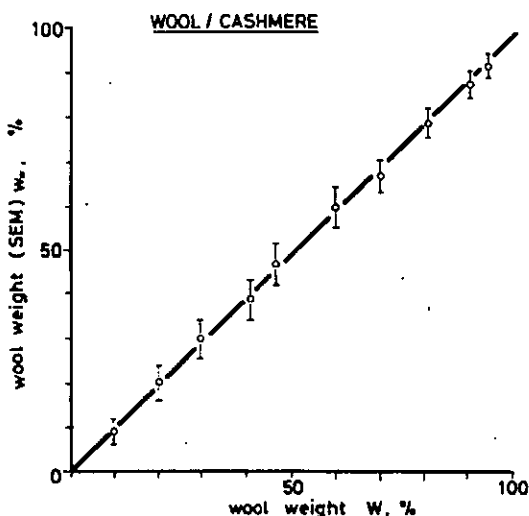


FIGURE 4. Wool weight fraction in wool/cashmere blends as determined by the SEM procedure  $w_w$  with the 95% confidence limits, plotted against the predetermined composition  $W$  of the samples.

blends, and less than a third of the mean confidence distances. The fact that the confidence distance  $q$  for both the wool/mohair and the wool/cashmere blends is considerably larger than the mean absolute error can be attributed to the high coefficient of variation (CV) for the mean diameters. Although the high CV value indicates a certain fraction of large diameter coat hair [12], especially for the cashmere, the accuracy of the analysis is largely unaffected.

For all 19 mixtures, the true composition lies within the confidence limits of the analysis calculated from the error progression of Equation 1. They are hence reliable measures for the limits within which the true composition can be expected. For both kinds of blends and for the experimental procedure, the mean absolute error of about 1% compares well with the accuracy of the chemical methods of fiber blend analysis [1] and will satisfy any directives that might reasonably be set forth for the control of the composition of textiles [14].

The value and the direction of the error  $\Delta$  is independent of the composition for both kinds of blends and hence unsystematic. This is reflected in near to zero mean values for  $\Delta$  in Table III.

The remaining error  $\Delta$ , beyond the already achieved accuracy of the method, can be attributed to purely random variations. Possible sources for systematic errors are the ellipticity of the fibers, differences in their moisture contents or densities, etc., or the operator. From the data we can conclude that, for the blends investigated and the experimental procedure used, systematic errors of any kind can largely be excluded.

With other specialty fibers, some of these assumptions (e.g., circular cross section [3] and equal and uniform densities) cannot be expected to apply. Angora rabbit hair is a typical and somewhat extreme example of such a fiber, where the large range for the degree of ellipticity, the content of coarse coat hair, and the various degrees of medullation make these assumptions somewhat doubtful. In our current investigations, we are trying to determine the contributions of the different factors to the errors of the analysis, especially for different specialty fibers such as camel, llama, alpaca, and angora rabbit.

### Conclusions

Using cuticle scale height as an objective criterion for distinguishing between sheep's wool and specialty animal fibers (mohair and cashmere) provides a reliable test for the quantitative analysis of blends of wool with these fibers by scanning electron microscopy. With an experimental procedure analogous to the relevant standard methods, an accuracy is achieved that com-

parens well with the accuracy of the usual chemical methods.

#### ACKNOWLEDGMENTS

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**The Experience of CCMI in Cashmere Testing****Unione Industriale Pratese*****Kashmir: andamento del mercato e tutela del prodotto***

19 gennaio 2007

Auditorium della Cultura e dell'Economia

Palazzo dell'Industria, via Valentini 14 - Prato

***L'Esperienza del CCMI nel Controllo dei Capi con Fibre Nobili***

When CCMI was founded in 1984 the issue which was foremost on the minds of the then weavers of cashmere, camel hair and blend fabrics in the US was the mislabeling of garments purporting to contain high percentages of cashmere or camel hair fabric. At that time the problem was centered around men's and ladies' overcoats and tailored clothing. A typical man's overcoat might be labeled 92% camel hair, 8% nylon but, in fact, contained a blend of recycled wool and nylon with a very small proportion of fine animal hair which was more than likely recycled. As you can well imagine this was a very great concern to the legitimate weavers of cashmere and camel hair fabrics.

At that time mislabeling was relatively unknown in knitted garments because cashmere and camel hair were still luxury, very high quality niche market products.

CCMI began testing these woven garments that were found in numerous retail outlets in the United States. Very shortly we learned we had a problem in the testing community. There were one or two laboratories in the United States whose results correlated with well-known European laboratories who were involved with the International Wool Textile Organisation and were clearly knowledgeable in fine animal hair testing. Other US laboratories, while well-versed in standard textile testing technology, were ignorant of the science of identifying fine animal hair and distinguishing it from wool. Furthermore they had no practical experience. The laboratories rubber-stamped the representations made by garment suppliers, contributing to the problems and headaches confronted by CCMI in unraveling the problem of mislabeled fabrics and garments.

The dean of the science of fiber analysts in the US at that time was Dr. Samuel Golub who was associated with the Albany International Research Laboratories of Dedham, Massachusetts. Dr. Golub had long experience in the science and technology of fiber identification by light microscopy and also drew on the works of Harry Appleyard, from the United Kingdom. CCMI was fortunate to draw on the work of Dr. Golub in several instances proving mislabeling against US retailers and garment manufacturers. This put those testing laboratories who were honest on alert that they would have to upgrade their skills in identifying wool and fine animal hair or leave that testing to those who were competent to do so. Albany International Research Laboratories also did extensive work with the scanning electron microscope, and was the only laboratory in the US to do so with reference to wool and fine animal hair.

Dr. Golub and his colleagues were in contact with Prof. Franz Josef Wortmann, who was then

with the Deutsches Wollforschungsinstitut, and other members of the team, which developed the Scanning Electron Microscopic method for distinguishing wool and fine animal hair. CCMI was also in close contact with those developments. At the same time CCMI, under the leadership of its United Kingdom members, Dawson International, Johnstons of Elgin, and Z. Hinchliffe & Sons, worked closely with the British Textile Technology Group on the development of a technique for identifying and distinguishing different animal hair fibers by the use of DNA. As we know, this method achieved practical fruition in the qualitative analysis of fibers and detection of contamination. However, to this day we are on the cusp of accomplishing quantification of fine animal hair blends by DNA extraction and analysis.

Producers of legitimate woven cashmere, camel hair and blend fabrics are still vexed by unfair competition from cheap mislabeled fabrics, primarily produced on the carded system, some even containing recycled wool, and mislabeled as to both wool and man-made fiber content. Our successes in such cases as the voluntary recall of ladies outerwear coats by the German retailer, Peek & Cloppenburg, simply points to the on-going dimensions of this problem.

However, in the last ten or fifteen years a new dimension to the fine animal hair mislabeling problem has manifested its self in very strong terms. The problem of mislabeling in knitwear has come on with the entry of China into the cashmere garment market. When the production of cashmere yarn was confined to a relatively few very high quality spinners, primarily in Europe and Japan, and these spinners knew well their sources of supply and the quality of the raw material, the consumer could shop for cashmere knitwear with a reasonable degree of assurance as to the product he or she was getting. With the huge upsurge of garment production from China, compounded by the 2005 removal of quotas for cashmere knitwear and all other textile products, there has as everyone knows, been a tremendous influx of cheap cashmere knit-wear in the US, European and Japanese markets. Prices for some of these garments are unheard of, £20 from a supermarket in the United Kingdom and \$59 at a discount chain in the US. These are not your grandmother's cashmere jumpers, investment products that lasted a lifetime and got better with reasonable care. They are light, loosely knit, poorly constructed of short length, high micron thickness cashmere, if in fact they are cashmere and not some percentage of sheep's wool. Obviously we have a certain amount of knowledge about the supply of cashmere fiber in the world and we must ask ourselves if the number of garments on sale in China, the Western markets and Japan, labeled cashmere, comports with worldwide raw material, as we know it. The answer points to the inevitable conclusion that other goat fibers, particularly from Central Asia and Siberia, and wool can be found throughout the production chain for lower priced knitwear. Wool may have been chemically altered to make it more difficult to distinguish from cashmere. Fine micron wool produced by a Chinese native sheep, which some in China have the unmitigated gall and effrontery to call "sheep cashmere", is unfortunately painful and hard to distinguish by light microscopy. All of these products present challenges to the qualified laboratories able to identify these fibers.

In 2005, twenty-eight testing laboratories from around the world agreed to participate in a CCMI sponsored "round trial" to ascertain their accuracy in detecting the quantity of fine animal hair present in six fiber or yarn samples of known composition. We tabulated the results, sample-by-sample, lab-by-lab, and, for the purposes of this analysis, assigned a "pass" or "fail" grade based on correct identification within three percentage points. This is say, for a fiber present as 50% of the total fiber composition, a lab reporting from 47% to 53% was deemed as having passed the test. The three percentage point tolerance was selected because it is the tolerance permitted under the U.S. Textile Products Labeling Act.

Twenty-four labs used light microscopy (LM) exclusively. Two labs used the scanning electron microscope (SEM) exclusively. One lab reported using BIO

testing. And one lab used LM for sample 6 and DNA for the balance of the testing.

Three labs using LM correctly identified all six samples. Four labs using LM correctly identified five of the six samples.

The four worst performing labs were one using LM that failed to correctly identify any of the samples and three using LM that each correctly identified only one of the six samples.

**Sample 1 contained 80% cashmere and 20% wool.** Thirteen of 28 labs correctly (within the 3% tolerance) identified the fiber composition. Of the 15 failing labs, five were off by ten or more percentage points.

**Sample 2 contained 50% cashmere and 50% wool.** Twelve of the labs correctly identified the fiber content of this sample. Of the 16 that failed this test, 11 were off by ten or more percentage points.

**Sample 3 contained 100% cashmere.** Twenty-three labs correctly identified the fiber content of this sample. Of the five that failed, two were off by ten or more percentage points.

**Sample 4 contained 100% yak.** Seven of the 28 labs correctly identified the fiber content. Of the 21 labs that failed on sample 4, 16 were off by ten or more percentage points. Nineteen incorrectly reported the presence of wool. Other fibers incorrectly identified as being present were: cashmere (5 labs), camel hair (4 labs) and mohair (one lab).

**Sample 5 contained 100% yak.** Twenty-four of the 28 labs correctly identified the fiber content. Of the four that failed to correctly identify this sample, three were off by ten or more percentage points. Four labs incorrectly identified the presence of cashmere in quantities ranging from 4% to 100%. One lab also incorrectly reported the presence of wool (80%) and one incorrectly reported the presence of camel hair (96%).

**Sample 6 contained 10% cashmere and 90% wool.** Thirteen of the 28 labs correctly identified the fiber content. Of the 15 labs that failed to correctly identify this sample, three were off by ten or more percentage points.

We observe that the labs did the best (24 passing) with sample 5, which was 100% yak. However, sample 4, which was also 100% proved the most challenging, with merely seven of the labs passing and many of the labs incorrectly identifying wool, cashmere, and camel hair in this sample.

As regards detection of genuine cashmere fiber, we note that the labs did very well at identifying the 100% cashmere sample. Merely five failed (greater than three percentage points off) and of those just two were grossly off (ten or more percentage points off). Cashmere/wool blends, however, presented more of a challenge. Both the high cashmere content blend (80%) and the low cashmere content blend (10%) were incorrectly identified by 15 of the 28 labs.

Technology scrambles to keep up with the resourcefulness of the cheaters but we still rely to a large degree on optical microscopy. We also extensively use scanning electron microscopy particularly here in Europe. DNA testing is being made available not only from the British but

also from Japan and several other sources in Europe. Near infrared spectrography is another technique that is coming to the fore at this time. Also there is the method being sponsored and developed by CCMI with the cooperation of the Italian research laboratory, CNR, which is based on bio-immunological response.

We believe the industry stands on the brink of major technological breakthroughs in identification and quantification of animal fibers using one or a combination of the methods to which I have referred. We believe that objective testing will be simple, accurate and sure and that this will happen within the next twelve months. We believe that cashmere is a valuable brand and a unique luxury product which must be protected from fraud and contamination.

Furthermore, we believe that the world community and the international trading community has awoken to the dangers to our businesses, our markets and our economies from mislabeling, counterfeiting and unfair trade practices of this nature. CCMI will have the support of the international trading community as it continues to fight these unfair practices with evermore-sophisticated technologies at its disposal.

Thank you very much. I look forward to taking your questions.

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## **EXHIBIT 3**



## MICROSCOPE ANALYSIS OF ANIMAL FIBRE BLENDS

### TRAINING OF OPERATIVES

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**Liqin Zhang<sup>2</sup>** (MA,ACIM)

### SUMMARY

The SGS animal fibre testing laboratory in Bradford, UK, has been involved in the measurement of animal fibre blends for over 20 years and has been working with and approved by the Cashmere and Camel Hair Manufacturers Institute (CCMI) for over 10 years.

Traditionally Operative training has been carried out by a peer to peer method, which has proved successful in the past.

However SGS now operates 3 laboratories based in Europe and China, and has up to 15 operatives measuring and/or training at any one time. Under these circumstances the traditional methods are more difficult and expensive to operate.

A strategic decision has been taken to co-ordinate the training and operation of these laboratories by use of a centralised facility based in the UK.

The main objective is to ensure consistent results throughout the SGS operation by the standardisation of operational and training methods, and the provision of traceable training and reference material in the form of both physical and electronic means.

This paper outlines the approach used.

<sup>1</sup>SGS Cashmere Labs, Project Manager

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## 1. INTRODUCTION

Measurement of the composition of cashmere and other animal fibre blends is a difficult and subjective procedure. It relies on the identification of individual fibres, primarily from the scale characteristics, but also from pigmentation and other visual characteristics plus the objective measurements of features such as diameter, scale density and scale thickness.

There is also a problem with substitution with lower value fibres such as wool and yak. This substitution can take place at all points along the processing chain from the raw cashmere to the final yarn spinning process.

There exist two current "commercial" methods – Optical Microscope Method and Scanning Electron Microscope (SEM) Method.

### Optical Microscope Method

The **Optical** method relies on an operator identifying magnified fibres from their scale structure and other features. Methodology is outlined in a number of sources, including ASTM-629, AATCC-20A, Wildman and Bray, which include some photographs and descriptions of the main identifying features of different fibre types. When differentiating between cashmere and wool fibres there are areas of overlap of characteristics and correct training of operatives is crucial to maintaining reasonable accuracy and consistency of results.

HOWEVER, the method remains primarily subjective and is further complicated by the increasing introduction of modified wool fibres, other animal fibres of similar characteristics (Yak, cashgora, other goat fibres, etc.) and the different sources of cashmere (e.g. Iranian cashmere compared to Mongolian cashmere).

Further difficulties in identification arise from treatment of the fibres, which can hide the surface characteristics. This is particularly prevalent with finished garments/fabrics that have undergone extensive chemical and physical processing, and in some cases make it impossible to identify individual fibres.

The basic operation of Optical methods is limited by the resolution of the microscopes (best resolution of about 0.3 microns) and the small depth of field (1 to 2 microns) which means that surface characteristics are not in focus. Also the Optical microscope views the transmitted light, so interference with scale shadows from both sides of the fibre occurs. The big advantage is that the internal structure (pigments) of the fibres can be seen.

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### Scanning Electron Microscope (SEM) Method.

The scanning electron microscope uses secondary electrons to "view" the surface characteristics of a fibre. Unlike Optical microscopes the fibre interior is not viewed, indeed the surface is usually coated with a thin layer of gold to assist speedy display of the scanned surface.

The major advantage of the SEM is its very high resolution (down to 2 nanometres) and the relatively large depth of field. This enables the complete surface of a fibre to be seen in high detail (usually limited by the resolution of the digital display) thus enabling a better discrimination of the surface characteristics. However no internal information (i.e. pigmentation) is available as with Optical Microscopes.

This enables clear measurement of topographical features of the fibre such as scale height and density (ratio of scale length to fibre diameter) which can also be useful in objective discrimination of fibres.

The measurement of composition by SEM has been standardised as IWTO-58, which uses the scale height to differentiate between Wool and Speciality fibres (cashmere, mohair, Llama/alpaca, camel etc.). Wool is defined as animal fibres with a scale height (thickness) greater than 0.55microns. The identification of the "speciality" fibres is then determined from other topographical properties such as scale shape, density, micron etc. In this sense the procedure relies on clear separation on scale height of wool from the others. Identification of the others is then on subjective assessment of surface characteristics. For this training of operatives is still essential.

Figure 1 below shows examples of the images obtained from Optical and SEM microscopes.

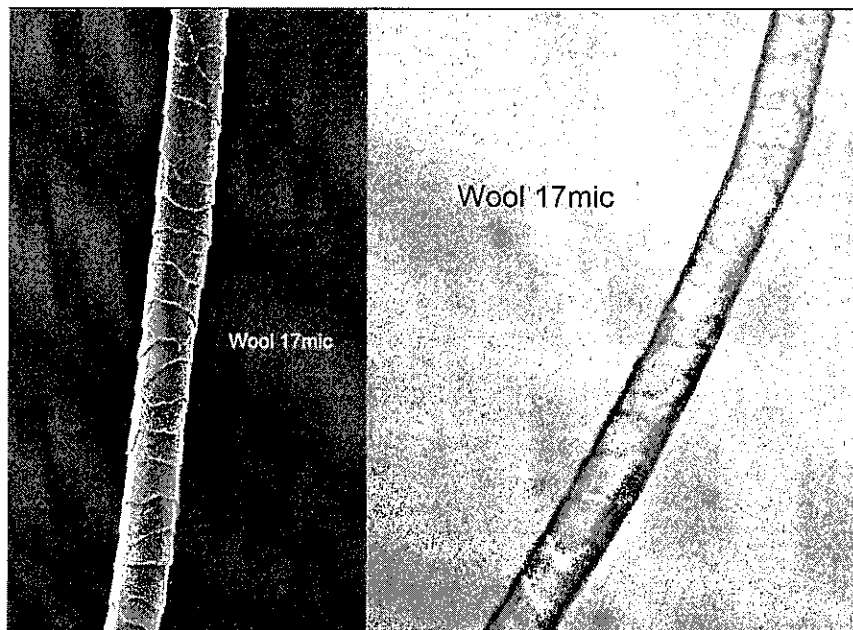


FIGURE 1: COMPARISON OF OPTICAL AND SEM IMAGES OF A WOOL FIBRE



## 2. CRITICAL AREAS FOR MEASUREMENT OF COMPOSITION

There are a number of critical areas that need to be addressed to ensure consistency. One of the major concerns of clients is the differences that are observed between laboratories. Some of these are unavoidable due to the nature of the testing - subjective assessment of the morphology of magnified fibres - but others are undoubtedly caused by poor or inconsistent training of operatives. Such problems can occur with both optical and SEM techniques.

These critical areas can be divided into Technical Procedures and Fibre Identification.

### 2.1 Technical Procedures

Technical procedures includes both sampling techniques and measurement techniques.

**Sampling** is an important area, which is often neglected, and refers to the selection of a representative, unbiased sample. This covers such things as **Global Sampling** (core sampling, yarn packages, fabric) and **Subsampling Procedures**. Consistent procedures and equipment will greatly reduce errors from this source.

Where possible SGS uses standard published procedures, and staff are trained in their application. Extra procedures are written for clarification or where a suitable procedure does not exist.

### Measurement Procedures

Another important area relates to the test methods and procedures used for measurement. Aspects covered include Slide/Snippet Preparation, selection of fibre snippets to ensure that a length biased sample is obtained, and defining how Micron is measured and composition calculated.

Again SGS used published test procedures wherever possible, or bases its written procedures on the best practice as indicated from the published methods.

### 2.2 Fibre Identification

Probably the most important and difficult aspect is to ensure that operatives are properly trained to identify the fibres correctly. This applies to both Optical and SEM microscopy. It is probably true to say that consistency only comes with experience.

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### 3. OPERATIVE TRAINING

#### 3.1 Background

Training at SGS, and I suspect elsewhere, has been carried out using peer to peer training methods, where an experienced operator is placed with a new operative and is shown how to carry out the procedures and how to identify the different fibres. This requires long sessions with the microscope gaining experience, and ties up a qualified operative for long periods.

Experience shows that a new operative can take between 3 to 6 months to train and that, in some instances, the operative never becomes skilled enough in fibre identification to carry out commercial tests.

In the case of SGS where 3 separate laboratories are in operation, and up to 15 operatives can be active at any time, this can become a big problem, especially as there are large distances between the laboratories and the requirement to train new staff when trained personnel leave.

For this reason SGS made a strategic decision to co-ordinate the activities of these laboratories through a centralised facility based in the UK. The main objective of this project is to ensure consistent results throughout the SGS operation by the standardisation of operational and training methods, and the provision of traceable training and reference material in both physical and electronic forms.

#### 3.2 Training

SGS is developing standardised training of Operatives in 3 basic areas: Technical Procedures, Fibre Identification, and Monitoring of Performance.

##### 3.2.1 Training in Technical Procedures.

This relates to the technical procedures as discussed in section 2.1 and preliminary training in the standardised procedures is carried out in the normal manner of study, demonstration and practice. In all cases training of staff is relatively straight forward as it involves following written procedures which are clear and objective.

Once completed this enables operatives to select and prepare samples and specimens, and to operate the microscopes (Optical or SEM) in the required manner. Staff can always refer to the written procedures if necessary.

#### 2: Fibre identification

Training operatives to identify different animal fibres requires that operatives are exposed under standard conditions to images of **known** fibres and instructed by an experienced operative on the basis for distinguishing the different fibre types. For this purpose most laboratories have a collection of

"known" fibre samples which form the basis for training and comparison purposes.

### Traceable Animal Library

With the objective of having a sound basis for standardise training, SGS has undertaken the collection of fibres direct from **known** animals. In this way a completely **traceable** fibre collection is being formed, where we can **certify** exactly from which animal the fibres originate.

A detailed procedure has been designed for collection of these fibres to ensure that an appropriate range of animals from a herd or flock are collected, and the details of the animal recorded and certified by an SGS employee or Agent. To date approximately 60 animal samples, predominately cashmere from the different areas of China, have been collected. Figure 2 shows the type of information collected on each animal sampled.

This is an ongoing program using SGS Offices to obtain samples from all relevant countries and animal types.


Reference Number:		Contact Name:	Cao Yue Zhao
Animal Filename:	jefflian-001.bmp	Farm Name:	
Animal Sampled:		Farm Address:	Wangcun, Doupoxiang, Xi county, Shangxi Province
Animal Colour:	White	Telephone:	0357-7368112
Harvest Method:	Combed	Facsimile:	
Fibre Type:	Cashmere	Email:	
Country:	China	Sampled By:	Jeff Lian
Province:	Shanxi	Date Sampled:	16-Feb-05
Animal Type:	Goat	Sampling Notes:	Feeding with cornstalk, it's a crossbreed of cashmere goat from Liaoning and local cashmere goat after 10-15 years.
Animal Breed:	Cashmere		
Animal Sex:	Neuter		
Animal Age:			

FIGURE 2: DETAILS OF ANIMALS FROM WHICH SAMPLES ARE COLLECTED.

### Traceable Fibre Library

From each known source, SGS intends to build up a Traceable Fibre Library of both SEM and Optical Images, which will then be used for both reference and training purposes.

It is intended that each fibre image will be measured for the following properties:

- ✓ Mean Fibre Diameter (and perhaps Variation within fibre snippet)
- ✓ Scale Ratio (measured as Scale Length to Diameter)
- ✓ Scale Height (Thickness - SEM images only)
- ✓ Scale Angle

And the following descriptors also noted:

- ✓ Fibre treatment (e.g. greasy, scoured, carbonised, bleached etc.)
- ✓ Fibre Damage (e.g. split, abraded, fibrillated etc.)
- ✓ Fibre Pigmentation (Optical only, relative levels of pigmentation)
- ✓ Colour Depth (for dyed fibres, optical only)
- ✓ Medullation (relative levels of medullation)
- ✓ Scale Shape (basic description such as coronal, smooth edged etc)
- ✓ Scale Flaking (relative level of flaking)
- ✓ Damage (relative level of damage)

This information is stored in a database (Figure 3) along with the fibre image to facilitate its use for both training of operatives and reference purposes.

Over a period, representative images of a full range of fibres/sources will be collected.

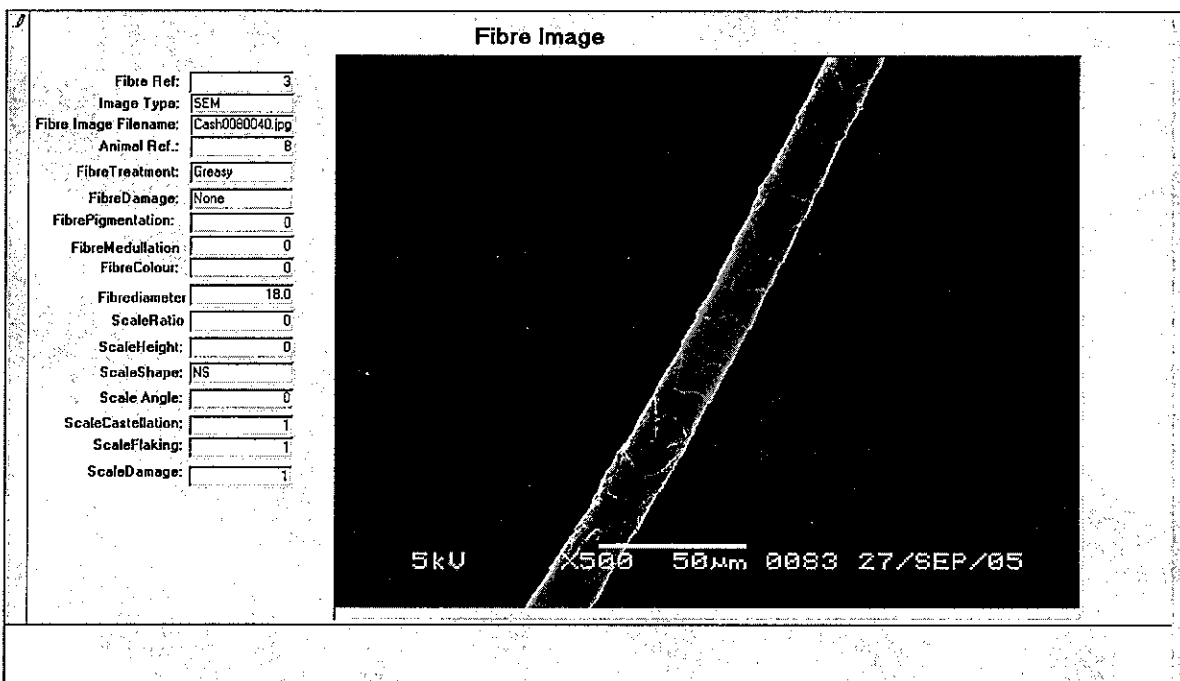


FIGURE 3: DATABASE OF FIBRE IMAGES

### Operative Training - Fibre Identification

The Fibre database is intended to be used for the training of operatives as follows.

First, using specially selected images, the main features of the different fibres are shown and explained to the new Operatives. For this purpose SEM photos are used to provide the best images and these are correlated with Optical Images. In this way Operatives can better interpret the surface characteristics as viewed using an optical microscope.

For example Figure 4 below shows a comparison between a cashmere fibre and a wool fibre of the same diameter as taken by a SEM microscope and Figure 5 below shows a similar image comparing cashmere to wool using an Optical microscope.

While the identification of the fibres is still very obvious, the differences between the two images enable better interpretation of the optical image by comparison with the distinct surface image of the SEM microscope.

The effects of low depth of field, and the mixture of surface and internal features caused by the transmitted light of the Optical Image, are clearly shown.

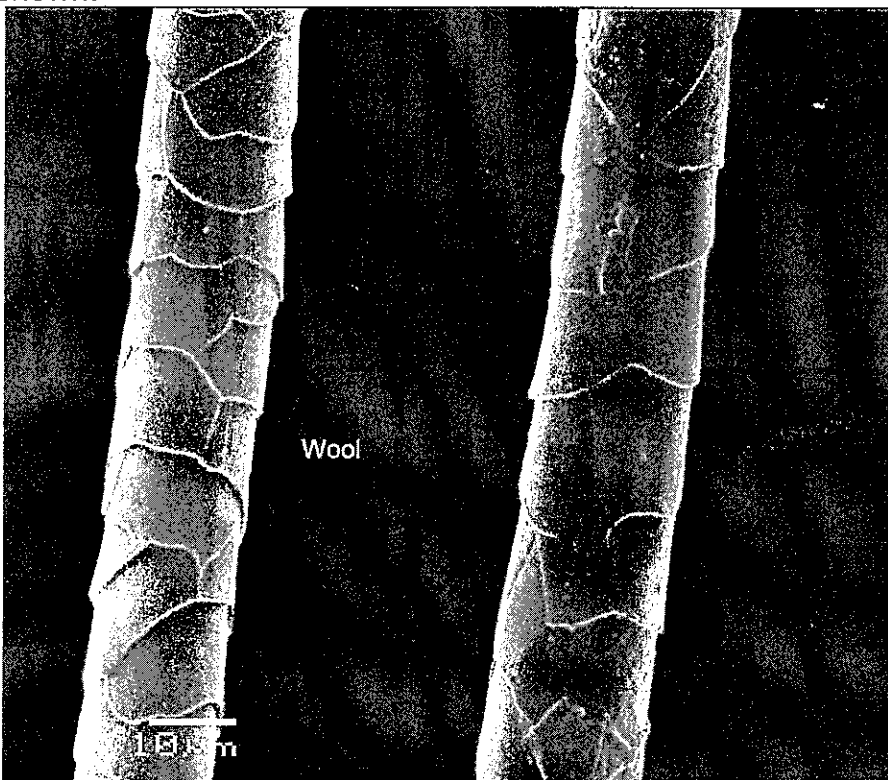


FIGURE 4: TYPICAL CASHMERE AND WOOL FIBRE - SEM IMAGE

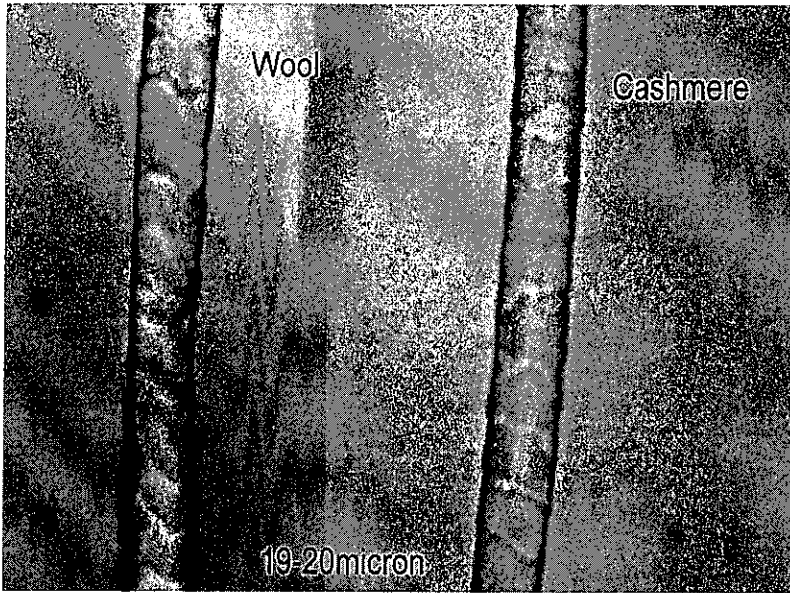


FIGURE 5: CASHMERE AND WOOL FIBRE - OPTICAL IMAGE

By selection of appropriate images the observer can quickly be introduced to the fundamentals of Fibre Identification. Even more importantly this can be done over a wide range of fibres, for example, cashmere fibres can be compared to wool fibres of the same diameter over a range of diameters so that Operatives can understand the effect that diameter has on the scale structure/ fibre image.

Figure 6 below shows a comparison for fine fibres, and Figure 7 for relatively coarse fibres.

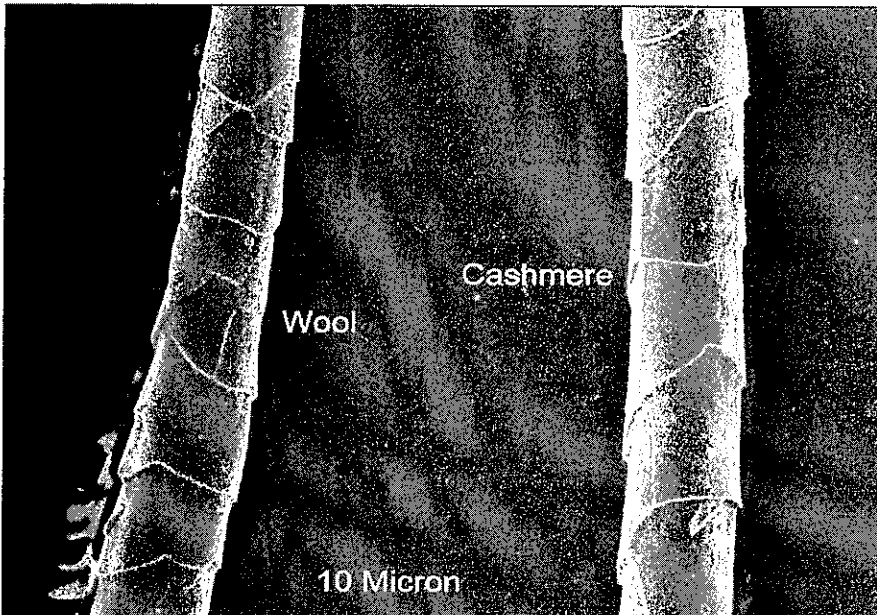


FIGURE 6: SEM IMAGE OF 10 MICRON CASHMERE AND WOOL FIBRES.

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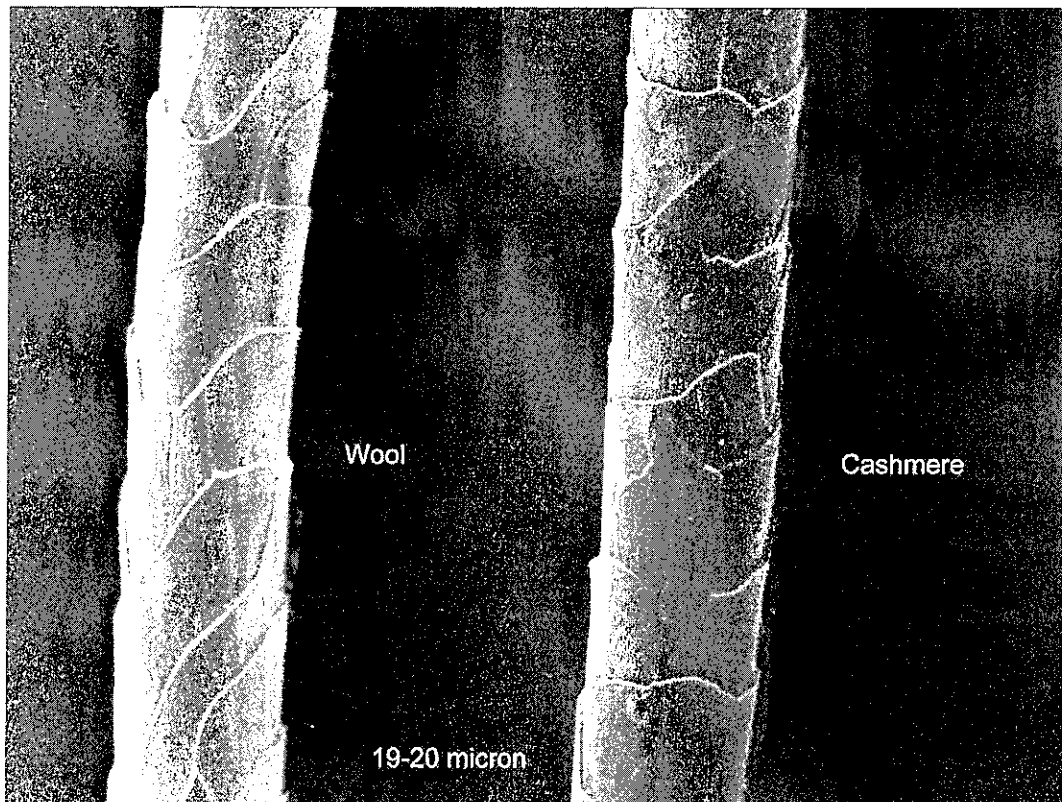


FIGURE 7: SEM IMAGES OF 20 MICRON CASHMERE AND WOOL FIBRES.

The Operative then practices identification on selected sets of fibre images which cover a range of fibre diameters and sources. The operative views an image (either SEM or OPTICAL) and makes an identification. This is supported by feedback as to the correctness of otherwise of the identification.

If a fibre is misidentified then the Operative is shown other images to demonstrate the error. For example he could be shown a fibre image which agreed with his identification for comparison with the original fibre, and possible a series of fibres (all of similar diameters) of the two fibre types to reinforce the visual differences.

Once an operative has reached a suitable level of expertise on "set" training exercises, further experience would be gained using "random" testing from the fibre database.

Finally the Operative would carry out training on actual samples to see how well the "electronic" experience transfers to the real world. **This is of course not a trivial step.**

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With **SEM** procedures the images used for training will be virtually identical to that used in an actual test so the abilities learnt should transfer well.

However for the **Optical** microscope, **which is the bulk of the testing done today**, the images viewed in real time are not identical with the images used for training. In real time the focus of the fibre can be varied to bring different aspects into focus, and the fibre can be viewed along its full snippet length if necessary.

This effect is demonstrated in Figure 8, where several different points of focus are shown for the same fibre. From the left to the right we are focussing on the upper surface, the fibre edges (as required when measuring diameter) and at the bottom surface.

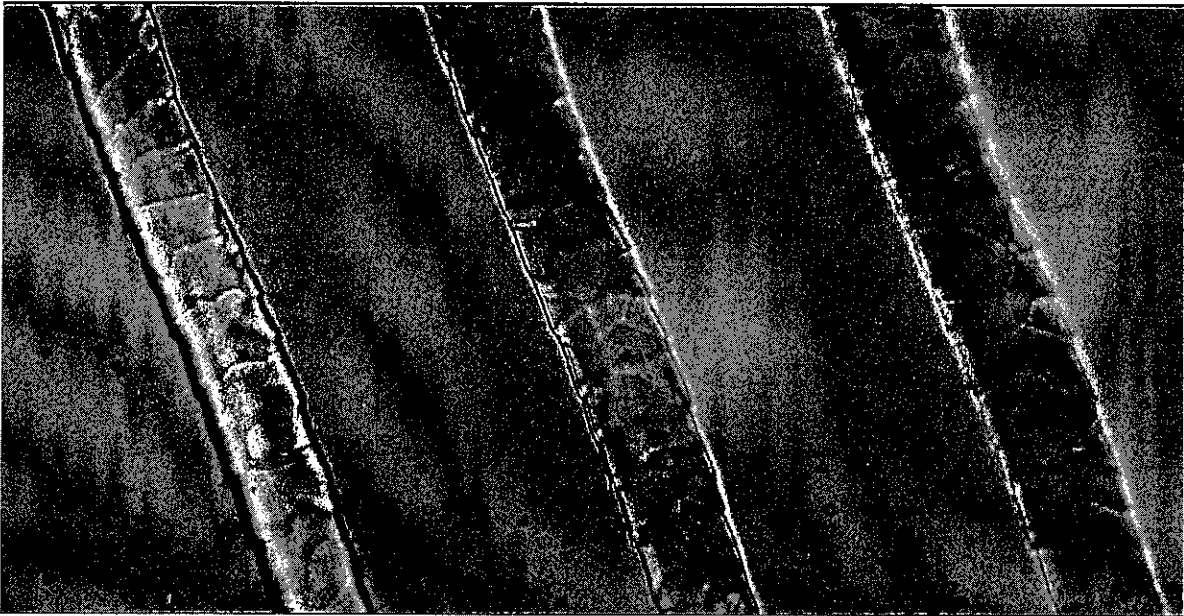


FIGURE 8: FIBRE IMAGE AT 3 FOCUS POINTS - OPTICAL MICROSCOPE

In essence the Operative has more information to work with, which will hopefully make the transition to "live" microscopy more accurate even if more difficult.

It cannot be emphasised enough that the procedures proposed above still require Operatives to gain experience, and it will still take a long period to become expert enough to carry out real analysis.

The advantages in using such an approach are:



- **Standardisation** of the training, both in **how** and **what** Operatives learn.
- Fibres used for training are **traceable**. That is we can certify exactly the samples we use.
- The procedure can be provided as a **computer package**, greatly reducing the time required by trained staff for training purposes. Hopefully also reducing training time for new operatives.

### 3: Monitoring of Operatives.

No matter how much training is carried out, or how detailed and dedicated it is, it is still necessary for Operatives to demonstrate their abilities before commercial testing can be carried out.

For this purpose SGS has instigated internal proficiency testing of all operatives. Provision of Quality Control, in the form of proficiency testing trials leading to an internal accreditation of **individual operatives**, is an essential ongoing requirement to ensure the quality of results.

These trials are designed to monitor and/or train **OPERATIVES** with regard to identification of cashmere/wool/other fibres and measurement of fibre diameter.

Each Operative is provided with a test specimen and is required to **independently** carry out **all** the operations for measurement of fibre composition. The sample may be loose fibre, sliver, yarn or fabric. Each operative at each laboratory is required to independently identify, and measure the diameter of, the cashmere and/or wool fibres on each of 2 slides, using specific procedures. **It is essential that each operative acts in isolation from other trained operatives so that performance of each operative can be assessed independently.**

A summary report of the results is provided to each laboratory with indications of any problems that arise. Where appropriate, differences between Operatives will be highlighted and suggestions regarding possible corrective actions made. This takes the form of a standard EXCEL analysis with appropriate graphs covering both %Composition and Mean Fibre Diameter measurements.

Results are compared to either the "nominal" (or technical) value or the average of the results (excluding outliers).

Examples of the Cashmere% Results for a round trial re shown in Figure 9 below. (Note that a similar analysis of micron measurements is also carried out.)

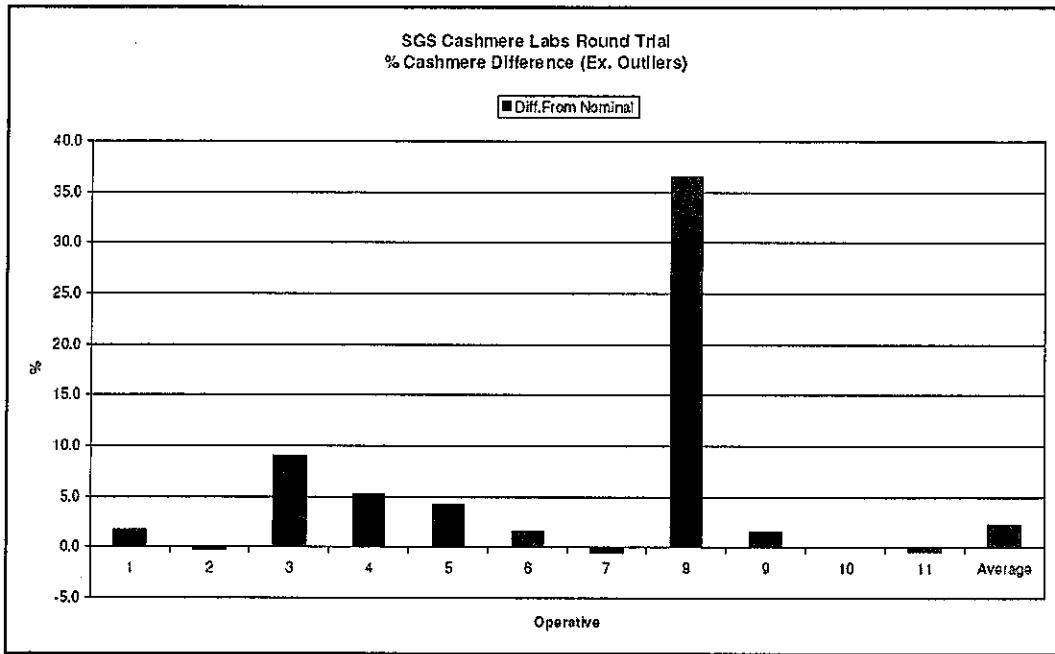


FIGURE 9: ROUND TRIAL RESULTS

This shows that Operative 8 (a new trainee) had serious problems with this sample, and that 3 and 4 were borderline. All other operatives had acceptable results.

The results of all trials for each operative are also monitored as shown below in Figure 10.

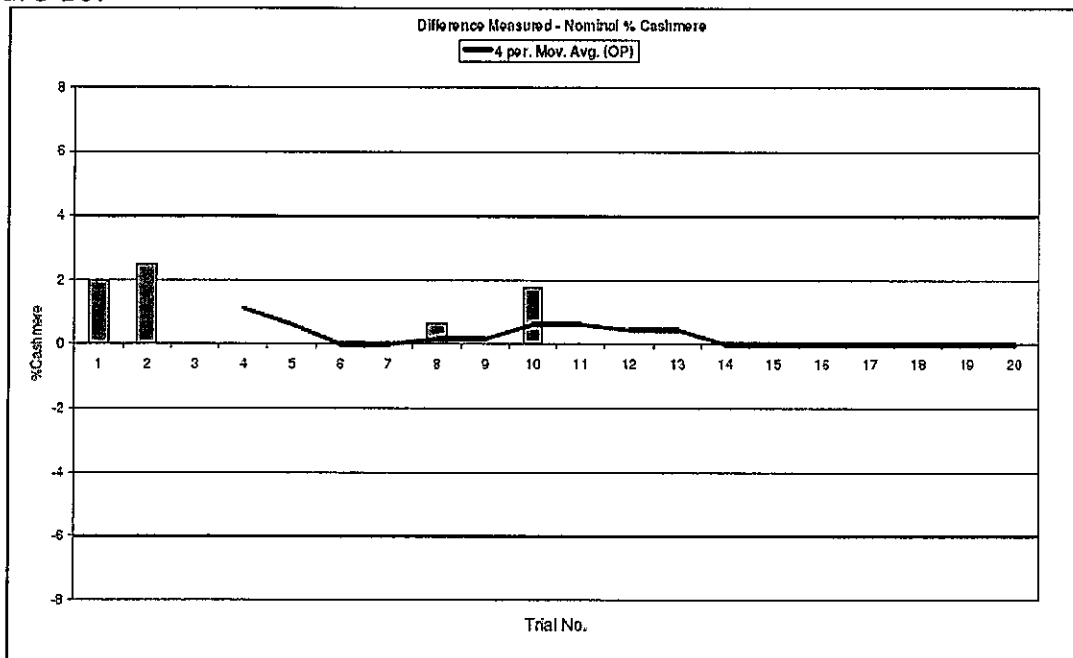


FIGURE 10: SUMMARY RESULTS FOR A "GOOD" OPERATIVE

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In this manner we can check that operatives are performing within acceptable limits compared to other operatives, and over time. For example we would expect a good operative to be within 4% of the Nominal Value and with a Moving Average (that is the average over the last 4 trials) be within 2% of the Nominal Value.

SGS also monitors in a similar fashion the micron measurements of each operative.

### **SUMMARY**

Measurement of cashmere and animal fibre blends currently rely predominantly on Optical Microscope and Scanning Electron Microscope methods.

Both methods rely heavily on training of operatives to identify the individual fibres, and this is a source of within and between laboratory differences.

To ameliorate this problem a SGS has decided to co-ordinate the training and operation of these laboratories by use of a centralised facility based in the UK. This training is based on the development of:

- Standardised technical procedures
- Traceable electronic and physical fibre samples
- Computerised training for fibre identification
- Ongoing monitoring of operatives.

In this manner the consistency and accuracy of results should be improved, and the cost and time for training of operatives reduced.

## **EXHIBIT 4**

# Quantitative Fiber Mixture Analysis by Scanning Electron Microscopy<sup>1</sup>

## Part VI: Possibilities and Limitations of the Analysis of Binary Specialty Fiber/Wool Blends in View of Test Method IWTO-58

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### ABSTRACT

Test Method 58 was established by the International Wool Textile Organization in 2000 to analyze textile products made from specialty fibers, sheep's wool, and their blends using the scanning electron microscope. This part of the series critically examines the design of the analytical approach for quantitative analysis of binary blends specified in IWTO-58. The 95% confidence range is established as a suitable measure for the precision and accuracy of the method. The most critical, legal, and technical issue of quantitative analyses of binary wool/specialty fiber blends is the requirement of 3 weight-% accuracy. This accuracy is not consistently reached with the current analytical protocol, that is, for 50:50 blends, though the course of the analysis already operates at its feasible technical and economical limits. Extended analysis protocols are investigated showing the requirements for meeting this pre-set accuracy. The results show that a substantially higher number of diameter measurements for the fiber components and a substantial increase in the number of identified fibers are required for routine analysis, which is beyond the reach of the current operator-based and thus rather tedious methodology.

To analyze blends of specialty fibers with sheep's wool, the International Wool Textile Organization (IWTO) established Test Method 58 [12] in 2000. The method specifies the use of the scanning electron microscope (SEM) for distinguishing sheep's wool on the one hand from all specialty fibers on the other. Corroborating a previous paper [32], our current objective is to elucidate in some detail the technical and economic possibilities and limitations of the methodology for quantitative analyses of binary blends, that is, by addressing issues related to the precision and accuracy parameter implemented in IWTO-58. Furthermore, some current and future practical and legal consequences are considered.

### Principles of IWTO-58

Analysis of a textile consisting in total or in part of specialty fibers according to IWTO-58 is based on the following principles: Short fiber snippets (0.4 mm) are obtained from the material according, for example, to IWTO-8 [9]. These samples are length-biased [23], in that longer fibers in the sample have a proportionally

higher chance to be represented as snippets than shorter fibers. These snippets are the basis for SEM analysis of the sample according to IWTO-58 [12].

Wool fibers can be differentiated in the SEM from all other specialty animal fibers by the cuticle scale height (CSH) criterion. The height at the distal edge of the wool cuticle cells reaches a value of 0.6  $\mu\text{m}$  or more, whereas it is around 0.4  $\mu\text{m}$  or less for all other specialty fibers [15, 25]. Furthermore, other characteristics such as scale pattern, scale frequency, and diameter are useful for rapid and economical fiber identification [19].

Three different cases are distinguished during the progress of sample analysis, which define the specifics of the analytical procedure. These cases derive either from the anticipated composition of the sample, being either pure or a blend, or from the technical and economical limits for quantifying minor fiber admixtures.

At this point, it is important to note that the principles outlined in this as well as in the previous paper [32] apply equally to any other analytical method that uses length-biased snippet samples, fiber identification, and individual fiber diameter measurements to determine the composition of a fiber blend, provided the fibers are identified correctly.

According to the procedures specified in IWTO-58, the analysis starts by scanning the first stub in the SEM in

<sup>1</sup> This paper is based in part on report no. STG01 for the Technology & Standards Committee of the International Wool Textile Organisation in Nice, France, November 2000.

a specific manner, identifying the fibers appearing on the monitor as belonging to one of two types, specialty fiber or wool.

*Case 1* applies, when after 150 identifications, only one fiber type is found; 300 further fiber snippets on the next two stubs are then scanned. If no fiber of a second type is found for a total of 450 fibers, the sample is declared pure. This procedure is called a "purity check."

*Case 2* applies if two fiber types have been found after checking 150 fibers, where the number percentage of the second component is below 3% ( $<5/150$ ). The second fiber type is regarded as representing a minor component; 300 further fibers are then checked from the next two stubs, and the amount of the minor component is reported in terms of its number percentage. This procedure is called "two-component analysis by number."

*Case 3*, that is, the quantitative analysis of binary blends, is the topic of this paper. *Case 3* applies when the number percentage of the second component on the first stub is equal to or above 3%. If five or more fibers have been found after checking 150 fibers, the sample is considered to be a blend. On each of six further stubs, 150 fibers are identified and their diameters are measured for the first twenty fibers of each fiber type (or of all fibers of that component, if less than twenty).

The weight percentage of the component identified as wool  $W_w^q$  is calculated by applying the Wildman/Bray (WB) formula [23]:

$$W_w^q = \frac{n_w(\bar{d}_w^2 + s_w^2)\bar{\rho}_w}{n_w(\bar{d}_w^2 + s_w^2)\bar{\rho}_w + n_s(\bar{d}_s^2 + s_s^2)\bar{\rho}_s} \times 100\% \quad (1)$$

where  $n_w$  = number of wool fibers,  $n_s$  = number of specialty fibers,  $\bar{d}_w$  = mean diameter of wool fibers,  $\bar{d}_s$  = mean diameter of specialty fibers,  $s_w$  = standard deviation for  $\bar{d}_w$ ,  $s_s$  = standard deviation for  $\bar{d}_s$ ,  $\bar{\rho}_w$  = mean density of wool fibers (1.31 g/cm<sup>3</sup>), and  $\bar{\rho}_s$  = mean density of specialty fibers (1.31 g/cm<sup>3</sup> for all specialty fibers except Angora rabbit hair, 1.15 g/cm<sup>3</sup>). The weight fraction of the specialty fibers  $W_s^q$  (e.g., cashmere, mohair. . .) is

$$W_s^q = 100\% - W_w^q \quad (2)$$

Equation 1 implies the assumption of circular cross sections for the component fibers. Due to the vacuum in the SEM, weight fractions of the dry fiber components can be determined. Following this procedure, 1050 fibers are thus identified for a sample, and 120 measurements of fiber diameter are made for each component.

In the previous paper [32], we analyzed in some detail the consequences of the thresholds separating the three cases, as well as defining critical parameters, such as the

number detection limit and the number quantification limit. Furthermore, we discussed in detail the specifics of the performance of IWTO-58 for Cases 1 and 2. That is, for various kinds of fiber admixtures of practical relevance, we determined their potential, maximum, and minimum numbers and their weight fractions for various analytical protocols.

The WB formula implies that for quantitative blend analysis, accurate identification of the component fibers is only one, though the most important, requirement for accurately determining their weight fractions. Taylor [22] states, "Quantitative measurements are always estimates of the values of the measure and involve some level of uncertainty. The measurements must be made such that the limits of uncertainty can be assigned with a stated probability. Without such an assignment, no logical use can be made of the data." Looking at the widespread practice of analytical laboratories, who report their blend analysis results without a measure of uncertainty, emphasizes the importance of the last sentence of the statement.

#### Precision

The uncertainty, that is, the *precision* of the result, for instance, for  $W_w$ , depends on the variabilities of the parameters of the WB formula. Staying in the tradition of Wildman and Bray's work [23], the confidence interval of the analysis result, determined at the 95% confidence level [34], is identified as a suitable measure of precision [24, 29, 31].

The three relevant contributions to the magnitude of the precision parameter are related to the variabilities of fiber counting and the diameters of the fiber components. The experimentally determined standard deviations are measures for diameter variability. Fiber count variability is given by the standard deviation of the binomial distribution [24]. Assuming the analysis steps to be stochastically independent, the rules of Gaussian error progression are applied to the WB formula (Equation 1) to yield, through partial differentials for the parameters, the value for the 95% confidence range  $q$  as a measure of precision [24, 29].

Giving detailed consideration to various aspects of error propagation [3, 31], we have developed a step-by-step calculation procedure to enable straightforward determination of the value of the confidence range for a given analysis appended to IWTO-58.

In a two-component blend, the confidence ranges are equal for both components, regardless of blend composition. The value of  $q$  yields the upper 95% limit for the confidence interval for the fraction of component  $i$  in a blend ( $W_i + q$ ) and the lower limit ( $W_i - q$ ). An alleged blend composition is regarded as being correct if

it falls within the confidence interval of the analysis. The use of  $q$  to specify a range for the wool or the specialty fiber weight fraction on a predetermined confidence level closely corresponds to the concept of *expanded uncertainty*, as specified in DIN V/ENV 13005 [7, 13, 31].

To document the practical expectation range for  $q$ -values, we calculated them for binary fiber blends of a 21  $\mu\text{m}$  wool ( $CV = 25\%$ ) with specialty fibers of mean diameters ( $CV = 20\%$ ) between 10  $\mu\text{m}$  (e.g., shah-toosh) and 40  $\mu\text{m}$  (e.g., coarse mohair). Figure 1 summarizes the  $q$ -values in a contour plot. Equal  $q$ -values are joined by iso-lines. The plot gives a view of the overall analysis precision for a wide range of fiber blends. The  $q$ -values decrease with a decreasing amount of the second blend component. The maximum follows a ridge in the range of the 50:50 composition by weight. A minimum occurs along the ridge for the case of equal mean diameters of the components.

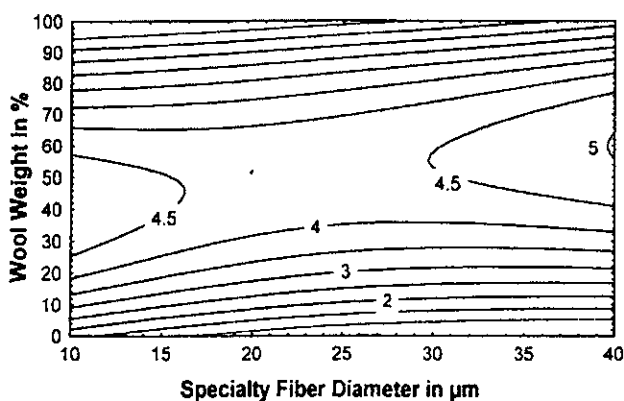


FIGURE 1. Least squares surface contour plot for the 95%-confidence range for wool weight percentages for blends of 21  $\mu\text{m}$  wool ( $CV = 25\%$ ) with a practical range of specialty fibers ( $CV = 20\%$ ).

Precision thus changes markedly with the geometric properties of the components and the blend composition. This characteristic of microscopic blend analysis is in pronounced contrast to chemical tests of fiber composition, and it indicates the principal differences between the factors controlling the analytical errors of the two methods.

Figure 1 further shows that the  $q$ -values approach 5% for 50:50 blends with very dissimilar fiber types in terms of mean diameter. Remember that there is a substantial range of blend ratios of primary commercial relevance where the test method design leads to precision limits that well exceed those limits of 3 weight-%. This can generally be considered as part of trade customs and legislative considerations [5, 6, 8].

To reflect the precision of the test method, we calculated  $q$ -values for a number of blends of the fiber types specified in Table I, chosen to cover even extremes in the range of practical relevance. The data are given in Table II, which is identical to Table A2 in IWTO-58 [12].

TABLE I. Various kinds of wool and specialty fibers, specified in IWTO-58 [12], to be used for evaluating their quantitative admixture levels in the microscopic analysis.

Fiber type	Acronym	Mean diameter, $\mu\text{m}$	CV, % <sup>a</sup>
Wool	W19	19	20
	W23	23	25
	W30	30	30
Specialty fiber	S16	16	20
	S23	23	25
	S35	35	30

<sup>a</sup> CV is the coefficient of variation given by  $CV = \text{standard deviation}/\text{mean diameter} \times 100\%$ .

TABLE II. Values for the 95% confidence range calculated for various blends of wool and specialty fiber types as specified in Table I.

Blend type	Ratio of wool/specialty fiber blends by weight-%				
	10:90	30:70	50:50	70:30	90:10
W19/S16	2.3	3.8	4.1	3.4	1.9
W19/S23	1.9	3.6	4.3	3.9	2.4
W19/S35	1.7	3.8	4.9	5.0	3.4
W23/S16	2.7	4.2	4.4	3.6	1.8
W23/S23	2.2	3.9	4.4	3.9	2.2
W23/S35	1.8	3.9	4.8	4.6	2.9
W30/S16	3.5	5.0	4.9	3.8	1.7
W30/S23	2.6	4.4	4.7	3.9	2.0
W30/S35	2.1	4.1	4.9	4.4	2.5

In view of the error progression of the WB formula, we suggest that the confidence limits can be reduced by increasing the numbers for fiber identification and diameter measurements. Figure 2 summarizes  $q$ -values for a 50:50 model blend by weight of wool (21  $\mu\text{m}$ ,  $CV = 25\%$ ) and cashmere (19  $\mu\text{m}$ ,  $CV = 20\%$ ) with increased numbers of identified fibers and for various cases of increased numbers of diameter measurements for both components. Courses of precision are specifically elucidated in Figure 2 for  $N = 100$  and 1000 by empirical lines through the data. The results show that a significant improvement of precision, that is, a decrease of  $q$ , can be achieved through substantial expansion of fiber identifications and diameter measurements. Increasing the number of diameter measurements from 100 to 300 already has a strong effect, which can further be enhanced by doubling or tripling the number of fiber identifications. For 1000 diameter measurements per fi-

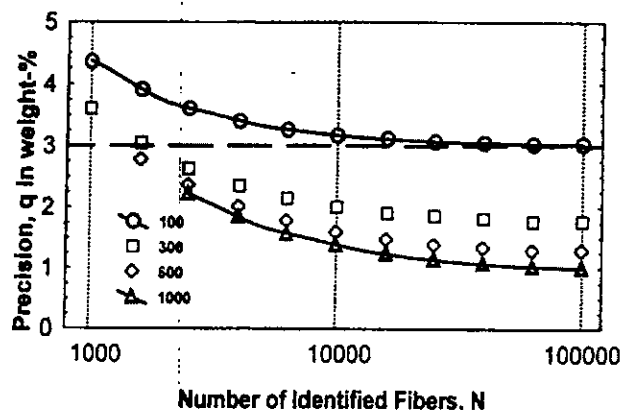


FIGURE 2. Changes in the precision of the analysis of a 50:50 wool/cashmere blend by weight (see text), when increasing the number of identified fibers and the number of diameter measurements for each fiber component from 100 to 1000 (see legend). Solid lines are empirical and meant as a guide for the eye. The 3 weight-% level, reflecting a technical as well as legal issues, is indicated.

ber type  $q$  approaches, for large numbers of fiber identifications ( $>100,000$ ), a value of about 1 weight-%.

For the standard procedure of IWTO-58, that is, 1050 fiber identifications and 120 diameter measurements, precision for the model blend is 4.2%. Considering a tripling of the analytical efforts, just technically feasible under special circumstances, the precision becomes 2.4% and thus drops well below the "magical" level of 3%.

#### Accuracy

Our test method proposes determining the height of the distal edges of cuticle scales on the fiber surface at a suitable magnification as a specific criterion for fiber identification. These heights are around  $0.4 \mu\text{m}$  for the specialty fibers and about  $0.8 \mu\text{m}$  for wool [25, 29]. For this test method, the threshold for fiber discrimination was introduced in IWTO-58 at  $0.55 \mu\text{m}$ .

Though emphasis is placed on cuticle scale height as a tool for fiber discrimination, special reference is made to the importance of other fiber surface characteristics, enabling the experienced operator to rapidly identify fibers. The specific role of cuticle scale height for the selectivity of the method has been emphasized by Robson [19], and is consistent with the characteristic differences in surface roughness observed for, e.g., wool and mohair [33]. Furthermore, even harsh chemical processing has only a very limited influence on the selectivity of the CSH criterion for fiber identification [15, 30]. Appendix B of IWTO-58 contains SEM photos of the most important textile animal fibers. The photos were chosen to show the typical appearance of the fiber types.

In view of the selectivity of the method, we have assumed that wool fibers on the one hand and specialty

fibers on the other can safely be discriminated so that no *a priori* systematic error enters the analysis result. Potential and practically realistic sources of analysis errors are systematic differences in the mean diameters, which are determined for the fiber components during the course of the analysis. Such biases may either result from differences in instrument performance or may be laboratory specific. However, since it is safe to assume that a diameter bias will affect both fiber types in the same way, the related effects will cancel out each other when introduced into the WB formula. An analytical bias may, in principle, be introduced if one fiber component is strongly noncircular and/or medullated through systematic errors in determining fiber diameters and by uncertain values for fiber densities. These effects have been shown to be only of very limited influence [26, 29]. Consequently, the WB formula is very robust.

Against the background of these considerations, we can assume that the issue of a bias originating from diameter measurements or density estimates is not critical for the accuracy (trueness) of the blend analysis. No systematic error is expected to enter an analysis result along this path.

The 95% confidence limits derived from the error progression of Equation 1 reflect the random errors occurring during analysis of a sample in a laboratory and are thus primarily a measure of precision.

The SEM method or any other method for animal fiber blend analysis can be used for samples for which, due to their preparation, the composition is known with much higher accuracy than that of the analysis method. Such samples can be considered as having a *correct composition* (reference samples). In a round trial with such samples, the analysis results of a laboratory are correct if the composition of such a sample is included in the confidence limits of the result.

Initial investigations conducted on this basis in our laboratory [24, 26] and various round trials conducted under the auspices of IWTO [29], the International Mohair Association [28], and CCMI [16, 17] have shown that analyses can consistently be conducted within the precision limits of the method. No bias is observed, and for this reason, no component of interlaboratory variance, either from random or systematic sources, is considered as an acceptable, additional component of analysis accuracy. The 95% confidence limits of an analysis result, corresponding to the concept of expanded uncertainty [7], are expected to cover unsystematic as well as minor though unspecified systematic errors [13], and are thus taken to represent the precision as well as the accuracy of the test method.

## Conclusions

Our aim of this and our previous [32] investigation is to take from various angles an in-depth look at IWTO-58, that is, at the background of its specifications. The protocol for IWTO-58 was devised by considering the current technical and economical feasibility of the method, with a perspective for the majority of practically relevant analytical problems, developed through intensive discussion at, for example, IWTO meetings [14, 16, 17, 18, 25, 31].

The most critical, legal, and technical issue of quantitative wool/specialty fiber blend analysis, specified as Case 3 in IWTO-58, is the requirement of 3 weight-% accuracy for the important U.S. market [8]. The results show that this is routinely out of reach of the current operator-based and thus rather tedious methodology. Note that this limitation applies equally to analyses based on light microscopy and following the relevant ASTM and AATCC standards [1, 2].

Our results emphasize the need for an automatic method, able to rapidly but safely identify and measure large fiber numbers, analogous to the successful implementation of, for example, automated wool fiber diameter measurement systems [10, 11]. Various steps in this direction have been taken by various authors and with very variable success, using combinations of microscopy and image analysis together with statistical techniques [4, 19, 20, 21, 27].

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**CERTIFICATE OF ELECTRONIC SERVICE**

I hereby certify that on the date set forth below, I electronically filed the foregoing Declaration of Joshua R. Slavitt with the Clerk of the Court using the CM/ECF system which will send notification of such filing to the following:

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