

## **Notes on the Determination of the Fibre Fineness of a Merino Wool Sample.**

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THE technique adopted in South Africa for determining the fibre fineness of a sample of wool, although essentially that described by Duerden (1929), has certain modifications which are here outlined.

In the present contribution it is intended to deal with the method of preparing a wool sample for mounting and with its measurement. The results dealing with the sampling of the merino fleece, both on and off the live sheep, will be dealt with in another paper.

### PREPARATION OF THE SAMPLE FOR MOUNTING.

(a) *Preliminary degreasing of sample.* The greasy sample is washed in cold benzene so as to remove most of the impurities. The other method of mixing is subsequently used and this removes the residual impurities.

(b) *The cutting of the fibres for mixing.* Owing to the variability in fibre fineness along the length of fibres, it is necessary that an average of several points along the fibre bundle be taken, for which purpose the method described by Duerden (1929) is being used. It consists in either cutting the sample into fragments along the entire length of the fibre bundle, or else, removing the fragments at equal intervals of approximately a  $\frac{1}{4}$  inch along the length. Malan, Carter and van Wyk (1938) showed that there was no difference between the two methods in the case of a small sample which was cut at three points and also along its entire length.

Methods for cutting the fragments have also been investigated and those of other workers tested. Wildman (1936), Wildman and Daniels (1937) and later Malan, Carter and van Wyk (1938) gripped the fibre bundles between strips of paper. Huberty (1938) described a method of cutting fragments with a razor blade, a method which it is claimed, results in shorter and more uniform fragments. This method was tested and it was found difficult to cut, with a single

stroke, all the fragments from one region, an essential condition in the case of raw wool, as will be clear from the following considerations.

It has been shown (Bosman, 1937), that such factors as differences in the plane of nutrition of the sheep, period of lactation of the ewe, etc., can cause marked differences in the fibre fineness of different regions of the staple. Where fragments are therefore removed at equal intervals from the staple, care is necessary to ensure that all regions contribute the same number of fragments to the total.

After the fragments have been cut at intervals, they can quite easily be clipped into shorter pieces with the aid of a pair of finely pointed scissors, but for raw wool this method has the disadvantage that the originally cut longer fragments probably contribute a greater proportion of smaller fragments to the total than do the shorter original fragments, and, if longer fragments are cut in any special region, the result is biased in favour of that region. If, with Huberty's method it is not possible to cut the ultimate fragments for mounting with a single stroke of the razor blade so as to ensure an equal number of fragments from each region, the method is not suitable for raw wool.

In routine practice, the method of gripping the wool bundle between the fore-finger and thumb has given very good results and the method has been adopted for general use in our laboratories. With practice, and the necessary care, routine assistants are able to use this method to better advantage than has been the case with other methods. It has also been found that uniform short fragments give a better mixture than do the uneven longer ones and very good results have been obtained with a little training. Slides made by this method have shown an even fibre distribution without any fibre clusters on the slide.

There is a limit to the shortness of cutting the length of fibre fragments, since the scissors distort the ends of the fragments. It has been found that fibre lengths of from  $\frac{1}{3}$  to  $\frac{1}{2}$  millimeter can be cut with ease and give satisfactory results.

The objection to this method, that several fragments from the same fibre are included and consequently that the same fibre may be measured more often than once, has been investigated. It was found that the bundle contains many thousands of fibres while the number of points at which fragments are taken seldom exceeds 10, so that the chance of measuring the same fibre more often than once is exceedingly small.

#### MOUNTING THE FRAGMENTS FOR MEASUREMENT.

The cut fragments are transferred to a test tube of about 2 cm. diameter and 6 cm. deep containing ether. The fibres are shaken, giving a uniform dispersion of fragments which are allowed to settle. Successive clumps, taken at random by finely pointed forceps are placed in rows on a black cardboard where the ether is allowed to evaporate.

A thin layer of Euparal mounting medium\* (which has been found to be superior to other mounting media such as Canada Balsam, Cedar wood oil, etc.), is spread on a glass slide and the clumps of fragments, taken at random from the cardboard, stirred by the aid of dissecting needles, into the Euparal until the fragments are well distributed.

A cover slip is finally pressed down over the slide, excess Euparal being removed at the edges of the cover slip by means of blotting paper. A few fragments of fibres are removed with the excess Euparal, but the remainder is sufficiently representative of the original. With practice it is possible to use just the right amount of Euparal for a minimum loss of fibre fragments and also for a complete absence of air bubbles in the slide.

The question as to whether successive clumps of fragments taken from the test tube are similar, has been tested. Each successive clump that was placed on the cardboard, was mounted on a slide and measurements taken. The results are summarised in Tables 1 and 2.

TABLE 1.  
*The Fibre Fineness of successive Clumps taken from the Ether Test Tube.*

Clump No.	Mean Fibre Fineness. $\mu$
1.....	17.45
2.....	17.55
3.....	17.76
4.....	17.69
5.....	17.52
6.....	17.81
7.....	17.68
8.....	17.97
9.....	17.51
10.....	17.32
20.....	18.13
30.....	18.11
40.....	17.98
50.....	17.97

TABLE 2.

Variance.	Standard Dev.	Loge S.D.	Z.
Between slides.....	2.0215	.7041	.2659
Within slides.....	1.5503	.4382	( $n_1 = 13$ ) ( $n_2 = 3486$ )

\* The Technical Committee of the International Wool Textile Organisation at its 1939 meeting decided that for routine purposes not requiring the preparation of permanent slides, cedarwood oil was preferable.

This aspect has, however, been carefully investigated in our laboratories and it has been found that for studies of wool from production aspects, such as in breeding experiments and others, where permanent slides for reference purposes are essential, the use of Euparal is preferred. Furthermore, cedarwood oil does not give the same clear fibre outline as Euparal.

According to Fisher, the analysis of variance indicates no significant difference between the variances between and within slides. The fact that several clumps, taken at random, made up one slide, justifies this method of preparing slides.

An alternative method of mounting fibres was described by Malan, Carter and van Wyk (1938). The fragments were mixed in ether in a shallow dish, excess ether was poured off and the remaining ether allowed to evaporate. Clumps of dry fibres were shaken out by tapping the cut fragments over the slide that had a thin layer of Euparal. Another thin layer of Euparal was then placed on the cover slip which was pressed down over the slide. It was found that, by this method, fragments of fibres were prevented from being forced out with excess of Euparal.

While this method gave satisfactory results, it has not found favour in our laboratories, the reason being that dry fibres are difficult to handle this way, and there is a possibility that the lighter, finer fibres may be blown away during the tapping process.

#### HUMIDITY.

All slides are mounted in a room maintained at constant humidity and temperature, and in this way variations due to swelling of the fibres are eliminated. Tests were, however, made for conditions outside the Constant Humidity Chamber, so as to be applicable for cases where the controlled conditions were not available. The test, carried out over a period of 19 days, consisted of cutting up a sample of wool into fragments and mixing in ether. Clumps of fragments were placed on a black cardboard and dried in a desiccator for a few days. The clumps of fibres were then placed in a room where the relative humidity was determined at intervals by a whirling hygrometer. Two slides were prepared from the fragments at different humidities and, as a check on the moisture content, a standard sample, which had been washed in ether, was weighed in proximity to the fragments. The results are given in Table 3.

TABLE 3.

Relative Humidity.	Temperature.	Mean Fineness ( $\mu$ ).	Regain of Standard Sample.
Per cent.			Per cent.
13.....	32° C.	20.4	6.8
18.....	27° C.	20.2	8.6
30.....	29° C.	20.0	9.6
60.....	25° C.	20.5	12.8
70.....	21° C.	20.6	16.2
76.....	22° C.	20.8	16.9
80.....	22° C.	21.0	17.9

The difference between the values at 13 per cent. and 18 per cent. R.H. is not significant but the difference between the values at 13 per cent. and 30 per cent. R.H. is just significant at the 5 per cent. probability level. Such irregularities may be ascribed to

several causes, such as the large variations in temperature, the fact that some humidities were reached under adsorption conditions and others under desorption conditions, and the lag in taking up or losing moisture at each humidity. The results show the necessity for preparing the slides under controlled conditions.

#### THE INFLUENCE OF ETHER.

It has been stated by Daniels (1938) that the values obtained from permanent slides made from ether and Euparal were consistently lower than those made from cedar oil, and this was attributed to the contraction of the fibres by ether.

A series of tests, using different treatments and mounting media, was therefore carried out.

A sample was cut into fragments which were mixed in ether. Fibre clumps were withdrawn from the mixture and laid on a black cardboard in the usual way. When the ether had evaporated, every second clump was placed in a test tube containing distilled water. The fragments were mixed and shaken in water until all had been thoroughly wetted. Clumps were then withdrawn and laid on a card. Both sets of clumps, the ones mixed in ether only and the ones mixed in ether and water, were placed in a desiccator for 24 hours and then exposed to an atmosphere of constant humidity and temperature. Slides were made of each set of clumps, using Euparal, for the measurement of 500 fibres from each set. This procedure was followed with two samples, the results being summarised in Table 4.

TABLE 4.

Sample.	MEAN FINENESS ( $\mu$ ).		Difference ( $\mu$ ).	S.E. of Diff. ( $\mu$ ).	" t " = Diff./S.E. of Diff.
	Mixed in Ether.	Mixed in Ether and washed in Water.			
1.....	20.012	19.732	0.280	$\pm$ 0.2385	1.174
2.....	17.292	17.152	0.140	$\pm$ 0.1852	0.756

The values obtained from fibres mixed in ether are not significantly different from those mixed in ether and water (when  $n=500$ ).

A further series of tests based on 15 pairs of slides, in which some fragments had been mixed in ether only and some in water only, was carried out. The results are summarised in Table 5.

TABLE 5.

MEAN FIBRE FINENESS OF SLIDES.		Difference.	S.E. of Diff.	" t ".
$\mu$	$\mu$			
Mixed in Ether.	Mixed in Water.			
20.696	20.411	0.285	$\pm$ 0.075	3.80

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These results show a significant difference between the fibres mixed in ether and those mixed in water. It is shown that the fibres mixed in ether are 1 per cent. thicker than those mixed in water and it appears that the ether caused the fibres to swell.

Tests were also made on fibres mounted in cedar wood oil and those mounted in Euparal. The results are given in Table 6.

TABLE 6.

Treatment.	Mean Fibre Fineness ( $\mu$ ).		Difference.	S.E. of Difference.	"t".
	Mounted in Euparal.	Mounted in Cedar Wood Oil.			
Mixed in ether....	20.696	20.406	0.290	$\pm$ 0.1201	2.84
Mixed in water....	20.411	20.281	0.130	$\pm$ 0.0576	2.26

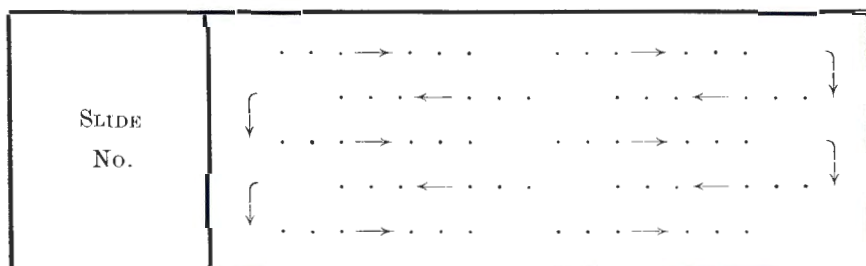
In both cases the fibres mounted in Euparal gave significantly higher values than those mounted in Cedar wood oil, the difference again being of the order of 1 per cent. These figures also suggest that, when slides are made with fibres mixed in ether and mounted in Euparal, the results are about 2 per cent. higher than those mixed in water and mounted in Cedar wood oil. In all these tests the fibres were dried in a desiccator before being exposed to the constant humidity conditions, so that swelling took place under conditions of adsorption.

THE MEASUREMENT OF THE FIBRES.

The measurements were made on Zeiss Lanameters. The magnification on the screen of each instrument was found to vary from the centre outwards and it was necessary to use only an area that could be accurately standardised. A circle of radius 4 cm. was drawn in the centre of the screen, the magnification of fibres falling within this circle being adjusted to exactly 500. In routine measurements only fibres lying within this circle are measured.

Each slide containing the wool fragments is systematically read in the manner shown in Figure A. Dotted lines represent the regions where fibres are measured while arrows indicate the direction in which the slide is moved.

FIG. A.



There are ten regions of measurement and one tenth of the desired number of readings are taken in each region. By this method measurements are made over the whole slide.

As a check on the uniformity of the distribution of the fragments over the slide, the results are recorded in successive groups of 25 measurements.

An analysis of variance is made comparing the variance between groups of 25 with the variance within groups of 25. Cases which occurred in a sampling experiment during the measurement of 10 slides are shown in Table 7.

TABLE 7.

*Comparison of Variance between and within Groups of 25 Readings.*

Variance.	D.F.	CASE 1. CASE 2. CASE 3. CASE 4. CASE 5.				
		Mean	Mean	Mean	Mean	Mean
		Square.	Square.	Square.	Square.	Square.
Between groups of 25 readings...	99	4.8411	4.1002	3.6678	4.0997	4.5157
Within groups of 25 readings...	2,400	4.3529	4.1076	4.3512	3.9925	4.2757
TOTAL.....	2,499	4.3722	4.1073	4.3241	3.9967	4.2852

These results show that the fineness distribution of the fragments over the slide is satisfactory.

In routine practice, duplicate slides are usually made and half the total number of readings taken from each slide. The difference between the slides has to conform to the demands of statistical theory.

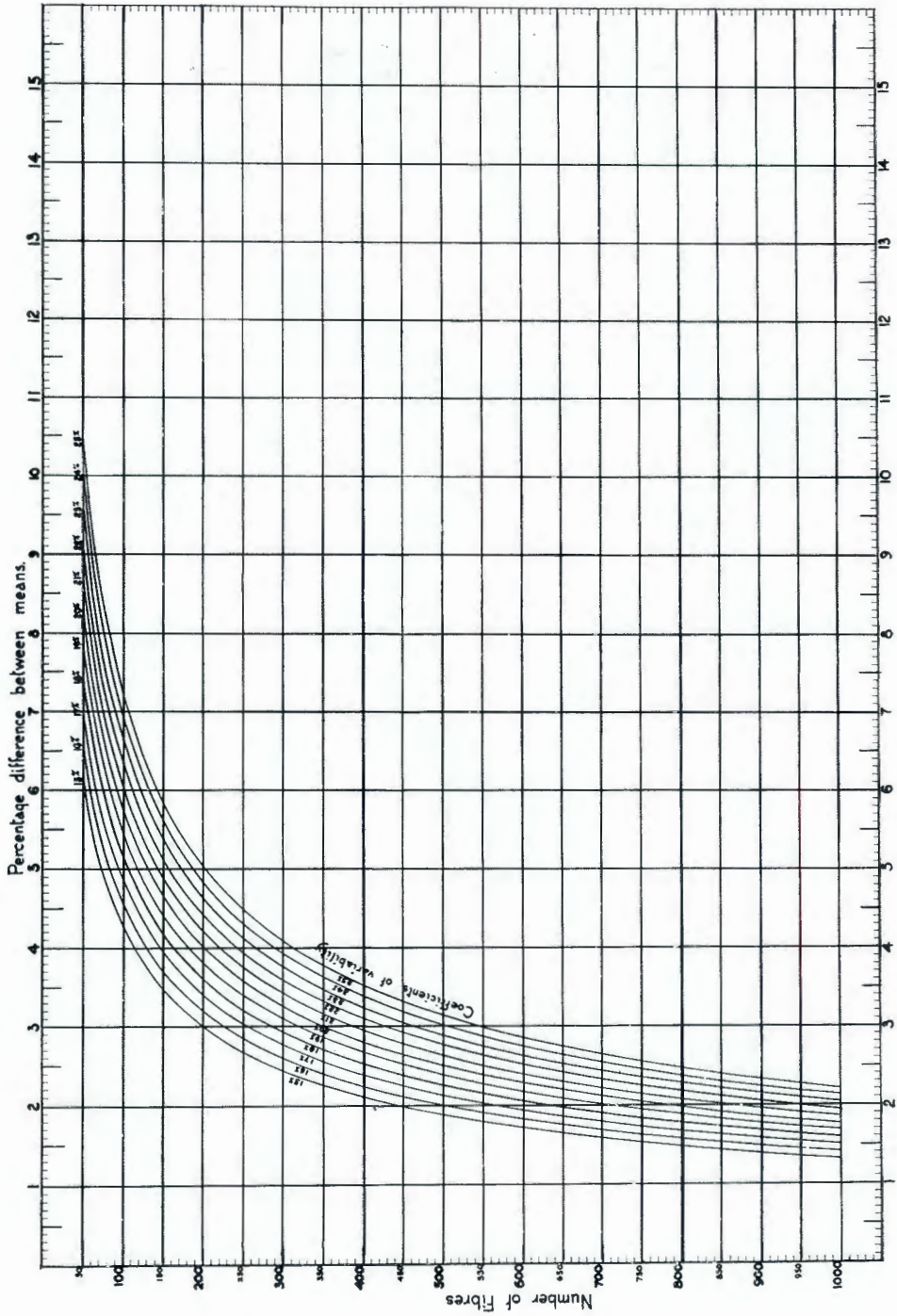
#### THE NUMBER OF FIBRES MEASURED.

Since the number of measurements necessary from any one sample depends on the variability within the sample, the statistical constants serve as a guide for determining the number of measurements to be made. It has been shown that for Merino wool, 250 measurements are generally sufficient to bring the permissible error to within 3 per cent. of the mean and although many of the samples need less than 250 measurements, it is simpler and quicker to measure 250 straight off than to measure a number less than this for a preliminary statistical test.

The procedure thus is to measure 250 fibres on all Merino samples and then work out the statistical constants. It is very seldom that more than this number need to be measured.

A convenient chart (Fig. B) showing the relationships between the statistical constants and the number of fibres necessary for measurement is used and has proved very valuable for routine work.

Fig. B.—The relationships between the statistical constants and the number of fibres necessary for the measurement of a sample of merino wool. The values are given for a 5 per cent. probability level of significance.





## THE HUMAN ELEMENT AMONG OBSERVERS.

Serious differences have been noticed among different observers, and special investigations on this aspect have been undertaken in our laboratories. It has been observed that although differences among observers are often constant, they are not necessarily always so.

Three sets of slides measured by four observers are here summarised, each observer measuring 125 fibres from each slide.

The results are given in Tables 8 and 9.

TABLE 8.  
*Mean Fibre Fineness ( $\mu$ ).*

	OBSERVERS.			
	A.	B.	C.	D.
1st Set (26 slides).....	18.84	18.46	18.76	18.69
2nd Set (43 slides).....	27.14	26.67	27.31	27.04
3rd Set (36 slides).....	23.62	22.82	23.92	23.65

TABLE 9.  
*Differences between Observers A, B, C and D (in  $\mu$ ).*

	B.	C.	D.
A. 1st Set.....	<b>0.38</b> $\pm$ 0.0907	0.08 $\pm$ 0.0661	0.15 $\pm$ 0.0765
A. 2nd Set.....	<b>0.47</b> $\pm$ 0.0942	-0.17 $\pm$ 0.1194	0.10 $\pm$ 0.1242
A. 3rd Set.....	<b>0.80</b> $\pm$ 0.0847	- <b>0.30</b> $\pm$ 0.0599	-0.03 $\pm$ 0.0533
B. 1st Set.....		- <b>0.30</b> $\pm$ 0.0676	- <b>0.23</b> $\pm$ 0.0769
B. 2nd Set.....		- <b>0.64</b> $\pm$ 0.1263	- <b>0.37</b> $\pm$ 0.1280
B. 3rd Set.....		- <b>1.10</b> $\pm$ 0.0954	- <b>0.83</b> $\pm$ 0.0794
C. 1st Set.....			0.07 $\pm$ 0.0496
C. 2nd Set.....			<b>0.27</b> $\pm$ 0.0921
C. 3rd Set.....			<b>0.27</b> $\pm$ 0.0450

Significant differences are in black type.

The results show that there are significant differences between observers except between A and D, where the differences are not significant. It is also shown that the differences are not constant.

An important fact, that was also established, is that, when slides are measured continuously without the observers having a change on to some other work and without a periodic check on the observers,

serious differences can develop. It is shown in the tables that, in general, there is a tendency for difference between observers to be greater in the third set of slides except between observers A and D. A closer study of the results has led to the conclusion that a lack of concentration developed during the reading of the third set. This was largely attributed to the factor of fatigue.

Special tests were also made on the systematic differences between observers. A hundred synthetic fibres were mounted parallel across the slide and the four observers were made to measure the hundred fibres carefully at the same spots. The averages of three such sets of readings, each taken at a different level of the slide, are summarised in Table 10.

TABLE 10.

*Differences between Observers on Standard Slides (in  $\mu$ ).*

	B.	C.	D.
A.	$\left. \begin{array}{l} + 0.26 \\ 0 \\ - 0.15 \end{array} \right\} 0.04$	$\left. \begin{array}{l} 0.04 \\ 0.14 \\ - 0.27 \end{array} \right\} -0.12$	$\left. \begin{array}{l} + 0.28 \\ - 0.06 \\ - 0.02 \end{array} \right\} -0.11$
B.		$\left. \begin{array}{l} - 0.22 \\ - 0.14 \\ - 0.12 \end{array} \right\} -0.16$	$\left. \begin{array}{l} - 0.02 \\ - 0.06 \\ - 0.13 \end{array} \right\} +0.06$
C.			$\left. \begin{array}{l} - 0.20 \\ - 0.20 \\ - 0.25 \end{array} \right\} +0.22$

The results of Table 10 show that the differences between observers were small and insignificant and a closer study of the observers showed that differences to a large degree depended on the fact that, when the edges of the fibres, presented for measurement, fell midway between the scale divisions on the lanameter, judgment as to which division the measurement belonged, differed among individuals. Such differences may possibly be reduced by the use of smaller scale divisions and larger magnifications, but then in the case of Merino wool this would tend to make measurements more difficult because of the serrated edge of the wool fibres.

In order to minimise differences between observers in our department dealing with fibre fineness measurements, a system was introduced whereby all measurements were made by pairs of observers, one of the pair taking half the required number of readings on a slide, while the other enters the readings, the pairs being chosen in such a way that the averages of the measurements of each individual of the pair counterbalances differences between the pair. It was found that the effect of the factor of fatigue was considerably reduced by each pair measuring the 125 readings in turn. Frequent intervals of rest are also prescribed in the form of changes to some other type of work.

The results now obtained from different pairs of assistants are very satisfactory, differences among observers being reduced to a minimum.

The results of 42 slides are given in Table 11.

TABLE 11.

*Differences between Observers—Averages of 42 Slides (in  $\mu$ ).*

	B.	C.	D.
A.	0.030 $\pm$ 0.0737	0.015 $\pm$ 0.033	0.093 $\pm$ 0.0657
B.		0.015 $\pm$ 0.0691	0.123 $\pm$ 0.0879
C.			0.108 $\pm$ 0.0693

These differences are below the possible error due to sampling. The system whereby pairs of observers take measurements of the same slide serves as a check on the observers and a system of entry of the results has been instituted whereby the results can be summarised at a glance.

In addition, duplicate slides are usually made of the same lot of fragments and it was found that, when one observer of a pair makes half the required number of measurements from one slide and the other observer the remainder on the other slide, the making of the slides as well as the observers are under control.

#### THE WEIGHT-LENGTH METHOD OF DETERMINING FIBRE FINENESS.

It has been suggested (Wilsdon, 1938) that in cases where errors are likely to occur between the measurements of different observers, the weight-length method should be used for arbitration purposes.

A comprehensive set of tests was carried out on samples of Merino wool for ascertaining the relative merits of the weight-length method and the projection or microscope method.

The following conclusions were arrived at:—

1. The weight-length method depends on determinations of fibre length as straight fibre length. In this respect tests have shown that different observers obtain different results on the same sample of wool and even on the same fibres, due to a personal element. Because of this the weight-length method cannot be said to be more reliable than the projection method.

2. In the weight-length method (Roberts, 1930) it is assumed that the specific gravity of all wool is constant at 1.30. It has, however, been found that there is a variation of 1.3 per cent, in the specific gravity of South African wool (van Wyk and Nel, 1939). On a Merino wool, therefore, with a fibre fineness of  $20\mu$ , errors of up to  $0.14\mu$  may accrue from the assumption that all wool has the

same specific gravity. The weight-length method is, therefore, at a disadvantage unless the specific gravity of each sample analysed, is also determined. This, however, is impracticable.

3. All wool samples do not adsorb moisture to the same degree (van Wyk, 1939) hence the use of a standard sample for dry weight determinations will give rise to error.

4. The weight-length method gives a weight-biassed result (Roberts, 1930) while the microscopic or projection method gives a length-biassed result (Wildman, 1936) and it has been found that the two methods do not give the same measurement.

The weight-length method is based on the dry fineness of the fibres when the dry weight is obtained. Determinations on 56 Merino wool samples by the two methods showed that the microscopic method gave values which were 5 per cent. higher than those obtained by the weight-length method, the latter being on the dry weight basis. This difference is presumably due to the swelling of the fibres with adsorption of moisture.

If the weight used is based on a certain humidity and temperature, errors will be introduced by differences in the specific gravity, moisture adsorption and swelling of the different types of wool, factors which have to be controlled where extreme accuracy is needed.

5. In the case of Merino wool the average weight of a fibre from a 3-inch staple is about .04 milligrams. This necessitates the counting of a large number of fibres for the weight-length determinations, to ensure any degree of accuracy in the weighing.

#### SUMMARY.

The technique for determining the fibre fineness of a sample of wool as used in South Africa is outlined.

The methods of cutting the fibre bundle into fragments and the mounting of these on a slide are described.

The effect of relative humidity during the preparation of the slide shows the necessity for controlled moisture and temperature conditions.

The effect of the ether used for mixing the wool fragments and the Euparal mounting medium are described.

The method of measurement, the system of recording the results and the number of fibres to be measured in Merino wool are recorded.

The question of the personal errors of different observers has been studied. The differences between observers are not necessarily constant, the factor of fatigue being of special importance. Methods for reducing these errors to a minimum are described.

The weight-length method for determining fibre fineness, suggested for arbitration purposes, is discussed and difficulties peculiar to this method are enumerated.

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