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FUNDAMENTAL PRINCIPLES OF FIBRE FINENESS MEASUREMENT

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This document is a collation of a series of articles by the author originally published in the AWTA Ltd Newsletter. This document was expanded as each additional article was published. The intention of this series is to provide a single comprehensive resource on this most important topic – most important because fibre fineness is the primary determinant of the value of wool. The series is now complete

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THE TECHNICAL & COMMERCIAL REQUIREMENTS OF

WOOL TESTING SYSTEMS

Objective measurements now provide the primary information used to determine the market value of greasy wool. They ensure that wool producers get paid a fair price and that processors are able to purchase greasy wool and then manufacture tops, yarns and fabrics of a specified quality.

The important parameters that are now measured and certified for most of Australia's wool are:

- Wool Base;
- Vegetable Matter Base and Hardheads & Twigs;
- Mean Fibre Diameter & Coefficient of Variation of Diameter; and
- Staple Length, Strength & Position of Break.

A small proportion of the clip is also certified for colour. Other, non-certified information, such as curvature and vegetable matter base, is also available.

Table 1: Objective Criteria for Comparing Testing Systems

Criterion	Numerical Measure
1. Precision	Absolute Standard Deviation Relative Standard Deviation Coefficient of Variation Variance
2. Bias	Absolute Systematic Error Relative Systematic Error
3. Sensitivity	Calibration Sensitivity Analytical Sensitivity
4. Detection Limit	Blank plus 3 times the standard deviation of the blank
5. Range	Limit of quantitation (LOQ) to limit of linearity (LOL)
6. Selectivity	Coefficient of Selectivity

The Test Methods and associated technologies for determining these parameters have been developed and refined over the last 30 years by the International Wool Textiles Organisation. This has involved technical input from engineers and scientists from all around the world, and commercial input from wool producers, wool agents, wool buyers, wool traders and wool processors, thereby ensuring that the Test Methods are technically sound while at the same time meeting, as far as is reasonably practicable, the commercial requirements.

The technology used for IWTO Certification has also found application in providing information for selecting animals, although the testing systems or protocols used have not been standardised.

Increasingly, alternative technologies for measuring some parameters are becoming available, and expenditure for research into and development of as yet unknown but hopefully less expensive new technologies is also being considered. Before the commercial implications of using these new technologies can be understood it is necessary to understand the criteria (Table 1) for establishing their equivalence to those they are designed to complement or supplant.

Sampling - the Number One Issue

Objective determination of defined characteristics of materials usually involves measurements based on a small proportion of the total material of interest. In materials that are **homogeneous**, obtaining a representative sub-sample of the whole is a relatively simple problem. Where there is **heterogeneity**, obtaining a sub-sample that is representative of the whole is a much more difficult task.

TABLE 2: Defining the Precision of Analytical Methods

Terms	Definition
Absolute Standard Deviation	$s = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$
Relative Standard Deviation	$RSD = \frac{s}{\bar{x}}$
Standard Deviation of the Mean,	$s_m = \frac{s}{\sqrt{N}}$
Coefficient of Variation	$CV = \frac{s}{\bar{x}} \cdot 100$
Variance of the mean	s_m^2
Confidence Level (95%)	$CL = \pm 1.96\sqrt{s_m^2}$

x_i = numerical value of the i^{th} measurement

$\bar{x}$ = the mean of N measurements = $\frac{\sum_{i=1}^{N} x_i}{N}$

Wool is clearly a **heterogeneous** material, both in the bulk or when still on the sheep's back. The sampling procedures for sale lots or consignments of wool have been carefully developed to ensure that the sample represents the bulk with a predictable degree of error. The requirements for sampling the bulk also extend to further sub-sampling of the sample itself, in order to measure a specific characteristic. The theory and practice of these sampling regimes will not be considered in detail here. Suffice to say the same theory and practice must also be applied when sampling individual animals.

Generally, modern analytical instruments provide increased speed, more ease and convenience of use, and often less skill is required of the operator. However, particularly in the case of analysis of greasy wool, results provided by such instrumentation are diminished in value unless an appropriate sampling regime is defined and strictly followed. Sampling is the first and most important step in any wool testing system.

Precision

Precision describes the reproducibility of results - that is, the agreement between numerical values of two or more replicate measurements, or measurements that have been made in exactly the same way. Generally, the precision of a testing system can be obtained simply by repeating the measurement, using the same technique, a number of times.

Precision is often confused with **accuracy**. Accuracy simply describes the correctness of a result. Strictly speaking, the only type of measurement that can be described as completely accurate is one that involves counting objects. **All other measurements contain errors and are really only approximations or estimates**.

Three terms are widely used to describe the precision of a set of replicate data:

- standard deviation,
- variance and
- coefficient of variation.

These terms have statistical significance and are defined, together with some related terms, in Table 2.

The main objective in standardising any testing system is to ensure a predictable and commercially acceptable precision of the measurements.

Bias

Analysts are concerned with two types of errors:

- random or indeterminate errors; and
- systematic or determinate errors.

The error in the mean of a number of replicate measurements is equal to the sum of these two errors.

Random or indeterminate errors impact upon precision. Bias may have little or no effect on precision, but it has a significant effect upon accuracy.

Bias is a result of systematic or determinate errors. Systematic errors always act in one direction, resulting in a consistently larger or a consistently smaller result than that provided by the reference measurement. In general, bias can only be determined by reference to measurements provided by primary measurement systems (i.e systems based on direct reference to primary metric standards such as length and weight). Bias can exist between measurements provided by secondary measurement systems (systems calibrated against primary systems), but unless the bias can be confirmed by reference to a primary measurement system, the analyst may never be sure whether one or both of the secondary measurement systems are responsible for the bias. Bias can result from several causes, and generally, these can be classified into one of six groups.

- **Sampling**: Inadequate design of sampling systems can result in a sample that is biased. A biased sample may still be useful depending on the intended use of the measurements made on the sample. Samples taken from a defined location on sheep will almost certainly be a biased representation of their fleeces. If the purpose of these samples is to obtain information to assist in ranking sheep for breeding purposes, the bias can be acceptable, provided it is similar across all sheep to be ranked. However, if the purpose of the samples is to obtain information to predict characteristics in classed lines of wool produced from the sheep, then the bias may be unacceptable.
- **Differences in fundamental assumptions**: In the case of wool fibre fineness, different assumptions about the geometry of the fibre by different instrumental methods, may lead to bias.
- **Personal Errors:** Bias can also be the result of blind prejudice. Most of us, however honest, have a natural tendency to estimate scale readings in a direction that improves the precision of a set of results, or causes the results to fall closer to a preconceived notion of the true value. When sampling wool this source of bias is particularly important. Measurement of staple length and strength requires the selection of a representative set of wool staples. In the early stages of the development of the IWTO Test Method, it was observed that staff with wool knowledge generally selected a set of staples that were longer than those selected by staff with little or no wool knowledge.
- **Instrumental Errors:** Bias can be caused by instrument drift, or by assumptions made by the technology used in the instrument. The OFDA 100 instrument, used for determining the mean fibre diameter distribution characteristics of wool has been shown to exhibit biases in either Mean Fibre Diameter or Standard Deviation of Diameter, depending upon how the calibration samples are prepared. The instrument must use separate calibration systems for unbiased estimates of either parameter.
- **Method Errors:** An example of this type of bias is the failure to maintain rigid control over the environmental conditions that impact upon the measurement (for example temperature and humidity, or measuring fibre diameter without removing attached grease, wax and suint).
- **Interferences**: Bias can also be caused by interferences that arise from the constituents of the sample. In fibre measurements, where most methods use physical measurement techniques, bias from this source is unlikely, provided the sample is prepared appropriately. In the case of fibre diameter, the presence of extraneous material such as a synthetic fibre, or very fine vegetable matter, is an example of this effect.

Bias may be constant over the range of variation of the characteristic being measured, or it may vary over this range. One of the objectives of standardising wool testing systems is the elimination or at least the

minimisation of bias. Where bias cannot be eliminated, provided it is not level dependent, the measurement technology may still be useful.

Sensitivity

Sensitivity of an instrument or a testing system refers to its ability to discriminate between small differences in the material being analysed. In wool testing three factors limit sensitivity:

- the slope of an instrument's calibration curve;
- the precision of the instrument; and
- the error in the sampling system.

If two instruments have equal precision the one having the steeper calibration curve will have the greater sensitivity. Conversely if two instruments have calibration curves with identical slope, the one having the greater precision will have the greater sensitivity. In testing wool, the errors arising from sample variation are generally so large that they mask any differences in sensitivity between measurement instruments.

Detection Limit

The detection limit is a minimum value of the characteristic being measured that can be detected at a known confidence level. This is not an important issue, for example, when measuring mean fibre diameter, because wool fibres never approach zero fineness, and most measurements are conducted within ranges that exceed the probable detection limit by factors greater than three. However, if attempts currently underway to produce ultra fine flocks succeed (see May 2001 Newsletter) then this may become an increasingly important factor. It is already a very important factor to be considered in developing instruments to measure dark fibre contamination in wool, because the minimum quantity of such fibres generally considered to be important is extremely low.

Range

The useful range of an analytical method can be defined as the lowest point at which a measurement can be made (the detection limit or the LOQ), to the point at which the calibration departs from linearity (LOC). However, some measurement systems have non-linear calibration functions. The useful range in these instances is more difficult to define.

Selectivity

Selectivity refers to the degree to which the analytical method is free from interferences by other species in the sample matrix. This is generally not a major issue when testing wool. However, as indicated previously, it may be an issue for measurement of fibre diameter if extraneous synthetic fibres or very fine vegetable matter is present in the sample.

Equivalence of Testing Systems

In qualitative terms two wool testing systems can be said to be technically equivalent provided they have the same overall precision (encompassing sampling and measurement), the same bias, the same sensitivity, the same detection limit, the same selectivity and operate over the same range. From a commercial perspective the same criteria will apply.

This does beg the question of how "sameness" is to be determined. However, as indicated in Table 1, each of these characteristics can be quantified.

The capability of any new and as yet undiscovered technological systems for measuring the commercially important characteristics of greasy wool must also be judged against these criteria.

UNDERSTANDING FIBRE DIAMETER MEASUREMENT

FUNDAMENTAL CONCEPTS

In the next review in this series we briefly describe the various technologies that the wool industry has explored for measuring the fibre diameter of wool. In later reviews we intend to provide more detailed discussion of these technologies, particularly those that have found commercial use. However, before doing so we need to set the scene by defining quite clearly what we mean when we talk about wool fibre diameter.

The language of today's wool industry employs the term **diameter** to describe a characteristic once described as **fineness**. The word "diameter" is derived from the Greek word "*diametros*", consisting of the prefix "*dia*" (through or across) and "*metron*" (measure). Its common meaning in English is "*a straight line passing from side to side through the centre of a body or figure, especially a circle or a sphere*". In a more general context "diameter" can mean a transverse measurement, width or thickness. In geometry the term "diameter" is exclusively used to describe the maximum transverse dimension of a circle or a sphere.

Wool fibres are not circular in cross-section. The cross sectional shape is irregular. Some fibres are nearly circular, some are roughly elliptical, some are ovoid, and some can be visualised as elongated ovals or shapes that approximate ovals with concavities (Figure 1). The most common geometrical shapes that are ascribed to wool fibre cross-sections are circles or ellipses. The technical literature is replete with both terms, particularly since 1950. It is clear that this is a simplification of the reality (Figure 2). At best, the cross-sectional shape can be described as a circle that has been deformed to differing degrees about its radii.

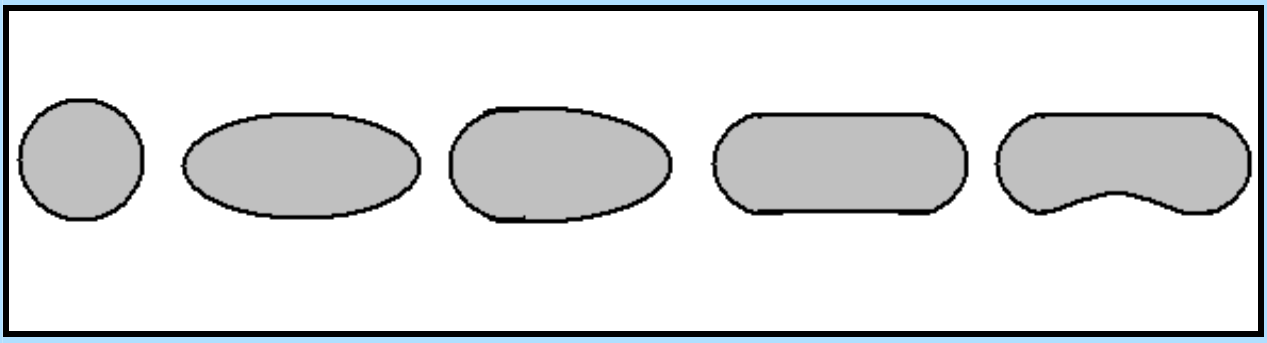

FIGURE 1: **Wool fibre cross-sections can approximate circles, ellipses, ovoids, ovals or other shapes exhibiting concavities**

The concept of circularity or ellipticity is useful in developing theoretical models to explain the influence of the morphometry of the fibre on the various measurement systems that have been investigated. These models will be discussed in detail in later editions of this newsletter. The fact remains that it is the average **fineness** of wool fibres that is the dominant dimensional characteristic of the material immediately affecting its value for manufacturing purposes. There is a nice distinction between the meaning of fineness and diameter. Fineness does not imply a specific geometrical shape for the fibre cross-section. Diameter generally does imply a specific geometrical shape. An interesting feature of the literature on this subject, has been the gradual transition from the term fineness to the term diameter. This is almost certainly related to the development of standard test methods relying on measurements of projected transverse dimensions such as the Projection Microscope. It is probably also related to the development of the Airflow system, where the theoretical model used by the wool industry to explain the physics of this instrument assumes circularity of the fibres.

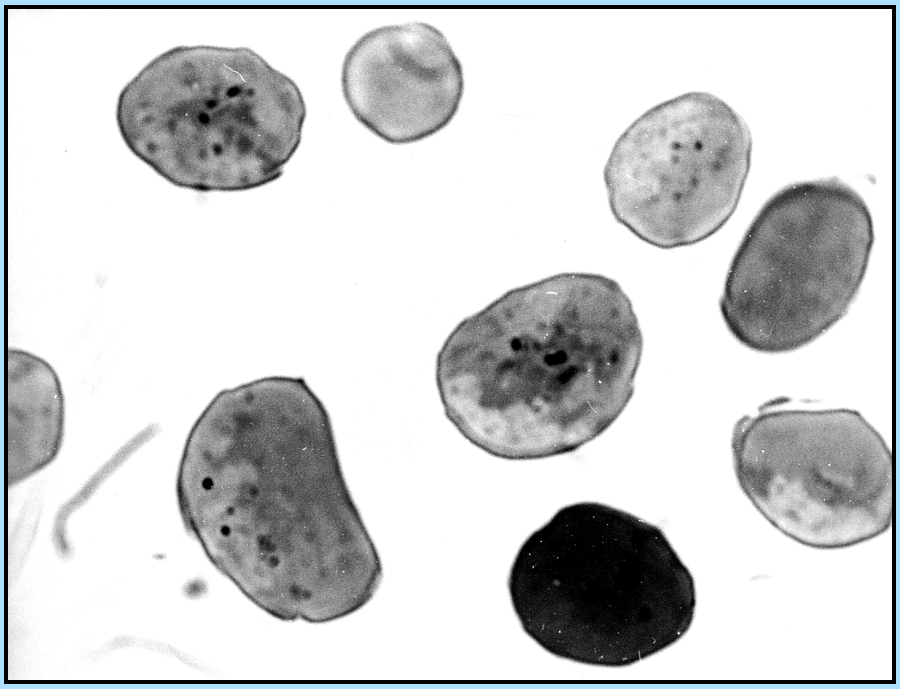

Figure 2: **Wool Fibre Cross-Sections at high magnification illustrating the range of shapes that occur[1]**

Given that the wool fibre cross-section is not a regular geometric shape, the fibre fineness is best described in terms of its cross-sectional area or its weight per unit length. Cross-sectional area or weight per unit length avoids any presumptions about geometrical shape. If a relationship to a circular geometrical shape is required it is relatively simple to transform a cross-sectional area into a circle of equivalent area. The desired dimensional characteristic (diameter) can then be calculated.

Alternatively, one can adequately specify fibre fineness by measuring the specific surface[2]. This is the method used by the Cotton Industry. However, the mature cotton fibre differs substantially from the wool fibre. The immature fibre is hollow and very nearly circular. It collapses as it matures to form a ribbon-like cross-section. The temptation for researchers to ascribe a geometrical shape to the fibre cross-section to the cotton fibre is therefore diminished. It follows that the Cotton Industry generally reports specific surface as an estimate of fineness instead of diameter.

Although it may appear otherwise, the direct measurement of the cross-sectional area, the weight per unit length or the specific surface of textile fibres is not a simple task. This will become increasingly clear in later articles in this section of the newsletter. However, this problem is not unique to the Textile Industry. Defining the fineness of powders, the fineness of fibres, and the size of particles in granular beds, is important for a wide range of industries. Indeed many of the techniques that have been applied by wool technologists to this problem have been adapted from other industries.

Clearly, the definition of the characteristic to be measured is of critical importance. Equally important is the definition of the measurement system and of the principles on which it is based. In other analytical sciences, such as chemistry and physics, a method that is capable of directly measuring a fundamental characteristic such as cross-sectional area or specific surface, by direct reference to primary metric standards is called a

[1] Courtesy of Peter Turner, CSIRO Division of Wool Technology, Belmont, Victoria, Australia.
[2] Various technologists have used the term *Specific Surface* differently. In this instance the *Specific Surface* is the ratio of the surface area of the fibre to its mass.

Primary Measurement System. A method that measures these characteristics indirectly, either by a calibration technique or by directly measuring another parameter that is an estimator or indicator of the required characteristic, is called a **Secondary Measurement System**.

This distinction has rarely been clearly stated in the wool metrology literature. This is not saying it has not been understood. It is a critical distinction if one is to understand the relationship between methods of measuring fibre fineness. In 1970 Murray Andrews & Phil Irvine (CSIRO Division of Wool Technology) pointed out the importance of calibrating test methods against a direct method, which they defined as "*a method requiring no calibration against secondary standards"*. In 1985 Lunney and Browne (also CSIRO Division of Wool Technology) stated "*only two methods ever used to measure the transverse dimensions of wool fibres are clearly connected to primary metric standards and therefore may be considerer absolute* (ie. Primary Measurement Systems)*: the **gravimetric method** and the **Projection Microscope***".

As outlined in the September 2001 newsletter, the gravimetric method relies on the assumption that the density of wool is constant, which in fact is not the case. One could therefore argue that this method is not a Primary Measurement System. If the Gravimetric Method included a step to directly measure the mean density of the sample being measured then the gravimetric method would become a Primary Measurement System.

One could also argue that the current IWTO Projection Microscope method is not a Primary Measurement System given that it provides an estimate of fibre fineness by measuring the transverse dimensions of individual wool fibres. Transverse dimensions can only be related to fibre fineness by making assumptions about the shapes of the cross-sections defined by the transverse dimensions. This view conflicts with that expressed by some technologists, but the difference hinges on the definition of fineness that is used. If fineness is defined in terms of the mean transverse dimension, as measured by the Projection Microscope, then the conflict evaporates, but a spectrum of attendant difficulties is introduced.

However, Projection Microscope measurements of fibre cross-sectional areas derived from examination of thin cross-sections can possibly be considered a Primary Measurement System, because in this case the areas measured are directly connected to primary metric standards. Unfortunately, due to the variability of fibre fineness along and between fibres, such measurements are impossibly difficult.

The availability of Primary Measurement Systems is essential for the calibration of Secondary Measurement Systems, and for establishing a reference point to determine the equivalence of different Secondary Measurement Systems. The importance of these distinctions was recognised and reported by Alan Stearn in 1969 and expressed in mathematical terms. Stearn observed that "*to compare the results from various methods for measuring the diameter of textile fibres, one has to consider the basic geometric parameters used to define the fineness in each case*".

Consider a measurement system where the fineness estimated from measurements of the cross-sectional area. Assume that the system measures the total length l of the fibres in the sample, the total mass m and the mean density ρ. The estimate of fineness or the equivalent mean diameter, D_g in this instance, can be expressed as follows:

$$D_g^2 = \frac{4}{\pi}\frac{m}{l\rho} \qquad 1$$

Further let us assume that there is a method for measuring the surface area S of the fibres in the same sample, where the total mass is again m and the density is ρ. The estimate of fineness or the mean diameter D_s in this instance can be expressed as follows:

$$D_s = \frac{4m}{S\rho} \qquad 2$$

Now let us consider a method where the equivalent mean fibre diameter is usually obtained by making a large number of measurements on individual fibre snippets. The mean distance between parallel tangents of a randomly oriented area whose circumference is wholly convex is equal to the circumference divided by π. This statistic is called Feret's Statistical Diameter. Assume that the rate of change of cross-section is sufficiently small, as is the case for wool fibres. One can show, by consideration of many infinitely thin

sections of the sample, that the estimate of fineness or the mean diameter in this instance, estimates the average circumference P divided by π or the surface area per unit length divided by π. Thus, the total surface area S can be described as follows:

$$S = Pl = \pi D_m l$$

And therefore,

$$D_m = \frac{S}{\pi l} \qquad 3$$

Combining equations 3 and 2, we obtain

$$D_s D_m = \frac{4m}{S\rho} \frac{S}{\pi l} = \frac{4m}{\rho \pi l} \qquad 4$$

This is the square of the mean diameter obtained in equation 1. Hence,

$$D_s D_m = D_g^2 \qquad 5$$

Stearn also demonstrated that in the case where the shape of the cross-section includes concavities, equation 4 does still apply, but only by redefining the meaning of S in the second example. However in this instance, as the cross-section departs from convex (i.e. if concavities exist) then D_m increases relative to D_g, and D_s decreases relative to D_g.

Note that D_s, D_m and D_g are constructs – they are estimates of fineness, expressed as diameters and derived by transforming the estimates of mean cross-sectional area of the fibres into circles of equivalent area. Note further that equation 4 is quite independent of the size distribution of fibres in any sample considered. This is because in each case the parameters measured are the totals.

This may seem a somewhat trivial exercise. It is not. Stearn demonstrated that:

> ***The equivalence of different methods for determining fibre fineness, each measuring a different geometrical feature of the fibres, depends on the definition of fineness, and on the definition of the particular geometrical characteristics measured, being the same in each case***.

In 1947 Palmer introduced a notation to describe mean transverse dimension of wool fibres, later extended, in 1960 by Monfort. Briefly, let (X, Y) represents the mean value of Y, when it is distributed in proportion to X. In this terminology the mean transverse dimension weighted proportionally to length is defined by equation 5.

$$(l, d) = \frac{\sum_i \sum_j d_i n_{i,j} l_j}{\sum_i \sum_j n_{i,j} l_j} \qquad 6$$

where

$i =$	class interval for the transverse dimension;
$j =$	class interval for fibre length;
$d_i =$	magnitude of the i[th] class interval for the transverse dimension;
$n_{i,j} =$	number of fibres in the i[th] class interval for diameter and the j[th] class interval for length;
$l_j =$	magnitude of the j[th] class interval for length; and
$\sum_j n_{i,j} l_j = .$	the total length of all fibres of diameter d_i.

This is mathematical definition of a fundamental characteristic of wool fibre fineness. Wool fibres vary in length as well as in diameter. If a sample is separated into all the individual fibres, and each separately measured, then the simple arithmetic mean of all the separate measurements will only represent the true mean of the entire sample if two conditions are satisfied:

- the transverse dimension is uniform along the length of the fibre; and
- the lengths of all the fibres are identical

These conditions do not occur. Not only do the fibres vary in length; their transverse dimension also varies along the fibres and between the fibres. Any numerical definition of fineness must consider this.

Thus the contribution of individual fibres to the mean of the estimate of the transverse dimension must weighted according to their lengths.

It follows therefore that, if an estimate of the fineness is to be made from measurements on individual fibre specimens obtained from the bulk, then the sampling of the fibre specimens must be proportioned to the fibre length.

If the transverse dimension is assumed to be circular, then it is possible to show that

$$\left(l, d^2\right) = \left(l, d\right)^2 \left(1 + C^2\right) \qquad 7$$

and

$$\left(ld, d\right) = \left(l, d\right)\left(1 + C^2\right) \qquad 8$$

where C is the coefficient of variation of the distribution. These equations provide a means of converting mean fineness defined by measurements of fibre thickness to those defined by cross sectional area or surface area.

Regretfully Palmers notation is no longer in common usage. The notation is somewhat unwieldy in complex mathematical expressions and no doubt this has contributed to its demise. But it is a useful reminder of the different definitions of fibre fineness, and of the requirements of methods based on measurement of samples taken from the bulk.

In the case of sampling wool tops, where the fibres have been aligned with each other, but are randomly located according to their longitudinal displacement, simply using two parallel blades to cut across the top, will obtain a length proportioned sample of fibre snippets. In the case of raw wool, where the fibres are randomly orientated in the bulk, an approximately length distributed sample can be obtained using a circular core tube. Once this length-proportioned sample has been obtained, we need only estimate the fineness of each snippet, and average these measurements to obtain the mean fineness of the bulk.

In subsequent reviews we will discuss the development of the various technologies used for estimating the fineness of wool fibres and consider just how closely they conform to these fundamental concepts.

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TECHNOLOGIES FOR MEASURING THE FINENESS

OF WOOL FIBRES

Over the last 200 years the wool industry has been quite innovative in its efforts to develop better technologies for measuring the diameter distribution characteristics of wool. Here we will briefly review some of the technologies that have been investigated.

It is important to realise before selecting a technology for possible evaluation that the technology must be capable of measuring a characteristic that can be directly or indirectly related to one or more of the geometries that actually define fibre fineness, viz:

- the area of the cross section;
- the width of a 2-dimensional projected image;
- the area of the surface; or
- the area of a 2-dimensional projected image.

Direct Measurement

In SI units the primary unit of length is the metre. A number of devices such as the micrometer and the micrometer calliper are available for measuring the thickness, in fractions of a metre, of various fine materials. In suitable materials measurements of the order of 0.01 micrometres are possible. The thickness is determined by using an arrangement of high precision screws to adjust the physical distance between two parallel jaws, which grip the material transversely. The screws provide a method of amplifying the scale and to make the fine adjustments necessary to adjust the gap between the jaws to the thickness of the material.

The first recorded application of this technology to measuring wool fibre diameter was in 1921. Given the tediousness of this approach and inherent sampling problems it was soon abandoned.

Optical Microscopy

Using a microscope to measure fibre diameter first occurred in 1777. In the ensuing 150 years various iterations of this approach appeared. The final iteration, the Projection Microscope, was developed in the period from 1927 to 1949, and in 1950 the American Society of Testing Materials published a tentative test method based on this instrument. An IWTO Specification was published in 1954. However little substantive development to the instrument, apart from improved optics, has occurred since 1950.

The Projection Microscope is the only primary reference method for determination of the diameter distribution characteristics of wool. **It is the reference method against which all other methods are now calibrated**. However, due to the tediousness of the technique and the high cost incurred in achieving an acceptable precision, more rapid and cost effective instrumentation is increasingly being used for routine measurements.

Gravimetry

In the early 1930's IWTO adopted its unit of fibre diameter as the weight in milligrams of 10 metres of wool fibres at a regain of 18.5%. The method relied on weighing a definite number of fibres cut to a certain length and expressing the mean diameter in terms of the weight of a standard length at a standard regain.

Subsequent applications of the gravimetric method used the relationship between mass, volume and density to define the fibre fineness in terms of its cross-sectional area.

The gravimetric method has never been advanced to a standard test method. Nevertheless it was widely used in the period 1930 – 1950.

Its basic limitation rests with the measuring or the length of the individual fibres. This limits the precision of the method because of the uncertainty surrounding the amount of stretching that occurs during this measurement. Furthermore, owing to the necessarily few fibres that can be measured in a reasonable time, the sampling error limits the precision.

Optical Diffraction

Optical diffraction was first applied to the measurement of wool fibre diameter in 1884. Interest in applying this phenomenon continued until 1932, when it inexplicably ceased. Interest emerged again in 1959 with the publication of scientific paper describing the physics on which measurement instruments based on the phenomenon relied. This culminated in development in 1971 of the Mikronmeter, the first and only hand-held instrument for on-farm measurement of Mean Fibre Diameter. Unfortunately, the precision of this instrument was soon shown to be unacceptable.

The experience with the Mikronmeter appears to have sounded the death knell for this technology, with very little interest being shown since 1972. However active development of the technology has continued in other industries and diffraction techniques are currently being applied to estimate diameters of optical fibres. The abandonment of the technology by the wool industry is possibly a good example of how an immature technology can loose favour very quickly if it is released too early into the market.

Porosity

The flow of air through a bulk assembly of wool fibres, with a standardised mass and volume, is related to the average diameter of the fibres. This fact is the basis of the Airflow Instrument, which was the Wool Industry's favoured technology for measuring wool fibre diameter from 1960 to 2000.

Porosity of bulk assemblies of fibres is actually related to the surface area of the fibres. For a given mass, fine fibres have a larger surface area than coarser fibres. The cotton industry preceded the wool industry in using this principle. Development of instruments commenced in the cotton industry in 1940 with the wool industry entering the field in 1942. The basic research upon which the wool industry's Airflow Instrument rests occurred in 1947. The first IWTO Test Method for wool sliver based on the instrument was approved in 1960, and a test method for greasy wool was approved in 1971.

An on-farm version of the instrument (the Sonic B) was produced by CSIRO in 1974. This version used sound to generate an oscillating flow of air through an assembly of wool fibres, and some of these instruments are still in use on farms today. However, the samples still need to cleaned and carded before being measured, and this preparation limits the usefulness of the instrument for on-farm applications.

Harmonics

The musical notes produced by stringed instruments, are the result of standing waves being established along the strings, either by plucking or bowing the strings. The frequency and the amplitude of these waves,

and hence the sound they produce, is determined by the thickness and density of each string, the tension applied and its length.

Likewise, standing waves can be generated in a string by placing the string in the path of an oscillating sound source. If the string is maintained at a constant tension and length, and the frequency of the sound source is varied the string will be observed to vibrate, with a standing waveform observed along the fibre at specific frequencies, depending upon the diameter, density, tension and the length.

This principle was first applied to the measurement of wool fibre diameter in 1947. Its major limitation is that it is restricted to single fibre measurements, and consequently has found little favour since.

Radiometry

Radiometric instruments utilise the phenomenon associated with the decay of radioactive substances, and the emissions of sub-atomic particles that is associated with this process, to monitor either rates of decay, or the concentration of the source of the emission.

In measuring the diameter of wool fibres this technology relies upon the adsorption of radioactive isotopes on the surface of wool fibres, then measuring the concentration of these isotopes in a solution in which the wool is subsequently dissolved. Thus it is actually measuring the surface area of the fibres.

The then statutory AWTA evaluated this technology during 1970's, with a view to utilising the method for flock testing services, but this work was abandoned before 1980. The advantage of the technology in this particular application is the possibility of automated analysis of large numbers of samples, where the major application of the data is for ranking animals. The Department of Agriculture, New South Wales, Australia used the technology for many years, in the Department's Trangie laboratories

Conductometry

Conductometry is a general term, encompassing a range of measurement systems, which utilise the phenomenon of the electrical conductivity of solids and liquids.

A Coulter Counter uses conductance to measure particle size. A suspension of particles, suspended in a conducting liquid, which is inert with respect to the particles, is metered through a small orifice. Electrodes are located on each side of the opening, and the electrical resistance of the path from one electrode to the other varies proportionally to the volume of the particle passing through the orifice. More exactly the resistance changes proportionally to volume of conducting liquid displaced by the particle while it is passing through the resistance path.

From 1962 to 1969 it was demonstrated that this instrument could also be adapted to measure wool fibre diameter. However, the simpler Airflow instrument became available and interest in the technology waned.

Sedimentometry

Sedimentometry is the measurement of rates of settlement of particles or fibres in a fluid, where the differential settling of the particles or fibres is a function of their dimensional characteristics. From 1948 to 1968 this phenomenon was applied the measurement of the diameter of wool tops, using three distinctly different approaches, but it was never seriously pursued, again probably due to the development of the Airflow Instrument.

Photometry

Photometry is the analytical use of the properties of light to measure the physical and chemical properties of solids, liquids and gases, and mixtures or solutions thereof. Wavelengths in the infrared, visible and ultra-violet portions of the electromagnetic radiation spectrum are generally used in photometric measurements. Photometry is probably the most extensively used of all analytical technologies.

In the USA photometric techniques were first applied to the measurement of wool fibre diameter in the mid 1950's. Over the last 20 years considerable efforts have been made in New Zealand and in Australia to apply Near Infrared Reflectance photometry to the measurement of wool fibre diameter, but the inexactness of this technology for this particular application has meant that it delivers inadequate precision.

The one successful application of photometry is the Sirolan™ Laserscan instrument, developed by CSIRO, and adopted by AWTA Ltd last year as its standard system for determining the fibre distribution characteristics of wool.

Of all the technologies currently available the Laserscan instrument most closely emulates the results produced by the industry's primary reference system, the Projection Microscope, and its performance in international round trials has demonstrated that it is the most precise. There is considerable scope to further develop this technology and extend the information it can provide.

Optical Image Analysis

Image analysis is broadly defined as using an image of a sample of the material of interest as a basis for determining a particular characteristic. Image analysis was first used by the wool industry in the mid 1930's. However, it was the advent of computer technology that enabled real progress to be made.

The current incarnations of this technology, the OFDA 100 and ODFA 2000 can trace their genesis to 1980, when AWTA Ltd first began to examine the potential of the technology for wool fibre diameter measurement. AWTA Ltd abandoned further development in 1990 in favour of the Laserscan technology. Around the same time BSC Electronics Pty Ltd released the OFDA 100 instrument.

Since the 1980's, facilitated by the development of high-speed computers and electronics, image analysis technology has found many industrial applications and there is still considerable potential for the use of this technology by the wool industry. Although in this review the Laserscan has been classified as a photometric device, the discrimination system it uses to select fibres for measurement is essentially an image analysis system, and therefore it could be viewed as a hybrid of both technologies.

Summary

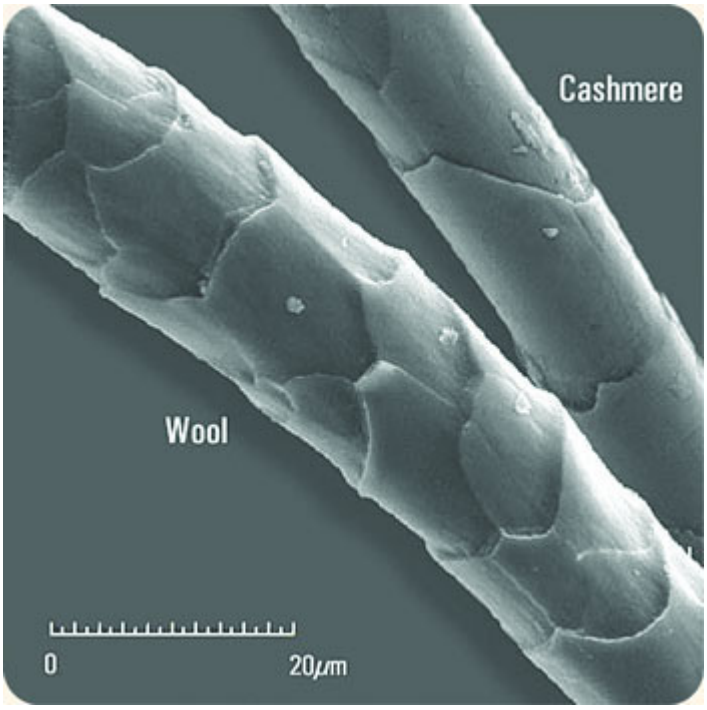
There is a considerable body of research in the literature describing these various technologies, and also the reasons why most have been rejected by the wool industry. In the most cases this is simply because they were not capable of providing the precision required. In other cases it was simply because they were uneconomic. All testing systems are a compromise between cost, precision and timeliness. These factors have to be appropriately balanced. Otherwise the systems will not find commercial acceptance. Nevertheless, it is always worthwhile revisiting the technologies that have been evaluated in the past because new developments may well render what was unsuitable yesterday fit for purpose today.

THE PROJECTION MICROSCOPE

Early interest in the fineness of wool fibres was centred on wool top. The International Wool Textile Organisation (IWTO) initially defined fibre fineness in terms of the weight in milligrams of 10 metres of wool fibres at a regain of 18.25%. The method used (called the Gravimetric Method) relied upon weighing a defined number of wool fibres cut to a known length, and expressing the mean fineness in terms of the weight of a standard length at a standard regain.

This method, and consequently this definition, was subsequently found to have a number of limitations. In the period 1932 - 1954 an increasing emphasis was placed on the use of the Projection Microscope, which defines wool fibre fineness in terms of the mean width of the projected image of the fibre. The Projection Microscope was more precise than the Gravimetric Method and moreover it also provided information about the fineness distribution.

The American Society for Testing Materials (ASTM) produced a draft specification for the measurement of wool fibre fineness, based on the Projection Microscope, in 1950. The first IWTO Specification for the Projection Microscope was approved in 1954, following a series of international laboratory round trials in 1947 and 1948.



Direct Measurement of the width of magnified images of animal fibres remains the only primary method for determining fibre fineness, and the method against which all other methods must be calibrated

It is readily acknowledged that the Projection Microscope is of limited usefulness. Firstly it is a very slow and labour intensive technique. Secondly it is very imprecise when a single operator conducts measurements within one laboratory. High precision is only attained by using several laboratories and many operators.

Notwithstanding these difficulties, no alternative method exists for the direct measurement of the Mean Fibre Diameter of wool. *Consequently, the projection microscope remains the reference method against which all other instrumental methods, specifically the Airflow, SIROLAN-LASERSCAN and OFDA 100, must be calibrated*

Principle

The are two separate systems for estimating fibre fineness using optical microscopes:

- examination of the dimensions of cross-sections of fibres; and
- examination of the transverse dimensions of fibres.

Both systems enable estimation of the mean diameter and of the standard deviation in diameter, of the fibre population.

The first system requires obtaining thin sections across the transverse dimension of the fibres. In this instance, great care must be taken to avoid cutting at an angle to the longitudinal axis. Failure to do this will increase the cross-sectional area and increase the fineness estimate. The cross-sections can be viewed in either the transmission or the reflection mode. There are several techniques for mounting the cross-sections on a glass slide prior to measurement. The area of each fibre cross-section, magnified as a projected image, is measured using a planimeter or a similar device. Because of the difficulties inherent in sample preparation, there are no commercial standard test methods based on this technique.

The standard test methods developed by IWTO (IWT0-8) and ASTM (D2130-90) are based on measurements of the transverse dimensions of fibres. These require the distribution of a random sub-sample of the fibre assembly onto a glass slide, or the distribution of snippets, prepared from the fibre assembly using a microtome or a similar device, onto a glass slide. These fibres or snippets are distributed in a mounting medium, under a cover plate and generally viewed in the transmission mode, projected onto a screen. A number of techniques have been developed for estimating the physical dimensions. In general terms, these usually involve a graduated linear scale. The observer is required to classify the transverse dimension of each fibre into one of 40 or more class intervals, where each class interval is 2 microns. Thus a frequency histogram of the transverse dimensions of the fibres is developed. The sampling of the snippets is designed to obtain a length-proportioned sample and hence the measurement can be said to equate to the length-proportioned mean of the bulk.

The test methods are designed to ensure that measured snippets are selected at random, and that each snippet is measured only once at a single point located randomly along its length. Great care must also be taken to ensure that the snippets are in focus when being measured.

To minimise the effect of operator bias IWTO-8 requires the measurement to be conducted by at least two observers, each measuring 300 snippets.

The mean, $\bar{d}$, and the standard deviation, s , of the sample is calculated from the resultant histogram data.

$$\bar{d} = \frac{\sum_{i=1}^{m} n_i d_i}{\sum_{i=1}^{m} n_i} \qquad 1$$

$$s = \sqrt{\frac{\sum_{i=1}^{m} n_i d_i^2 - \left[\left(\sum_{i=1}^{m} n_i d_i \right)^2 \Big/ \sum_{i=1}^{m} n_i \right]}{\sum_{i=1}^{m} n_i - 1}} \qquad 2$$

where $n_i =$ the number of measurements assigned to the i[th] class interval

$d_i =$ the diameter, in microns of the i[th] class interval

$m =$ the number of class intervals

$i =$ an integer, 1,2,3,4…… m

A magnification factor of 500:1 is considered ideal.

Development

Early measurements of wool fibres were confined to measurements of single fibres. The first recorded use of a microscope was in 1777 when Daubenton measured the fibre thickness by comparison with lines drawn on a piece of quartz, which was also placed under the microscope. Adopting this technique Voightlaender (1815) and Winekler (1821) were the first to measure multiple fibres on the one slide. The fibres were mounted parallel to each other on a special frame, which was then placed under the microscope.

In 1860 Parry criticised Daubenton's method and was probably the first to actually measure the image of the fibre as shown by the microscope. About the same time Rohde introduced an eyepiece, equipped with micrometer.

In 1886 McMurtie described the Dollond Eriometer. This instrument, an adaption of Daubenton's technique, enjoyed wide usage in the early part of the 19th century and for some time it was considered the basis for comparison, the unit of measurement being the Dollond unit.

Doehner (1929) described an apparatus consisting of a microscope, with a mechanical stage. The stage was adapted to take a special cell consisting of a metal frame divided into three compartments, and carrying two glass plates between which the wool sample, previously cleaned by brushing over with ether, and mounted in thinned cedar oil, was distributed. A wooden box stood in front of the microscope and in a tight connection with it. The front of the box carried a matt viewing screen provided with a light protecting cap for daylight use, and a measuring disc or apparatus for photographing the projected image. If the matt screen was removed the image could be projected onto a wall for the benefit of a number of observers. The magnification for viewing on the matt screen was 60:1 and standards for comparison were provided by means of diapositives kept in a slide holder beside the apparatus.

These permitted the sample to be classified roughly. If more precise information was required, the thickness of single fibres was measured by means of a rotatable disc, calibrated in millimetres. In this case, the microscope was arranged to project an image magnified by 500:1 onto the graduated disc, and the width of the fibre at a given point was measured in millimetres. The calculated fibre width measurements were classified and the classified widths plotted as an abscissa, with the frequency of each classification as the ordinate. With practice, approximately 100 measurements could be made every 10 minutes with this apparatus. Barker (1931) designed a double optical system, which projected two images side by side, a test sample as well as a standard sample, for comparison.

Von Bergen (1935) commented that the old methods of measuring the thickness of the fibres through a microscope with a micrometer were too tedious and not sufficiently accurate. He too favoured projecting the image of the fibre onto a screen at high magnification and measuring its width, and developed a wedge ruler to simplify the measurement process. The width of the image was recorded on the wedge ruler in such a manner as to automatically sort the fibres according to their width.

At a lecture at Roubaix in 1935 Rasuch summarised the situation regarding fineness measurement, prior to the 1936 conference of IWTO, where Germany was proposing to discuss in full, methods for estimating the properties of wool. In his opinion the projection method, based on Doehner's Lanometer, was the most satisfactory.

Bernhadt (1938) reported that the speed of the measurement was increased by using a plain frosted screen in the lanometer and by measuring the fibre thickness on this by using a transparent celluloid rule.

In 1938 IWTO decided that any satisfactory type of apparatus would be recognised for measuring fineness in cases of arbitration. However IWTO had adopted as one of its primary objectives the drawing up of standard methods, based on generally accepted procedures, which would serve to measure independently all the characteristics of wool entering into the assessment of quality.

Henning (1940) reported on progress by an IWTO technical committee in establishing a standard technique for measurement of wool fibre fineness based on the Projection Microscope.

Wollner, Tanner & Spiegel (1944) described a modification of Von Bergen's wedge method for estimating fibre thickness using projected images. The authors had developed a wedge rule base on a calibrated spiral. They also reported the preparation of very short snippets (approximately 75 micron in length) for measurement. This provided a compact single layer of fibre snippets on the microscope slide, minimising the need to refocus the instrument during measurement.

Anderson and Palmer (1947) provided evidence that measurement of mean width of fibre snippets by the Projection Microscope was sensitive to the snippet length. They examined snippet samples of two tops (both with a mean diameter in the range 32 - 38 micrometres), where the snippet length ranged from 50 to 1600 micrometres (0.05 mm to 1.6 mm).Results for very short snippets were significantly higher than for longer snippets. They attributed this to a tendency for very short snippets to come to rest on the slide, under the influence of gravity, with their major axis parallel to the slide. They concluded: **"it appears that for fibres of non-circular cross-section a section length of 300 microns is too small and it may be worth while adopting a minimum length of 800 microns, though this may be too small for some fibres. For merinos on the other hand, this effect is not likely to be so pronounced"**. WIRA (1955) published additional data, using tops of a similar diameter, confirming this effect and suggesting a minimum snippet length of 800 micrometres (0.8 mm) was required.

The effect of water absorption on the radial dimensions of wool fibres was also being extensively examined as it has implications for any wool fineness measurement system. The first published work was by Hirst (1922) who carried out microscopic measurements of a single wool fibre at a number of different regains and demonstrated the increase in the dimensional characteristics of the fibre as the regain increased. King (1926) conducted some quantitative experiments and was able to calculate the radial swelling of the fibres for a range of increasing regains. Warburton (1947) demonstrated that increasing the regain from 0% to approximately 32% increased the radial dimensions by approximately 17%.

Cassie (1945) reported a study of the absorption isotherms of water into wool fibres. He explained an observed hysteresis effect in the adsorption-desorption process in terms of a mechanical hysteresis of the fibres. The implication of this work is that measured fineness of wool fibres is influenced by the mode of equilibration with water. Conditioning from the dry side produces a different effect to conditioning from the wet side.

Semple (1947) considered the interaction of the absorption of moisture and the buffering capacity of the mounting medium on Projection Microscope measurements. He suggested that there was merit in heating the mounting medium rather than attempting to maintain control of either the water content in the medium or the conditioning of the wool.

Anderson & Palmer (1948, 1951) considered this issue in some detail. They concluded that there were two ways of mounting wool fibres for Projection Microscope measurements that are both satisfactory, in principle, for diameter measurements:

- condition the wool and mount in a medium such as cedar wood oil that has a low water buffering capacity; or

- do not condition the wool and mount the fibres in a medium such as glycerine or water that has a high buffering capacity.

Mediums of intermediate buffering capacity should be avoided. If wool is mounted in mediums of high buffering capacity, then the final regain of the fibre will be determined by the medium, and not at all by the initial regain of the wool. If wool is mounted in a medium of low buffering capacity, such as cedar wood oil, then the final regain of the fibre will be the same as when it was mounted, irrespective of any moisture content the oil might have. Anderson & Palmer also suggested that the refractive index of the medium was not a critical factor in determining the fibre diameter. In spite of this they suggested that some mediums might give less observer error than others owing perhaps, to easier focussing.

In 1947 a round trial was conducted, to evaluate a Projection Microscope developed by the Wool Industry Research Association (WIRA) in the UK. The trial used 5 tops ranging from 21 to 37 micron and involved laboratories in UK (WIRA), Belgium, Canada, USA and Italy. No special effort was made to standardise the procedures to be used in the participating laboratories.

This particular trial indicated that differences between the laboratories were not significant, and that most of the variation in the measurements seemed to be due to between operator variances within laboratories. The standard deviation of the results was 0.53 microns, or a precision of ±1.06.

A more extensive trial, involving 15 laboratories was organised in 1948 and the results reported by Palmer (1948). The objective of this was to test the reproducibility of the measurements when all laboratories strictly followed the same procedure.

- It is worth noting the special points in the procedure that were adopted in the 1948 trial.
- A standard snippet length of 800 microns (0.8 mm) was adopted.
- The fibre pieces on the slide were brought into equilibrium with an atmosphere of 65% R.H. and mounted in a medium of low buffering power such as cedar oil.
- Selection rules were designed to ensure that the observer measured the fibres at a place absolutely independent of any observer choice.

The precision of the mean diameter measurement was ±1.26, a little higher than the 1947 trials. Palmer observed: **"Neither of these could be regarded as satisfactory, because an error the size of the smaller means that two laboratories will differ by 1 micron or more about 1 time in six"**. However Palmer also observed that for the diameter measurement 6 of the 15 laboratories were "out of control" in that their deviation from the others was statistically significant.

The 1947 and 1948 trials were major milestones in the development of Projection Microscope standards. Shortly after the completion of these trials a tentative ASTM specification for determination of wool fibre fineness of raw wool, top and yarn by the Projection Microscope was published. By 1954 the Projection Microscope method was progressed to a standard test method by IWTO. However, while these standards have continually improved, little substantive development to the instrument, apart from improved optics, has occurred.

The human factor has always been one source of variation in the method. A comprehensive study of operator bias and its day-to-day variation was reported by Kritzinger et al (1964).

Precision

The precision of the Projection Microscope for the measurement of fineness of wool top and greasy wool is defined by IWTO-8 (IWTO, 1989). The standard states that **"...in the absence of more definitive data, the estimates of the variance components calculated by Andrews and David (1978) are the best available"**. These data are shown in the Table on the next page.

Variance components of Mean Fibre Diameter by Projection Microscope

Component of Variance	Symbol	Value (for raw wool)	
Between Bales	s_t^2	0.125	(μm^2)
Between cores	s_c^2	0.083	(Australian Wools)
Between laboratories	s_l^2	0.082	
Between sub-samples	s_s^2	0.024	(for d = 22 μm)
Between specimens	s_k^2	0.011	
Between operators/slides	s_o^2	0.058	
Between fibre snippets	s_f^2	25	(for d = 22 μm)

Using these data an estimate of the variance of the method for a 22 micron lot of raw wool can be made from the formula:

$$S^2(d) = \frac{s_c^2}{n} + s_l^2 + \frac{s_s^2\left(1-\frac{q}{5}\right)}{q} + \frac{s_k^2}{k} + \frac{s_o^2}{kj} + \frac{s_f^2}{ijk} \qquad 3$$

where $n =$ total number of cores taken from the lot (each bale being equally cored)
$q =$ subsamples taken from the total sample of n cores after blending
$k =$ test specimens taken
$j =$ slides prepared from each test specimen
$i =$ fibre snippets measured from each slide by one of 2 operators

The 95% confidence limits or precision are then given by:

$$95\%\ CL = 1.96\sqrt{S^2(d)} \qquad 4$$

For raw wool, where the samples have been obtained by core sampling, the confidence levels are therefore ± 0.87 for a 22-micron lot. IWTO-8 provides additional equations to allow estimates of the precision for a range of Mean Fibre Diameters. A similar calculation can be done for estimating the precision when measuring wool top (sliver).

The between laboratories component of variance is the largest single component, accounting for over one third of the total. If 400 or more snippets are measured, the effect on the between snippets components on the variance of the mean is outweighed by the combined contributions of the other components, particularly the between laboratories component. Beyond a certain point it is unproductive to attempt to reduce further the over-all variance of the mean by increasing the number of snippets. It follows therefore that in order to improve the precision of the Projection Microscope estimate of mean diameter it is necessary to replicate the testing in more than one laboratory.

Lunney (1980) considered the effect of random errors of observation on estimates of mean diameter. The projection method requires the observer to categorise individual fibre measurement into class intervals of 2 micron. Random errors of observation result in measurements being placed in an adjacent class interval.

Lunney simulated this by perturbing the distribution. He moved one quarter of the elements in each class interval to the interval immediately below, and one quarter to the interval immediately above. This simulation showed that resulting increase in between-fibre variance may be neglected. Lunney concluded that random errors of observation of individual fibres do not contribute significantly to the variance of the method.

Sheppard (1898) suggested that variances of continuous distributions calculated from frequencies assigned to discrete classes of identical class interval, over-estimated the variance (and hence the standard deviation) of the distribution. Sheppard suggested that a quantity h2/12, should be subtracted from the variance, where 'h' is the class interval, to remove this bias. David (1992) used computer simulation to determine whether this correction should be applied to Projection Microscope measurements on wool. He concluded that the bias does exist, but that it is variable, apparently at random. Panov (1995) reviewed David's paper, pointing out that the problem in using the Projection Microscope was the range in error in measuring transverse dimensions of individual fibres. Sheppards correction of 0.333 is negligibly less than the class interval (2 microns), and less than the lower detection limit (LDL) of the Projection Microscope, estimated to be approximately 1 micron. Consequently the error in measurement is greater than the bias introduced by using grouped data to determine the mean and the variance.

Commercial Importance

Although the Projection Microscope is now rarely used as a basis for the commercial trading of wool, its importance to the industry, commercially and technically, cannot be overstated. It remains the only standard method that can provide both a mean transverse dimension and a standard deviation in that dimension, by direct measurement. It is the only such method in current usage, for which an international standard exists, and is therefore the primary reference method for the industry. It is the basis for calibration of all alternative commercial instruments.

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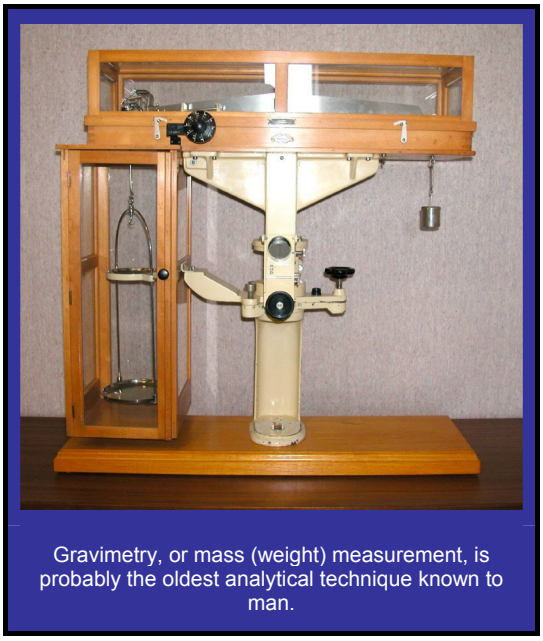
GRAVIMETRY

Principle

IWTO initially adopted its unit of fibre fineness as the weight in milligrams of 10 metres of wool fibres at a regain of 18.5% (Von Bergen, 1932). The method relied on weighing a definite number of fibres cut to a certain length and expressing the mean fineness in terms of the weight of a standard length at a standard regain. Subsequent applications of the gravimetric method used the relationship between mass, volume and density to define the fibre fineness in terms of its cross-sectional area.



Gravimetry, or mass (weight) measurement, is probably the oldest analytical technique known to man.

$$Density = \frac{Mass}{Volume} = \frac{Mass}{Length\ x\ Area}$$

Therefore $$Area = \frac{Mass}{Density\ x\ Length}$$

By assuming a circular cross-section, and a uniform density, the fineness can be expressed in terms of the mean diameter of a circle of equivalent cross-sectional area.

$$D_g^2 = \frac{4}{\pi} \cdot \frac{m}{\rho l}$$

Where D_g = mean diameter of the equivalent circle;

m = mass of the fibre sample;

l = total length of the fibres in the sample; and

ρ = mean density of the sample.

The fibre fineness can then be defined as the *root mean square diameter,* i.e.

$$D_g = \sqrt{\frac{4m}{\pi \rho l}}$$

For the gravimetric method, although the measurement is based on a sample consisting of a discrete number of fibres, each fibre is effectively represented at all points along its length. In other words, if we imagine all the fibres in the sample to be laid end on end the method effectively measures the average cross-section over the whole length, and then calculates the average diameter on the assumption all the fibres are circular. The measurement is therefore an estimate of the mean for the bulk.

In contrast, as we have already seen, the Projection Microscope profile method, measures individual fibre snippets at a single point randomly located along the length of the snippet. Providing that the sample is length biased, and each snippet is measured only once, the profile measurement also estimates the mean thickness of the fibres, D_p, in the bulk. Thus if we assume circularity, and d is the diameter at any point along a fibre,

$$D_g = \sqrt{\sum d^2 / n}$$

and $$D_p = \frac{\sum d}{n}$$

Now $$\sum d^2 = n\left(\frac{\sum d}{n}\right)^2 + n\sigma^2$$

where $\sigma =$ the standard deviation of d.

From these equations it follows that:

$$D_g = \sqrt{\left(D_p^2 + \sigma^2\right)}$$

or the root mean square diameter

$$D_g = D_p\sqrt{1 + c^2}$$

where $c =$ fractional coefficient of variation of d.

In Palmers notation (see For Technophiles - January 2002) this is defined as $\sqrt{\left(l, d^2\right)}$.

Development

The gravimetric method has never been advanced to a standard test method. Nevertheless it was widely used in the period 1930 – 1950.

Von Bergen (1932), reported the results of comparisons of gravimetric measurements on wool tops, compared with measurements based on fibre cross-sections and fibre widths determined by optical examination through a microscope. A selection of the data he reported is summarised in Table 1.

Table 1: Comparison of Gravimetric Measurement of fineness of top with two Microscope Methods

Quality Number	Gravimetric Method		Width Method
80's	19.6		19.5
70's	20.4	20.7	20.8
64's	22.3		21.9
60's	24.3	24.4	23.5
58's	25.7		24.8
56's	28.1	27.7	26.9
50's	31.1		30.4
48's	32.9	33.8	33.0

Von Bergen remarked, "....*there was an astonishing conformity of results*".

Palmer (1948, 1951) reported the results of the 1948 inter-laboratory diameter and length experiment involving 15 international laboratories and using 6 tops. This followed an earlier experiment on a smaller scale conducted in 1947, which was designed to test the reproducibility between laboratories of three different methods, one involving optical measurements by microscope and the remaining methods being two different gravimetric methods.

One of these methods, developed by WIRA (Wool Industry Research Association) obtained a sample of fibres by a cut squaring procedure. The length of each fibre was measured by stretching between two pairs

of forceps. The measured fibres were collected, cleaned, conditioned and weighed. The weight of the fibres was multiplied by a constant and divided by the total length of all the fibres measured. The square root of this gave the length proportioned root mean square diameter.

A modification of this method involved measuring the length of each fibre under constant tension, by hanging each fibre with a constant mass attached to the free end. The purpose of this was to determine whether the different amount of stretch applied to each fibre by different observers using the forceps technique, was an appreciable source of error.

The alternative method, developed by Maillard and Roehrich, involved sorting the fibres into length groups. Cutting known lengths of fibres from each group and determining their weight enabled the root mean square diameter of each length group to be calculated. The root mean square diameter of the whole material was determined by calculating the weighted mean of the results for the separate length groups.

Palmer concluded that the *modified* WIRA gravimetric method improved the precision of the measurement within and between laboratories, and that the variable amount of stretch applied to the fibres by different operators using the forceps method was an appreciable source of error. He concluded that the Maillard-Roehrich method gave more variability within laboratories, and suggested the major sources of this variation arose from stretching of the fibres when the constant length sections were prepared from each class interval, and from the preparation, conditioning and weighing of the fibres. The trial confirmed that the WIRA gravimetric method and the modified WIRA gravimetric method gave good agreement with the Projection Microscope, for the root mean square fibre diameter, with the Maillard-Roehrich method giving results approximately 0.5 - 1.0 microns higher (Table 2).

Palmer noted that not all laboratories involved in the trial were within statistical control. The error limits quoted in Table 2 are for all laboratories and are therefore slightly higher than for those laboratories that were in control. This particular experiment marked a significant milestone for IWTO in that it was the beginning of the formal development of IWTO standard methods for estimating the fineness of wool fibre.

TABLE 2: Comparison of mean diameter determined by the four methods used for the 1948 Inter-laboratory Diameter Experiment (Palmer, 1948, 1951)

Top Identification	Projection Microscope	WIRA Gravimetric Method	Modified WIRA Gravimetric Method	Maillard-Roehrich Gravimetric Method
CG	19.52	19.69	19.62	20.46
BL	20.52	20.56	20.59	21.08
FJ	21.14	21.56	21.62	21.92
AD	21.46	21.69	21.82	22.20
HI	22.12	22.44	22.56	22.60
EK	24.41	24.56	24.60	25.04
Error (all labs)	0.63	0.60	0.63	0.84

Andrews and Irvine (1969) proposed a method for measuring the gravimetric diameter by using small snippets instead of full-length fibres. The novelty of the method was that the fibres were cut into snippets short enough to be easily specified. Since the number of length measurements required for a test then became too large to be practicable, an estimate is obtained of the total length of snippets in the weighed sample. Firstly the total number of snippets, *N*, was counted using a Coulter Counter. Secondly, the individual lengths in another, much smaller, sample of the snippets were measured with a Projection Microscope and averaged. The product of *N* and the average snippet length is an estimate of the total length of fibre in the sample. In such a numerical sample of snippets, cut from the original sliver or assembly, the total length of snippets in each small interval of diameter must be proportional to the total length of fibres in the same diameter interval in the original assembly. Each fibre is therefore represented in proportion to its length, as is the case with the intact fibre gravimetric method. The precision for the method was reported to be better than 0.2 µm.

Technical Issues

Gravimetric methods do not provide an estimate of the standard deviation (or coefficient of variation) of the estimated fibre diameter.

Their basic limitation rests with the measuring the length of the individual fibres. This limits the precision of the method because of the uncertainty surrounding the amount of stretching that occurs during this measurement. Furthermore, owing to the necessarily few fibres that can be measured in a reasonable time, the sampling error further limits the precision. This is the same limitation that applies to the Projection Microscope. Although Andrews and Irvine (1969) did demonstrate that the method is capable of improvement, little further progress has been made, and for wool the method remains relatively underdeveloped. However, gravimetric measurement is widely used for estimating the fineness of synthetic textile fibres, and in such cases is often the only practical method given the enormous divergence from circularity of many synthetic fibres.

The method does rely on the presumption that the density of wool fibres is relatively constant. This is clearly not the case with medullated fibres, and this limits its general applicability. There is evidence that the fibre density of individual farm lots can vary by small but significant amounts from the generally accepted value of 1.310 g/cm^3 (Van Wyk and Nel, 1940, Connell & Andrews, 1974). This means that for very precise work it may be necessary to measure the density of the sample in order to reduce small differences in estimates arising from density differences alone.

However, if the density of the sample is also measured, then the gravimetric method is one of only two methods that approach the status of primary measurement systems. Also, the gravimetric method provides a totally unambiguous definition of fineness, in that the reported diameter is independent of the shape of the fibre cross-section.

Commercial Issues

The absence of a standard test method is the major commercial limitation of the gravimetric method. Also, the cost of measurements based on gravimetric methods severely inhibit its commercial usefulness, in the same way as the costs of the Projection Microscope measurement have limited the commercial application of the Projection Microscope standard method. Furthermore gravimetric methods do not provide distribution data.

However the gravimetric method does have the potential to provide a primary measurement system, linked directly to SI units, for wool fibre fineness measurement, and thereby provide standard reference material that is traceable to the SI standards for weight and length. This does depend upon the availability of a suitable technique for accurately determining the fibre density. The commercial benefit would be a more fundamental basis for calibrating any appropriate secondary test procedure for use in determining the conformity of deliveries to contract specifications.

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DIRECT MEASUREMENT

Principle

In SI units the primary unit of length is the metre. A number of devices such as the micrometer and the micrometer calliper are available for measuring the thickness, in fractions of a metre, of various fine materials. In suitable materials distances of the order of 0.01 micrometres are possible. The thickness is determined by using an arrangement of high precision screws to adjust the physical distance between two parallel jaws, which grip the material transversely. The screws provide a method of amplifying the scale and to make the fine adjustments necessary to adjust the gap between the jaws to the thickness of the material.

The micrometer and the micrometer calliper are available for measuring the thickness, in fractions of a metre, of various fine materials.

Development

Hill (1921) used a machinist's calliper in measuring the thickness of a wool fibre.

Burns (1935) described the use of the micrometer calliper and expressed his view that it was preferred to other methods then available for the measurement of the thickness of wool fibres. He claimed that the micrometer calliper method provided information on fibre diameter variability, with the entire fibres as units, whereas cross-sectional methods altered the identity of individual fibres. There was little crushing action in the micrometer measurements. A resolution lower than that obtained using microscopic methods was claimed, results were provided demonstrating high correlation with measurements made using length to weight ratios.

Technical Issues

Since this initial work there has been little interest in this technique. There is almost no data on the precision of the method, and it was probably made redundant by the rapid development of methods based on the optical microscope in the period 1930 to 1940. Consequently few technical issues have been adequately documented.

However, the limitations that apply to the Projection Microscope would almost certainly apply to this technique. Individual fibres must be sampled at random locations along their length and in proportion to their length in order to obtain a length-biased sample. A large number of such measurements would be required for an acceptable precision. It must be expected therefore that the technique would be slow and tedious.

Commercial Issues

Within the wool industry, this mode of measurement has never been applied commercially, largely because faster and less expensive measurements systems have been developed.

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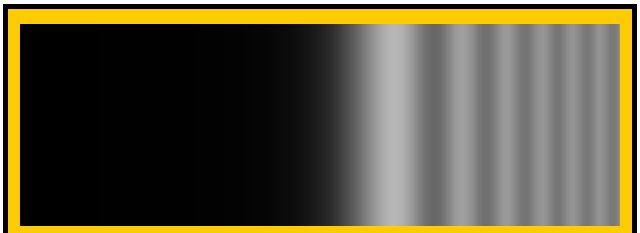
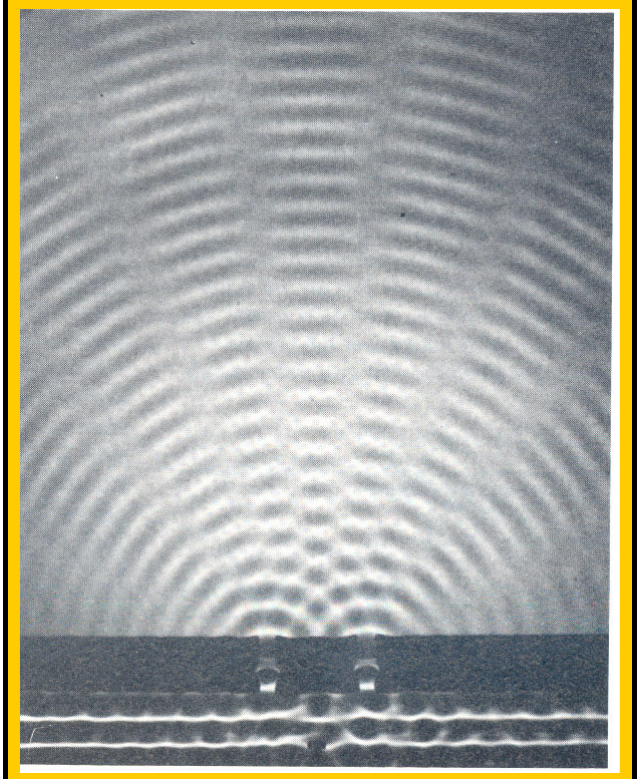
OPTICAL DIFFRACTION

Principle

Diffraction is a change in the direction, or bending, of a wave into a region where it would normally be obscured (the geometric shadow). All wave phenomena, including electron beams, which can exhibit wave-like behaviour, are subject to diffraction. It is easily observed in water waves, which can bend around an obstruction in the water.



Diffraction pattern produced by a straight edge.



Diffraction pattern produced by water waves passing through two parallel slits.

The effect is especially important in the case of visible light, as it affects the design and performance of optical instruments. There are two major cases in which light diffraction is observed. In the first, light that passes through a small aperture does not form a sharp image of the aperture on a screen; the image is diffuse, and a series of bright and dark rings, or fringes, outline the image and fall within the predicted geometric shadow of the aperture. This effect is directly observed only if the size of the aperture is no wider than a few wavelengths of light, or less than a millimetre. The second case occurs when light is bent around the edge of a smooth object (such as a wool fibre). In the region of the geometric shadow there is a series of fine bright and dark fringes instead of the predicted sharp shadow edge.

Diffraction is considered a wave phenomenon, and its explanation by Augustan Fresnel in 1814 played an important part in establishing the wave theory of light. The basis for the wave theory is traced to Christian Huygens (1629-1695), who proposed that each point on a wavefront may be regarded as a new source of waves. Thus, each point on a wavefront is the resultant of the many contributions of secondary waves from the previous wavefront. Toward the centre of the beam these secondary waves combine in such a way as to transmit the light in straight lines. Diffraction results from the obstruction of a portion of the light, which removes some secondary waves. These ordinarily would cancel other waves that travel into the geometric shadow; thus some light is observed in this region.

For historical reasons diffraction phenomena are classified into two types: Fraunhofer and Fresnel diffraction. Fraunhofer diffraction treats cases where the source of light and the screen on which the pattern is observed are effectively at infinite distances from the intervening aperture. Thus, beams of light are parallel, or the wavefront is plane, and the mathematical treatment of this type of diffraction is simple and elegant. Fresnel diffraction treats cases in which the source and the screen are at finite distances and therefore the light is divergent. This type of diffraction is easier to observe, but its complete mathematical explanation is considerably more complex.

The theoretical application of diffraction to measure the mean diameter and the standard deviation in diameter of fibres was discussed by Onions (1959). Onions showed that when monochromatic light is

incident on a slit, in a direction perpendicular to the plane of the slit, the expression for the intensity of the light at an angle θ may be written:

$$I = A_o d_w^2 \cdot \left(\frac{Sin^2 \beta}{\beta^2} \right) \qquad 1$$

where $A_o =$ a constant;

$d_w =$ the width of the slit;

$\beta = \dfrac{\pi d_w \sin\theta}{\lambda}$;

$\theta =$ angle of diffraction; and

$\lambda =$ wavelength of the light.

In a group of fibres, all approximately parallel to the slit, different fibre elements will generally vary in diameter and will simultaneously intercept different proportions of the light beam. Onions assumed that the arrangement of fibres approximated a group of equivalent slits. From this he showed that for a case where the fibre diameter is normally distributed then the radial distribution in intensity is given by:

$$I_{T,\theta} = A_0 \int_0^\infty D^2 \cdot \left(\frac{\sin\beta}{\beta} \right)^2 \cdot \frac{N}{s\sqrt{2\pi}} \cdot \exp\left[-\frac{\left(D - \overline{D}\right)^2}{2s^2} \right] dD \qquad 2$$

where $I_{T,\theta} =$ the intensity of the light at an angle θ;

$D =$ the fibre diameter;

$\overline{D} =$ mean fibre diameter;

$s =$ standard deviation in diameter; and

$N =$ the number of fibres in the specimen.

From this it is not difficult to show that:

$$I_{T,\theta} = \frac{B_o}{\phi^2} \left[1 - \cos\phi \cdot \exp\left(-\frac{\left(D - \overline{D}\right)^2}{2s^2} \cdot \phi^2 \right) \right] \qquad 3$$

where $\phi = \dfrac{2\pi\overline{D}\sin\theta}{\lambda}$; and

$B_o = 2NA_o\overline{D}^2$

Based on this theoretical model, Onions proposed a design of an instrument that could measure Fibre Diameter and also the Standard Deviation in Diameter.

Development

Young (1824) was the first to adapt the phenomenon of light diffraction to the measurement of fibre diameter. Ewles (1928) made an instrument based on the principle, which consisted of a portable tube, but gave no experimental information about the comparative data in measurements obtained with this instrument

Duerden (1921) reported experiments with a laboratory diffraction apparatus. He made a large number of measurements, using microscopic and diffraction methods, and found a very close agreement.

Burns (1930) reported a few measurements with the Ewles instrument as compared with the micrometer calliper, and found that the micrometer measurements were on average about 5 micrometres finer than the readings taken by the Ewles instrument.

McNicholas and Curtis (1928) reviewed the history of diffraction instruments and described an improved device called an eriometer. They made an extensive study of the accuracy and adaptability of the eriometer in averaging a wide range of diameters, as distributed in a sample of fibres. They found the average fineness obtained with their eriometer agreed closely with the microscope and concluded that "… *the diffraction method offers considerable opportunity for the further development of instruments to include other features that are desirable in the study of wool and other textile fibres*."

Mathews (1932) reported that long straight fibres are the easiest to measure by the diffraction technique. *"One must be careful to prepare the wool sample so that the fibres are parallel, doing away with the fuzziness of the bands that are so prevalent when the fibres are crossed over one another."*

Von Hertzog (1932) gave a description of a light interference method for the estimation of fibre thickness, firstly by means of polarised light, and secondly by means of a special polarisation apparatus.

The reporting of studies of light diffraction techniques applied to wool metrology suddenly disappeared from the literature until Onions wrote his paper describing the physics of the system in 1959. Onions' theory was discussed by Whan and Paynter (1967). Boshoff and Kruger (1971) described the Mikronmeter, an instrument based on the original design by Ewles (1928) and Onions (1959). The instrument measured the circular diffraction pattern produced by a sample of randomly oriented fibres. The authors claimed that a well trained operator could measure fibre diameter very accurately while randomly chosen operators could determine fibre diameter with a confidence interval of ±0.8 micrometres. However occasionally, some operators could not use the instrument correctly.

Lynch and Thomas (1971) examined the diffraction patterns produced by wool and other fibres by single fibres in a helium-neon laser beam and suggested that a possible application was the determination of fibre diameter.

Edmonds (1988) reported results obtained using a diffraction instrument where the diffraction patterns were recorded as photographic images and later analysed. In this device the samples were randomly oriented, but the diffraction pattern was obtained by rotating the sample in front of the slit in the instrument. Edmonds found a correlation of 0.95 with the Projection Microscope method but only a 0.5 correlation for standard deviation.

Fouda, El-Dessouki and El-Farhaty (1988) reported a study using a laser as a source of coherent light to examine the diffraction patterns produced by a range of synthetic fibres. They examined three techniques, and found the forward light scattering technique the most satisfactory.

Technical Issues

The Mikronmeter was commercialised shortly after Boshoff and Kruger's paper was published. The instrument arrived on the market almost at the same time as work was commencing in Australia to extend the testing of greasy wool to farm lots prior to auction. The target market for the instrument was wool growers, wool brokers, wool buyers and wool processors. The instrument was then available for A$135.

David & O'Connell (1972) reported the results of a trial to evaluate the precision of the Mikronmeter. They found the same difficulty with some individual operators that were reported by Boshoff and Kruger. They concluded that contrary to the data reported by these authors a precision of only ±2 micrometres could be achieved and only then if 5 sub-samples were measured.

The experience with the Mikronmeter appears to have sounded the death knell for this technology, with very little interest since 1972, accept for the two studies reported above. However active development of the technology has continued in other industries and diffraction techniques are currently being applied to estimate diameters of optical fibres. The abandonment of the technology by the wool industry is possibly a good example of how an immature technology can loose favour very quickly if it is released too early into the market.

Commercial Issues

Optical diffraction has never found a successful commercial application in the wool industry. However, with the increasing interest in Australia by wool growers in testing their flocks prior to shearing, and using the data to assist in classing, the technology is currently being revisited.

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Huygens lived from 1629 to 1695. Following the death of Descartes, Huygens was the most celebrated mathematician in Europe. He developed the wave theory of light, from focusing on the diffraction and refraction properties he could observe. "Huygens's wavelets" were the formal basis for explaining these phenomena

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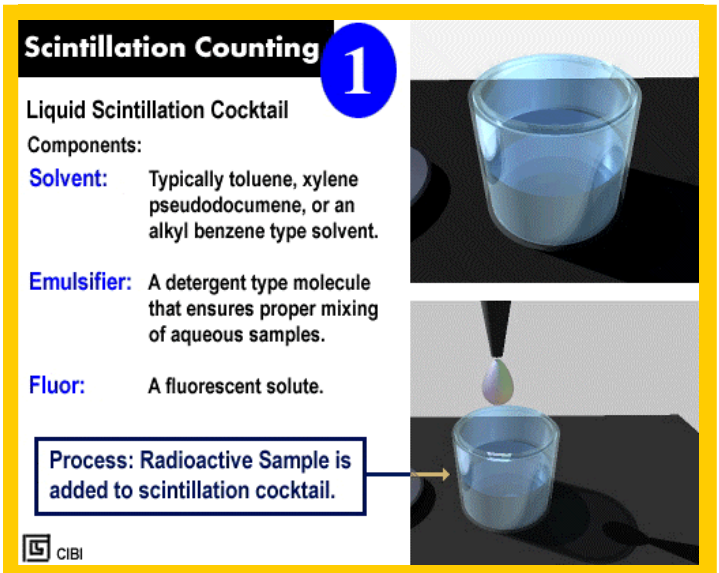
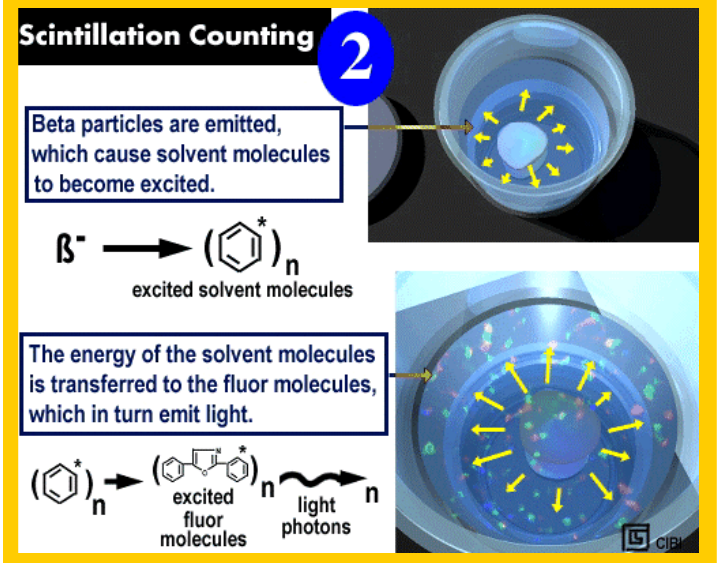


The process of liquid scintillation involves the detection of beta decay within a sample via capture of beta emissions in a system of organic solvents and solutes referred to as the scintillation cocktail. This mixture is designed to capture the beta emission and transform it into a photon emission, which can be detected via a photomultiplier tube within a scintillation counter. The cocktail must also act as a solubilizing agent, keeping a uniform suspension of the sample.

The scintillation counting system consists of three primary components: The radioactive substance, the solvent, and the solute (or fluor).



The solvent is the first compound in the scintillation cocktail to capture the energy of the beta particle. The solvent molecule achieves an excited state, and the excess energy is transferred from solvent molecule to solvent molecule. The solvent remains in the excited stated for an extended period of time, decaying into the ground state without the emission of light. The solute then absorbs the excitation energy of the solvent, and quickly returns to the ground state by emitting light. If a secondary solute is used, that solute absorbs the signal of the first solute and emits a second burst of light at a longer wavelength.

RADIOMETRY

Principle

Radiometric instruments utilise the phenomenon associated with the decay of radioactive substances, and the emissions of sub-atomic particles that is associated with this process, to monitor either rates of decay, or the concentration of the source of the emission.

The luminescence produced when radiation strikes a phosphor represents one of the oldest methods of detecting radioactivity and X-rays, and one of the newest as well. Liquid scintillation is one of the techniques relying on this phenomenon. Liquid scintillation instruments detect scintillations in a suitable liquid such as p-terphenyl in toluene, produced by low energy beta radiation from radioisotopes such as carbon-14, sulphur-35 and tritium.

The sample is generally dissolved in a solution of the scintillating liquid. A vial containing the solution is then placed between two photomultiplier tubes housed in a light tight container. The output from the two tubes is then fed into a coincidence counter, an electronic device that records a count only when pulses from the two detectors arrive simultaneously. The coincidence counter reduces background noise from the detectors and amplifiers because of the low probability of such noise affecting both sensors simultaneously.

The application of this technology to the measurement of the fineness of wool relies on the fact that the surface area of a wool fibre increases as the fibre diameter increases. This affords the possibility of absorbing the active isotope from a standard solution onto the wool fibre, separating the fibre from the solution and measuring the concentration of the isotope in the liquid. Alternatively the fibres can be labelled by immersing them into a suitable solution containing the active radioisotope, waiting until the isotope is distributed uniformly thoughout the fibre, and then measuring the beta emissions directly, with the fibre immersed in a suitable scintillator. The beta particles from the isotope within the fibre will be absorbed and therefore not detected. The visible emissions will originate only from those atoms that are located in an annulus under the surface of the fibre. The fibres can then be oxidised and re-measured to determine the concentration of isotope within the fibre. The ratio of counts before and after oxidation can be shown to be proportional to diameter as follows:

$$\frac{C_{raw}}{C_{oxid}} = \frac{4K\left(RD_s - R^2\right)}{D_s^2} \qquad 1$$

where

$C_{raw} =$ count rate of the raw fibre

$C_{oxid} =$ count rate of the oxidised solution of fibre

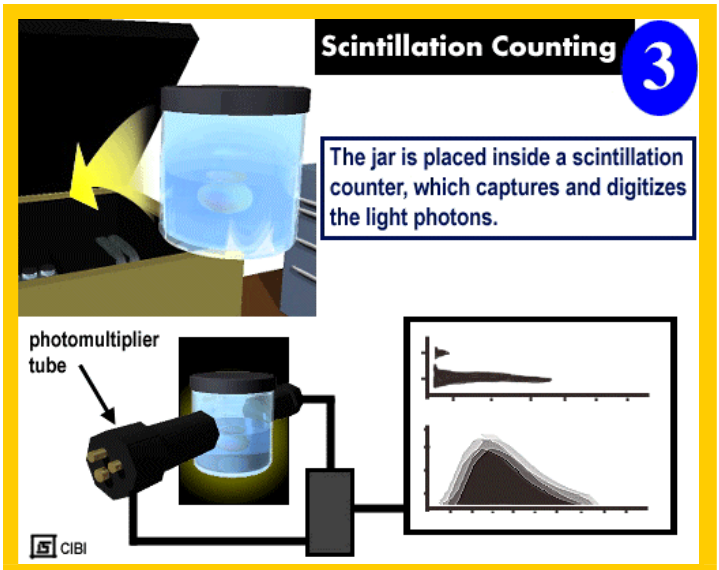
$D_s =$ diameter of the fibre

$K =$ a constant

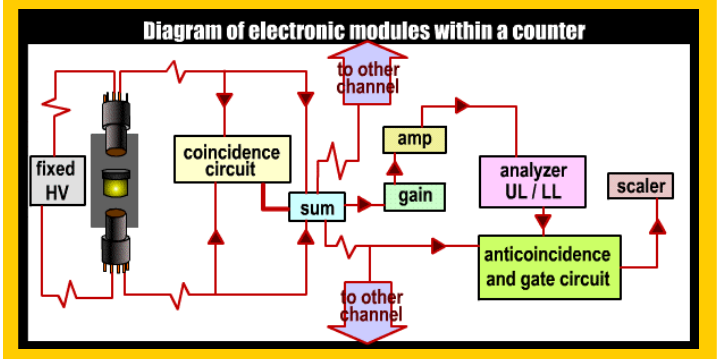
$R =$ the thickness of the annulus

Beta particles are low range and therefore the thickness of the annulus will be very much smaller than the diameter. Equation 1 therefore simplifies to

$$\frac{C_{raw}}{C_{oxid}} = \frac{K}{D_s} \qquad 2$$



Soon after the discovery of the basic principles of liquid scintillation in 1950, instruments designed for counting began appearing, with the first commercial model becoming available in 1954. A schematic diagram of a scintillation machine can be seen below:



Most commercial scintillation counters are coincidence systems utilizing Photo Multiplier Tubes (PMT's) in tandem to monitor for a photon event. A pulse is not registered unless both PMTs view the incident photons within the predetermined time interval usually 20-30 nsec. If a pulse is recorded by the two PMTs within the 20-30 nanosecond window, a coincidence pulse is recorded that is a meaure of the number of single events which occurred during the window. If an event occurs within only one of the PMTs, a coincidence pulse will not be recorded.

Source: Principles of Autoradiography, Dr S Gambhir, UCLA.

Development

Downes and Till (1963) described the application of the liquid scintillation technique to analyse the concentration of tritium, carbon-14 and sulphur-35 in wool. They reported their finding that direct measurement of previously labelled samples, counted only those isotopes that were absorbed into an annular layer on the surface of the fibre and proposed that this phenomenon could enable estimates to be made of the fibre diameter.

In a further study in 1965 these authors reported on the effects of fibre length, fibre diameter, moisture and air bubbles on the efficacy of counting scintillations produced by wool labelled with tritium and sulphur-35. They reported that tritium provided more analytical sensitivity than sulfur-35 for the estimation of diameter. They also described an investigation showing that the method could be used to estimate the degree of yellowing of the fibre. The chemical reactions associated with the yellowing process quenched the beta emissions from the labelled samples to an extent that correlated with the degree yellowing.

Downes and Till (1968) reported further studies, using formic acid labelled with carbon-14, to measure the diameter of wool. They observed a linear relationship in the count ratio with increasing fibre diameter. This fall in count ratio was small (about 1% per micron) but the sensitivity was sufficient to suggest that the technique could be suitable for the rapid measurement of mean fibre diameter for a large number of samples. They identified some disadvantages of the method:

- Pigmented and/or medullated fibres would likely interfere with the measurement and produce a bias.

- The method depended on the volume or mass per unit length of fibre and therefore the derived diameter value would be volume biased, a factor that could cause unusual results for samples with unusual fibre diameter distributions.

In a further extension of their work Downes and Till (1968) examined wool samples labelled with tritium and carbon-14 by aqueous reaction with iodoacetic acid that had been labelled with one of these isotopes. The counting rate was measured firstly, with the wool suspended in the liquid scintillation solution (the direct method) or secondly, after oxidising the same sample and dissolving the product in another liquid scintillation solution. The counting rate determined by the direct method depended on the fibre diameter, because of self-absorption of beta particles by the wool whereas the counting rate after oxidation was constant. With the wools labelled with carbon-14 the ratio of the counting rates changes by 35 % for a diameter change from 14 to 35 microns. For the tritiated samples the analytical sensitivity was considerably greater, with a 100% change in the ratio of the counting rates, for the same range in diameter. The authors reported that the results were the basis for a new method for measuring mean fibre diameter.

Finally Downes (1971) described a method using liquid scintillation for determining the mean fibre diameter of wool. A precision of 0,2 microns was reported. The instrument was calibrated against samples where the mean diameter had been previously determined by Airflow.

Technical Issues

The liquid scintillation system is a calibrated system, where the diameter obtained is directly related to the mean surface area of the fibres. It cannot provide information about distribution. It is unique among the methods that have been developed in that it is the only method that is directly related to surface area. The Airflow system also has a relationship to the surface area of the fibre, but it is less direct. Although Downes (1971) did not explicitly state it, this is probably why the Airflow measurements were used to calibrate the method.

The fact that the system relies on radioactive isotopes, may be of concern today, particularly with regard to occupational health and safety. However the isotopes that were used are handled routinely and safely in many clinical laboratories all over the world, and provided normal laboratory practice is followed they can be handled quite safely

The precision quoted by Downes is really quite amazing, but no documented studies based on inter-laboratory studies have been located. The Australian Wool Testing Authority evaluated the system during 1970's, with a view to utilising the method for flock testing services, but this work was abandoned before 1980.

The advantage of the system in this particular application is the possibility of automated analysis of large numbers of samples, where the major application of the data is for ranking animals. The Department of Agriculture, New South Wales, Australia used the method for many years, in the Department's Trangie laboratories.

Commercial Application

The technology has never been used for the commercial trading of greasy or semi-processed wool, although on the limited data available it does appear to have adequate precision. Its use for testing of Fleece Samples has also ceased, simpler, faster and more precise technologies now being available.

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HARMONICS

Principle

Fourier (1768-1830) showed that any periodic motion could be built by adding sinusoidal harmonic components of waves in proper proportions. This process applies to all oscillating systems, such as electromagnetic waves and sound waves.

The musical notes produced by stringed instruments, are the result of standing waves being established along the strings, either by plucking or bowing the strings. The frequency and the amplitude of these waves, and hence the sound they produce, is determined by the thickness and density of each string, the tension applied and its length.

Likewise, standing waves can be generated in a string by placing the string in the path of an oscillating sound source. If the string is maintained at a constant tension and length, and the frequency of the sound source is varied the string will be observed to vibrate, with a standing waveform observed along the fibre at specific frequencies, depending upon the diameter, density, tension and the length.

$$D_v^2 = \frac{K}{\nu^2} \qquad 1$$

where $D_v =$ the diameter of the string

$K =$ a constant depending on density, tension and length

$\nu =$ the harmonic frequency

Development

Gonsalves (1947) described a method for measuring a diameter of a fibre by using an instrument he called a vibrascope. Dart and Peterson (1949) using a similar instrument showed that by varying the frequency, tension, or length, the diameter of a fibre can be measured providing two of these variables are maintained constant.

Buchanan & Bolin (1952) described a simple vibrascope they had developed for the measurement of fibre diameter of single fibres from individual sheep. They found that the diameter of a vibrating wool fibre could be determined from the following equation.

$$D_v^2 = \frac{T \times 980 \times 10^8}{L^2 \nu^2 \rho \times 3.1416} \qquad 2$$

where $T =$ the applied tension in grams

$L =$ the length of the fibre in cm

$\rho =$ the density of wool

$\nu =$ the frequency of the oscillation.

The authors reported that the method correlated with measurements obtained from cross-sections examined under the microscope and was considerably faster, with forty to fifty fibres being measured in one hour.

This system measures root mean square diameter because the fibre is assumed to be circular. However the system also assumes uniform fibre thickness along the length of the fibre and it is well documented that this is not the case in almost all instances.

Technical Issues

The major limitation of this technology is that it is limited to single fibre measurements, which means that establishing a mean fibre fineness estimate for a commercial consignment would be slow and consequently expensive. The precision of the method has never been documented, which probably is an indication that it is less than the precision of alternative methods, simply because of the sampling difficulties.

Commercial Issues

The Vibrascope has never found a commercial application in wool testing and its use has generally been confined to applications where precision has not been as critical as that normally expected in commercial transactions. Within Australia the technology has been re-examined for possible applications by woolgrowers in assisting them to select animals and class the fleeces during shearing, but the cost and inherent sampling problems very quickly ruled the technology out for this application.

The Vibrascope is still used for the estimation of fineness of synthetic fibres, where variation along and between fibres (cut from the same production batch) is considerably less, and the sampling variation is much reduced.

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CONDUCTOMETRY

The Coulter Counter

Principle

Conductometry is a general term encompassing a range of measurement systems that utilise the phenomenon of the electrical conductivity of solids and liquids. It is a widely used technique in the science of analytical chemistry.

Application of the technique to the measurement of the fineness of wool has been limited. However the Coulter Counter, an instrument designed to measure particle sizes and particle size distributions has been applied to the measurement of fineness of wool tops and core samples from greasy wool.

Berg (1958) has described the Coulter Counter. A suspension of particles, suspended in a conducting liquid, which is inert with respect to the particles, is metered through a small orifice. Electrodes are located on each side of the opening, and the electrical resistance of the path from one electrode to the other varies proportionally to the volume of the particle passing through the orifice. More exactly the resistance changes proportionally to volume of conducting liquid displaced by the particle while it is passing through the resistance path. The electronics of the instrument is designed to produce a voltage pulse with the passage of each particle. The size of the pulse is the measured variable, and by calibration with particles of known dimensions the equivalent volume of unknown particles can be determined. If the calibrating particles and the measured particles have the same dimensional characteristics in at least two dimensions then the third dimension of an unknown can be inferred. In the case of roughly cylindrical particles, such as snippets of wool fibres the calibration simply relies upon ensuring that the fibre snippets of the calibrating material and the measured material are approximately the same.

Development

O'Connell and Martsch (1962) described an application of the Coulter Counter to the measurement of the fineness of wool top. The data reported by these authors is shown in the following table. The Projection Microscope measurements on the comparative tops were based on round trials between a large number of laboratories using the ASTM method.

O'Connell and Martsch concluded that the average fineness of wool tops could be measured with the Coulter Counter with a reproducibility equivalent to or better than estimates made by expert operators using the microscope. Under a set of standard conditions, they expected that the Coulter Counter would produce a higher degree of reproducibility among operators or laboratories than the projector method because operator judgement would not be a source of error.

Comparison of the Coulter Counter with Projection Microscope

Top	ASTM Projector		Coulter Counter		Differences	
	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev
57-1	20.44	4.46	20.15	4.84	-0.29	0.38
57-3	21.69	4.84	21.44	5.33	-0.25	0.49
57-4	23.60	5.78	23.13	5.94	-0.47	0.16
259-P	24.43	5.05	23.96	5.48	-0.47	0.43
57-5	26.25	6.29	26.26	6.70	0.01	0.41
233-P	26.55	6.92	27.03	7.14	0.48	0.22
57-2	27.71	6.60	27.72	6.51	0.01	-0.09
57-6	29.84	7.72	29.37	7.37	-0.47	-0.35
57-7	31.52	7.48	31.00	7.47	-0.52	-0.01
53-11	32.68	9.10	31.35	8.57	-1.33	-0.53
239-P	32.85	8.82	32.27	8.67	-0.58	-0.15
57-8	34.36	8.55	33.47	8.29	-0.89	-0.26
51-119	37.98	9.31	38.02	9.54	0.04	0.23
58-27	21.49	5.00	21.63	5.32	0.14	0.32
Lot F	30.08	8.08	29.96	8.08	-0.12	0.00
Mean	28.10	6.93	27.78	7.02	-0.31	0.08

Bloch and Gusack (1963) described the performance of the instrument in measuring denier distributions in synthetic fibres. They summarised the advantages and disadvantages of the instrument as follows:

Advantages	***Disadvantages***
Measures a large sample size (approximately 1000 fibres).	Accuracy of the results depends on the accuracy of the fibre density or alternatively on constant density.
Independence of measurements from particle shape.	Accuracy depends on the length uniformity of the sectioned fibres.
Efficient and relatively rapid procedure.	
Suitability of data for automatic processing.	
Minimising of operator fatigue and bias.	

Buras and Penoyer (1968) described an investigation into methods for rapidly preparing snippets from wool top and greasy wool cores for measurement in the Coulter Counter. They developed a system that involved shrink-wrapping the fibre bundle, and wetting the bundle to encourage the fibres to swell and provide increased support for each other. The bundle was then placed in a restraining mechanism and advanced with a precision micrometer, while the sections were taken by cutting with a double-edged razor blade. Approximately 20 minutes was required to prepare each sample.

A novel application of the Coulter Counter was reported by Andrews and Irvine (1969). This involved using the Coulter Counter simply as a counting device in an improved gravimetric method.

The novelty of the method was that the fibres were cut into snippets short enough to be easily specified. Since the number of length measurements required for a test then became too large to be practicable, an estimate is obtained of the total length of snippets in the weighed sample. Firstly the total number of snippets, *N*, was counted using a Coulter Counter. Secondly, the individual lengths in another, much smaller, sample of the snippets were measured with a Projection Microscope and averaged. The product of *N* and the average snippet length is an estimate of the total length of fibre in the sample. In such a numerical sample of snippets, cut from the original sliver or assembly, the total length of snippets in each

small interval of diameter must be proportional to the total length of fibres in the same diameter interval in the original assembly. Each fibre is therefore represented in proportion to its length, as is the case with the intact fibre gravimetric method. The precision for the method was better than 0.2 μm.

Technical Issues

Many of the technical issues that must be considered for other measurement systems must also be considered with the Coulter Counter. Obviously, because the instrument measures a volume from which fineness must be inferred, it is sensitive to the preparative systems. Uniformity in the length of the snippets is important, much more important than in other systems that are based on an estimate of the dimensions of snippets. For the same reason the density is also important. The technique has not been adequately developed to assess the effects of conditioning, but the same importance this assumes in other systems will apply.

Commercial Issues

Interest in the Coulter Counter for estimation of wool fibre fineness has not been substantial, and the instrument has remained a tool for researchers rather than a commercially used instrument.

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SEDIMENTOMETRY

Principle

Sedimentometry is the measurement of rates of settlement of particles or fibres in a fluid, where the differential settling of the particles or fibres is a function of their dimensional characteristics. Sedimentation of particles in a fluid is described by their Stokes Diameter d_s.

$$d_s = \sqrt{\frac{\mu h t}{(\rho_p - \rho_l) g}} \qquad 1$$

where:

μ = the dynamic viscosity of the fluid

h = height fallen in time t

ρ_p = density of the particles

ρ_l = density of the liquid

and g = acceleration due to gravity

A powder sedimentometer used for determining particle size distributions in fine powders.

Development

Le Compte (1948) described a method where fibres were cut into uniform lengths (not exceeding 200 micrometres), suspended in a liquid and allowed to settle. Four deposits of fibres were removed at spaced intervals and weighed, thus determining the proportions of four grades of fineness in the samples. The author described the cutter and sedimentation apparatus and reported that the method could deal with 100,000 to 200,000 fibres at a time and was rapid and objective.

Uno, Shiomi and Yanagawa (1966) devised an apparatus for measuring fibre fineness by horizontal airflow. A mass of cotton fibres was separated into single fibres in an opening box. The opened cotton was fed through a guide tube in the measuring chamber carried by a horizontal air current and progressively deposited on a board. The distribution of fibre along the board was related to fineness. A theoretical model based on the assumed Stokes diameters was evaluated and found to give good agreement with practice. The authors claimed the method compared favourably with the Micronaire.

Onions and Townhill (1968) described a method based on Photo-extinction Sedimentometry. The cut fibres were dispersed in a liquid and the settling rate monitored by turbidimetric measurements over a period of time. The instrument was calibrated by constructing extinction curves for the IWTO tops. The time at which 50% extinction occurred for each of the tops was determined and used as the criterion for defining the mean diameter of the calibration top. Using this parameter the calibration curve was constructed. Various suspension media, fibre lengths and sample concentrations were investigated. The author claimed a precision of +/- 0.5 micrometres was achieved, using 100% xylene as the suspension medium.

Technical Issues

This technique has never been seriously developed for testing wool. Consequently very little is known about the technical issues associated with the method.

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POROSITY

Principle

A modern Airflow instrument used for the determination of wool fibre fineness

The theory underlying the physics of the flow of air through porous beds of fibres is founded on the work of a French Mathematician in 1840. Poiseuille's Law describes the relationships governing the flow of fluids though capillaries.

$$u = \frac{d_e^2}{32\eta} \cdot \frac{\Delta P g}{L_c} \qquad 1$$

where $u =$ the face velocity

$d_e =$ the diameter of a circular capillary

$\eta =$ the viscosity of the fluid

$\Delta P =$ the pressure difference along the capillary

$g =$ acceleration due to gravity

and $L_c =$ the length of the capillary.

Much of the later developments have rested on the assumption that the flow of fluid through porous beds is analogous to the flow of fluid through a network of capillaries.

Darcy (1856) derived an empirical relationship to describe the flow of water through sand filter beds.

$$u = K \frac{\Delta P}{L_c} \qquad 2$$

where in this case:

$K =$ a constant

$L_c =$ the depth of the bed,

and the terms u and ΔP are effectively the same as above.

The first extension of Darcy's relatively simple model was by Dupuit (1863). Dupuit realised that the face velocity, u, must be less than the actual velocity in the pores. If the pore space in the bed is evenly distributed, then the porosity of a layer of infinitesimal thickness normal to the direction of flow must be equal to the porosity ε of the bed as a whole. For such a layer the fractional free volume must be equal to the fractional free area and the pore velocity must therefore be u/ε. He expressed this mathematically as follows:

$$u = \varepsilon \cdot K_1 \cdot \frac{\Delta P}{L_c} \qquad 3$$

where $$\varepsilon = \frac{V_c - V_m}{V_c} \qquad 4$$

and $V_c =$ the volume of the bed

$V_m =$ the volume of the bed material

Greenhill (1881) developed a theorem that described the flow of viscous fluids through pipes or channels. He demonstrated that the flow of fluid through a linear channel of defined length and cross-section can be described by complex hydrodynamic equations. The essence of his theorem is the conclusion that the solutions to these complex equations, when expressed as a ratio of the volume of the channel to the area exposed to the fluid, **do not depend critically on the shape of the channel**. He demonstrated that

$$u = \frac{\phi}{\eta} \cdot \left(-\frac{dP}{dr} \right) \cdot \left(\frac{V}{S} \right)^2 \qquad 5$$

$\phi =$ a surface shape factor

$\frac{dP}{dr} =$ rate of change of pressure with the direction of flow

$V =$ volume of the channel

and $S =$ surface area of the channel per unit volume of fluid.

Blake (1922) and Kozeny (1927) used Darcy's law and Greenhill's Theorem. They expanded on the concept of porosity introduced by Dupuit, and developed a generalised equation describing the flow of fluids through porous beds. Kozeny, in particular, assumed that a granular bed is equivalent to a group of parallel, similar channels, such that the total internal surface and the total internal volume are equal to the particle surface and the pore volume respectively. Kozeny assumed

$$m = \frac{area\ normal\ to\ flow}{perimeter\ presented\ to\ fluid} = \frac{\varepsilon}{S} \qquad 6$$

where $m =$ the mean hydraulic radius (for a circular pipe $m = diameter/4$)

The general equation that Kozeny formulated takes the form

$$u = \frac{\varepsilon^3}{k\eta S^2} \cdot \frac{\Delta P g}{L_c} \qquad 7$$

where $$S = \frac{6(1-\varepsilon)}{\phi d_e} \qquad 8$$

$k =$ the Kozeny Constant

$d_e =$ the effective diameter of the particles.

All the other terms in equation.7 have been previously defined. For a bed of spheres, equation 8 takes the form

$$S = \frac{6(1-\varepsilon)}{d_e} \qquad 9$$

The shape factor ϕ is a correction for particles that are non-spherical, and is usually experimentally determined. This term is a surface related factor, which is unity for spherical particles, and since a sphere has a minimum specific surface, the value of ϕ must be less than unity for all other non-spherical particles. The effective diameter d_e is the diameter of a sphere with the same surface area.

The extension Kozeny made to Greenhill's theorem was to recognise the equivalence between the surface area of the channel per unit volume of fluid and the surface area of the particles in a porous bed to the volume of the bed. The term S is equal to the surface area of the particles per unit volume of the bed.

Carman (1937) also recognised that the effective bed depth is greater than the actual depth (see also Fair and Hatch, 1933). Carmen theorised, and demonstrated experimentally that the Kozeny Constant could be replaced by a more exact expression such that the Kozeny Equation becomes

$$u = \frac{\varepsilon^3}{k_o \eta S^2} \cdot \frac{\Delta P g}{L_c} \qquad 10$$

where $$k_o = k \cdot \left(\frac{L_c}{L_e} \right)^2 \quad (11)$$

where $L_e =$ the effective path length.

He observed that in packed beds of glass spheres the fluid tended to flow diagonally across the bed and suggested that as an approximation

$$L_e = L_c \sqrt{2} \quad (12)$$

The literature on the study of beds of particles and powders reports values for the Kozeny constant (k) in the range 5.0 - 6.5. However experiments where the effective path length is taken into account report values of approximately 2.5 for the modified constant k_o.

Various workers have investigated the validity of the Kozeny equation in describing the factors that determine the flow of fluids through porous beds. Applications extend to engineering studies of the permeability of rock strata to liquid and gaseous fluids, water filtration, surface areas of fine powders, chemistry of catalysis in packed towers, the specific surface of cotton fibres, and of course the mean fibre diameter of wool.

Carman (1938), while applying the Kozeny equation to the study of the surface area of very fine powders, recognised that the value of S, the surface area per unit volume of the bed, is very difficult to determine experimentally. It requires knowledge of the size, shape, volume and packing of the particles. If the size and shape of the particles are constant, then

$$S = (1 - \varepsilon) S_0 \quad (13)$$

where the **Specific Surface**[1]

$$S_o = \frac{A_m}{V_m} \quad (14)$$

and $A_m =$ surface area of the particles

$V_m =$ volume of the particles

By assuming specific dimensional characteristics of a porous bed, it is relatively simple to derive the more familiar general expression of the Kozeny equation from equation 7 or equation 10.

The flow of fluid through a porous bed is a function of the face velocity and the cross-sectional area of the bed. Therefore

$$Q = u A_c \quad (15)$$

Substituting 13, 14 and 15 into equation 7 gives the following general expression.

$$Q = \frac{1}{k} \cdot \frac{\Delta P g A_c}{\eta L_c} \cdot \frac{\varepsilon^3}{(1 - \varepsilon)^2} \cdot \left(\frac{V_m}{A_m} \right)^2 \quad (16)$$

[1] This is a different definition to that commonly used in the textile industry, where specific surface is defined as area per unit mass

There has been an enormous amount of work reported in the scientific literature concerning the generality and validity of the Kozeny Equation. There are three significant limitations that have been reported (Carman, 1937, 1938, 1939).

- Initial studies were limited to beds consisting of grains of uniform size. When there is a distribution of grain size, the porosity of the bed increases, compared with a uniform bed of the same specific surface and volume. The equation does not explain this, although for distributions of particles with regular geometrical shapes it can be predicted.

- Under certain conditions, the pressure drop will rise more rapidly than the rate of flow, all else remaining equal. This is the result of turbulent rather than laminar flow of the fluid though the pore spaces. Thus the Kozeny equation only applies under conditions of laminar flow.

- Under certain conditions the flow will actually increase faster than the pressure. This is attributed to slippage of the fluid at the molecular interface at the surface of the bed material. This behaviour can cause a departure from linearity in the flow vs. pressure relationship, particularly at relatively small flows.

Cassie (1942) was the first to investigate the application of the Kozeny equation to the determination of the mean fibre diameter of wool, and it is from his original work that the Airflow system has developed into an IWTO standard test method. The principle of the air-flow instrument is based on the observation that plugs of randomly oriented wool fibres, behave in much the same way as porous beds of powders, and that the flow of air through a plug of wool can be described by the equation

$$Q = K_a \cdot \frac{A_c}{L_c} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2} \cdot \Delta P \cdot \left(\frac{V_m}{A_m} \right)^2 \qquad 17$$

By making an assumption about circularity it is relatively easy to show that

$$Q = K_b \cdot \frac{A_c}{L_c} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2} \cdot \Delta P \cdot d^2 \qquad 18$$

where d = the mean diameter.

Development

The earliest work that applied the Kozeny equation to fluid flow through fibres was by Wiggins, Campbell and Maas (1939). These authors studied the flow of liquids through beds of glass wool, and celanese yarn and found the Kozeny equation adequately described the flow behaviour. They varied the length to diameter ratios from 50:1 to 200:1 with no apparent effect, and reported that distributions in diameter also had no apparent effect, provided that the range in diameter did not exceed 5:1 and the proportions of each diameter class were approximately equal.

Fowler and Hertel (1940) studied the flow of air through plugs of cotton, wool, rayon and glass wool fibres. They found a substantial compliance with the predictions of the Kozeny equation using a gaseous fluid (air) instead of liquid fluids. Fowler and Hertel used the equation to estimate the density of these fibres, obtaining data in the range 1.26 - 1.33 for wool. The authors observed that the constant factor in the Kozeny equation depended upon the shape and orientation of the fluid passages. This is in fact a misinterpretation of the fundamental basis of the Kozeny equation, which is actually based on the assumption that the shape of the passages is not a critical factor (Greenhill, 1881). This confusion seems to have entered into the literature because the general law for laminar flow through a channel does assume a form very close to the Kozeny equation and the constant in that expression is theoretically dependent on the shape of the cross-section of the channel. This theory predicts that for a circular channel the predicted constant will be 2.0. However there are many systems to which the Kozeny equation has been applied which give a value of k = 2.0, and where it is generally recognised that the cross-section of the pores is not circular (Carman, 1937).

Sullivan and Hertel (1940) investigated the airflow through plugs of cotton. As a consequence they proposed that the fibre surface per gram be used as a measure of fibre fineness. They found that the Airflow method determined the surface area per gram within a standard error of 3%, and that the mean from a single Airflow test gave the same accuracy as did the mean from the microscopic examination of 2500 fibres.

Grimes (1942) applied the method reported by Sullivan and Hertel to 36 cottons, and found that the method could be used satisfactorily for the measurement of the fineness. Grimes particularly emphasised the advantage of the method over more traditional approaches with regard to time and consequent cost.

Cassie (1942) reported the first detailed study of the Airflow technique to estimate the fineness of wool. Cassie measured the flow of air through small plugs of top, where the fibres in the plug were approximately parallel, and the pressure difference across the plug was maintained constant. Cassie rearranged the form of the Kozeny equation reported by Carman as follows:

$$\frac{Q}{A_c} = \frac{1}{k\eta S^2} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2} \cdot \frac{\Delta P}{L_c} \qquad 19$$

where the terms are defined as previously. Cassie repeated the mistake made by Fowler and Hertel in describing the Kozeny constant in this equation as a "*dimensional shape factor depending on the size and the shape of the pores*".

Cassie then assumed that the fibres in the plug were circular in cross-section, and hence the surface area per unit mass could then be defined in terms of the mean diameter. Neglecting the areas at the end of the fibres the surface area then becomes $\pi d\ell$ where ℓ is the length, and the volume is $\pi d^2 \ell / 4$; or

$$S = \frac{4\pi d\ell}{\pi d^2 \ell} = \frac{4}{d} \qquad 20$$

Cassie further rearranged equation 19 by expressing the porosity n terms of the mass and the density of the fibre.

$$\varepsilon = 1 - \frac{m}{\rho V_c}$$

where $m =$ the mass of the plug

$V_c =$ the volume of the plug

and $\rho =$ the density of the wool.

By expressing the flow in terms of the volume v of air that flows through the plug in time t it follows quite simply that

$$d^2 t = \frac{16k\eta v}{\Delta P} \cdot \frac{\rho V_c L_c}{A_c} \cdot \frac{m^2}{(\rho V_c - m)^2} \qquad 21$$

Cassie also observed that it would appear from equation 21 that the system will be sensitive to variations in density between samples, and he showed that for small changes in density the effect on mean diameter could be described by equation 22

$$\frac{\Delta(d^2 t)}{(d^2 t)} = -\frac{2\rho V_c + m}{\rho V_c - m} \cdot \frac{\Delta\rho}{\rho} \qquad 22$$

On this basis a variation of density of 1% would give an error of 2.5% in the mean diameter in the situation where $m = \frac{1}{2}\rho V_c$. If the mass becomes greater than this then the error rapidly increases.

In his experiments, Cassie found that the estimated diameter was not dependent upon the volume of the plug, provided that the mass to volume ratio was constant. He found that a wool density of 1.35 g/cc had to be assumed instead of the accepted value of 1.315 g/cc to get consistent results. Following from equation 22 Cassie observed that unless the density of wool from different sources is constant the method was unlikely to give consistent results. Furthermore he noted that any oil on the fibres must be removed prior to measurement, and that the measurements would be sensitive to regain. It is worth noting at this point that Cassie, and all the earlier workers, were not working with a calibrated instruments, because at that time no calibration material was available. Table 1 shows estimates made by Cassie for the density of wool ranging from 10.0 to 32.9 micron as well as the value of the Kozeny constant obtained under his experimental conditions.

TABLE 1: Estimates of Density and of the Kozeny Constant (Cassie, 1942)

Micron	L_c = 5/16 inch		L_c = 5/8 inch	
	Density	k	Density	k
32.9	1.37	2.17	1.31	2.47
29.0	1.36	2.22	1.35	2.10
27.1	1.42	3.11	1.35	2.25
23.7	1.39	2.47	1.37	2.32
22.7	1.35	2.32	1.35	2.17
19.5	1.38	2.47	1.37	2.47
19.1	1.35	2.10	1.39	2.60
19.0	1.305	1.79	1.36	2.40
Mean	1.366	2.33	1.356	2.35

The values obtained for the Kozeny constant by Cassie are quite variable. The mean value however is within the range reported by Carmen for k_o. and substantially less than the values commonly reported for k. This is likely due to the fact that Cassie arranged the fibres in parallel alignment, and hence $L_c \approx L_e$.

Anderson and Warburton (1949) demonstrated that that this was in fact the case. They investigated the effect of randomising the orientation of the fibres in the plug by using packed beds of cut and randomised fibre pieces. They also attempted to correct for slippage effects. They found that the randomised arrangement reduced the coefficient of variation in the estimate of the Kozeny constant from 19% to 4%. The Kozeny constant increased from approximately 2.3 to 6, which is much closer to the values reported for powders and granular beds (Carman 1937,1938, 1939; Rose, 1945; Arnell, 1947; Carmen & Arnell, 1948). The results of the Anderson and Warburton estimates using parallel and randomised orientations are shown in Figure 1. The range of k for the randomised arrays is actually not substantially less than for the parallel arrays. However the coefficient of variation is substantially less because of the higher mean value of k.

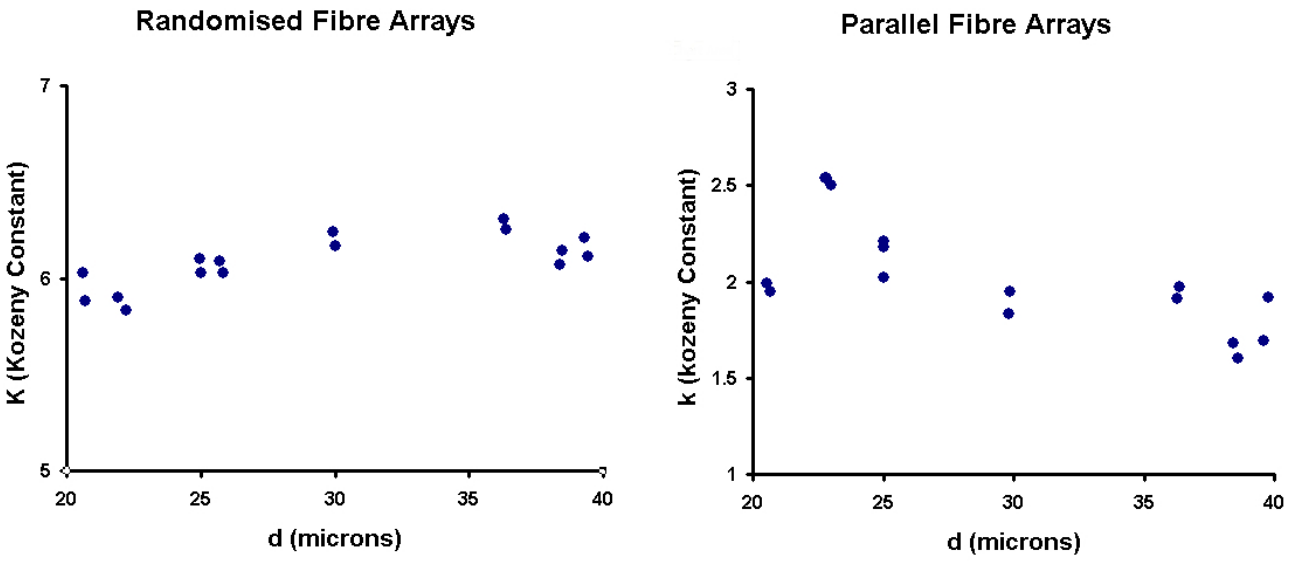


FIGURE 1: The effect of fibre orientation on the value of the Kozeny Constant (Anderson & Warburton, 1949)

Note: These graphs have been redrawn from Anderson & Warburton's original paper. However, the authors used different scales on the vertical axes, which made the value of k obtained from the parallel arrays appear much more variable than those from the randomised arrays. The vertical scales in the above have been made the same. The key point is the large difference in the value of k, although the parallel arrays are still more variable.

Anderson and Warburton also made the same error as Cassie in referring to the Kozeny constant as a shape factor. The close association obtained by Anderson and Warburton with the results of Carmen et al, where the shape characteristics of the materials are very different, simply highlights this. The Kozeny constant is not directly dependent on the shape of the pores between the particles or fibres, and a particular value for the constant does not imply a particular shape for the pores (Carmen 1937).

Anderson and Warburton also examined the relationship between fineness estimates derived from Projection Microscope measurements and fineness determined by the Airflow technique. They pointed out that the fibre fineness of wool is variable between fibres and along fibres, and accordingly the approximation of **uniform** circularity implied by equation 4.5.20, is too simplistic. More rigorously

$$S = \frac{\int 4\pi d\,\partial\ell}{\int \pi d^2\,\partial\ell}$$

where the integrals are then over the total length of the fibre in the plug. It can be shown that

$$S = 4 \cdot \frac{\bar{d}}{\bar{d}^2}$$

where $\bar{d}$ is the length proportioned mean diameter as determined by the Projection Microscope (note that this still assumes circularity of cross-section) and $\bar{d}^2$ is the similarly proportioned mean square diameter. Anderson and Warburton then demonstrated the familiar relationship

$$d = \bar{d}\left(1 + C^2\right) \qquad 22$$

where $C =$ the fractional coefficient of variation in $\bar{d}$

Finally Anderson and Warburton derived an equation from which they could determine the mean fibre diameter of fibres in a randomly oriented plug with a precision of 2.5%. Note that this was a direct determination based on Airflow measurements and empirically determined constants.

TABLE 2: Comparison of Projector and Micronaire (Palmer 1948)

Top	Projector	Corrected Micronaire	Original Micronaire	Difference (uncorrected)	Difference (corrected)
CG	19.06	19.97	19.68	0.62	-0.29
BL	20.1	20.96	19.83	-0.27	-1.13
FJ	20.58	21.69	20.9	0.32	-0.79
AD	20.98	21.97	21.26	0.28	-0.71
HI	21.58	22.68	21.51	-0.07	-1.17
EK	23.52	25.33	23.35	-0.17	-1.98

The Cotton Industry continued its development of the Airflow technique separately, the Micronaire instrument appearing in 1947. Measurements derived from this instrument were included in the 1948 round trial reported by Palmer (1948, 1951). The data obtained by the instrument compared with the Projection Microscope and gravimetric techniques also included in this round trial are shown in Table 2. There are two points of interest here. Firstly the agreement is excellent considering the relative immaturity of the Micronaire. Secondly the correction of the Micronaire results, using the coefficient of variation as suggested by Anderson and Warburton (equation 22), actually made the differences between the two instruments worse rather than better. Palmer suggested that perhaps the manufacturer had already included the correction in the calibration of the instrument.

Calkins (1950) reported on comparisons between different instruments, designed on the Airflow principle, and used for estimating the fineness of cotton.

Brown and Graham (1950) reported an investigation into different ways of calibrating the instruments. Their data showed that when the instruments were calibrated in terms of weight per unit length (i.e. fineness) the curves for different varieties of cotton were quite different. This was due to the fact that the resistance to Airflow through a fibre plug correlated closely to the surface area of the fibres in the plug rather than their fineness. The surface area of cotton varies variety and maturity, independently of the fineness.

Lord (1955) published the results of an extensive study of the Airflow system, using cotton, viscose rayon, cuprammonium rayon, wool and silk. He systematically examined all the variables in the Kozeny equation, and thoroughly demonstrated the validity of the theory on which it was based. Lord (1956) later reported a detailed study of the various factors influencing measurements on cotton obtained by the Micronaire instrument. His study showed that both fibre fineness and maturity affect the results and described a calibration of the scale of the instrument using a simple function of these two quantities. The interpretation of the Micronaire results was discussed, and applications of the method to mill checking of cotton quality, and to cotton breeding, were discussed, with particular reference to the problems arising from the dependence of Micronaire test values on fibre fineness and maturity. The nature of variation in Micronaire values associated with differences occurring between samples, between bales and within bales was investigated. Estimates of the contribution to the precision of various sources of variance were also discussed.

The relationships defined by the Kozeny equation suggested that two types of instruments were possible. Consider equation 18 again.

$$Q = K_b \cdot \frac{A_c}{L_c} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2} \cdot \Delta P \cdot d^2$$

Clearly in any instrument the variables A_c, L_c and ε can be maintained constant, simply by deciding on fixed dimensions for the chamber and a constant porosity. The porosity is related to the mass and the density, so by setting a constant mass and assuming constant density, the only variables that remain in the relationship are ΔP, Q and d, and equation 4.5.19 simplifies to

$$Q = K_c \cdot \Delta P \cdot d^2 \qquad 23$$

Therefore a suitable instrument can be designed in two ways:

- Constant flow and variable pressure
- Constant pressure and variable flow

Two such instruments, Version A (constant flow, variable pressure) and Version B (constant pressure and variable flow) were described by Anderson (1954), together with a procedure for preparing the samples, calibrating the instruments with tops of known diameter, and conducting the measurements. It is of interest that Anderson clearly infers that the former was non-linear and the latter was approximately linear. From the form of equation 23 one would expect both instruments to be non-linear[2]. However the flow meter for these instruments is manufactured so that the height of the rotameter in the flow meter is related to the square of the flow, which in the constant pressure device makes the flow meter scale linear to diameter (James, 1970)[3].

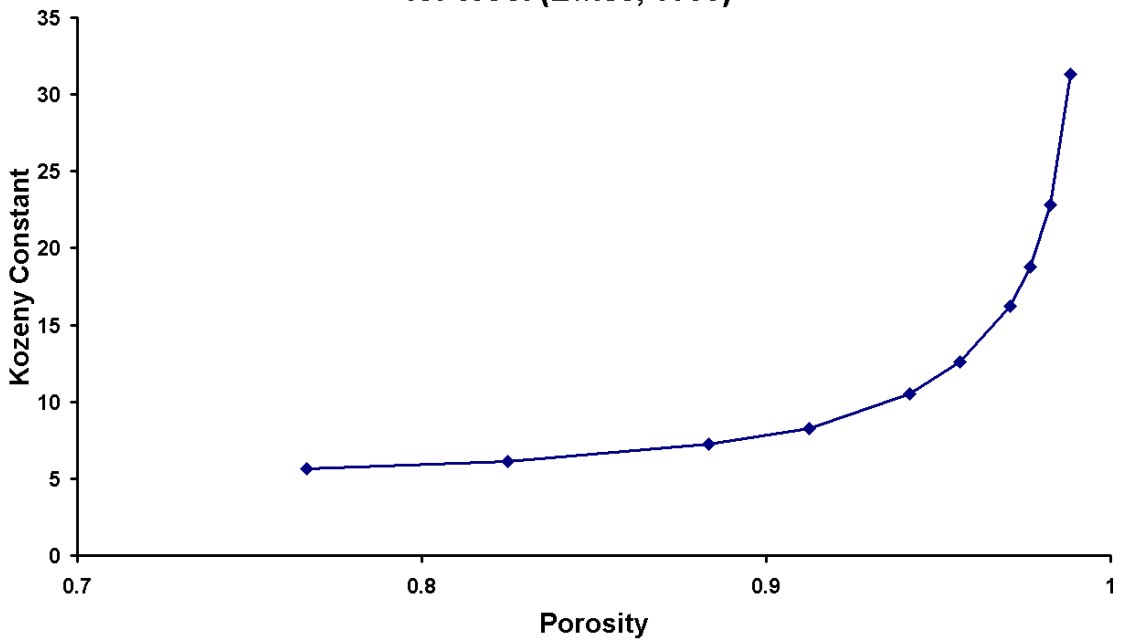
Richards (1954), using a Version B Airflow instrument examined the effect of oil on the fibres and changes in porosity on the Airflow measurement of the fineness of wool. She remarked, " *There is evidence to suggest that, when the fibres are comparatively widely spaced, their resistance to fluid flow is more nearly described in terms of the aerodynamic resistance of a number of circular cylinders. This and possibly changes in density caused by medullation for coarse fibres lead to a nearly linear relation between Airflow and fibre diameter, which is not expected of the Kozeny equation. However, it appears that under the conditions previously described for wool fibre diameter measurement, the Kozeny equation gives a sufficiently accurate description*".

Lord (1955) also observed the effect of porosity on the Kozeny constant (see Figure 2). This work has not been widely cited but the curve in Figure 2 demonstrates the departure from the Kozeny equation that one would expect with increasing porosity. Unlike fine powders and sands, fibres exhibit a high degree of elasticity. At high porosities the interstitial spaces between the fibres will increase so much that the capillary model that is the basis of the Kozeny equation will no longer apply, and the flow will certainly begin to approach the circular pipe behaviour described by Richards. The interesting feature of Figure 2 is the way the curve asymptotes to a value of k equal to 5. This is identical to the values reported by Carman (1937) for fine powders. It simply indicates that if the porosity of the plug is greater than 0.7 then the Kozeny constant for the system is in part being affected by the elasticity of the fibre and the fact that the fibre has not been completely compressed. This will cause departures from Kozeny's assumption concerning the mean hydraulic radius of the pores

[2] The linearity or otherwise of the flow diameter relationship in the constant pressure instrument has been a source of confusion for 40 years. In a letter to the Journal of the Textile Institute, Mann (1970) suggested that the regression equation in IWTO-28 should reflect the theoretical model. Anderson (1970) responded to this and suggested that a logarithmic function would be more suitable than the quadratic expression in the standard. It remained for James (1970) to remind both Mann and Anderson that the flow meter selected for the Airflow instrument was designed so that the height of the rotameter was proportional to the square of the flow, and thus it was directly related to the diameter. In a lively rejoiner Anderson pointed out that the flow meters, when manufactured in bulk, did not always adhere to this design and small departures from linearity in the height vs flow relationship may be observed. For this reason Anderson expressed his preference for the constant flow instrument. In more recent years Baxter, Brims and Taylor (1992) have also suggested that the constant pressure Airflow instrument is non-linear, suggesting that they too did not understand the basis of the design of the instrument

[3] This letter from James to the Journal of the Textile Institute is the only reference the author has been able to locate where this fact about the design of the instrument has been clearly stated.

FIGURE 2: Effect of Plug Porosity on The Kozeny Constant for Wool (Ewles, 1955)



.These effects were investigated and explained in detail by Sommerville (1998).

Monfort (1954) reported on the industrial application of Anderson's Version A instrument. "*We have examined Anderson's apparatus (Version A) and seen this used in industrial conditions. It has given entirely satisfactory service and it has replaced the lanometer for 90% of the fineness measurements by Peltzer and Fils*." Monfort remarked that it was unfortunate that the instrument did not provide estimates of coefficient of variation, but this measurement was rarely used in any event. Monfort confirmed that the instrument was very precise in the industrial application and also demonstrated the instrument behaved very much as predicted by the theory.

A description of the Version A instrument was provided by WIRA (1955). The method of calibration and sample preparation and measurement was also described. It is clear that Anderson (and therefore WIRA) preferred the Version A instrument to the Version B, despite the fact that it was non-linear. However Anderson had found that the between instrument reproducibility of the Version B could be improved by first calibrating the flow meter of the instruments and then calibrate the diameter directly to the flow, rather than to the height of the flow meter.

Settle (1955) described an application of the principles of the Airflow instrument to the rapid estimation of the diameter or cross-section of single fibres.

Anderson (1955) published a conversion table for the Airflow results for different percentages of relative humidity of the surrounding air and demonstrated the validity of this adjustment for tops.

Robinet and Franck (1958, 1959) applied the Airflow instrument to the measurement of the fineness of wool from skins of lambs, adult sheep and slipe wool. They used a Projection Microscope to verify the results, and the recommended IWTO method was used in setting up the Airflow. The authors found that the Airflow was up to 7% lower than the microscope. They suggested this was due to the tapering of the tip of the fibre. They also reported difficulty in measuring slipe wool. These authors were apparently unaware of the possible effects of fibre density, originally identified by Cassie (1942) which would have also contributed to these differences as medullated or kemp fibres are frequently present in large numbers in samples from these sources.

Monfort (1960) summarised the results of an inter-laboratory trial in which 10 tops were distributed to 17 laboratories, 6 to be used for calibration and 4 to be measured. From this data Monfort calculated the sampling and testing components of variance. Monfort also proposed some tolerances to be used to determine the acceptability of Airflow measurements of fineness of wool tops (see Table 3)

TABLE 3: Tolerances between routine measurements by Airflow (Montfort 1959)

Micron Value	Probability of a value exceeding specified tolerance			
	3 chances per 100		1 chance per 100	
	2 measurements	3 measurements	2 measurements	3 measurements
20	0.27	0.25	0.35	0.32
25	0.42	0.40	0.54	0.51
30	0.61	0.57	0.79	0.74
33	0.85	0.81	1.10	1.05

In 1960 the International Wool Textile Conference implemented IWTO-6, the first IWTO standard for the WIRA[4] Airflow instrument. It is of interest that WIRA chose to commercialise the constant pressure version of the instrument (Version B) rather than the constant flow version (Version A). Anderson (1970) reported that this was a commercial rather than a technical decision. "*It is interesting to note that the constant-flow type of air-flow instrument does not require any intermediate flow meter calibration, since only one portion of the float is used. Furthermore the heights of the manometer menisci above a datum point may be considered fundamental units of pressure. This was realised at the time, and, in fact, this type was the laboratory prototype of the Airflow method.* ***However it was reluctantly decided that the very marked non-linearity of the scale made it much less acceptable to industry, so the constant pressure type was chosen for the commercial instrument***."

The development of IWTO-6 prepared much of the ground for the extension of the Airflow technique to the measurement of fineness of greasy and scoured wool. However some work still remained.

Greuel and Sustmann (1961) considered the influence of atmospheric temperature and pressure proposed a correction to be applied whenever these conditions differed widely from the conditions of calibration.

Anderson (1963/2) examined the use of the shirley analyser to open scoured wool prior to measurement and reported the existence of systematic differences.

James and Bow (1968) described a satisfactory procedure for determining with Airflow the mean fineness of greasy wool sampled from bales by pressure coring. These authors used a wool model Shirley Analyser to remove extraneous vegetable matter, and dirt from a scoured sample from the core sample. They found that the Shirley Analyser blended the wool fibres and actually improved the precision of the measurement. The Shirley Analyser, once set up correctly, and provided an excessive number of passes of the sample was avoided, removed less than 2% of the fibre from sample and did not have an effect on the mean fibre diameter. A precision of ±0.5 micrometres for wool cores ranging from 16 to 27 micrometres was achieved. However it was found that it was necessary to process the calibration samples through the analyser to achieve these results. In effect the calibration material needed to have much the same length distribution as the core samples, and the fibres needed to be randomised and mixed to the same extent in the Shirley Analyser.

[4] In the United States the Micronaire and the Port-Ar instrument were also utilised for measurement of the fineness of wool. Kirby, Johnson and Larson (1976) reported comparative study of the Port-Ar instrument and the WIRA instrument which showed that while the instruments were highly correlated there was a diameter dependent bias in the range 22 to 32 micrometres with the WIRA instrument becoming progressively finer as the diameter increased. Hourihan, Terrill and Wilson (1970) reported a comparison of the Micronaire, Port-Ar and Projection Microscope methods and concluded either of the Airflow instruments were satisfactory.

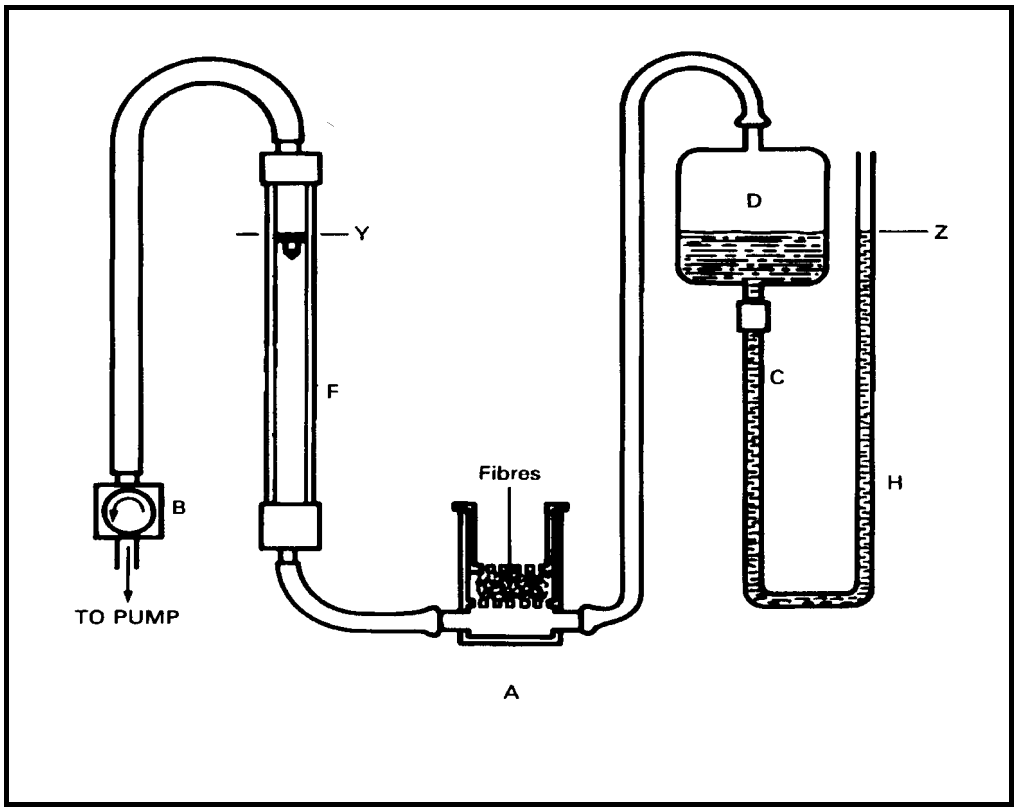


FIGURE 3[5]: The design of the constant pressure Airflow apparatus used in IWTO-6 and IWTO-28 was developed by WIRA. Most of the early development work on the method was done using the constant flow instrument. However the fundamental principles on which both instruments are based are the same. The illustration shows in a schematic form the operation of the instrument. The apparatus consists of a constant volume chamber (A), a manometer (H) connected by tube (C) to a fluid reservoir (D), a flow control valve (B) connected to a vacuum pump, and a flow meter (F). The plug of wool fibres is placed in the chamber A and compressed to a constant volume by a perforated plunger. The valve B is opened until the flow is stable and the manometer liquid level is stabilised at a constant point Z. The height Y of the rotameter in the flow meter is then recorded.

It is noteworthy that the usefulness of the Airflow system for a wide range of textile fibres continued to be investigated. Sinha and Bandyopadhyay (1968) described the application method to the measurement of fineness of jute and mesa. They found that the predictions of the Kozeny equation were also valid for these fibres. However a parallel array of fibres was preferred. As a consequence of this work Sinha and Bandyopadhyay developed a simple constant flow instrument for use in rural areas of India.

Downes and McKelvie (1969) described the effects of vegetable matter sand and dust on fineness measurements using the Airflow instrument. The impetus for this work was the growing interest in the method for fineness measurements on greasy wool. Downes and McKelvie found that provided the amount of vegetable matter did not exceed 5%, and coarse sandy dirt is not present in an amount exceeding 1%, it was likely that the error in the indicated diameter resulting from the combined effects of these contaminants would fall in the range 0 to +4.2%. In short the error in the fineness measurement would not exceed ± 2.1% at the 95% confidence interval. In a convoluted way these authors simply confirmed that contamination by vegetable matter and/or sand could significantly effect the measurement of fineness by Airflow.

[5] The diagram in Figure 3 is extracted directly from IWTO-6, courtesy of the International Wool Textile Organisation

Henning and McKelvie (1969) confirmed the earlier work by James and Bow (1968). In this instance the authors compared the results of Airflow measurements with Projection Microscope measurements on core samples obtained from 27 lots of scoured wool. The core samples were hand carded before the Airflow tests were conducted. The sub-samples for the Projection Microscope were cut into snippets, 1 mm long in order to obtain a length-biased sample. The Airflow instrument was calibrated with reference tops in five different forms. The results showed that there was a systematic difference between the Airflow and the Projection Microscope measurements of mean diameter of scoured wool when the Airflow instrument was calibrated with reference tops in their original state. A better agreement was obtained by hand carding the test samples of the reference tops, but the closest agreement was obtained by cutting, washing, and hand carding the calibration samples so that their condition was similar to that of the core samples. Provided this type of calibration was applied the mean diameter of the scoured wool could be determined by Airflow testing the cored and hand-carded samples.

Roberts (1959) proposed that a correction for the effects of coefficient of variation on fineness measurements by Airflow should be made. This proposal was based on the relationship derived by Anderson and Warburton (1947) (see equation 22). Assume a calibration top has a mean diameter d_o and a coefficient of variation C_o, determined by the Projection Microscope. From equation 22 the Airflow diameter, d_a, of this top will be

$$d_a = \bar{d}_o \cdot \left(1 + C_o^2\right)$$

If another top is measured which has a Projection Microscope value of $\bar{d}_1$, a coefficient of variation C_1 and the same Airflow diameter d_a, then, according to Roberts, the equivalent Projection Microscope value of this second top will be obtained as follows[6]:

$$\bar{d}_1 = \bar{d}_o \cdot \frac{1 + C_o^2}{1 + C_1^2} \qquad 24$$

James and David (1968) stated that differences between the results obtained for the fineness of wool tops measured by Airflow and the Projection Microscope can arise because the coefficient of variation of the measured sample is different from that of the tops used to calibrate the Airflow instrument. However the authors did not proceed to demonstrate this effect[7]. Rather they simply reanalysed information reported by Ott[8] in an attempt to develop a correlation between mean fibre diameter of tops, and standard deviation, and thereby provide a means of correcting the Airflow results for the effect of coefficient of variation. The aim of this work was to establish criteria for selection of tops, based on standard deviation. If the calibration tops were broadly representative of commercial tops in terms of coefficient of variation, then biases in individual tests arising from the effect of coefficient of variation would be minimised.

The original work of Anderson and Warburton (1947), and later work by Ewles (1955) showed that the Airflow through a plug of wool fibres is sensitive to the arrangement of the fibres. James and Bow (1968) and Henning and McKelvie (1969) had demonstrated the importance of preparing the calibration standards in the same way as the samples to be measured. Downes and McKelvie (1969) considered these issues in more detail. These authors investigated the shift in the values obtained from the Airflow instrument produced by subjecting tops to a variety of treatments:

- Cutting;
- Coring;
- Hand washing;
- Passage through the Shirley analyser; and
- Steaming

[6] Since Anderson and Warburton originally proposed this relationship it was finally verified experimentally in 1997. Roberts alluded to experimental data supporting the theory, but it has never been published. Despite this absence of verification for nearly 50 years numerous authors have cited the relationship.

[7] The practical difficulties in actually detecting this effect are probably the major reason why its verification has taken so long. The theory predicts a variation of 3% in coefficient of variation, at a mean diameter of 20 microns, will cause a bias of only 0.2 microns in the Airflow diameter.

[8] R. Ott, Bulletin Inst. Text. France, **77**, 63, 1958

These were applied singly or in certain combinations. In all cases, the treatment produced an apparent increase in mean fibre diameter i.e. increased air permeability, the magnitude of the increase rising with increasing diameter. The change in apparent diameter was considered to arise from an increase in pore size distribution in the fibre mass, which was the result of disordering the fibre arrangement[9].

James (1970) reported the results of an inter-laboratory trial, involving eight laboratories, in which the laboratories measured the fineness of core-samples of greasy wool. The laboratories all used a standardised procedure, based on a WIRA constant pressure Airflow instrument, the essential elements of which were as follows:

- The reference wool tops for calibrating the instruments were cut into 0.75 inch lengths, hand mixed and then passed twice through the shirley analyser. These reference samples were conditioned in a standard atmosphere for at least 4 hours, and three sub samples of each reference top were measured. Readings of centimetre height on the flow meter were recorded. These calibration results were fitted by the least-squares method to quadratic equations within each laboratory and printed out as tables of values for diameter in microns against 1 mm intervals of flow meter height.
- The core samples were washed with water and detergent, dried and then conditioned to 25-33% regain before processing through the shirley analyser. A standardised procedure for passing the sample and the reject was adopted.
- The carded samples were dried to a bout 5% regain and then conditioned for 4 hours in a standard atmosphere. Three sub-samples from each conditioned and carded sample were measured
- The measured sub-samples were washed in alchohol, re-conditioned and remeasured
- Flow readings were recorded to the nearest mm of flow meter height and converted to mean fibre diameter using the calibration table.

As a result of this trial tentative limits of precision of the method were established. The mean difference between any two laboratories ranged from 0.2 microns at 20 microns mean diameter to 0.5 micrometres at 28 micrometres mean diameter. This trial formed the basis for IWTO-28, which was accepted by IWTO in 1971.

Sonic Airflow

Any review of the development of the Airflow system would not be complete without reference to the Sonic Airflow developed by CSIRO in 1971. Stearn (1970) described the theoretical basis of this variant of the instrument. The fundamental principles are identical to the WIRA instrument and other derivatives. However the flow of air through the plug of fibre is produce by creating a fluctuating pressure above the fibre plug, using an oscillating sound frequency of 50 hertz (hence the term “Sonic”). The resistance of the fibre plug attenuates the pressure fluctuations in an air space below, in a way that is proportional to the mean diameter of the fibres in the plug. The attenuation in the pressure can be detected electrically, and displayed in a calibrated instrument directly as micrometres.

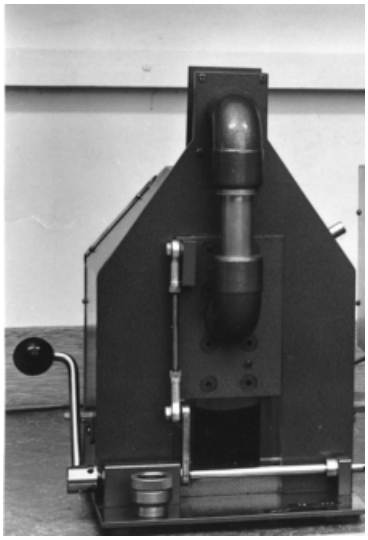


FIGURE 4: The Sonic Airflow Instrument

Consider a container in which, on top of an atmospheric pressure P_0 is superimposed a pressure fluctuation of $P_1 Sin\omega t$. Another container, of volume V is connected to the former via a porous resistance to flow R. Superimposed on the atmospheric pressure in this vessel is a fluctuation P derived from the flow through the resistance R. If we consider the first container, and assume the pressure fluctuations within it to be adiabatic, then

[9] In a theoretical model Carmen (1937) demonstrated that an increase in pore size distribution increased the apparent permeability of porous beds. Stearn (1971) published an elegant theoretical explanation of this effect. Stearn suggested that the alignment of fibres increased the number of fibre to fibre contacts. Disorientation of fibres reduces the number of contacts per unit length, thereby increasing the porosity of the fibre mass.

$$\gamma P = \frac{PV}{\nu}$$

where $\gamma P =$ the adiabatic elasticity of an ideal gas

$\nu =$ volume of air compression required to give the increment P

Let $m =$ the mass of air in the volume V

And $m' =$ the amount at any instant that has flowed into V

It follows that $\frac{\nu}{V} = \frac{m'}{m}$

Therefore $P = \frac{P_0 \gamma m'}{m}$

From Darcy's law (equation 2)

$$\frac{P_1 \sin \omega t - P}{R} = \frac{kT}{P_0} \cdot \frac{\partial m'}{\partial t}$$

Where $k =$ the universal gas constant

$T =$ mean temperature

$\frac{kT}{P_0} =$ a factor to convert mass flow into volume flow.

From these expressions, and using the equation for an ideal gas $P_0 V = mkT$, it is possible show that

$$R\tau \frac{\partial P}{\partial t} + P = P_1 \sin \omega t$$

where $\tau = \frac{V}{\gamma P_0}$

The steady state solution to this equation is

$$P = \frac{P_1 \sin(\omega t - \psi)}{\sqrt{1 + \omega^2 \tau^2 R^2}}$$

where $\psi = \tan^{-1}(\omega \tau R)$

If $\omega \tau R > 1$ then $P \propto -\frac{P_1}{\omega \tau R} \cdot \cos \omega t$

It follows that P is inversely proportional to R provided all other factors are constant. This relationship was utilised by CSIRO in designing the Sonic A fibre fineness tester.

James and Stearn (1971) described the prototype of this instrument. David and Ward (1973) reported on commercial trials of the instrument and found the Sonic Fibre Tester "*to give fibre diameter values which do not vary seriously from those obtained on the same wool samples using the WIRA Airflow instrument. The observed differences are, in fact, no greater than are likely to be found between two WIRA instruments. When used under routine testing conditions the Sonic tester has been found to provide some operational advantages*." The authors recommended that IWTO consider the inclusion of the instrument as an acceptable method for the measurement of mean fibre diameter both for wool tops and wool core samples.

The Sonic A machine was never put into commercial use by any of the major test houses. Stearn, Andrews, Bloos, Bow and Harley (1974) described the Sonic B machine. Irvine (1973) developed a simple mass comparator for use with this instrument, which was specifically intended for measurements of fibre diameter in such applications as ram selection and flock culling. The instrument was calibrated with full-length hand carded wool and used a 2 gram test specimen. An accuracy of ±0.5 microns was claimed provided that the appropriate corrections were made for temperature and atmospheric pressure. An interesting feature is that each instrument can be produced commercially with identical calibration characteristics.

Jackson and Engel (1980) described a method for calibrating the Sonic instrument.

Technical Issues

Precision

The Airflow instrument has become the de facto standard for commercial trading of greasy wool, scoured wool and top. IWTO-28 is its major application in terms of total tests conducted each year, largely because of the general application of the method for determining the fineness of greasy and scoured wool. The components of variance for the current version of the method are shown in Table 4 and the confidence limits in Table 5[10]. Similar data can be found in IWTO-6, the Airflow method for fineness of tops.

As with IWTO-8 the confidence limits reflect the relationship with diameter reported by Andrews and David (1978)

TABLE 4: Components of variance for Airflow Measurements and Confidence limits for 2x2 Determinations

	Single Meter Method		Two Meter Method	
	<26 µm	>26 µm	<26 µm	>26 µm
Between Laboratories s_L^2	0.60	0.105	0.020	0.037
Between meters s_l^2			0.021	0.027
Between specimens s_S^2	0.018	0.058	0.026	0.052
Between Readings s_R^2	0.006	0.008	0.004	0.005
Variance of 2x2 determinations	0.0705	0.136	0.0445	0.0778
Confidence limit of 2x2 determinations	0.52	0.72	0.41	0.55

TABLE 5: 95% Confidence Limits for a given Mean Diameter

Mean Fibre Diameter (µm)	95% Confidence Limits (µm)
15.0	+/- 0.33
20.0	+/- 0.45
25.0	+/- 0.57
30.0	+/- 0.68
35.0	+/- 0.80
40.0	+/- 0.92

[10] Extracted from IWTO-28

The IWTO method employs a quadratic equation to relate the millimetre heights on the flow meter the mean fibre diameter of a set of calibration (Interwoollab) tops. This equation takes the form:

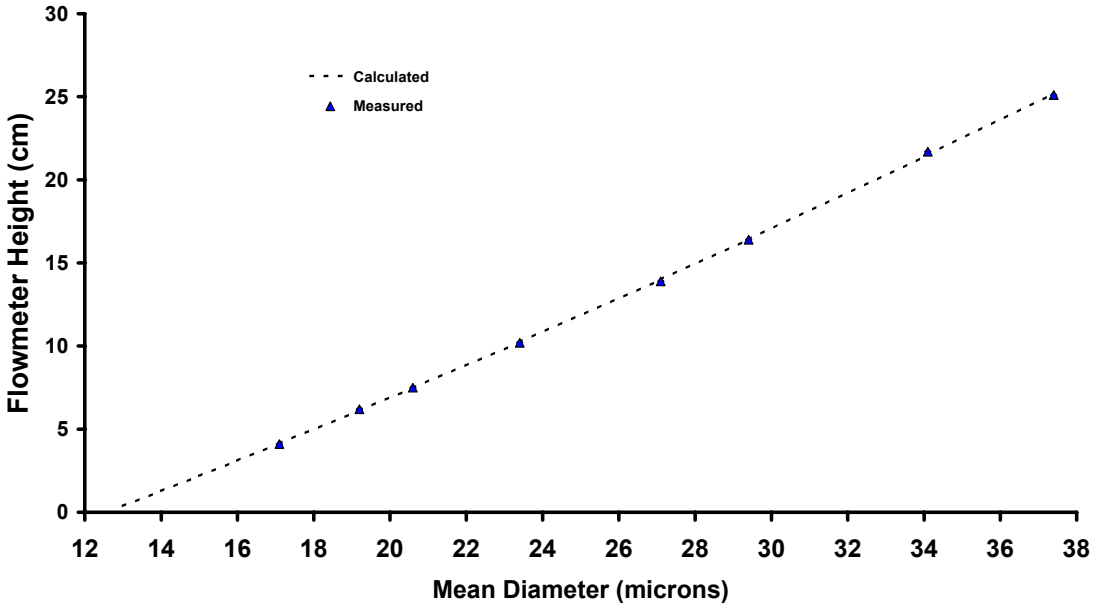
$$h = a + bd + cd^2$$

Various authors (Baxter, Brims & Taylor, 1991) have commented on the non-linearity of the Airflow system, because of this, and the ability to extrapolate the calibration curve to enable the instrument to measure the fineness of samples which do not lie within the range of he calibration has been challenged. It must be noted that the constant in the quadratic term is very small, and effectively the calibration is linear. The quadratic has been included to maximise the precision of the instrument and also to compensate for the small departures from linearity that do occur in individual flow meters (James, 1971). An example of a typical calibration curve for a constant pressure Airflow is shown in Figure 3. The actual calibration points are shown, overlayed by the regression equation that has been fitted to the points i.e.

$$h = -10.4412 + 0.7676d + 0.004961d^2$$

This has been extrapolated to 13 microns. For fine wools the extrapolation of the Airflow calibration is really not a major problem. The flow meter height is really what determines the range of the instrument[11].

FIGURE 3: Typical Airflow Calibration Curve (IWTO-28)



Limitations of the Airflow Technology

The technical limitations of the Airflow method are clearly defined in the terms that make up the Kozeny equation.

$$Q = K_b \cdot \frac{A_c}{L_c} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2} \cdot \Delta P \cdot d^2$$

[11] Sommerville (1998) examined the issue of extrapolation of Airflow calibrations in considerable detail. He concluded that extrapolation errors could be a cause for differences between Airflow, Laserscan and OFDA for very fine wools.

where $K_b = \frac{g}{k\eta}$

The important variables in this equation are:

- Viscosity of the fluid
- Cross-sectional area of the chamber
- Length of the chamber
- Pressure difference across the plug
- Porosity of the fibre mass
- Volume to surface area ratio of the fibre mass, or the fineness of the fibre
- Effective path length through the fibre mass

The variables that form the basis of the Airflow measurement, namely flow and pressure, need not be considered as limitations. However large fluctuations of atmospheric pressure, compared with the pressure at which the instruments are calibrated, is a possible source of bias (Greuel & Sustmann, 1961).

Some of these factors can be controlled quite simply. This is certainly the case with the dimensional characteristics of the chamber. However the dependence of the instrument on the chamber dimensions is a possible source of bias in instruments that are heavily utilised and in which, as a consequence of wear, the chamber dimensions may slowly change over time. Most commercial laboratories guard against this possibility through various quality control measures, including inter-laboratory round trials such as the ILRT[12], and internal reference checks.

The viscosity of air is a variable that is dependent on humidity, temperature and atmospheric pressure. However this too is amenable to control.

The constant factor in the Kozeny equation is susceptible to variation under certain conditions. Recalling that

$$k = k_o \left(\frac{L_e}{L_c} \right)^2$$

it is clear that this is the major reason for the orientation effects reported by Anderson and Warburton (1947), Ewles (1955), James and Bow (1968), Henning and McKelvie (1969), Downes and Mackie (1969) and Hunter, Gee and Braun (1978). Thus the Airflow system is sensitive to the preparation of calibration samples and samples to be tested. The need for a standardised system of preparation is clearly recognised in both IWTO-6 and IWTO-28, where a substantial part of these standards is directed towards sample preparation.

Other Variables Influencing Porosity

Effect of Density

The porosity factor has been defined previously

$$\varepsilon = \frac{V_c - V_m}{V_c} \qquad 4$$

Cassie (1942) raised this issue in his original paper. He restricted his observations to speculation about the potential effects, rather than empirical evidence of real effects. However Cassie did demonstrate theoretically that the predominant variable that effects the porosity is fibre density (equation 22).

[12] The ILRT (Inter-laboratory Round Trial) involves replicate testing of greasy wool samples by the major Southern Hemisphere Test Houses. The samples are tested twice per week and the data reported regularly to IWTO

$$\frac{\Delta\left(d^2 t\right)}{\left(d^2 t\right)} = -\frac{2\rho V_c + m}{2\rho V_c - 2m} \cdot \frac{\Delta\rho}{\rho}$$

There is very little data concerning the variation of density between farm lots. Most of the measurements have been made on wool tops, which by their nature are blends of many farm lots and therefore an average. Because of this it is improbable that large variations in density between wool tops are likely to occur, except in particular instances where medullation is a factor. Medullation effects on Airflow have been studied by many authors (Robinet & Franck, 1958, 1959; Hunter, Smuts and Gee, 1986; Van Luijk, 1984 and more recently Baxter, 1993). This effect is well known and the test method explicitly warns about it, and describes certain types of wool, which are particularly likely to show biased results because of medullation.

Van Wyk and Nel (1940) reported the details of density measurements on South African merino wool. They reported the determination of density on 54 samples and found that real differences occurred. The values varied from 1.298 to 1.313 but the coefficient of variation was small, indicating that the density may be regarded as on of the least variable aspects of merino wool. Connell and Andrews (1974) attempted to quantify the effect on Airflow measurements. They measured the density of a number of New South Wales and Western Australian wools and summarised their data and that of Van Wyk and Nel (see Table 6)

TABLE 6: Specific Gravity of Australian and South African Wools

	Australian	South African
Number of Samples	174	54
Grand Mean	1.3042	1.3052
Std Deviation between samples	0.0028	0.0035
Coefficient of Variation (%)	0.15	0.27
Standard Error	+/- 0.0010	+/- 0.0012

Connell and Andrews expressed equation 22 in a slightly different form:

$$\frac{\Delta d}{d} = -\frac{2\rho V_c + m}{2\rho V_c - m} \cdot \frac{\Delta\rho}{\rho}$$

and by assuming the dimensions of the WIRA fineness meter where $V_c = 8.0$ cm^3 and $m = 2.500$ grams, and an average density for wool $\rho = 1.3$ g/cm^3 they expressed the above equation in the form

$$\Delta d = 1.27 d \frac{\Delta\rho}{\rho}$$

From this Connell and Andrews deduced, by treating all the wool samples collectively, assuming a mean diameter of 20 microns, and using an average standard deviation in density of 0.0028, then the density of 95% of wool lots will lie in the range 1.0342 +/- 0.0056. Hence

$$\Delta d = 1.27 \times 20 \times \frac{0.0028}{1.30} = 0.06 \text{ micrometres.}$$

This is seemingly small particularly since the error in the measurement on individual farm lots is somewhat larger. Unfortunately Connell and Andrews under estimated the effect. Firstly their simplification of Cassie's equation (equation 22) was incorrect. It should be

$$\frac{\Delta d}{d} = -\frac{2\rho V_c + m}{2\rho V_c - 2m} \cdot \frac{\Delta\rho}{\rho}$$

and hence $$\Delta d = 1.47 d \frac{\Delta \rho}{\rho}$$

Furthermore, using the standard deviation is somewhat misleading, because in quoting the likely effect on 95% of lots, one is ignoring the very real effect on the other 5%. The range of Van Wyk and Nel's data was 0.015. The range of Connell and Andrews data, allowing for the standard deviation in the individual results, was 0.011. Using the average of these two ranges, ie. 0.013 then

$$\Delta d = 1.47 \times 20 \times \frac{0.013}{1.30} = 0.29 \text{ microns}$$

This is significantly larger than the figure quoted by Connell and Andrews, albeit, likely in only a small percentage of cases. However it is important to note that the bias caused by density is diameter dependent. It decreases for fine wools and increases for coarser wools.

It is possible therefore that variations in density, may produce small differences between test methods that are independent of density, and Airflow, which does have a density dependence. These differences are less likely in tops, which are blends of farm lots.

Effect of Coefficient of Variation

The other variable that theoretically can effect porosity is standard deviation or coefficient of variation. It is curious that although the theoretical effect, first defined by Anderson and Warburton (1947), has been widely cited, until 1997 (Sommerville, 1997; Lindsay & Marler, 1997) it had never been experimentally verified. Andrews and Irvine (1972), in a report on the relationship between Projection Microscope and Airflow, observed "*for some tops the degree of agreement is surprising, in view of the atypical values obtained for their coefficients of variation in diameter*". Baird, Barry and Marler (1993) reported contradictory effects when they attempted to explain consistent small differences between Laserscan and Airflow measurements by correcting the Airflow measurement for coefficient of variation as proposed by Roberts (1959). Again Baird, Marler and Barry (1994), after an extensive investigation of these differences concluded "*the standard deviation of the test specimen relative to the standard deviation of the calibrating material might give rise to the observed discrepancies between Laserscan and Airflow."* Edmunds (1993) suggested on the basis of the theory, that "*significant uncertainty arises in deriving the true value of the mean fibre diameter from Airflow measurements. This has important consequences for the new measurement methods such as LASERSCAN and OFDA with regard to their calibration and comparison against Airflow values.*[13]"

Anderson and Warburton's relationship predicts that for wools with the same Projection Microscope diameter, if the relative coefficient of variation increases then the corresponding Airflow diameter will increase. The converse also applies. The mechanism for this can only be an increase in the porosity of the fibre plug. Carman (1947) produced a mathematical proof of the following proposition:

Flow is greater through parallel channels unequal in size than through channels of even size, with the same internal volume and internal surface, that is, with the same average mean hydraulic radius.

Consider one large circular pipe, diameter, d, and n smaller pipes, diameter rd with $r \leq 1$. Then, according to Poiseuille's law, the total flow through the pipes, at constant pressure drop, is given by

$$Q = K\left(d^4 + nr^4 d^4\right) = Kd^4\left(1 + nr^4\right)$$

where $K =$ a constant.

[13] Edmunds did not provide a definition of the "true" diameter of wool.

Consider a series of circular pipes, all of the same diameter, d_1, and with the same aggregate values for the internal volume and the internal surface. Then

$$d_1 = \frac{d^2 + nr^2 d^2}{d + nrd} = d\left(\frac{1+nr^2}{1+nr}\right)$$

and the number of tubes,

$$m = \frac{d + nrd}{d_1} = \frac{\left(1+nr^2\right)^3}{1+nr^2}$$

whence the total flow through the tubes, Q_1 is given by

$$Q_1 = Kmd_1^2 = Kd^4 \cdot \frac{\left(1+nr^2\right)^3}{\left(1+nr\right)^2}$$

It follows that in the ratio

$$\frac{Q_1}{Q} = \frac{\left(1+nr^2\right)^3}{\left(1+nr\right)^2\left(1+nr^4\right)}$$

Q_1 is greater than Q if

$$\left(1+nr^2\right)^3 > \left(1+nr\right)^2\left(1+nr^4\right)$$

that is, $3r + 3nr^2 > 2 + nr + r^3 + 2nr^4$

that is, $n\left(3r^3 - r - 2r^4\right) > \left(2 + r^3 - 3r\right)$

But this is impossible because n is positive and $0 < r < 1$, so the right hand side of the inequality is always positive and the left hand always negative. It may be concluded, therefore, that Q_1 is always less than Q

This is an idealised model, which provides a mechanism to explain why, in the Airflow system, the coefficient of variation can affect the readings obtained. For a given fibre mass, with a defined Projection Microscope mean diameter, the number of channels of uneven size will increase as the coefficient of variation increases, and therefore the apparent Airflow diameter will increase. However the flow of air through plugs of wool is not an identical situation to that considered by Carmen. Wool is compressible and the maximum compression is not applied to the plug in the chamber of the instrument. The values of porosity ε that are attained in the Airflow instrument are generally much higher than for powders. For wool in an Airflow machine, ε is greater than 0.6, whereas for powders and sands it is less than 0.5. It is possible that this is the reason why the system seems to be less sensitive to coefficient of variation effects than the theory predicts.

The effect of an increase in the coefficient of variation, while maintaining the mean diameter constant, is to increase the variation in the pore size within the fibre mass. In the Airflow instrument this results in an increased flow, even though the diameter remains constant. The same will occur in powders. However in the Airflow instrument the porosity has not been minimised, and indeed compared with powders it is quite large. In this instance small increases in coefficient of variation are unlikely to produce the effects predicted by the theory. Very large deviations will show an effect but these are unlikely to occur in consignment lots or conventionally classed lines, because of the variation between fleeces and between lots that naturally occurs. However lines that are classed with the assistance of objective measurement of the fineness of the individual fleeces may show the effect when measured by Airflow compared with other methods that also measure fineness distribution.

Despite this Sommerville (1997) and Lindsay & Marler (1997) independently verified that variations in CVD did influence the diameter as measured by the Airflow instrument. Sommerville prepared samples with the same mean fibre diameter but with significantly different CVD's by blending tops where the distribution details had been determined by Projection Microscope. When measured by Airflow the variation in the Airflow diameter from the Projection Microscope diameter was very close to that predicted by the theoretical equation:

$$\bar{d}_1 = \bar{d}_o \cdot \frac{1 + C_o^2}{1 + C_1^2}$$ (see equation 24)

Marler and Lindsay used a statistical approach, utilising a large number of tops where the Projection Microscope data was known, with similar results.

Table 7 (Marler & Sommerville, 1997) shows the magnitude of the errors in diameter measured by Airflow, compared with technologies that are not sensitive to CVD, that could be expected for differing Standard Deviation (SD) values. The corresponding CVD is shown in brackets.

TABLE 7

Sample SD (microns)	Sample MFD (microns) 19.0	20.0	21.0	22.0	
2.5	18.5 (13)				Airflow is finer
3.0	18.7 (16)	19.6 (15)	20.5 (14)		
3.5	18.9 (18)	19.7 (18)	20.6 (17)	21.5 (17)	
4.0	19.0 (21)	19.9 (20)	20.8 (19)	21.7 (18)	
4.5	19.3 (24)	20.1 (23)	21.0 (21)	21.9 (21)	
5.0	19.5 (26)	20.4 (25)	21.2 (24)	22.1 (23)	Airflow is coarser
5.5		20.6 (28)	21.5 (26)	22.3 (25)	
6.0				22.6 (27)	

In the case of the 20.0 micron wool the error in the Airflow measurement arising from the effect of CVD will be near zero (-0.1 microns) at a SD of 4.0 microns (i.e. CVD 20%). One would expect a bias of -0.4 microns when the SD was 3.0 microns (i.e. CVD 15%) and a bias of +0.4 microns when the SD was 5.0 microns (i.e. CVD 25%).

Commercial Issues

The Airflow method is the most important test method used by the wool industry for the estimation of fibre fineness. A considerable degree of confidence in the method has developed since 1960, largely because of the performance of the system in practical situations.

As far as CVD is concerned the magnitudes of the potential errors arising from this are small, but they can be commercially significant. The commercial risk has been minimised by the fact that commercial consignments are generally made up of farm lots sourced from a number of properties and often from different regions. This averages any CVD effect. However, this source of bias was possibly important for

individual woolgrowers, where selection decisions resulting in increases (or decreases) in CVD may result in changes in Airflow diameter of their farm lots to their commercial disadvantage. However, it must be stressed that such effects are very small, and generally less than the testing error.

The same argument applies to effects of density variation.

The amount of testing of the system that has occurred since the introduction of IWTO-6 and IWTO-28 is substantial. David (1979) reported that for greasy wool tested by laboratories in Australia, New Zealand and South Africa the standard deviation in mean diameter between laboratories was 0.065 microns. David also showed that the 95% confidence level for expected differences between two laboratories for tops was 0.6 micron for a 21 micron top and 1.2 microns for a 31 micron top.

Ward and Douglas (1976) examined 122 consignments of greasy wool processed between 1964 and 1973, and compared the mean fibre diameter of the top compared with the greasy wool. They found an average difference of +0.3 microns with a standard deviation of 0.53 microns. This contrasted with an expected difference of 0.5 microns when coretesting of greasy wool first commenced

Jackson, Marler and Morgan (1989) provided an analysis of the core to comb comparisons for 498 consignments processed though 32 mills as part of the TEAM 1 and TEAM 2 trials. The results are shown in Table 8.

TABLE 8: Core/Comb Comparisons (Top-Core). TEAM-1 and TEAM-2

	Number of Mills	Number of Consignments	Mean Difference	Range of Mill Mean Differences	Standard Deviation of Differences	Range of Standard Deviations
TEAM-1	12	211	0.00	-0.44 to 0.25	0.29	0.13 to 0.38
TEAM-2	20	287	0.09	-0.41 to 0.33	0.23	0.10 to 0.33
Overall	32	498	0.05	-0.44 to 0.33	0.26	0.10 to 0.38

Analysis of a smaller number of consignments for 6 mills over a two year period gave similar results. For these mills the authors reported small significant trends for some diameter classes with time but these did not exhibit a consistent pattern.

The objective evidence suggests that the Airflow system has served the commercial interests of the industry well over the years since its introduction, despite its known limitations.

The objective evidence suggests that the Airflow system has served the commercial interests of the industry well over the years since its introduction, despite the known limitations. The introduction of alternative technologies such as Laserscan and OFDA, while not without their own limitations, has substantially improved the precision and accuracy of diameter measurement.

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PHOTOMETRY

Principle

Photometry is the analytical use of light (luminous) intensity to measure the physical and chemical properties of solids, liquids and gases, and mixtures or solutions thereof. Wavelengths in the infrared, visible and ultra-violet portions of the electromagnetic radiation spectrum are commonly used in photometric measurements. Photometry is probably the most extensively used of all analytical technologies.

In principle the application of photometry to the measurement of wool fineness is very simple. Photometers consist of a source of light of constant radiance, a sample cell and a photo-detector. In the specific case of wool the measurement of fineness is based on the principle of light scattering and presumes that the wool fibre is opaque. The impact of the light beam on the photo-detector in the absence of any interference will generate a detectable electrical signal. If a fibre at right angles to the beam intersects the beam the fibre will project a shadow onto the photo-detector, due to the light incident on the fibre being scattered. The shadow of the fibre will reduce the signal from the photo-detector by an amount that is proportional to the projected area of the fibre. If the length of the fibre intersecting the beam is constant then the output from the photo-detector will be proportional to the transverse dimension of the fibre[1].

However, this is a very simplistic description of the physics of photometry as applied to wool fibre diameter measurement. The specific details of the physics in particular instruments are dependent upon the instrument design.

When measuring wool fibre diameter the critical step required for photometric instruments is a technique for aligning the fibres so that they are always at right angles or near right angles to the beam of light, and of ensuring that the orientations of the individual fibres across the beam are similar.

There is an extensive literature describing aspects of photometric techniques for the measurement of the diameter distribution characteristics of wool i.e.

- Mean Fibre Diameter (MFD);
- Standard Deviation of Diameter (SD); and
- Coefficient of Variation of Diameter (CVD).

This is a very large topic. Because of this we will be considering the use of Photometry in several parts, each part focusing on key developments within specific time periods i.e.

- The First Efforts (1950-1970);
- Leaping Forward with Laser Optics (1970-1983);
- Resolving Problems (1983-1989); and
- The Sirolan Laserscan (1989-today).

[1] This is but one principle that can be applied, and is described here because it is the principle behind modern photometric instruments. However earlier instruments utilised the birefringent properties of wool and polarised light to create an image that could cause a response in a photo-detector.

The First Efforts (1950-1970)

The earliest development of a photometric instrument began in the United States. The issue of aligning the fibres appropriately was resolved in this instrument by incorporating a device for aligning fibres on a microscope slide called the Fibroalineator.

Developed in the early 1950's the Fibroalineator was an extension of a technique first reported by Larose (1947), for aligning fibres on a microscope slide by applying an electric field across the slide.

In 1957 the Sheep and Fur Animal Branch, Animal Husbandry Research Division, ARS, USDA in Beltsville Maryland commenced work on a prototype photometric instrument, which incorporated the Fibroalineator. The instrument became known as the Electronic Fibre Fineness Indicator (EFFI).

The instrument was an optical, mechanical and electronic system designed to automatically scan a microscope slide on which cut wool fibres had been mounted. The prepared slide was placed on a movable microscope stage equipped with high voltage electrodes, which by means of electrostatic forces caused the fibres to align parallel to the electrostatic field – the principle of the Fibroalineator. A beam of light from an incandescent lamp passed through a condensing lens and then a polarising filter. The polarised light then passed through the wool fibres on the slide on which the fibres were aligned by the electrostatic field. The wool fibres being bifringent rotate the light that passes through them. A microscope was located below the slide and magnified the image of the fibre before the light beam passed through a second polarising filter located below the microscope. This filter was mounted at right angles to the first, and blocked the unrotated light, allowing the rotated light to pass. Thus the microscope projected an image of the fibres against a black background onto a slit.

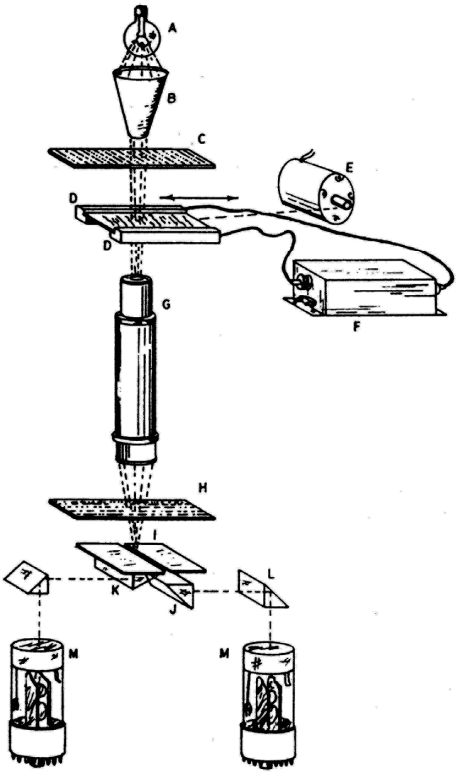


FIG. 1. Diagram of optical system of the EFFI: A = Lamp; B = Condensing lens; C = Polarizing filter; D = Electrodes; E = Stage drive motor; F = H.V. transformer; G = Microscope; H = Polarized filter; I = Slit; J = Prism "A"; K = Prism "B"; L = Prism; M = Photo-multiplier tube.

A synchronous motor was programmed to move the slide carrier from right to left and to move the slide carrier forward 1 mm at the end of each crossing. The slide moved at a uniform rate and the fibre images were detected on photomultiplier tubes and converted into electrical pulses the duration of which was proportional to the area of the fibres, and hence to the transverse dimension. Since the fibre alignment was not always complete, the image of the fibre was split by use of a pair of prisms. The two images were scanned by separate photomultiplier tubes, which were parallel to and in line with the length of the fibres. This arrangement caused a fibre that was parallel to the slit to cause an electrical pulse from both photomultiplier tubes at the same time. However a fibre image that was not parallel to the slit caused an electrical output from one photomultiplier, which was out of phase with the pulse from the second tube. By this means the instrument discriminated against fibres that were not correctly aligned.

Hourihan, Terrill, Neil and Mackey (1970) reported on the application of this instrument to the measurement of the diameter of wool tops.

The instrument was calibrated with IWS tops. Its performance against the projection microscope method for a series of validation tops is illustrated in the following table.

TABLE.1: Comparison of the EFFI and Projection Microscope

Top	Projection Microscope Data		EFFI	
	Mean	Standard Deviation	Mean	Standard Deviation
A	18.40	3.93	19.68	8.63
B	21.22	4.72	20.89	8.80
C	23.28	5.86	23.84	9.38
D	23.26	6.23	27.35	10.40
E	28.79	7.72	29.40	1059
F	31.26	8.42	30.34	11.80
G	31.61	8.38	32.50	12.69
H	36.49	9.43	34.75	13.10

The instrument had a clear diameter dependent bias, being coarser for the fine wools and becoming progressively finer as the diameter increased. The standard deviation was considerably larger. The authors recognised this but suggested the instrument was at least satisfactory for ranking results, and provided a much more rapid measurement than the projection microscope method, and thereby providing a useful tool for quantitative geneticists for ranking animals.

Thorn Bendix in co-operation with the Textile Department at the University of Leeds developed an instrument called the Fibre Diameter Analyser in 1969 (Anon. 1969). Details on this device are scanty but the principle was relatively simple.

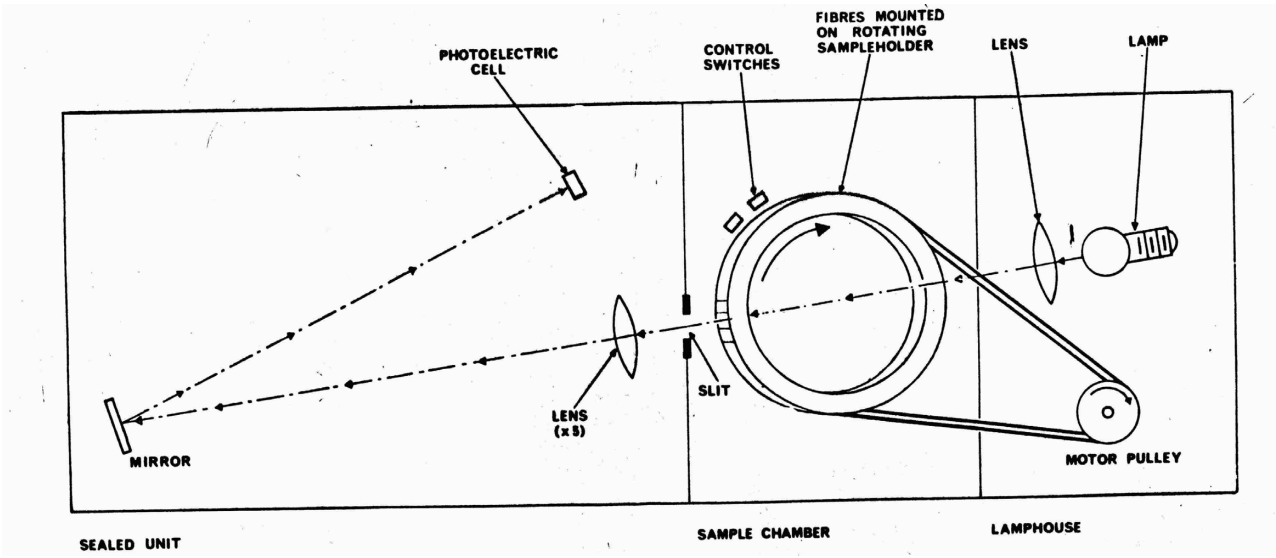


Figure 2
A Schematic of the Fibre Diameter Analyser developed by the University of Leeds in 1969

A sample batch of 300 fibres was mounted on the perimeter of a circular sample holder, parallel to the axis. The holder was rotated at 10 revolutions per minute so that each fibre in turn passed between stabilised light source and a photo-detector. The electrical pulses obtained varied in magnitude depending upon the diameter of the fibre. It was claimed that the instrument had a range of 10 to 70 microns, and a precision better than 0.5 microns. This instrument has since vanished into obscurity.

Leaping Forward with Laser Optics (1970-1983)

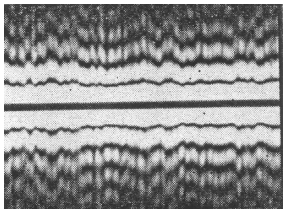


Figure 3:
Diffraction pattern produced by a Lincoln wool fibre

Lynch and Thomas (1971) examined the light scattering properties of single fibres of wool, hair and jute, and filaments of Nylon, Terylene and glass by scanning along their lengths with a Helium-Neon laser beam. The light scattering profiles of the natural fibres suggested uniformity in the optical properties of the fibre materials and therefore closely related to the geometry of the fibres. The optical properties of Nylon and Terylene on the other hand were observed to be very non-uniform along the filaments. Lynch and Thomas concluded that the optical diffraction profiles of single fibres offered the possibility of measuring continuously the variation in diameter and cross section along the length of the fibres at a length-interval resolution of at least 0.5 mm and possibly as great as a few diameters. They foreshadowed that although the diffraction profiles of natural fibres could not be used in conjunction with some of the simpler scattering equations to give absolute values of diameter and cross-section, an empirical calibration of the scattering angle of the first diffraction minimum against diameter, as measured by some other system, could be made with some precision.

Lynch and Michie (1976) described the design principles, construction and operation of an instrument designed for the rapid automatic measurement of fibre fineness distribution, and of course mean diameter. The instrument was based on the electro-optical measurement of the amount of light scattered from a directed beam generated by a laser by fibre snippets. The fibre snippets were transported through the beam dispersed in a moving liquid.

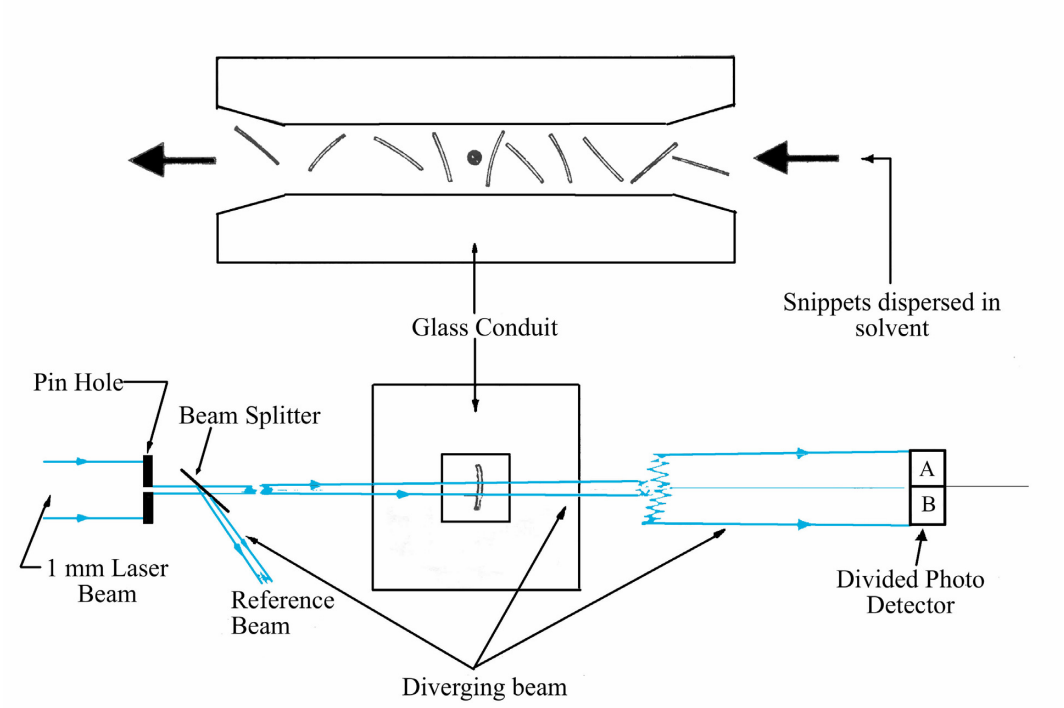


Figure 4:
Schematic of the method of fibre snippet presentation for measurement. The snippets are transported in a liquid through a glass conduit of square section through which the light beam passes.

Lynch and Michie's paper is a very carefully constructed description of the instrument and identifies the critical features in the design. These included:

- The geometry of the beam of light, including its shape and its area;

- The presentation of the fibres to the beam;
- The characteristics of the liquid used to transport the fibres;
- The orientation of the fibres in the beam;
- The discrimination of fibres that are suitable to measure from those that are unsuitable;
- The discrimination of non-fibrous material and fibre fragments from actual fibres;
- The discrimination of signals produced from a single fibre from those produced by multiple fibres;
- The stability of the electro-optics;
- The precise control of the temperature of the liquid transporting the fibres;
- The selection of the transporting liquid; and
- The desirability to calibrate the instrument to be in as close as possible agreement with the Projection Microscope.

The instrument utilised the unique correlation between the amount of light scattered from a directed light beam by a fibre and the fineness of the fibre. By using a very low angle of detection, the instrument avoided any problems arising from the fact that wool fibre is non-absorbing of light and is irregular in geometrical and material properties. By using a beam of light of circular symmetry and causing the fibre to intersect the beam at right angles to the direction of the beam, a unique situation occurred in transit when the maximum amount of light was scattered by the beam. This occurred when the axis of the fibre and the axis of the beam intersected, and because the beam was circular, it was independent of the orientation of the fibre in the plane of intersection. For a parallel beam of uniform irradiance the amount of light scattered is clearly proportional to the area of the projected shadow or image of the fibre. If this area is taken at a unique point of passage when the fibre is intersecting the beam's axis then the projection area or area of light extinction is maximum and defined by the equation:

$$A = \frac{D^2}{2}\left[\sin^{-1}\left(\frac{d}{D}\right) + \frac{d}{D}\sqrt{1 - \frac{d^2}{D^2}}\right] \qquad 1$$

where $A =$ the area of extinction

$D =$ beam diameter

and $d =$ the transverse dimension of the fibre

There is an almost linear relationship between A and d up to a value of $d = D/6$. By taking account of this and also allowing for curvature due to crimp the instrument was designed with a beam of 200 micrometres diameter. This was generated by directing the laser beam through a 200 micrometre pinhole and setting a circular 2 mm aperture in the far field of the 200 micrometre pinhole to accept only the central lobe of the resultant diffraction pattern. This produced a slowly diverging diffraction limited beam, of circular cross-section. The irradiance of the beam decreased radially. To compensate for this, the plane of intersection for the scattering of the beam by the fibres was chosen to be located at a point in the far field of the pinhole where the diameter was approximately 300 micrometres.

The fibres were presented to the beam dispersed as a liquid slurry, which was channelled through a squared-sectioned, fused glass conduit which confined the slurry to a laminar region 2 mm deep intersecting the beam.

With this arrangement not all fibres fully intersected the beam. To discriminate against such events, the instrument used a split circular photo detector. If the signals from the two semi-circular detectors did not match then the event was not recorded as a valid fibre. This method of detection also enabled the rejection

of signals produced by contaminating particles. The possibility of multiple fibres traversing the detector simultaneously was minimised by ensuring that the slurry concentration was low and a maximum count rate was not exceeded. In the event that two fibres crossed the detector simultaneously, the electronics was set to detect the multiple peaks above a base threshold that such an event produced, and then to reject the event.

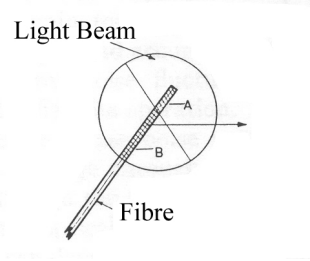


Figure 5a:
An incomplete intercept of the beam by a fibre snippet. Sections A and B of the beam correspond to the separate photodiodes of the detector.

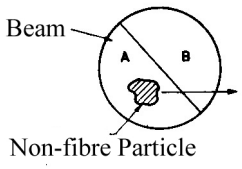


Figure 5b:
Transit of a non-fibrous particle through the scattering beam. Again sections A and B correspond to the separate photodiodes of the detector.

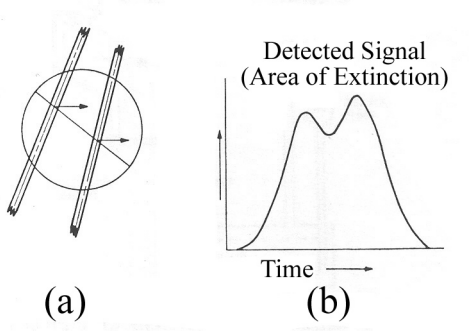


Figure 5c:
A coincident event: (a) when two snippets intersect the beam in the same measurement interval, and (b) the resultant light scattering pulse.

The stability of the laser power was critical to the instrument. This was achieved by a feedback loop whereby the actual measurement beam was sampled by a reference photodiode and the detected intensity used to regulate the laser discharge current. This regulation maintained long term stability and a suitable baseline reference.

Precise temperature control was required to minimise fluctuations of the optical properties of the transporting fluid and the air through which the laser beam travelled. Temperature fluctuations within discrete domains of either fluid can cause excessive noise in the instrument. Laser beams are particularly sensitive to this effect. The precise control of temperature required the instrument to be located in a temperature-controlled cabinet, and also required the transporting liquid to be recirculated. Thus a filter system was incorporated to remove the fibre snippets after they passed through the measurement cell.

The liquid chosen to transport the snippets was required to have a number of properties. It needed to

- be capable of dispersing the snippets;
- have as large as possible refractive index difference from that of wool;
- have a low coefficient of refractive index change with temperature;
- be transparent to the light beam;
- not cause undesirable changes in the physical properties of the wool such as unpredictable swelling of the fibres;
- be inactive chemically with wool;
- have low viscosity at room temperature; and
- be non-toxic.

Isopropanol was chosen because of all the available liquids it best fitted these criteria.

Lynch and Michie reported a preliminary evaluation of the instrument. They used 1.5 mm snippets because this length was long enough to ensure a good probability of the fibres fully intersecting the beam and not too long to cause entanglements and resultant blockages in the circulating system and conduits. They observed that preparation systems that caused felting of the fibre should be avoided. Felting caused measurement error due to the significant number of fibre snippets that remained in contact during measurement.

These authors finally concluded that the instrument was potentially effective for the measurement of fibre fineness. That is

- it was sufficiently reproducible for repeated measurement on single samples;
- it was sufficiently reproducible for a series of subsamples drawn from a well blended wool; and
- correspondence with fibre fineness measurements using Projection Microscope appeared to be statistically significant.

Lynch and Michie lamented the dearth of suitably calibrated reference wools, pointing out that these were critical for the optimum calibration of the instrument, and they foreshadowed the preparation of such reference tops by the CSIRO.

The instrument described by Lynch and Michie and physically constructed by CSIRO is now known as the Fibre Fineness Distribution Analyser (FFDA) or alternatively as the Fibre Distribution Analyser (FDA).

Figure 6:
The FFDA in operation in AWTA Ltd's Sydney Laboratory circa 1985

Irvine and Lunney (1979) described a procedure for calibrating this instrument in such a way as to bring its readings into conformity with the Projection Microscope method. The justification Irvine and Lunney presented for a physical calibration against wool fibres of known distribution was based on two factors:

- The physical design of the instrument was such that it was impracticable to develop a simple calibration function that converted signals generated from fibre events directly into diameter. Firstly, the laser beam varied radially in its intensity. Secondly, the way the light was scattered by wool depends in a complex way on the bulk geometrical and material properties of the fibre. Thirdly, the size of the 200 micrometre pinhole was difficult to standardise so it was probable that each instrument would have a slightly different calibration.
- The aim of the calibration was to cause measurements by the instrument to conform as closely as possible to the Projection Microscope, which was then and still is the accepted international standard for establishing the distribution of fineness in wool.

Lunney and Irvine (1979) also described a number of factors affecting measurements on wool top produced by this instrument. These factors influenced the choice of conditions under which calibration and measurement was carried out, and included:

- Method of cutting fibre snippets;
- Proportion of snippets actually counted;
- Physical slice length compared with actual snippet length;
- Water content of the isopropanol;
- Effect of snippet length on the measured diameter;
- The actual snippet length to be used for calibration and measurement;
- The rate at which snippets were counted; and
- The number of coincidence counts.

These authors recommended calibrating the instrument with 2% water content in the isopropanol. They also recommended preparing snippets for calibrating the instrument in the same manner as snippets would be prepared from unknown samples prior to measurement. They concluded that both calibration and measurement required some control of the count rate, and suggested a maximum raw count rate of 20 counts per second. Snippets less than 0.4 mm were found to give very fine results. For longer snippets (up to 3 mm), a 21 micron top was not affected by snippet length, while the mean of a 32 micron top reduced by about 0.8 micrometres for every added 1 mm in length.

In a subsequent report, Lunney and Irvine (1982) revised their earlier recommendation for water content in the isopropanol, increasing the recommended level to 8.5%. This followed practical experience that the moisture content gradually increased with time and the 2% limit required too frequent adjustment. They also adopted a higher count rate (50 counts per second) than previously recommended.

Figure 7"
Dr Leo Lynch, one of the developers of the FDA (FFDA) is now retired. He is pictured here, fifth from the left, during a visit to AWTA Ltd's Research & Development Division in Sydney on 11th January 2001, in the workshop where the modern Sirolan Laserscan is assembled.

Resolving Problems (1983-1989)

In addition to their recommendation to increase the proportion of water in the isopropanol, Lunney and Irvine also reported a that the FFDA exhibited a diameter dependent bias in mean diameter (See Figure 1 & Table 1) and also a diameter dependent bias in the standard deviation (Table 1) when compared with the Projection Microscope.

Figure 1
Difference in MFD Between PM and FFDA
(Lunny & Irvine - 1982)

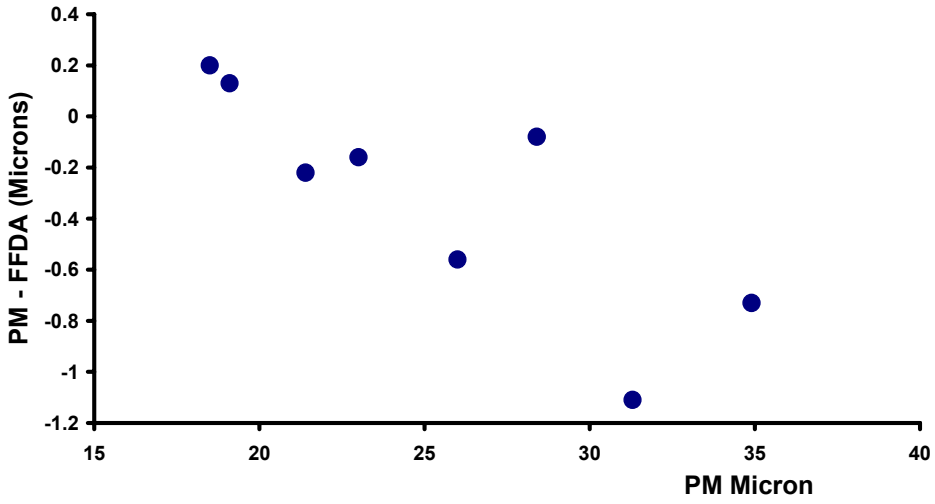


Table 1: Bias and Precision Exhibited by the FFDA

	At 21 Microns		At 30 Microns	
	Bias	**Precision**	**Bias**	**Precision**
Mean diameter	-0.05	±0.21	-0.61	±0.31
Standard Deviation	+0.20	±0.22	-0.07	±0.22
Coefficient of Variation (%)	+0.65	±0.98	-0.20	±0.66

The authors speculated about a number of possible sources of these biases but no conclusions were drawn. They pointed out that the biases reported were within the confidence limits for the measurements.

However, these particular problems were the focus of continuous research and much speculation by various parties for the next 7 years until it was finally resolved by CSIRO in 1989. But we will come to that later.

Sample preparation systems for the FFDA were improved substantially by Buckenham, Whiteley and Giri (1983), and they described designs of mechanised mini-coring and microtoming apparatus.

Lunney (1983)[2] used data generated by the FFDA to develop 10 statistics describing the distribution of diameter in commercial wool tops. These statistics were:

- standard deviation

[2] It is of interest to note that various researchers continued to use the FFDA to study the distribution characteristics of top and greasy wool, despite the then known biases inherent in the instrument. No doubt this was a reflection of the interest in the distributions, and the FFDA provided the only means available to quickly and cheaply provide comparative data.

- coefficient of variation
- skewdness
- Kurtosis
- % of fibres exceeding the mean plus ten micrometres
- % of fibres exceeding the mean plus two standard deviations
- number of standard deviations from the mean with 2% above
- decile range
- decile skewdness
- normalised decile skewdness

The precision of the FFDA in estimating these statistics was reported.

Marler and Lunney (1983) described the effects of dusty samples and fibre fragments on estimates of diameter provided by the FFDA. They reported that the instrument incorrectly registered these fragments as either fine or very coarse fibres, thereby spreading the distribution and biasing the mean and the standard deviation. They also noted that this occurred because the optical discrimination system, which we described in the last issue, was unable to discriminate against these events (see Figure 1).

Thompson and Teasdale (1984, 1985) summarised the results of an inter-laboratory round trial using the FFDA. These showed that it was possible to get means for FFDA and Projection Microscope to agree to within 0.5 micrometres for wools up to about 30 micrometres using the recommended calibration system. However the FFDA standard deviations appeared to be up to 0.7 micrometres too high and the FFDA means for wools coarser than 30 micrometres were more than 0.5 micrometres too fine. Van Luijk (1984) confirmed these observations, using New Zealand wools.

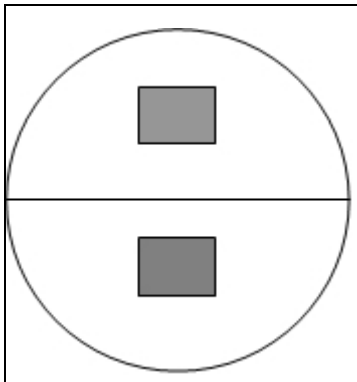


Figure 2

This is a simplified illustration of a failure of the optical discrimination system to identify fibre fragments, dust particles and small fragments of vegetable matter. The shaded rectangles represent the fragments, and the signal generated by each half of the split detector is incorrectly registered as a fibre. Clearly the probability of such events would depend significantly upon the concentration of the fragments, increasing with increasing concentration.

Various other researchers also reported difficulty in finding a calibration function that covered the full range of fibre diameters without introducing a bias in the measurement of individual wools.

Meanwhile Whitley, Thompson, Stanton and Welsman (1984) and Whitley and Thompson (1985) used the FFDA to report the distribution characteristics of raw wool sale lots, for merino and crossbred fleece wools.

In a report to the IWTO Fineness of Wool Working Group Marler and Irvine (1985) advised the members present that they had identified the reason for the bias in diameter. The problem arose from the fibre orientation in the measuring cell (this is one of the critical design features identified by Lynch and Michie). The details of this research were later presented by Irvine (1986) (Figure 3).

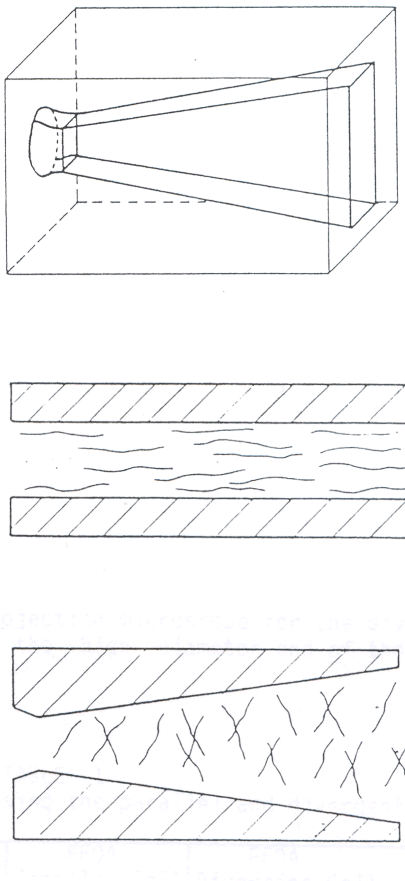


Figure 3:

The original cell had parallel sides and the fibres tended to orient themselves parallel to the horizontal axis. (i.e. the direction of flow). The new cell had two sides that expanded at an angle of 16 degrees.

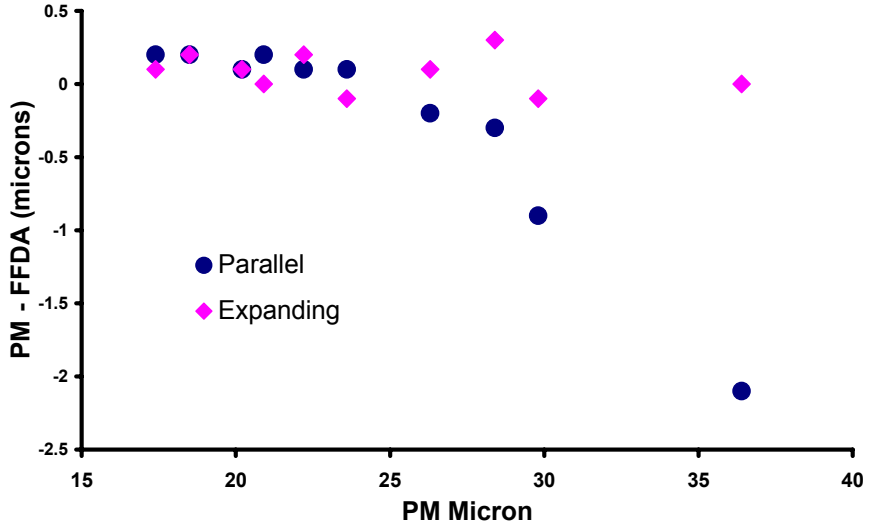
Irvine (1986) reported in detail on the evaluation of photographic images of fibres flowing through both cell designs using a flash of 10 microseconds. The rate of flow being 2.5 m/sec the distance travelled by the fibre snippets in this time was negligible. Clear photographs were obtained.

In the parallel cell the overall impression gained from the photographs was that the majority of fibre snippets were roughly aligned with the flow. Snippets only produced acceptable readings if the portion intercepting the beam lies across the beam diameter. If the snippets are largely aligned with the flow the probability of this occurring will be much greater if the snippet is curved so that part of the snippet is at an angle to the flow. The photographs showed that this was the case with finer wools which are generally more crimped.

The flash photography suggested that in the expanding cell the flow corresponds to the flow from a submerged orifice. The liquid above and below the jet is more turbulent and travels more slowly than the liquid in the jet. As the leading ends of the snippets move into the slower moving regions, the trailing end begins to "overtake" the leading ends, resulting in the snippets adopting a more acute angle to the flow direction. Measurement of snippets in the photographs suggested that the average snippet orientation is approximately 30 degrees to the direction of flow.

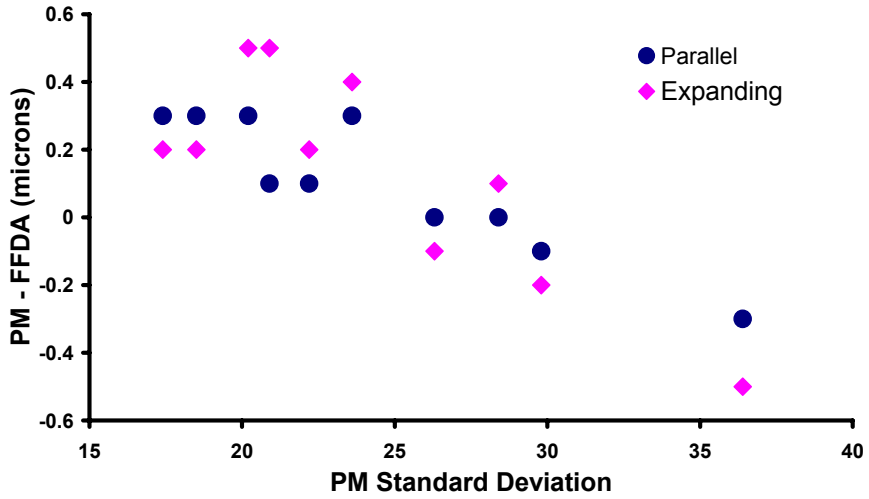
The improvement achieved in mean fibre diameter is illustrated in Figure 4.

Figure 4
Effect of Two Cell Designs on Mean Fibre Diameter
(Marler & Irvine - 1985)



The standard deviation data reported by Marler and Irvine (1985) in this instance was not the subject of comment in their report to the Fineness Working Group. The standard deviation differences are plotted in Figure 5 and clearly the earlier reported bias was still present for this parameter.

Figure 5
Effect of Two Cell Designs on Standard Deviation
(Marler & Irvine - 1985)



The authors also foreshadowed the possibility of calibrating the FFDA using a special graticule, with precisely etched lines of varying thickness, or by using wires of accurately known diameter.

The effect of medullation and the variability of fibre diameter on FFDA measurements was investigated by De Oliveira, Teasdale and Kennedy (1986) using an instrument fitted with the new cell. The principle aims of this study were:

- to evaluate the effects of medullation and standard deviation on mean fibre diameter as measured by the FFDA; and
- to develop correction equations taking account of medullation (as assessed by two methods) and standard deviation to improve fibre diameter estimates.

The second of these objectives assumed that medullation and standard deviation actually influenced the FFDA results. De Oliviera et al measured 242 wools by Projection Microscope and by FFDA. The medullation of the samples was assessed using the Projection Microscope (IWTO-12-64) and the SAWTRI medullameter. The resultant data was statistically analysed to determine the extent to which FFDA measurements were dependent upon Projection Microscope diameter, standard deviation and medullation. An attempt was made, assuming dependency was demonstrated, to correct the FFDA diameters to more closely reflect the Projection Microscope results.

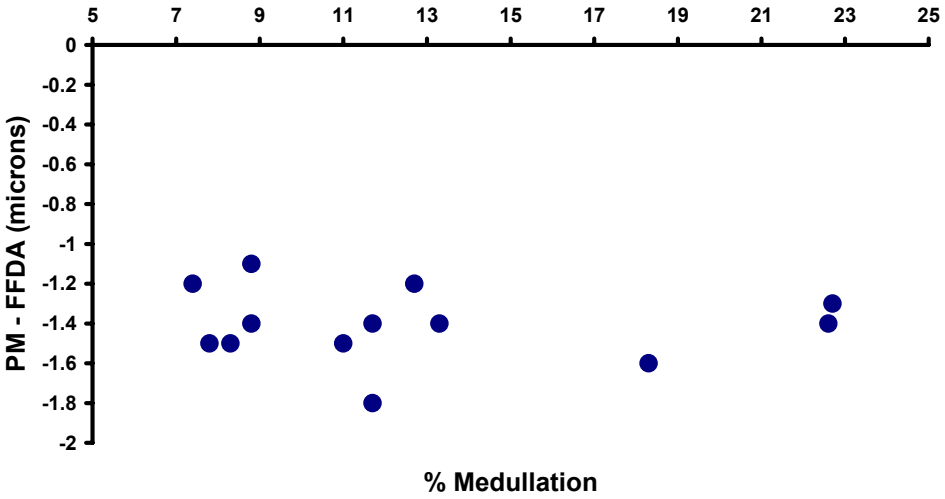
These authors reported that for wools free of medullation, the bias in the estimate of diameter by the FFDA increased substantially when the standard deviation of the samples was beyond the range reported for the calibration tops. Lower FFDA results tended to be produced on coarser wools, the same as reported previously. They observed that the expanding cell did not remove this bias, contradicting the findings of Marler & Irvine.

De Oliviera et al also reported that an increase in the proportion of medulated fibres also decreased substantially the FFDA mean diameter. The authors postulated that the medulated fibres may be seen as two fibres by the optical discrimination system, due to the passage of light through the medulla of the fibre, and were therefore rejected. This would have the effect of producing a negative bias in the measured result when compared with the Projection Microscope measurements.

In support of this hypothesis De Oliviera et al presented some results on some highly medullated wools, which showed the largest FFDA and PM differences. The mean diameter of both the medullated and non-medullated fibres in each of these samples was calculated from the PM measurements. The authors argued that the paired comparison of these data indicated that the FFDA diameter was based largely on the diameter of the non-medullated fibres.

If the hypothesis is valid then it would be reasonable to expect that increasing degrees of medullation would increase the differences between the Projection Microscope and the FFDA. The differences in diameter, as a function of percentage medullation are shown in Figure 6 and clearly there is no obvious association[3]. However De Oliviera et al conceded that the extent of the trends in the data they presented was confounded by biases due to the diameter biases in the coarser wools.

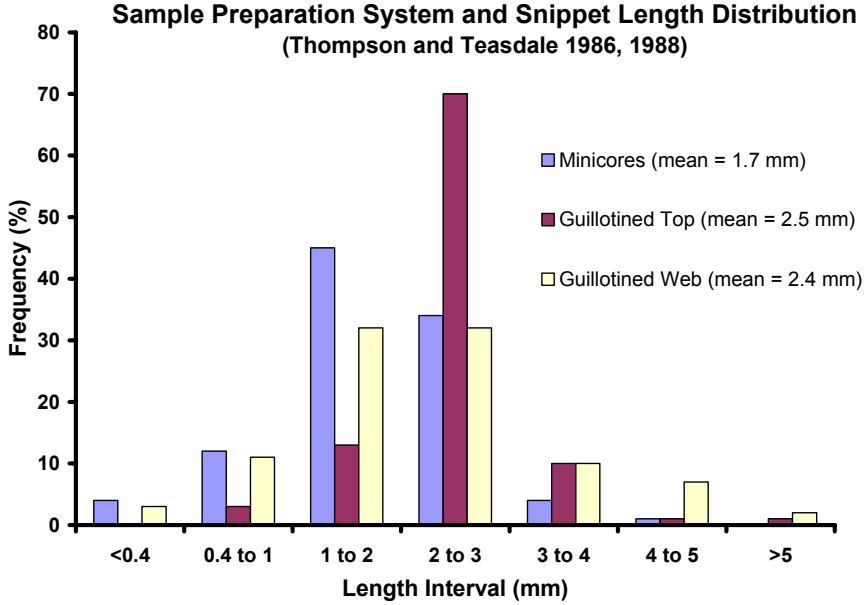
Figure 6
Effect of Medullation on the Difference between PM & FFDA
(De Oliveira, Teasdale and Kennedy - 1986)



Thompson and Teasdale (1986, 1988) considered the effect of different snippet lengths obtained by mini-coring as distinct from guillotining, comparing the parallel and the expanding cell. They found the results consistent with the previous results of Lunney and Irvine (1979) in that the mean decreased with increasing snippet length for coarse wools. This effect appeared to be less in the expanding cell. Lunney and Irvine had also reported changes in the standard deviation of 0.4 micrometres between 0.8 mm and 1.4 mm snippets for a 20 micrometre wool and 2 micrometres for a 32 micrometre wool. Thompson and Teasdale could not confirm these observations for either the parallel cell of the expanding cell.

[3] The author has examined these data in detail and cannot see the association claimed by De Oliviera et al. It is true that the picture is clouded by the diameter bias but there is no clear association with percentage medullation as indicated by Figure 6.

Figure 7
Sample Preparation System and Snippet Length Distribution
(Thompson and Teasdale 1986, 1988)



In comparing the snippet preparation techniques they found that fibre snippets obtained using a 2 mm diameter mini-core gave a shorter mean snippet length than snippets cut with a twin bladed guillotine with the blades set 2 mm apart (Figure 4.10.8). As a result of this difference they expected but did not confirm that when measuring the fibre diameter using a parallel cell, mini-cores would produce slightly coarser results than guillotined snippets for coarser wools.

Much of the work that was reported during this problem solving period was speculative. However, by 1989 on-going research by the CSIRO had identified the most dominant influence on the biases in the FFDA instrument was the optical discrimination system. Apart from the errors introduced by fibre, dust and Vegetable Matter fragments, first identified by Marler & Lunney (1983), the geometry of the FFDA discrimination system resulted in some fibres being selectively discriminated against, even though they were valid fibres. Likewise the system did not adequately discriminate against multiple fibres or certain arrangements of fibre ends.

To remediate these problems a new machine, called the SIROLAN LASERSCAN was designed. This incorporated a radical new approach to the operation of the optical discrimination system, to ensure that only **single** snippets that **fully intersect** the laser beam were selectively measured.

The principle of this device is illustrated in Figure 8. It consists of a ring of 16 fibre optic detectors surrounding a single fibre optic detector. The signal from each of these is continuously monitored. A high-speed computer program identifies when a decrease in signal from the central detector and two of the surrounding detectors occurs simultaneously and matches this event with the signal from the main detector. Events that do not match this selection criterion are rejected.

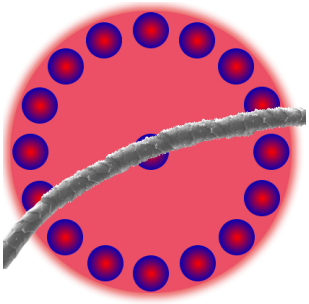
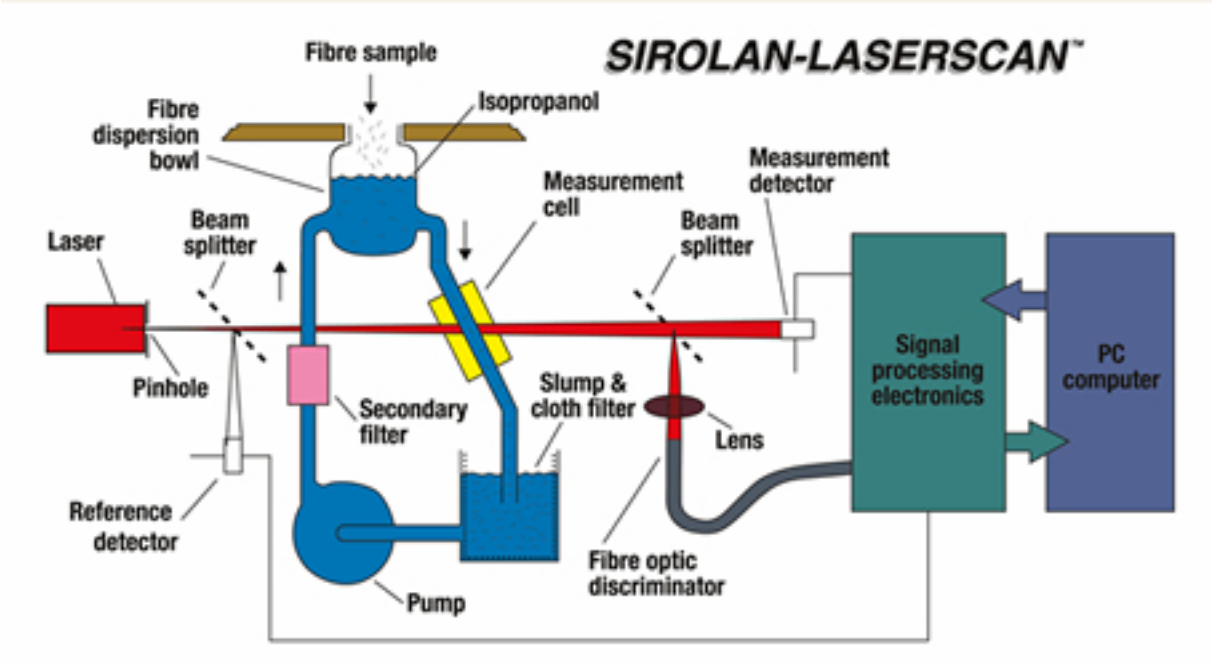


Figure 8: Schematic of the new optical discriminator.

In this regard, the LASERSCAN emulates the Projection Microscope, the primary reference system, in that only measurements on individual snippets are used to accumulate the Fibre Diameter Distribution. This is critically important in eliminating bias resulting from selective sampling from the total population of snippets presented to the instrument. Currently the LASERSCAN is the only commercial instrument, measuring fibre distribution characteristics utilising fibre snippets, that has this capability.

The new fibre optic discriminator also provides the instrument with the capability to measure curvature. The measurement is based on the physical dimensions and geometry of the discriminator itself.

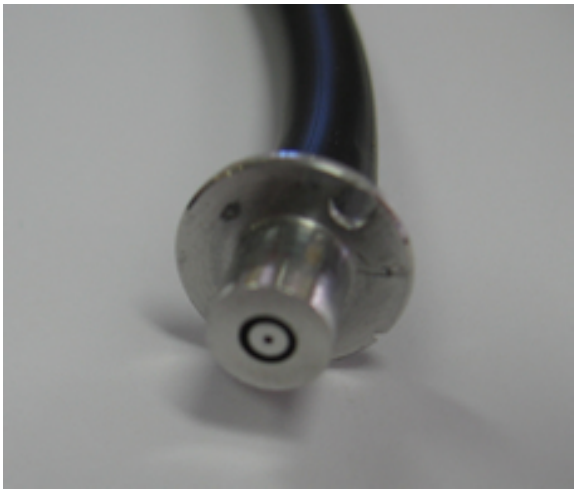


The Sirolan Laserscan (1989-today)

Evaluation of the Improved Optical Discriminator

By the end of 1989 research by the CSIRO had identified the cause of the biases in the FFDA instrument to be due to the optical discrimination system. A new machine, the SIROLAN LASERSCAN™ was designed, incorporating an improved optical discrimination system, and enhanced signal processing electronics and computer processing.

Dabbs and Glass (1992) presented a very detailed evaluation of the new discrimination system. CSIRO had suspected that the biases in the FFDA were due to failures of the discrimination system to correctly reject certain events. In order to identify situations which produced these failures a test bench was set up to record a video image of every fibre event, and whether or not the discriminator rejected or accepted the event. The images were viewed frame by frame and spurious positives were identified and classified. It was found that the following events were being accepted incorrectly.



The optical fibre discriminator developed by CSIRO practically eliminated the bias in mean fibre diameter and standard deviation of diameter that plagued the FFDA.

- The fibre did not completely span the detector, but usually spanned more than 90% of the detector. The FFDA incorrectly accepted between 3% and 7% of such instances. This tended to bias the distribution and hence the mean to finer diameters.
- Instances occurred (up to 4%) when two fibres spanning the detector were accepted. This increased as the count rate increased, registering as "broad" fibres.
- Some fragments were accepted as valid fibres.
- Approximately 20% of the events were incorrectly excluded for no apparent reason.

The new supplementary discrimination system, using fibre optics briefly described in the January 2006 Newsletter, was specifically designed to identify the fibre ends, multiple fibres and fragments missed by the FFDA.

The new detector was tested using the same test bench used to isolate the discrimination events incorrectly processed by the FFDA. The error rate was one sixth that of the FFDA, virtually independent of count rate and unaffected by sample preparation. The new system produced the same mean as manually discriminated diameter measurements over a broad range of wool and cashmere samples. On average the standard deviation was 0.2 micrometres higher than the manually discriminated value. This was due to 4 bad multiple fibres missed by the new system per 1000 valid fibre measurements.

Evaluation of Electronic Performance and Fibre Morphometry Effects

Bow, Van Schie and Irvine (1993) described an evaluation of a number of the performance aspects of the Laserscan. These included:

- The resolution and repeatability of the electronics and computer interface;
- Baseline drift;
- The optical discrimination system
- Response to medullation; and
- Response to fibre ellipticity.

Dabbs, Van Schie and Glass (1994) examined the effects of fibre

The term "*morphometry*" is derived from the Greek "*morphos*" meaning shape or form and "*metros*" meaning measure.

The term "*fibre morphometry*" describes the measurement of the shape characteristics of fibres. In the case of wool those characteristics of interest are the shape of a transverse cross section, which generally defines diameter, the surface area, which can also define diameter, and the degree of crimp or curvature along the length of the fibre.

curvature or fibre crimp using a complex theoretical model which was verified by experimental measurements.

Resolution and Repeatability

A wire wheel containing five wires with a range of diameters from 15 to 18 micrometres was rotated through the laser beam in the cell position. The system achieved a resolution of ±1 micron and missed only 0.1% of all possible counts.

Baseline Drift

The software controlling the Laserscan monitors the baseline signal and displays an alert if sudden fluctuations occur. If the baseline drifts by a small amount the software makes a correction for this. Changing the baseline signal for replicated measurements of three tops, at 10, 24 and 29 micrometres tested this software adjustment. There were no significant differences for the replicated measurements, demonstrating that the software was adequately correcting for the simulated drift.

Optical Discrimination System

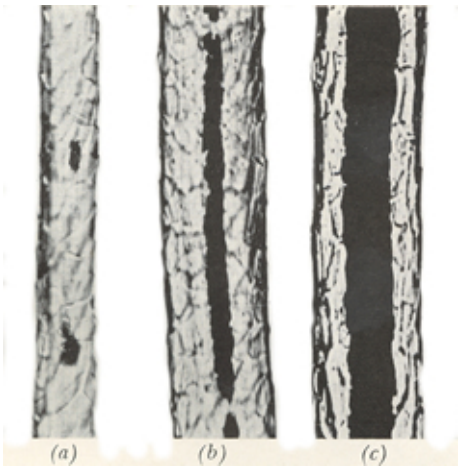
An experiment was conducted to verify the efficacy of the discrimination system. This involved forcing the instrument to monitor very high count rates and then checking the acceptance rate. As the count rate increased the acceptance rate decreased, indicating the effect of increased multiple fibre events produced by the high count rates. The effect predicted by Dabbs and Glass (1992) that this would also produce a small increase in the incidence of missed multiple counts was confirmed. The Laserscan software was constructed to stop counting if the count rate exceeded 100 counts per second to avoid this effect.

Medullation

In analysing the optics of the instrument Dabbs and Glass (1992) used a model that assumed the fibres were opaque. De Oliveira, Teasdale and Kennedy (1986) had postulated that medullated fibres may be transparent to the laser beam. Mounting both medullated and non-medullated fibres on a wheel, spinning the wheel in the laser beam, and measuring the percentage occlusion for each fibre tested this hypothesis. The average diameter of each fibre was measured independently using a microscope. A plot of the percentage occlusion for the medullated and the non-medullated fibres versus the diameter gave the same line for both fibre types. From this it was concluded that the medullation had no effect.

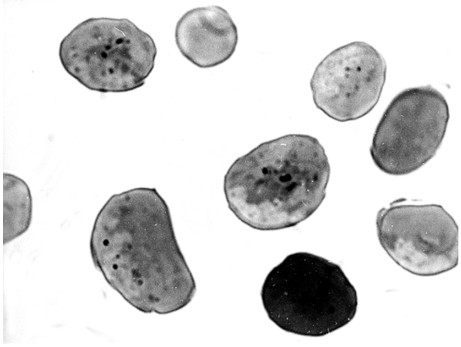
Ellipticity

The instrument relies on the orientation of the fibres to be randomised with respect to their projected dimensions as they pass through the laser beam. The fibres are transported through the beam in a moving liquid. The possibility that the orientation was not random and the instrument may produce a biased result for



Medullated fibres are known to cause bias in alternative measurement systems such as Airflow. Types of medulla that can occur in wool fibres are (a) fragmental, (b) interrupted and (c) continuous.

Source: Von Bergen's Wool Handbook Vol 1, 3rd Edition, Wiley Interscience 1963



Wool fibres, including merino wool fibres, exhibit a range of cross-sectional shapes. They may be approximately circular, ovoid, approximately elliptical or exhibit concavities.

This variation is known to cause bias in some measurement systems such as the Projection Microscope. In the instance the sample preparation is designed to minimise this effect.

A model of a wool fibre illustrating the crimp (or curvature), also showing the distribution of the two components of the cortex (othocortex and paracortex) along the fibre.

Source: Von Bergen's Wool Handbook Vol 1, 3rd Edition, Wiley Interscience 1963

samples with high ellipticity (contour ratio) was tested using a bean shaped elliptical fibre ("Nomex") with a contour ratio of approximately 2.3[4]. The mean diameter and distribution in diameter of a sample of this fibre was determined using the Projection Microscope cross-section method for both the major and minor axes and compared with the mean and distribution provided by the Laserscan. The distribution of the "Nomex" measured by the projection microscope was bi-modal, due to the effect of the fibre's ellipticity. However while the Laserscan distribution covered the range of the projection microscope data it was not bimodal, instead producing a distribution that was relatively square between the lower and higher peaks in the projection microscope distribution. This clearly demonstrated that the fibre snippets had no preferred orientation to the laser beam.

Fibre Curvature

The measured projected fibre curvature, combined with the resultant theoretically calculated diameter variation, indicated that the diameter measurement variations due to fibre curvature lead to insignificant (less than 0.1 micron) changes in the diameter distribution means and standard deviations.

Development of the IWTO Specification – IWTO 12

Agreement between the Laserscan, the Projection Microscope and the Airflow instrument was deemed to be very important commercially, even though there were good technical reasons to expect some differences to occur due to the different ways each instrument defined fibre diameter (for more detail on this refer to AWTA Ltd Newsletter, January 2002). Consequently throughout the development of the IWTO Test Specification a considerable amount of work was conducted examining the equivalence of the technologies, in addition to the basic metrology required by IWTO for a Test Specification to be accepted.

Baird and Barry (1992), examined the effects of sample preparation on, and the precision of the new instrument compared with the Projection Microscope and with the Airflow instrument. With regard to sample preparation they reported the following.

- While no difference was seen between Airflow and Laserscan for solvent scoured mini-cores, mini-cores taken directly from the oven-dried aqueously scoured samples required conditioning before measurement.
- Excessive manipulation of snippets by the operator, for example to remove vegetable matter particles, could lead to inaccuracy in the measurement.
- Whilst good agreement between Laserscan and Airflow measurements normally occurred for solvent scoured greasy mini-cores, occasional individual wools were not always adequately cleaned by the solvent scouring. These occurrences caused a significant difference from Airflow.
- The results from the Laserscan were not influenced by the length of the fibre snippets
- No clear effects on the measured results could be attributed to medullation in coarse wools.

With regard to the precision and accuracy of the instrument, Baird and Barry reported that results from Laserscan were found to agree well with the Projection Microscope for both mean fibre diameter and coefficient of variation for 29 tops ranging from 17 to 36 micrometres. Baird and Barry noted that the Mean Fibre Diameter as measured by Laserscan was 0.1 micrometres coarser than the Projection Microscope value, but this difference was not significant at the 95% level. On average the Coefficient of Variation was 0.5% greater. Although not stated by the authors this difference was significant at the 95% level.

Baird and Barry also reported that Laserscan results compared well with the Airflow method when snippets were measured for greasy mini-cores that had been solvent scoured, mini-cores from aqueous scoured wool and mini-cores from Shirley Analysed webs. The precision of measurement of Mean Fibre Diameter matched that of the Airflow method if 8000 fibres (ie. two test specimens of 2000 measurements for each of two sub-samples) were measured for scoured wool. Where samples were taken directly from greasy wool

[4] This is approximately twice that of wool.

by mini-coring at least 12000 fibres were required. There was no evidence of any bias between the Laserscan Mean Fibre Diameter and the Airflow Mean Fibre Diameter.

Irvine, Bow and Van Schie (1992) described the method of calibrating Laserscan, and reported that the instrument had been successfully calibrated with the Interwoollab Standard Tops. The calibration system differed from that of the FFDA. The FFDA required a decile calibration using the full diameter distribution of the calibration tops. The new instrument was calibrated using the means of the eight Interwoollab tops. The calibration function for the instrument was non-linear, taking the form

$$Diameter = \alpha + \beta(pulse\ height)^{\varphi}$$

Bow and Van Schie (1992) reported the results of two round trials looking at the within laboratory and between laboratory variability of the Laserscan for diameter measurement of tops and of core samples. The first trial was performed with five instruments in one laboratory and the second with instruments in five laboratories. The snippet samples were prepared by mini-coring the bulk sample and then solvent scouring the mini-cores. No significant differences were found between the two trials. The differences between the instruments were in most instances not significant. The greatest range on average between the instruments was 0.3 microns for mean diameter. Analyses of the data indicated the attainable precision limits shown in Table 1.

TABLE 1: Precision of the Laserscan (Bow & Van Schie, 1992)

	Top	Greasy Cores
Diameters < 26 μm	$\pm$ 0.3 μm	$\pm$ 0.3 μm
Diameters > 26 μm	$\pm$ 0.4 μm	

Spencer and Greatorex (1993) published the results of some acceptance trials on a new Laserscan instrument using tops. They found that there was very close agreement with the nominated Mean Fibre Diameter values, and reported a significant difference of +0.68% from the nominal Coefficient of Variation. This was very close to the value that was reported earlier, without comment, by Baird and Barry (1992)

Baird and Barry (1993) reported the results of an inter-laboratory round trial, specifically designed to establish the precision of a Draft Test Method for the Laserscan instrument. The method relied on mini-cores being taken from 200 pre-conditioned aqueous scoured sub-samples that arise in the normal yield test procedures. Each laboratory involved in the trials also tested each of the samples using the Airflow instrument. Eight laboratories participated but one laboratory failed to complete the work in time and one laboratory did not conduct the airflow measurements. Consequently the analysis was based on Laserscan data from seven laboratories and Airflow data from six laboratories. On average the Laserscan was 0.1 microns coarser than the Airflow, but this difference was not statistically significant at the 95% confidence level. The precision of the instrument was found to be equivalent to that of the Airflow and the authors recommended that the Draft Test Method be advanced to full Test Method status.

Baird and Barry (1993) reported the results of an international round trial based on a revised Draft Test Method for the use of the LASERSCAN, in which the snippets were obtained from mini-cores taken from preconditioned aqueous scoured sub-samples that arise from normal yield procedures. This trial showed that the precision of the LASERSCAN method was slightly better than the conventional Airflow Method (IWTO-28).

As a consequence of this research a “Standard Method for Measurement of the Mean and Distribution of Fibre Diameter using the Sirolan-Laserscan Fibre Diameter Analyser” was adopted by as a by IWTO in May 1993. However, in November 1993 the Standardisation Committee suspended IWTO-12. The reasons for this were political rather than technical, the decision made in the context of debates about the differences between the Laserscan and an alternative technology based on image analysis (OFDA100).

Marler (1994) advised the New Delhi meeting of the Raw Wool Group of IWTO that *"…the SIROLAN LASERSCAN instruments have been introduced into our* (AWTA Ltd's) *Random Micron Round Trials (RMRT), which have operated for Airflow instruments over many years. Recently we* (AWTA Ltd) *have noticed that there appears to be a tendency for a small, unexplained difference to arise at the superfine end of the Airflow calibration range"*. Baird, Barry and Marler (1994) subsequently reported on further experiments that were designed to confirm and explain this anomaly. Although the anomaly was confirmed it was not explained. These authors concluded *"…where differences exist between measurements made by Airflow and by SIROLAN-LASERSCAN, on the same wool, this difference is most likely caused by some physical characteristics of the wool"*. They proposed a "Cores Calibration" of the Laserscan to more closely align it with Airflow but also noted that such an approach also caused the Standard Deviation to increase by a small amount. *"As an interim measure, this serves the purpose of aligning two different instruments, but for the longer term, a better solution, based on an exact understanding of the cause(s) of the discrepancies between Airflow and SIROLAN-LASERSCAN is required"*.

The suspended Test Method for Laserscan was reinstated by IWTO at its Harrogate meeting 1995, following an extensive inter-laboratory round trial organised through IWTO on samples of wool tops and raw wool (Harig, 1995). Amendments, relating to the sampling and measurement procedures, were included at the same time. The Specification now required one subsample to be taken from each subsample tested for yield and 2 test specimens taken from each of these subsamples and 2000 snippets measured for each specimen.

In 1996 the precision limits of the Test Specification were updated, and the method amended so that only 1000 snippets needed to be measured on each test specimen.

Irvine and Barry (1997) developed an improved calibration function for the Laserscan, which was incorporated into the calibration software. IWTO-12 was amended to reflect this at the meeting in Nice that year.

Explaining Differences between Laserscan and Airflow

For more than 25 years the Airflow had been the industry's accepted baseline for commercial and technical evaluation of wool fibre fineness. This has been in the full knowledge that the Airflow does not closely emulate the Projection Microscope in all instances. IWTO-28 (Airflow) contains several clauses detailing instances where measurements provided by the instrument may not be reliable. Notwithstanding this research continued, directed and developing a better understanding of the differences observed, particularly for ultrafine wool.

Sommerville (1997) investigated differences between Airflow and Laserscan for superfine and ultra-fine wool. He confirmed that small differences existed, but the magnitude was diameter dependent, and for the wools examined the Laserscan gave a coarser result.

Sommerville (1998) repeated his earlier experiments using this new calibration function developed by Irvine & Barry (1997) and showed that differences from Laserscan still existed, but for wool less than about 15.5 microns the Laserscan was now finer than the Airflow. Knowles (1998) reported similar trends occurred for New Zealand superfine wool. However, Sommerville (1998) was also able to demonstrate that an extrapolation error associated with the Airflow calibration may be a contributing factor to these observed differences.

Marler and Lindsay (1997) and Sommerville (1997) discussed the effect of Coefficient of Variation on the estimates of Mean Fibre Diameter obtained via the Airflow. This is a fundamental factor, which in some specific instances produces small systematic differences from the "true" result (Marler and Sommerville, 1997). These authors confirmed for the first time the predictions of the theoretical equation (the Kozeny equation) describing the Airflow instrument. Coefficient of Variation of Diameter does cause very small but statistically significant biases in fibre diameter measurement by Airflow.

Theory also predicts that variations in wool density in non-medullated wool fibres will also affect the Airflow instrument but to date no experiments have been done to confirm this.

In 1999, in a background briefing paper to IWTO, in reference to the differences between the technologies, Sommerville noted:

"IWTO now has available four instrumental techniques for the determination of Mean Fibre Diameter of wool. These instruments have been extensively researched.

However, commercial participants in the Wool Industry can continue to have confidence in the underlying technology that has been developed to provide them with objective specifications of the fineness of wool fibre. In particular, they can continue to have confidence in the Projection Microscope as a primary reference method for the calibration of Airflow, Sirolan-Laserscan and OFDA.

Notwithstanding this, each of the calibrated techniques has its limitations. There is not always an exact one-to-one correspondence between them, ***in part because they each use slightly different definitions of fibre fineness****. For Australian greasy wool this correspondence is very close except for some specific instances.*

For Australian greasy wool, less than 30 microns, which is by far the greater proportion of the clip, it may be technically possible to use the results provided by the calibrated instruments interchangeably. However, in the longer term this is probably not commercially advisable, given the trading requirements that now exist."

Commercial Implementation of Laserscan

By 1998/99 AWTA Ltd had conducted approximately 2500 commercial tests using IWTO-12 (Laserscan) since the new calibration function was introduced. At that stage IWTO required all test Certificates for Mean Fibre Diameter by IWTO-12 to be accompanied by a corresponding Certificate using the Airflow Method (IWTO-28). Knowles and Marler (1999) presented the analysis of these data to IWTO at its meeting in Florence in May 1999. The corresponding differences plot showed no evidence of bias and that that the 95% confidence interval was ±0.5 microns, which, given the range of diameters involved, was very close to the precision limits of both methods.

AWTA Ltd introduced Laserscan for Certification of diameter distribution characteristics on 1st July, 2000

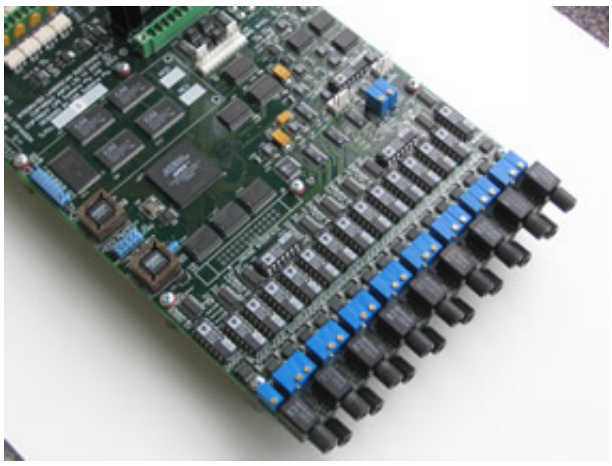
The then Managing Director of AWTA Ltd advised the meeting that AWTA Ltd would be introducing Laserscan as its primary method for IWTO Certification of Mean Fibre Diameter and Standard Deviation of Diameter from 1st July 2000.

For several months leading up to this transition AWTA Ltd tested all lots with both the Airflow and the Laserscan, and continued to provide an Airflow service for an additional fee after the transition. It became apparent that on average the Laserscan result was slightly coarser than the Airflow result.

Crowe and Marler (2000) recommended to the IWTO meeting in Nice in November 2000 a modification to the calibration procedure for the Laserscan instrument to require calibration snippets from the Interwoollabs calibration tops to be obtained by mini-core rather than guillotine, thereby bringing the calibration system for raw wool more in line with the measurement system. They anticipated that this change would make the Laserscan mean diameter very similar to the Airflow mean. In fact the effect was to make the Laserscan mean very slightly finer than the Airflow mean.

From 1999 AWTA Ltd began examining the use of water as the transport fluid instead of iso-propanol. Changes to IWTO-12 to allow this were approved by IWTO in Istanbul in November 2003. These changes enable the Laserscan to be used for measurement of diameter with negligible preconditioning of the sample, as the necessary absorption of moisture to obtain a stable degree of swelling of the fibre snippets occurs within seconds of the snippets being immersed in the transport fluid.

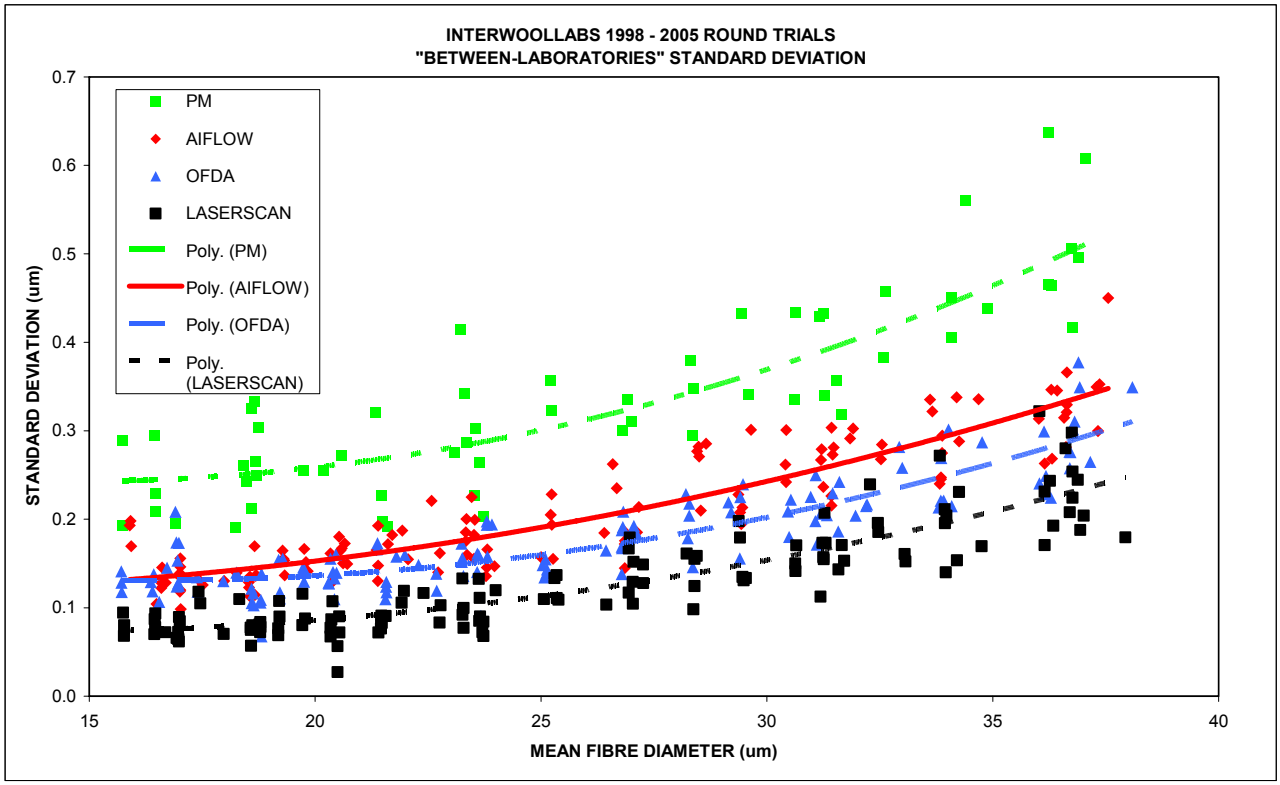
In parallel with this work AWTA Ltd has substantially improved the software and simplified the electronics by incorporating the 13 electronic boards in the original instrument onto a single board.



New electronics for the Laserscan Instrument

Technical Performance

Interwoollabs run a series of round trials every year on wool tops and the results are reported to IWTO each year. Laserscan has consistently exhibited the lowest between laboratories standard deviation of all the available technologies over this period. A summary of the results since 1998 was reported to IWTO in Cairo in May 2006 and this is reproduced below.



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Fundamental Principles of Fibre Fineness Measurement

Part 14

Image Analysis

Peter Sommerville

Corporate Development Manager

AWTA Ltd

June 2007

Originally printed in the April 2007 issue of AWTA Ltd's Newsletter this review article is the fourteenth of a series of articles on the fundamental principles of wool fibre fineness measurement.

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IMAGE ANALYSIS

Principle

In the preceding articles in this series I have attempted to place each technology under a broad category. As is often the case the distinctions between these categories are not always black and white. Image analysis is one example of this.

Image analysis is today and enormous field, due largely to the rapid advances in computer technology. Perhaps the most spectacular applications, simply because the impact on the general community, are in the many areas of modern medical tomography, such as Magnetic Resonance Imaging (MRI), Ultra Sound Imaging and Computerised Axial Tomographic (CAT) scans.

Another significant and well known application is in the X-ray imaging equipment now commonplace in security applications such as screening luggage and airports.

Image analysis is used extensively by NASA for researching the many bodies in our universe and routinely for analysing satellite photographs for estimating agricultural production around the world of a wide range of crops.

Image analysis can be broadly defined as deriving useful information from images. In many applications these images are constructed from complex electromagnetic spectral data using high speed computers, rather than directly from photographic images.

In the case of wool fibre diameter image analysis is generally defined as deriving an estimate of fibre diameter by examining a photographic, digital or projected image of the fibres, either manually, electronically or digitally.

When I discussed the Projection Microscope, I categorised this technology as Optical Microscopy. The Laserscan instrument was placed this under the category of Photometry. However both of these technologies incorporate elements of Image Analysis.

In the case of the Projection Microscope, the measurement is derived making manual measurements of fibres from a magnified image projected onto a screen. During the early development, images of fibre cross sections were also photographed and subsequently measured. Projection Microscopy can therefore also be categorised as the analysis of an image.

The Laserscan also uses image analysis, in the optical detector and in the discriminator. The optical detector analyses a diffraction pattern projected onto the detector by measuring a drop in the voltage output of the detector. The discriminator is a crude digital detector consisting of 17 “detectors” – the electronic circuitry uses the signal output from these detectors to decide whether or not a fibre image projected on to the discriminator meets the criteria established to determine whether or not the ‘image” cast onto the detector is to be measured. In a similar vein, the Electronic Fibre Fineness Indicator (EFFI), developed by Sheep and Fur Animal Branch, Animal Husbandry Research Division, ARS, USDA in Beltsville Maryland in 1957 also uses elements of Image Analysis.

In presenting the story of the different technologies explored by the wool industry in its search for more accurate and more efficient technologies for measuring this most important characteristic of wool the classification of both of these technologies under another heading always was a matter of convenience. Such is poetic, or should we say, scientific licence. However, it does mean that in outlining the development of image analysis as a tool for estimating fibre distribution characteristics of wool, we can start the story in comparatively recent times.

Development

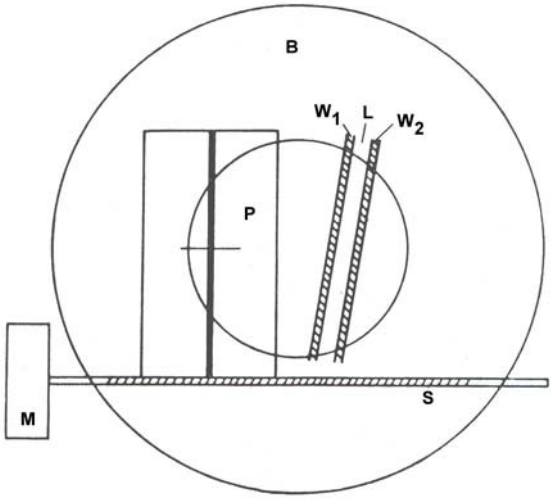
Automating the Projection Microscope

Given that Projection Microscopy marked the beginnings of Image Analysis for the purposes of determining wool fibre diameter it is appropriate to begin the story of the modern development of Image Analysis with

work by W.F. Du Bois & G.J.H Ten Cate reported in 1970. This was an early attempt to provide some automation of the microscopial measurement of cotton fibre dimensions.

These authors noted: *"The measurement of fibre dimensions with the aid of a microscope is a rather time-consuming affair, because these dimensions must be measured with the aid of a ruler or some other measuring device and the measurements must, in one way or another, be written down."*

To overcome these disadvantages Du Bois et al constructed device that was mounted over the ground glass screen of a projection microscope. It consisted of a bar with am extremely precise screw thread driven intermittently, but at a constant speed by a stepper motor (see below).



"Over the ground-glass projection disk, B, a bar, S, is mounted that is provided with an extremely precise screw-thread. This bar is driven intermittently but at a constant speed by a stepper motor, M. The number of steps per unit time is therefore constant. On the bar, a Perspex plate, P, is fitted and this is provided with a measuring line. This line moves stepwise but at a strictly constant speed of the (projected) microscopial images of the fibre(s).

Every time the measuring line passes a dividing line in this image (e.g. the outer wall of a fibre) a hand or foot operated switch is pressed. For instance in measuring the width of the lumen (L) and the diameter of a fibre (W1+L+W1) the switch is pressed four times for every single fibre. The number of steps between two switchings is transmitted electronically to three counters, at the same time marked on a paper recorder. Because of the constant speed of S, this number is a measure of the distance between the successive walls of the fibre.

When the measurement on a single fibre is completed, the counters are automatically switched back to zero."

"The pitch of the screw thread is 1.2 mm, and the stepper motor gives 48 steps for each complete turn of the bar, S. One step therefore induces a displacement of the measuring line of 0.025 mm i.e. 25 µm. At a magnification of 500x this means that one step conforms to 0.05 µm in the fibre dimensions. Every step is counted so the unit of measurement of this device is 0.1 µm.

There are no reports in the literature of this technique being applied to wool fibres, but the Du Bois and Ten Cate did indicate its potential for uses other than the measurement of cotton.

The earliest image analysis systems relied upon examining the area of fibre cross-sections. In the 1930's one common technique was to obtain thin cross-sections of a number of fibres, mount them on a microscope slide, take a photographic image of the slide through a microscope and then use a planimeter to estimate the area of each fibre cross-section. Philippen, Blankenberg and Merk (1971, 1972) described an instrument called a micro-image-analyser which was a refinement upon this technique, but which still relied on measuring fibre cross-sections.

Leaping forward to 1985 Hutchings & Ryder reported a more sophisticated technique for automating the Projection Microscope, this time with direct applicability to wool. Their apparatus consisted of a Gillet & Sibert conference microscope which projected an image upwards at about 45^0 onto an inclined mirror, instead of a screen, whereby it was reflected vertically downward onto a Summagraphics Bit Pad digitiser. The Bit Pad was mounted at a distance below the mirror such that the magnification was about 500x. This distance was not precisely controlled because the instrument was calibrated before each sample was measured.

The Bit-pad was controlled and the data from it processed by a computer. The system was calibrated by touching each end of a projected 300 µm scale with the Bit-pad stylus. Individual fibre measurements involved touching opposing edges of each projected fibre image with the stylus. The computer software

recorded each position from the Bit-pad output and calculated the diameter. All fibre measurements were retained, and at the end of the process mean, standard deviation and a histogram were calculated and output in a printed format.

Areas on the opposing corners of the Bit-pad were set aside to enable the operator to quickly record whether a fibre was medullated and/or whether it was pigmented. The remaining corners contained areas where the operator could reject a measurement and to end the process. Again, activation of these functions simply involved touching the relevant area with the stylus.

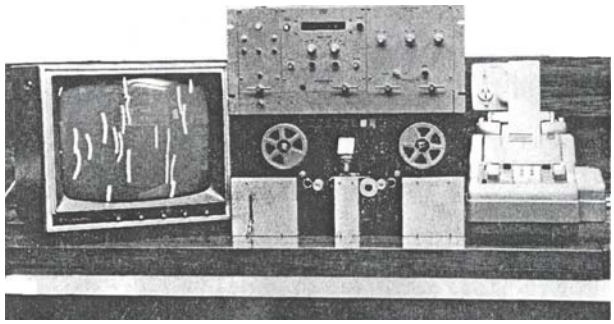
However, both of these innovations were primarily aimed at reducing the tedium involved in the projection microscope method by providing a means to automate some aspects of the procedure. Fundamentally the method itself remained the same.

Refining Photometric Techniques

A more revolutionary approach was announced to the world by Edmunds, Perry & Bedford in a paper published in 1973 in the Journal of the Textile Institute, titled "*FIDIVAN – an Automated System for the Rapid Measurement of Fibre Diameters*". This was an abridged version of an earlier report presented to the IWTO Technical Committee in Monaco in 1972.

The acronym FIDIVAN was derived from the name coined for the instrument – **FI**bre **DI**ameter **V**ideo **AN**alyser.

This instrument borrowed heavily on ideas incorporated Electronic Fibre Fineness Indicator (EFFI) developed in the USA in 1957 and the Fibre Diameter Analyser developed at Textile Department at the University in 1969 (see AWTA Ltd Newsletter, May 2006).



The FIDIVAN instrument

Degreased and washed fibres were first surface-stained with an aqueous iodine solution and rinsed. The still wet fibres were then cut with a semi-automated microtome to obtain 400 µm long snippets. These were dried and conditioned in a small sample bottle using a stream of conditioned air for about 3 hours. A pre-set volume of light-petroleum carrier liquid was then dispensed into each sample bottle.

The solvent and snippets were vigorously dispersed and the suspension dispensed by low pressure compressed air onto the emulsion surface of a slowly moving 16-mm film in the dark. While the snippets were slowly settling through the thin layer of the carrier liquid onto the film a 50 Hertz electric field was applied across the film causing them to become aligned parallel to one another. After a set period the film was exposed by an automatically triggered electronic flash. The exposed film was then processed in the normal way providing up to 100 30 mm long negatives, each containing thousands of images of transversely aligned fibre snippets.

The FIDIVAN instrument was then used to analyse these images to extract the fibre diameter information.

The film was cut into 30 cm lengths, these being spliced to form a continuous loop. One loop was then mounted on the reels of a film transport unit. The drive of this unit rotated the reels repeatedly in a stepwise manner. In this way 90 separate fields of view, each 3 mm x 2 mm were brought successively under a low powered microscope.

The microscope formed an image (magnified 4x) of the illuminated section in its field of view on the target of the tube in a high quality closed-circuit television camera, and the aligned snippet images contained in the field scanned at high speed by the raster of the camera. Since the image was a negative consisting of white fibre images on a black background, aligned at right angles to the direction of scanning, the output of the

camera was a sequential series of voltage pulses, each directly proportional to the diameter of an individual snippet at a point along its length.

FIDIVAN used a single channel pulse-width analyser to determine the width of each of these pulses. This was achieved by the instrument's Diameter Selector, which set up the pulse- width analyser to respond to a particular 2 µm range of fibre diameters. In all the Diameter Selector had a total of 35 positions corresponding to diameters ranging from 10 to 80 µm in 2 µm steps. The film loop was rotated a total of 35 times, once for each setting. At the end of each revolution the number of accepted pulses was printed out so that at the end of the 35 rotations a full frequency distribution was obtained.

Edmunds, Perry & Bedford were very optimistic about the potential utility of FIDIVAN, while acknowledging that there were aspects that required improvement. However, only two reports were presented to IWTO, both during 1972. There were no further reports in subsequent years, and it can only be surmised that the authors encountered technical difficulties that they were unable to resolve.

Computer based Analysis of Digital Images

The late 1970's and 1980's saw a revolution in computer technology and in digital camera technology. This enabled a completely new approach, leading firstly to the FIDAM (**F**ibre **I**mage **D**isplay **A**nd **M**easurement) instrument, and subsequently to the OFDA (**O**ptical **F**ibre **D**iameter **A**nalyser) family of instruments.

FIDAM

FIDAM was developed by AWTA Ltd. It's genesis occurred in 1981 when Mark Brims, then employed by AWTA Ltd as a Research Officer, built a prototype fibre image analyser system, which demonstrated the potential of image analysis for estimating the fineness of wool fibres (Mark is now the Director of BSC Electronics Pty Ltd, the manufacturer of the OFDA). The FIDAM instrument, as it evolved, became very different from this early prototype, but the fundamental principle was the same.

In 1987, after 6 years of development, McNally and Edmunds described the basic principles of this instrument. FIDAM consisted of a video camera, which viewed fibre snippets through a low powered (40x) microscope. The snippets were spread over the surface of a large glass slide using a mechanical spreader, and the slide was moved on a stage beneath the lens of the microscope. The focus of the microscope was fixed and therefore the fibres were generally slightly out of focus. A frame grabber was used to capture the image of each section of the slide as it was being viewed. These digitised images were then analysed by a computer program, using a series of algorithms to select a transverse width at points along the length of the fibre images, and to estimate the magnitude of these widths. The FIDAM instrument therefore defined fibre fineness in terms of the estimated width of an image of the fibre, which was generally slightly out of focus. The instrument relied on the computer program to correct for any errors arising from the lack of focus of the image and to reject images where these errors were too large to correct.

The FIDAM instrument.

Shown here is the microscope, camera and X,Y stage. The associated computer and other ancillary equipment are no longer available.

Further papers describing the instrument were published by Marler and McNally (1988) and Van Schie, Marler and Barry (1990). The final paper in this series concluded: "*The performance of the FIDAM*

instrument in measuring the mean fibre diameter of raw samples compares more favourably with the airflow technique than the FFDA. Further research is required to understand the differences in the fundamentals of the measurement process for single fibre measurements versus bulk fibre web properties before commercial acceptance could be considered. This should include the influence of medullation and the co-efficient of fibre diameter on both web and single fibre measurement systems. AWTA Ltd will not be commercialising FIDAM for raw wool testing".

The reason for this decision is that CSIRO had at that time solved the issues that had plagued the FFDA instrument, and were about to release a new enhanced version trademarked "Sirolan™ Laserscan". The Company decided to pursue the implementation of this technology instead of the FIDAM technology on the basis of 4 criteria:

- precision and accuracy
- speed;
- reliability; and
- cost.

OFDA

In 1991, Baxter, Brims and Taylor described the OFDA (Optical Fibre Diameter Analyser). The principle of this instrument was effectively the same as the FIDAM, although the software and the hardware were developed separately, and the system used different methods of data acquisition and analysis. The authors also submitted a Draft Test Method based on the instrument. A round trial based on this draft was conducted, producing an overall precision for tops, which matched the FFDA and was considerably better than the Projection Microscope. However it was noted that the estimate of Standard Deviation provided by the OFDA was higher by about 4-7% than the Projection Microscope estimates. The draft method was accepted by IWTO as a TME (Test Method under Evaluation).

In the following year the same authors reported the results of further studies based on the OFDA. In particular the authors focused on:

- the effect of contaminants such as grease;
- operator influences;
- snippet geometry; and
- the performance of the computer algorithms.

Baxter, Brims and Taylor reported that the instrument was sensitive to snippet length, for both Mean Fibre Diameter and Coefficient of Variation in Diameter, but these effects diminished as the snippet length approached 2 mm.

The OFDA100 instrument.

At the Nice meeting in 1992, Baxter reported on some round trials with greasy wool cores using the OFDA. This was a study of four sample preparation techniques and their effect on the precision of the TME. It showed that mini-coring the aqueous scoured core sample or mini-coring a sample prepared using a Waring Blender produced a level of precision equivalent to that of the Airflow for fine wools, but not for the coarse wools.

Baxter and Teasdale (1992) investigated the effect of calibrating with Interwoollabs tops on the precision of the OFDA system and reported that it was negligible. However, Lupke, Wright and Botes (1992) reported some comparisons of the OFDA and the Airflow for greasy wool samples from individual fleeces, which indicated a systematic bias of approximately 0.5 microns.

Subsequent round trials on greasy core samples did not confirm this bias (Edmunds, 1993 and Baxter and Brims, 1994), although, they did show significant differences between Airflow and OFDA on some very coarse wool samples. However, if these examples were excluded, the data suggested that on average, the OFDA estimates of Mean Fibre Diameter were marginally finer than the Airflow. A similar trial using wool tops (Baxter and Brims, 1994) also showed close correlation with the Airflow and with the Projection Microscope. However, data presented in this paper provided the first indication of a systematic pattern in the differences between Standard Deviation measured using the OFDA and using the Projection Microscope. This bias was subsequently confirmed by independent experiments.

In 2000 the OFDA100 range of instruments was extended with the release of the OFDA 2000. This was a portable version specifically targeting the on-farm fleece testing market. Based on image analysis of "micro-staples" drawn from full length fleece samples it provided estimates of MFD, SD, CVD, Curvature and Fibre Diameter Profiles along the length of the staples. .

The OFDA2000 instrument.

However, the instrument could also be configured in "OFDA100 Mode" and consequently IWTO permitted its use for IWTO Certification by IWTO-47.

Apart from its portability and additional functionality the major change in the instrument was the porting of system software from the DOS operating system of the OFDA100 to a Windows operating system. BSC Electronics, while continuing to maintain the OFDA100 is not doing any further development of this platform. As a consequence the instruments will become redundant because the computers and their operating systems are already redundant. It is not possible to simply transfer the OFDA2000 software across to an OFDA100 as the software on both instruments is hardware specific.

The OFDA range was further extended in 2002 with the release of a new instrument branded the OFDA4000. This instrument was targeted at wool combing mills. It reports the same parameters as the OFDA2000, but for wool tops and also provides an estimate of Hauteur. In this market the OFDA4000 competes with the Almeter, but has substantially more functionality, providing the mills with a single instrument for Fibre Diameter Distribution estimates as well as Hauteur. IWTO has at this stage approved a Draft Test Method for the instrument (DRAFT TM-62). However, development of this Draft Test Method has not been without controversy.

Development of the IWTO Specification – IWTO 47

The Draft Test Method for the OFDA100 was advanced to a full test specification (IWTO-47) in 1995, following an extensive inter-laboratory round trial on samples of wool tops and raw wool (Harig, 1995). In general this trial confirmed some of the discrepancies that had already been reported, and also that they were quite small. The pattern in the Standard Deviation referred to above was more clearly apparent in this data. However, in this instance, for raw wool, the OFDA Mean Fibre Diameter was slightly coarser than the Airflow. Later work by Sommerville (1997, 1998) and Knowles (1998) on superfine wool showed that for these wools the OFDA was generally finer than the Airflow.

Sommerville (1998) was also able to demonstrate that an extrapolation error associated with the Airflow calibration was a contributing factor to these observed differences.

Compared to other instrumental techniques, the OFDA was different in its calibration requirements. Turpie (1996) and Turpie and Steenkamp (1996) reported on the effect of relaxation of wool slivers on the measurement of Mean Fibre Diameter by Airflow, Sirolan-Laserscan and OFDA. Of the three instruments, only the OFDA exhibited any significant effect. The current IWTO Specification for the OFDA requires a specific calibration for greasy wool and another calibration for sliver. These calibrations use different preparation techniques for the calibration tops. This was not then required for the Sirolan-Laserscan.

At the Christchurch meeting of IWTO in 2000, the Independent Round Trials (ILRT) Group reported data showing that the ODFA100 was very sensitive to the sample preparation technique. The instrument could be calibrated for Mean Fibre Diameter, but this introduced biases in the Standard Deviation. If it was calibrated for Standard Deviation this introduced biases in the Mean Fibre Diameter.

Consequently IWTO-47 was amended to require separate calibrations when certifying Mean Fibre Diameter and Standard Deviation.

Resolving Differences

The differences between ODFA100 and Airflow, can to some extent be attributed to the same factors that also cause differences between the Sirolan-Laserscan and Airflow, so these factors will not be further considered here.

The systematic bias in OFDA100 measurements of Standard Deviation compared with both the Projection Microscope and the Sirolan-Laserscan has never been satisfactorily resolved.

Basically, when the OFDA100 is calibrated for diameter measurement, the SD measurements exhibit a highly reproducible diameter dependent bias. For very fine wools the OFDA100 SD is always higher. As the diameter increases the SD's of all the instruments converge, in some experiments becoming negative in the mid micron range. As the diameter increases further the difference becomes increasingly positive. When plotted over the full range the differences curve assumes a somewhat parabolic shape.

Likewise, there is a systematic difference in the shape of the distribution histograms. At the finer and broader ends of the range the OFDA100 distribution is somewhat normal, whilst within the intermediate range it assumes a skewed conformation, very similar to the Projection Microscope and the Sirolan-Laserscan.

Technically a resolution of this difference was found when the ILRT Group provided the data that showed that calibrating for Mean Fibre Diameter and SD separately mitigated these differences, and IWTO-47 was amended accordingly.

Scientifically this is probably less than satisfactory, as scientists always want to understand and explain such fundamental differences.

Some unpublished modeling work by AWTA Ltd conducted by its National Technical Manager, Mr. Jim Marler has suggested that part of the explanation can be found in the large error for individual fibre measurements made by the OFDA1000. The instrument uses a CCD camera where the pixel size is 4 µm square – comparatively large compared with the actual diameter of the fibres. Errors arising from this low resolution are compounded by the fact that the images captured by the instrument are also generally slightly out of focus and the software is required to apply an algorithm to adjust for this.

When allowance is made for these errors, and the output of the instrument mathematically modeled, the resultant distributions and standard deviations are comparable to those actually produced by the instrument.

Commercial Implementation

The ODFA100 is widely used in mill laboratories around the world for quality control purposes and also by many fleece testing laboratories.

AWTA Ltd has a number of these instruments and can provide test results using IWTO-47, but this service is an add-on rather than a default service. To facilitate the trading of wool if Certification according to IWTO-47 is requested, Certification by IWTO-12 (Sirolan-Laserscan) is always provided.

The best comparative measure of the precision of the OFDA100 is provided the Interwoollabs Round Trials which are reported each year to IWTO.

These can be viewed at the end of the chapter on Photometry which proceeds this chapter and demonstrate that the instrument in better than both the Projection Microscope and Airflow, but not quite as good as the Sirolan-Laserscan.

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