

6. Scoured Wool Quality and Testing

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Learning objectives

On completion of this topic you should be able to:

- Explain why testing is important to the industry
- Describe the sampling techniques appropriate to scoured wool testing
- Outline the yield test procedures, in particular the determination of wool base, ash content, vegetable matter content and solvent (i.e. ethanol and DCM) extractables
- Explain why and how various yield calculations are carried out, for presentation on a test certificate
- Describe briefly the modern instruments used for fibre diameter measurement, their operating principles and the results they produce
- Outline the colour measurement techniques applicable to wool, including preparation, instrument requirements and the form in which results are reported
- Explain the sampling, preparation and testing steps for the Length After Carding test
- Calculate the regain of a wool sample, given the original mass and the dry mass
- Outline the rapid tests used in the scour for moisture and residual grease measurement
- Describe how NIRA techniques have revolutionised in-scour quality management

Key terms and concepts

Objective measurement, sampling, yield, mineral (ash) content, vegetable matter content, alcohol (and DCM) extractables, woolbase, clean weight, micron, fibre diameter distribution, OFDA, Sirolan Laserscan, base colour, as-is colour, colorimeter, spectrophotometer, tristimulus values, illuminant, Length After Carding (LAC), carding, gilling, Almeter, regain, moisture content, near infrared reflectance analysis (NIRA), scour liquor, effluent

Introduction to the topic

Wool scouring is a process that adds value to wool, by removing the natural and acquired contaminants before yarn manufacture can begin. Because of the expectations of the client, it is essential that the scouring is thorough and consistent and a clean, bright product of the required moisture content is delivered. To achieve good scouring it is necessary to regularly check wool quality by testing in the scour. To provide firm evidence of quality to the client, certificated results are produced by a test house from the testing of a sample of scoured wool sent from the scour.

This topic looks at the full range of certificated tests that are routinely carried out on scoured wool consignments in Australia and New Zealand – yield, vegetable matter content and solvent extractables, fibre diameter, colour, regain and length after carding. In addition, the in-scour tests for quality are included – the rapid tests for grease (solvent extractables) and regain, and the use of NIRA technology. Tests are also available for woolgrease and effluent quality.

6.1 Introduction

Given the objectives of wool scouring it is obvious that the quality of the scoured product will be of concern to scourer and client alike. While some quality attributes can be assessed subjectively, the modern industry relies mostly on objective tests to ensure that the product meets the client's specifications and expectations with respect to quality

Testing is the term used for the various ways of:

1. Determining the physical properties of samples of greasy and scoured wool
2. Analysing scouring liquor, effluent, and woolgrease.

Commercial testing is done by accredited testing houses to facilitate the exchange of ownership of wool. The results issued by a test house are legally certified. Company staff also carry out in-plant testing during the scouring process, mainly to ensure that the quality of the scoured product is maintained and acceptable to the client.

Most of the commercial sampling and testing procedures have been developed through the International Wool Textile Organisation (IWTO) (<http://www.iwto.org/>) and the American Society for Testing and Materials (ASTM) (<http://www.astm.org/cgi-bin/SoftCart.exe/index.shtml?E+mystore>). The standard methods used for testing wool samples and the results presented on a test certificate are the basis for international commercial transactions.

Objective measurements of wool are needed to get accurate assessment of wool fibre properties. Measurements are objective where the results of scientifically-based tests are not open to different interpretation by different people. The testing and results are required for two reasons:

1. *The efficiency of modern methods of selling wool:* When wool is sold, buyers need to know the specifications of the wool before they buy it. By using recognised scientific standard testing methods, buyers can compare different lots of the same wool to see which will best suit their customers needs
2. *The use of modern textile machinery:* The further processing of wool is influenced by its type. The buyer needs specifications to buy the correct wool for the customer's processing system and to see which lot of wool will give the best processing yield.

Properly carried out objective, scientific tests will always be preferred to the subjective assessments based on the personal judgement of highly skilled wool buyers. This is true despite the fact that testing is generally more expensive.

The objective measurement of wool values comprises two steps: (1) sampling and (2) testing.

The sampling methods apply to both greasy and scoured wool, but different equipment may be used depending on whether the wool is scoured or not.

The downloadable document 'Testing the Wool Clip' (AWTA 2004) (http://www.awta.com.au/Publications/Marketing/Raw_Wool_Services/Testing_the_Clip.htm), is a useful source of information on wool testing.

6.2 Sampling principles

As with the testing of any material, the wool sample obtained for testing should:

1. Truly represent the lot of wool being sold or processed
2. Be drawn by trained operators who are independent of both buyer and seller, and
3. Be drawn using equipment and techniques that are approved and controlled by an authorised organisation.

If possible, sampling should be carried out by a machine that ensures consistency and eliminates the possibility of human bias.

Wool sampling is mainly carried out:

- a) In a broker's or wool merchant's store – greasy wool lots are sampled for testing prior to sale
- b) In a woolscour – scoured wool consignments are sampled for testing prior to dispatch.

Industry practice requires every bale in a greasy lot or scoured wool consignment to be sampled for testing. Only scoured wool sampling will be considered in this topic.

6.3 Scoured wool sampling and testing

Core sampling for scoured wool can be taken in the packing area of the woolscour. This can be done by:

1. A hydraulic coring unit for dense-baled scoured wool (Figure 6.1), or
2. Pushing a coring tube into the bale manually (only for conventional bales).

Figure 6.1 Core sampling in a woolscour. Source: Wood, 2006.



The IWTO Core Sampling Regulations require the coring tube to penetrate at least 93% of the length of the bale when machine coring, or at least 47% of the length of the bale if manually coring.

The core sample is used for in-house testing or is forwarded to a test house for certificated testing. Most tests applicable to greasy wool can also be carried out on scoured wool core samples, together with the additional tests for regain (or moisture content), pH and Length After Carding.

Since core samples cannot be used to provide a length measurement, in most NZ wool scours a special 'spear-head' probe is fitted to the dense-baling machines to extract a full length sample of scoured wool before the wool destined for a bale becomes highly compressed (Figure 6.2). The sample extracted by this device is sent to a test house for testing using the Length After Carding Test.

**Figure 6.2 Sampling Probe for the Length after Carding Test.
Source: New Zealand Wool Services International Ltd. (www.nzwsil.co.nz).**



The test house is supplied with the core samples or full length samples and other relevant information on the consignment of wool to be tested. This other information may include:

1. The total number of bales in the lot
2. The gross weight of the lot
3. The declared tare weight, that is, the weight of the packs, and
4. The net weight of the wool in the lot, that is, the gross weight minus the tare weight.

The tests to be considered in this topic are:

- Yield and residual grease
- Fibre diameter
- Colour
- Length after Carding
- Regain
- Testing in the scour
- NIRA

6.4 Yield testing

While yield testing (http://www.awta.com.au/Publications/Marketing/Raw_Wool_Services/yield.html) is carried out on greasy wool and scoured wool, the results will be quite different because scoured wool is virtually free of all contamination. Test results of around 100% are expected. The steps in the procedure are as follows:

Dry-scoured wool yield

Subsamples of the core samples, each weighing about 150 g, are scoured to determine the dry-scoured weight. The dry-scoured weight can be expressed as a percentage of the original weight to give the dry-scoured yield. It is calculated as follows:

$$\text{Dry-scoured yield} = \frac{\text{oven dried weight of sample}}{\text{original weight of sample}} \times \frac{100}{1}$$

By testing the dry scoured sample to determine:

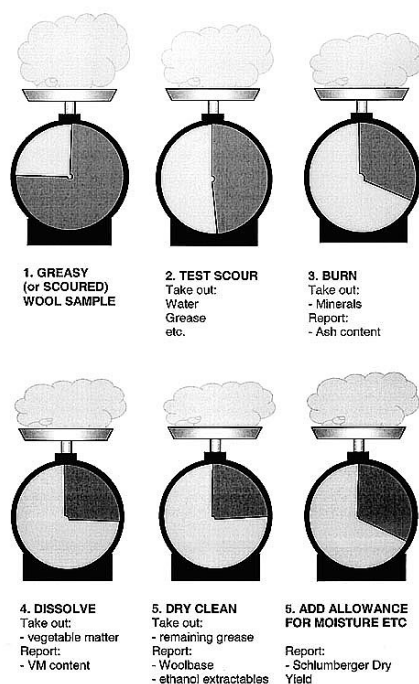
- a) the mineral or ash content
- b) the vegetable matter base or content (VM) (http://www.awta.com.au/Publications/Marketing/Raw_Wool_Services/major_vegetable.html), and
- c) the alcohol extractables, the *wool base* can then be calculated.

Knowing the wool base, various yields can be calculated. These indicate to a processor the yield that can be expected when the lot of wool has been processed. The yields used throughout the world for the commercial trading of wool include:

1. *IWTO clean wool content*: This is the yield of clean wool, allowing standard amount of residual dirt and grease of 2.27%, at a regain on 17%. There is no allowance for vegetable matter
2. *IWTO scoured wool yield at 16 or 17% regain*: The yield of clean wool plus vegetable matter, allowing a standard amount of residual dirt and grease of 2.27%
3. *ASTM clean wool fibre present (CWFP)*: Similar to (1) but uses a regain of 13.64%
4. *Japanese clean scoured yield*: The yield of clean wool, allowing a standard amount of residual dirt and grease of 1.5% and a regain of 16%
5. *IWTO Schlumberger Dry Top and Noil Yield*: The mostly used yield parameter for NZ wools (and the most complicated calculation, which is outlined later).

Figure 6.3 shows in a simplified for the steps for the yield test. Note that the diagrams depict greasy wool testing; for scoured wool the balance would show close to 100% at each stage.

Figure 6.3 Steps in the yield test. Source: Wood, 2006.



Three tests enable the above yields to be calculated.

(1) Mineral or ash content

A scoured sample is burnt in a furnace at 700 °C. The resultant ash is the sand and dirt not removed in scouring. The ash is weighed to the nearest 0.001 g.

(2) Vegetable matter base or content (VMB)

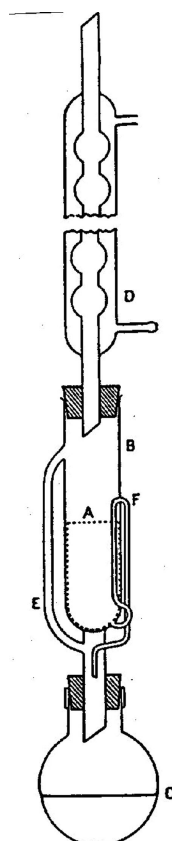
The test specimen is at least 40 g of the scoured sample. The specimen is placed in a 10% boiling solution of sodium hydroxide. The chemical formula for sodium hydroxide is NaOH, and its common name is caustic soda, which is an alkali. The wool fibres are dissolved in the solution, leaving the vegetable matter (VM). The VM is dried to a constant weight and then ashed at 700 °C. The weight of the ash is deducted from the constant weight of the VM. Allowances are made for the presence of hard heads (HH), which are twigs and hard pieces of VM that would fall out during processing. All weights are expressed in grams and taken to the nearest 0.001 g.

(3) Alcohol extractables

This is determined by the Soxhlet extraction of grease using ethyl alcohol as the solvent. Figure 6.4 shows a Soxhlet extraction apparatus in use. The porous thimble A is made of tough filter paper so that the solvent can filter through it. The sample of wool is put into the thimble, which is placed in the barrel of the apparatus B. The solvent is placed in the flask C. The flask of solvent is heated so that the solvent boils briskly. The solvent vapour passes through the wide tube E into the condenser D, where it condenses back into a liquid. The outside of the condenser has a constant flow of cold water in at the bottom and out at the top to ensure that the solvent vapour condenses.

The condensed vapour falls on to the sample in the thimble, and the barrel gradually fills with solvent. When the liquid reaches the top bend of the syphon tube F, it siphons over into the flask C, and thus removes the portion of grease extracted by the solvent. The liquid has become a mixture of solvent and dissolved grease. The liquid in the flask continues to boil and the process is repeated, with the solvent evaporating.

Figure 6.4 Soxhlet extraction apparatus. Source: Wood, 2006.



After 24 repeats of the siphoning cycle, the extraction process is stopped. The grease in the flask is separated from the solvent by evaporating the solvent out of the flask. The extracted grease matter is then weighed and expressed as a percentage of the weight of the original sample of wool before extraction.

Extraction tests for scoured wool often specify methylene chloride (or dichloromethane DCM) as the solvent rather than ethanol. This is because ethanol removes some lipid materials from the fibre as well as the surface contamination, thereby giving an over-estimate by about 0.8% of the residual grease present.

The IWTO standard grease tolerance for scoured wool is 1.25%, but to avoid soiling of fibres downstream the target level is generally set at around 0.3% (using DCM extraction).

Wool base

From the results of the three tests, (1) mineral or ash content, (2) VMB, and (3) solvent extractables, the percentage of clean, dry, ash-free, extractables-free, vegetable matter-free wool in the sample can be calculated. This is the Wool Base (WB), which is expressed as a percentage of the greasy sample, i.e.:

$$\text{WB} = \frac{\text{dry weight of wool} - \text{weight of all impurities}}{\text{greasy weight of core sample}} \times 100$$

Having calculated the wool base, other calculations, some of which use the WB, can be carried out.

IWTO clean wool content

Because a percentage of both mineral matter and greasy matter is left in the wool after scouring, a standard allowance is made for the two impurities. The IWTO clean wool content is made up of

WB	97.73 %
Standard allowance for impurities	$\frac{2.27 \%}{100.00 \%}$

From the wool base result, it is possible to calculate the various commercial yields that are likely to be requested. These are placed on the certificate to show the expected yields of the wool when it is processed. The following shows how the various yield calculations are carried out.

$$\begin{aligned}
 \text{IWTO clean wool content at 17\% regain} &= \text{WB} \times \frac{100}{97.73} \times \frac{117}{100} \\
 &= \text{WB} \times \frac{117}{97.73} \\
 &= \text{WB} \times 1.1972
 \end{aligned}$$

IWTO scoured wool yield at any required regain (R)

$$\begin{aligned}
 &= (\text{WB} + \text{VMB}) \times \frac{100}{97.73} \times \frac{(100 + R)}{100} \\
 &= (\text{WB} + \text{VMB}) \times \frac{(100 + R)}{97.73}
 \end{aligned}$$

ASTM clean wool fibre present (CWFP) at 13.64% regain, with allowance of 2.27% for impurities

$$\begin{aligned}
 &= \text{WB} \times \frac{100}{97.73} \times \frac{113.64}{100} \\
 &= \text{WB} \times \frac{113.64}{97.73} \\
 &= \text{WB} \times 1.1628
 \end{aligned}$$

Japanese clean scoured yield at 16% regain, with allowance of 1.5% for impurities:

$$\begin{aligned}
 &= \text{WB} \times \frac{100}{98.5} \times \frac{116}{100} \\
 &= \text{WB} \times \frac{116}{98.5} \\
 &= \text{WB} \times 1.1777
 \end{aligned}$$

Schlumberger dry top and noil yield (SCDY)

To understand the calculation of the Schlumberger dry top and noil yield, some knowledge of worsted processing is useful:

Noils are removed in the worsted spinning route when the fibres are combed. They consist of short and tangled fibres that, if left in the worsted yarn, would adversely affect its strength and appearance. A feature of worsted yarn is its clean, even appearance.

A *top* is a sliver of combed fibres after the noils have been removed by the combing machine. After the top is made, the fibre slivers are prepared for spinning into worsted yarn.

The SCDY is based on a uniform ratio between the top and noil of 8 top to 1 noil (8:1). This ratio is called *tear*. Tear is the ratio of the amount of clean or combed top to the amount of impurities and unusable fibre removed by combing. The standard tear used in calculations is 8:1.

This top and noil yield shows the buyer the expected yield from the lot of wool after it has been combed as part of the yarn making process.

IWTO theoretical dry top and noil yield is calculated as follows:

$$\text{Schlumberger dry combed} = \text{WB} \times 1.208$$

The conversion to estimated commercial top and noil yield is calculated. The calculation is based on the theoretical top and noil yield with an allowance (VA) for the vegetable matter (VM) content but without the hard heads (HH).

$$\text{The allowance VA is calculated as: } VA = 5.2 - \frac{40.6}{7.8 + (\text{VMB} - \text{HH})}$$

Hence the Schlumberger commercial dry top and noil yield is:

$$\text{SCDY} = 1.208 \times \text{WB} - (2.5 + \text{VA})$$

Calculation of clean weight of consignment

Using any of the yield calculations outlined above, it is possible to calculate the *clean weight* of the consignment of scoured wool being tested. This is done for each individual yield and is presented on the test certificate. The clean weight is calculated as follows:

$$\text{Clean weight} = \frac{\text{percentage yield}}{100} \times \text{net weight of lot}$$

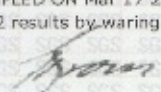
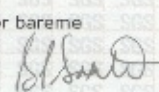
For example, if the SCDY result is tested to be 68.3% on a lot of 6500 kg (greasy weight), the clean weight is:

$$\begin{aligned} \text{Clean weight} &= 68.3 \times 6500 / 100 \\ &= \underline{4440 \text{ kg}} \end{aligned}$$

Reporting yield results

Figure 6.5 shows a section of a test certificate for a scoured wool consignment. The results for the various yields (as well as micron, colour, residual grease, regain and the conditioned weight of the consignment) are presented.

Figure 6.5 SGS Wool Testing Services Ltd. (www.wooltesting.sgs.com).

SGS		Wool Testing Services	
		Test No 12345678	
		IWTO TEST CERTIFICATE	
		No 1-12345678-X0	
		For IWTO 19/28/33/56/10	
		Of Scoured Wool	
Clients description/Shipping brand:-		Page 1	
		Date Mar 22 2005	
		Bales	20
		Gross Weight	8337.0 kg
		Declared Tare	99.0 kg
		Nett Weight	8238.0 kg
RE-NUMBERS 1-20			
YIELD TEST RESULTS (IWTO 19)			
Wool Base (4 Subsamples) 87.15%			
Vegetable Matter Base 0.1%			
(Inc 0.0% Hard Feed & Twigs)			
CALCULATED YIELDS			
IWTO S.W. @ 16%	103.5%	=	8530 kg
NZ PROCESS YLD	102.6%	=	8451 kg
IWTO S.W. @ 17%	104.5%	=	8608 kg
J.C.S.Y. @ 16%	102.6%	=	8456 kg
ASTM C.W.F.P.	101.3%	=	8349 kg
IWTO C.W.C.	104.3%	=	8596 kg
MEAN FIBRE DIAMETER (IWTO 28)			
(AIRFLOW 4 SPECIMENS)		=	36.5µm (Microns)
RESIDUAL GREASE (IWTO 10)			
@ 16% Regain		=	0.25 %
ADDITIONAL INFORMATION			
ASH TEST (IWTO 19)		=	0.70 %
CONDITION TEST (IWTO 33)			
Sample Weight	761.16 g		
Dry Sample Wt	675.83 g		
Oven Dry %	88.79 %		
Moisture Content	11.21 %		
Actual Regain	12.63 %		
CONDITION WEIGHTS			
@ 16.00% Regain	=	103.00 %	
	=	8485 kg	
@ 17.00% Regain	=	103.88 %	
	=	8558 kg	
COLOUR MEASUREMENT (IWTO 55)			
Mean of 8 Measurements			
C/2(w)* D65/10			
COLOUR X	=	65.8	67.6
COLOUR Y	=	67.4	71.4
COLOUR Z	=	67.4	61.3
Y-Z	=	0.0	10.1
SAMPLED ON Mar 17 2005			
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6.5 Fibre diameter measurement

The AWTA web site has an excellent downloadable document on this topic (http://www.awta.com.au/Publications/Research_Papers/Reviews/Fibre_Fineness_Measurement_Fundamentals.pdf).

Airflow method

The mean fibre diameter of wool has traditionally been measured by the Air-flow method. In this method the diameter of the fibre depends on the rate of air-flow through a porous plug of wool weighing 2.5 g. A standard pressure difference is maintained across the plug of wool and the rate of air-flow is expressed in microns (µm). A micron is one micrometre, which is one millionth of a metre (1/1,000,000). The finer the fibre, the greater the total surface area in the plug of wool, and hence the slower the rate of air-flow. This is because there will be more fibres in the plug of wool (hence a larger total surface area) than if the fibres were thicker. The instrument must be calibrated using wools with a range of accurately measured micron values. An AWTA newsletter

(http://www.awta.com.au/Publications/Newsletter/2004_March/Mar_2004_For_Technophiles.pdf) has an interesting article on the airflow test.

Despite its simplicity and its once universal use for commercial testing of wool, the Airflow test has several limitations. It provides only one result – the mean fibre diameter – rather than indicating the range of fibre diameters in a sample. Furthermore, it tends to be unreliable for certain types of wool, especially medullated wools and lambswool.

Modern instruments

Because of its limitations, the Airflow Test has now been replaced in most countries by two high-tech methods, OFDA (i.e. Optical Fibre Diameter Analyser) (<http://www.ofda.com/>) (Figure 6.6) and *Sirolan Laserscan* (http://www.awta.com.au/Publications/Technical/laserscan_technical/index.htm) (Figure 6.7).

Figure 6.6 The OFDA system, showing (clockwise from top) printed results, monitor showing video images, guillotine, spreader, microscope with video camera, computer.

Source: BCS Electronics.

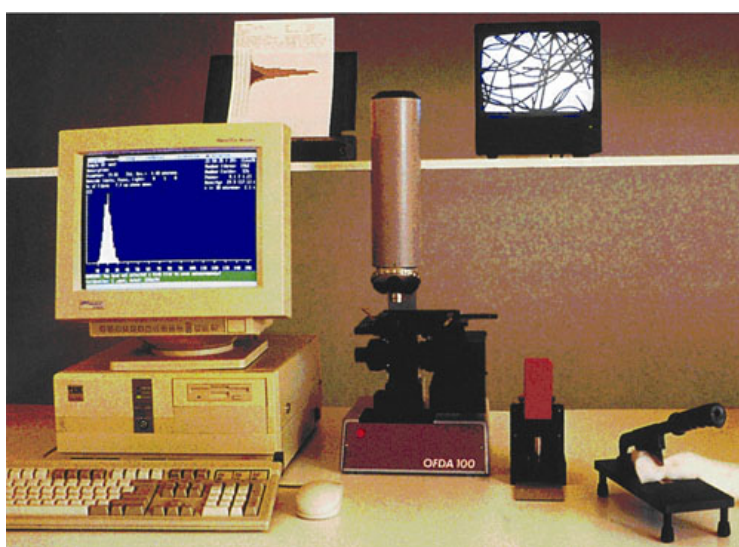
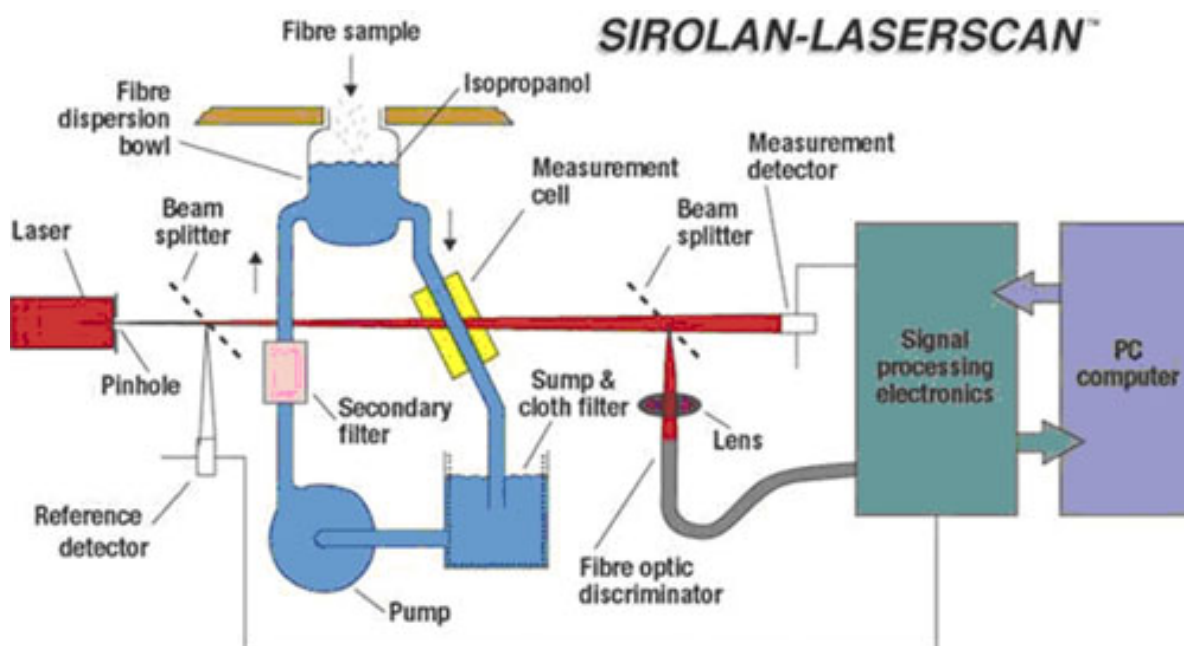


Figure 6.7 The Sirolan Laserscan system. Source : Australian Wool Testing Authority Ltd. (www.awta.com.au).



Both instruments measure the diameter of tiny snippets of wool fibres (but by two different approaches), and they rapidly provide the complete *fibre diameter distribution* of a sample of wool.

In OFDA the snippets are placed on a microscope slide by a 'spreader' device and the slide is then positioned on the platform of the microscope. As the slide is traversed under the microscope, a succession of images is captured by the video camera. These images are analysed sequentially by dedicated software to present the fibre diameter distribution of perhaps 3000 or more snippets in just a minute or two. Using the equivalent, manual instrument, the projection microscope, might take a day or more to perform the same task.

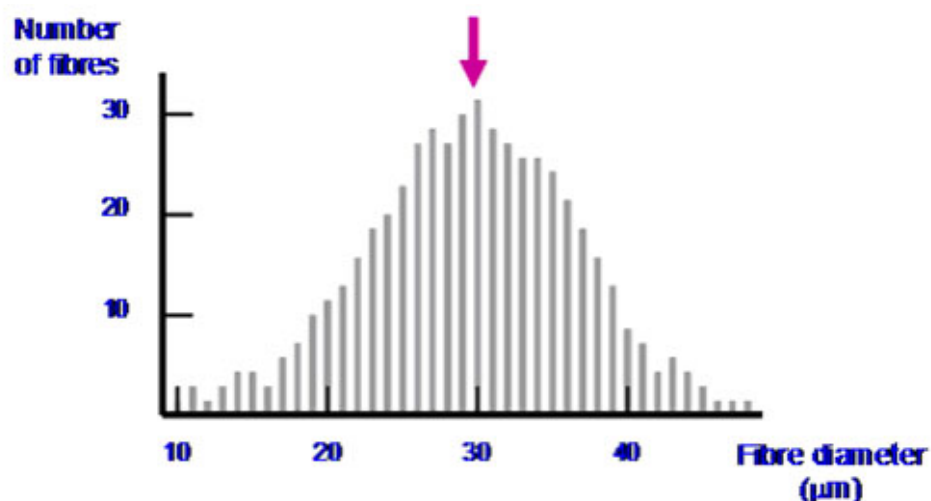
In Laserscan the individual snippets are transported past a fine laser beam by a stream of isopropanol. A snippet interrupts the beam and cast a shadow on an array of sensors, called an 'optical discriminator'. This produces an electrical signal that is proportional to the amount of light incident upon it. The fibre snippet reduces this signal by an amount that is directly proportional to the projected area, and therefore the diameter of the fibre. (More technical details of the Laserscan detection system are available by the downloadable document on *Sirolan Laserscan*) http://www.awta.com.au/Publications/Technical/laserscan_technical/index.html.

Figure 6.8 shows a typical fibre diameter distribution of a wool sample, as might be reported by OFDA or Laserscan. The distribution shows the diameters of the finest and coarsest fibres in a specimen tested, according to the proportions present in the specimen. Unlike the Airflow instrument, these instruments are reliable with all types of wool. As an additional feature, both OFDA and Laserscan are able to measure fibre curvature, which is closely related to the level of crimp in the fibres. Only OFDA is able to measure medullation content.

Fibre diameter is widely varying between breeds, between similar sheep in the same flock and even within the fleece of an animal. Hence the mean fibre diameter is mostly used to describe the fineness or coarseness of wool offered for sale, and partially processed wool (i.e. sliver and top). Information on the range of diameters is represented by the *coefficient of variation of diameter*.

Because (a) the price of fine wool is invariably much higher than coarser types, and (b) diameter is more critical in the manufacture of fine yarns and light fabrics, the measurement of diameter is more important on fine wools than coarse wools. This means that the client for a fine wool consignment (mostly for apparel) is much more concerned about its mean fibre diameter than the client for a consignment of coarse wool that will probably be converted into carpet.

Figure 6.8 A typical wool fibre diameter distribution. Arrow indicates mean fibre diameter (mean ~ 30 micron). Source: Wood, 2006.



6.6 Colour testing

The colour of scoured wool is of primary importance when assessing its usefulness in processing and its selling price. The colour of wool indicates what colours can or cannot be dyed on any particular lot. The colour results obtained by testing therefore indicate the suitability of the lot of wool for the shades that might be needed. If pastel shades are in fashion, then the whiter the wool, the higher its selling price.

Because of the variation in colour in New Zealand fleece wools, in comparison with the uniformly white colour that typifies fleece wools in the Australian clip, colour testing has always had greater importance in New Zealand.

An IWTO standard defines the test method for the measurement of colour in both scoured and greasy wool. Colour measurement is carried out on wool that has been scoured or re-scoured in the laboratory to ensure that no residual matter, which could affect the colour reading, remains on the fibre. The test for the *base colour* aims to determine the maximum potential colour of the wool being tested (i.e. using the cleanest possible sample). Thorough cleaning prior to measurement is therefore essential.

As-is colour is the result obtained when a sample of scoured wool is tested without an additional cleaning. Comparison of the base colour and as-is colour for a consignment provides an indication of scouring effectiveness, i.e. the closer the two results the better the scouring.

Given the need for an exceptionally clean fibre to be presented for colour measurement, the test house takes considerable care in the preparation of the wool sample. While the objectives are the same, sample preparation methods differ from test house to test house, with combinations of test scouring and mechanical carding generally being used. The small carding machine used here is the Shirley Analyser.

Principles of colour measurement

To understand how colour measurement works it is necessary to know a little about colour. When daylight strikes an object, the light can be wholly absorbed by the object. Then, the object appears black. If the daylight is totally reflected, the object appears white.

If the object absorbs some of the colours of the spectrum (<http://hyperphysics.phy-astr.gsu.edu/hbase/vision/specol.html>), then it appears as the colour of the light that is not absorbed. The spectrum of white light is made up of seven colours:

Red, Orange, Yellow, Green, Blue, Indigo, Violet

An object that absorbs say red, orange, green, blue, indigo, and violet appears yellow because the light it reflects has much more yellow than the other colours in the visible spectrum. However, if the light shining on the object is not daylight but contains a predominance of some section of the spectrum (i.e. it is 'coloured'), then the colour of the object will appear different from its colour under daylight. An example of this is when objects are viewed at night under the orange illumination of sodium vapour street lamps.

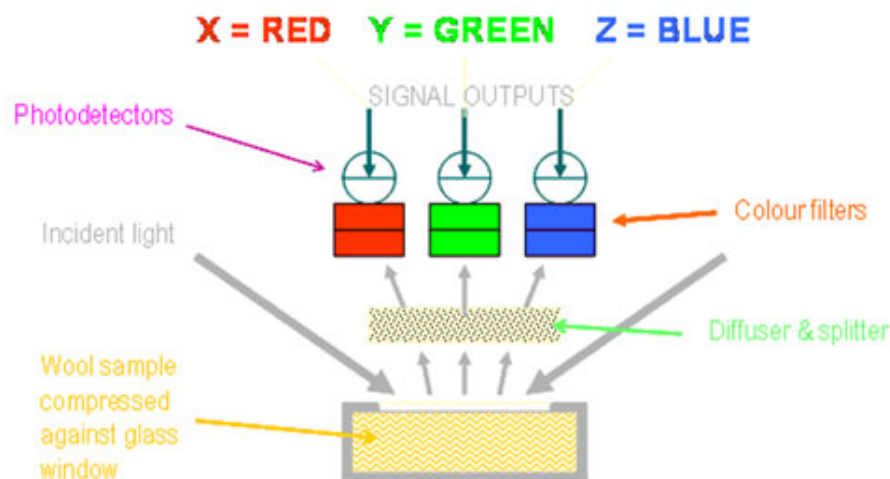
Hence, when the colour of wool is measured, a standard light source (or illuminant) must be used to ensure consistency and agreement between instruments.

Using a series of many coloured filters, it would be possible to measure the amount of light reflected from an object over all parts of the visible spectrum. This would give very accurate readings, but the result would be too cumbersome. To simplify the readings, the spectrum is divided into three zones. The zones are made up as follows:

1. The red/orange area, called X
2. The green/yellow area, called Y, and
3. The blue/indigo/violet area, called Z.

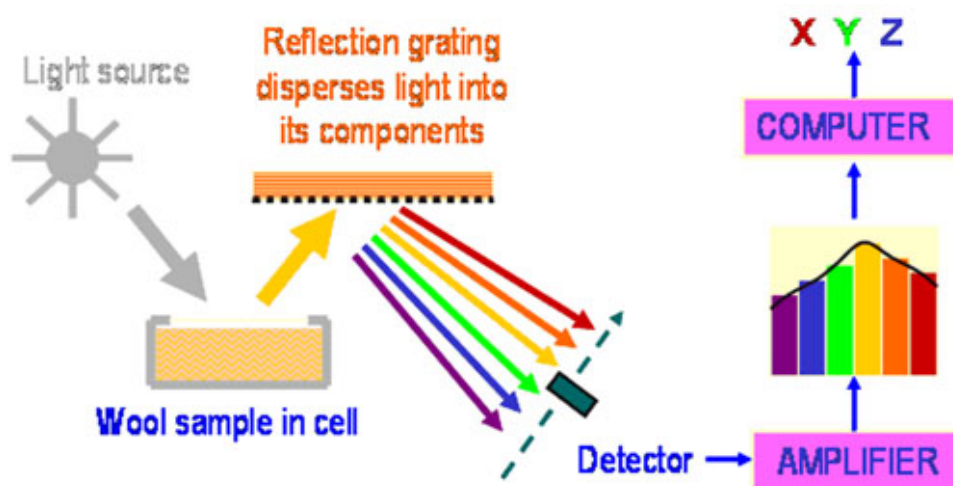
The measurements from these three zones are called the *tristimulus values* (<http://hyperphysics.phy-astr.gsu.edu/hbase/vision/colper.html>) and the instrument used to measure them is called a *tristimulus colorimeter*. Figure 6.9 is a schematic diagram of a colorimeter.

Figure 6.9 Layout of a simple colorimeter. Source: Wood, 2006.



A more sophisticated instrument, mostly used for wool colour measurement today is a *spectrophotometer* (Figure 6.10). This type of instrument does not use filters to separate the light into the X, Y and Z zones but instead produces a complete spectrum of the light reflected from the material, and determines the tristimulus values by rapid computer calculations.

Figure 6.10 Spectrophotometer for wool colour measurement. Source: Wood, 2006.



Colour measurement of wool

The prepared wool samples are compressed in a special cell which has glass windows at both ends and placed over the instrument port. A standard mass of wool is inserted in the cell at a fixed density, and one face of the cell is exposed to the light source of the colorimeter (or spectrophotometer) for a reading to be taken. Then the sample cell is reversed for a further reading to be taken using the other face. The process is repeated at least once with the wool rearranged in the cell, and all readings are averaged to give the certified results for X, Y and Z.

Tristimulus readings range roughly from 0 – 100, depending on the colour, texture and degree of gloss of the material. A black object has readings of X = 0, Y = 0, and Z = 0. In contrast, a pure white object viewed with the Illuminant C light has readings of X = 98.07, Y = 100.00, and Z = 118.22.

In 2001 the IWTO decided to change the illuminant used for wool colour measurement, from Illuminant C to Illuminant D65. This change has the effect of shifting X, Y and Z results.

Typical examples of tristimulus readings on different coloured objects are approximately:

	X	Y	Z
Yellow	59	61	40
Blue	36	40	61
Pink	54	47	42
Green	38	44	45

The interpretation of colour measurement results is more difficult than other wool measurements such as fibre diameter, which is a more 'absolute' result. This is mostly because colour measuring instruments are available which use different sources and with different illumination 'geometries' (i.e. the spatial relationship between the illumination source, sample surface and detector). Different calibration procedures (http://www.awta.com.au/Publications/Research_Papers/Colour/IWTO97_12_Report08.pdf) are also available. These will produce different results when measuring the same wool, leading to possible confusion.

However, it is possible to convert from one system to another, i.e. from Illuminant C to Illuminant D65 and vice versa. In the following examples and discussion, results derived from the Illuminant C system have been used.

A wool of good colour, might have tristimulus values of, say, $X = 62.5$, $Y = 64.0$, and $Z = 63.0$ and thus has a Y-Z value of 1.0. The wool would appear white and bright, as indicated by the relatively high Y value. Good colour (white) fine wools have Y - Z values typically around 0 – 1.0.

A poorer colour wool with tristimulus values of, say, $X = 57.5$, $Y = 58.5$, and $Z = 50.0$ is still reasonably bright (Y close to 60), but with a Y - Z value of 8.5 it will appear very yellow.

If all the X, Y, and Z values were low at around 50, the wool would appear dingy, grey, or dull.

The main purpose of colour measurement is to assist the dyer, when wool is being processed. Dyeing is an additive process, and so it is impossible to dye the wool lighter than its original colour. If a very light pastel shade is needed, the wool must be very white and bright. On the other hand, if only dark colours are needed, a stained and dingy wool could be successfully used.

All dyestuffs are produced with designated tristimulus values. To dye a batch of wool a certain colour, the wool must have tristimulus values numerically higher than those of the dye to be used. For example, a yellow dye may have tristimulus values of $X = 60$, $Y = 62$, and $Z = 40$. To dye a batch of wool to a yellow shade, the wool must have higher numerical tristimulus values than the dye.

A test certificate shows all three tristimulus values so that the client has the maximum information available regarding the dyeing potential of the wool. In practice, the X and Y values are very similar in wool measurement, and usually the X value is ignored. The two important aspects of wool colour are its lightness (or brightness) and yellowness. In this context, the Y result indicates the level of brightness, and the Y - Z result is an indicator of yellowness.

If the numerical value of Y is:

1. High, the wool will be bright, or
2. Low, the wool will appear dingy.

If the numerical value of Y - Z is:

1. Small, the wool will be white, or
2. High, the wool will be yellow.

Some examples of Y – Z values are:

Y – Z	=	-1 to 2	wool appearance is	white
		2 to 4	"	creamy
		4 to 6	"	showing yellow
		6 or >	"	yellow

The most commonly used colour parameter for assessing the quality of scouring, with respect to colour, is Delta Y (ΔY). This is defined as:

$$\Delta Y = Y_{\text{base}} - Y_{\text{as-is}}$$

Where: Y_{base} is the Y tristimulus value for the base colour and $Y_{\text{as-is}}$ is the Y tristimulus value for the as-is colour. The smaller the value of ΔY , the better the quality of scouring.

See the AWTA web page on colour measurement (<http://www.awta.com.au/Publications/Marketing/Colour/Colour.htm>).

See this web page (http://www.woolnews.net/news_item.php?ID=108) for information on recent changes in wool colour measurement.

6.7 Length after carding

The mean fibre length of a batch of wool changes during carding due to (a) fibre breakage (which causes a reduction in length) and (b) fibre losses on the card (which slightly increase the length of the wool remaining). The relationship between fibre length before and after carding is affected by the characteristics of the wool (especially the original fibre length and strength) and also by processing treatments prior to carding. The fibre length of wool after carding is a significant factor in subsequent processing efficiency and quality, and therefore has both commercial and technical significance.

The Length after Carding (LAC) test was developed to provide a result which indicates how a consignment of scoured wool will perform under commercial processing conditions (i.e. in yarn manufacture). The steps, which are conducted under standard, controlled conditions, are as follows:

1. Full length scoured wool samples are taken from each bale in the test lot using a spear head sampler (Figure 6.2). The spear removes clumps of wool from the dense-baling machine before each bale is compressed and these clumps are amalgamated to form the test sample
2. After conditioning and the addition of a standard quantity of processing lubricant, the fibre is carded on a standard sample card (Figure 6.11).

Figure 6.11 Carding step in LAC test showing the wool entering the machine.
Source: Wood, 2006.



3. The resulting batt of carded fibres is gilled three times to straighten and align the fibres into a sliver (Figure 6.12).

Figure 6.12 Gilling step in LAC test. Source: Wood, 2006.



4. The fibre length is then measured using an Almeter (http://www.awta.com.au/Publications/Newsletter/2003_December/for_technophiles.htm), which electronically scans the prepared specimen to produce a fibre length distribution. The key result is the Barbe value, which is the mean fibre length of the specimen, in mm.

Figure 6.13 shows the preparation unit of the Almeter system. This device extracts fibres from the gilled sliver and forms a 'beard' of aligned fibres. The beard, placed in position on the carriage of the Almeter measurement unit ready for scanning, is shown in Figure 6.14.

Figure 6.13 Almeter preparation unit. Source: Wood, 2006.



Figure 6.14 Almeter measuring unit. Source: Wood, 2006.



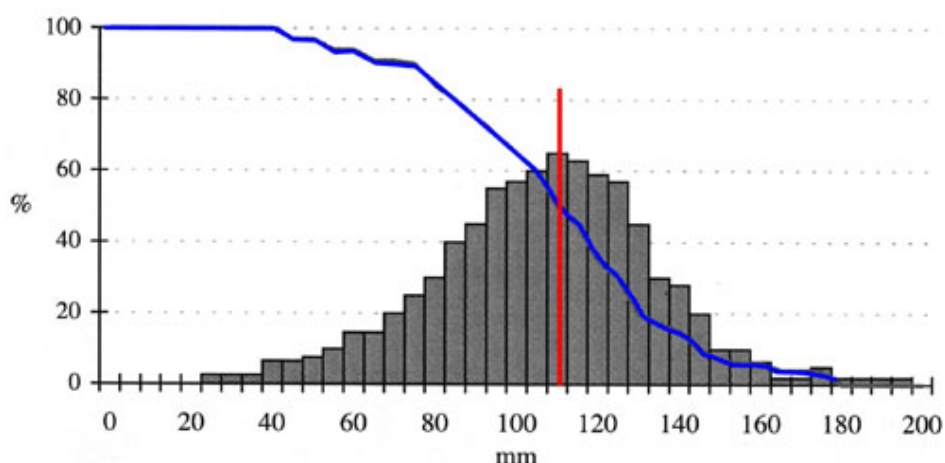
Two types of fibre length distribution are produced by the Almeter, termed *Hauteur* and *Barbe*. These are explained in the reading “*Notes on Length After Carding Parameters*” (Wood-Length After Carding.pdf). In brief, the Hauteur distribution shows the number of fibres recorded in each length interval, while the Barbe distribution shows the weight of fibres in each length interval. Hauteur (or, more correctly, mean Hauteur) is the preferred parameter for measuring the fibre length of tops, while Barbe is the key parameter reported from the LAC test.

Depending on the soundness of the wool, mean Barbe is 1.2 – 1.6 times larger than mean Hauteur.

A typical Barbe fibre length distribution produced by the Almeter is shown in Figure 6.15. The curve is the cumulative distribution which indicates the weight of fibres (as a percentage of the total) that are longer than a certain length. The bell-shaped bar graph is the Barbe distribution which indicates the weight of fibres in each length interval.

Note that the 50% value for the cumulative distribution corresponds to the mean Barbe value, which in this example is around 110 mm.

Figure 6.15 Typical Barbe distributions. Source: Wood, 2006.



The LAC test is primarily used for large consignments of scoured wool, the form in which most New Zealand wool is exported. The test has steadily grown in importance for the international marketing the New Zealand wool clip because it simulates commercial processing conditions and hence it provides a spinner with a reliable indicator of how his wool purchase is likely to perform on his machines. More recently, the LAC test has been introduced into Australia by AWTA for the commercial testing of the relatively smaller proportion of coarser wools in the local clip.

Card waste measurement

The LAC test can also be used as a test for the propensity for a wool to produce waste in carding. The simple procedure involves collecting all droppings from the card once the processing of a sample has been completed, weighing these, and expressing the weight as a percentage of the weight of the sample.

Measurement of entanglement

LAC is essentially a test for fibre breakage in carding, under standard carding conditions. As well as original fibre length and strength being factor in the level of breakage occurring, fibre entanglement as a result of the scouring process also contributes. There is no standard method for measuring the degree entanglement of scoured wool, although CSIRO have devised a research method. This test, for 'specific opening energy' (SOE), involves opening a sample of scoured on a specially adapted carding machine (Robinson 1986). The energy per kilogram required to fully card the wool (less the energy required to drive the unloaded machine), is the SOE. The higher the SOE the higher the degree of entanglement.

Downloadable documents on LAC are available from the AWTA website (http://www.awta.com.au/Publications/Fact_Sheets/Download/Acrobat/Fact_sheet_009.pdf) web site.

Regain

Regain is the percentage of moisture by weight that dry wool fibres must absorb to bring the fibres to correct condition for processing. Almost all wool scoured in Australia and New Zealand, especially for export, is tested for *regain*. This is to ensure that supplier and customer can agree on the quantity of useful fibre in a consignment and the customer expects to receive a certificated result for the regain of a scoured wool delivery.

The IWTO standard regain for scoured wool is 17%. Export wool is often scoured to a higher regain (i.e. 18 to 19%) to allow for moisture loss in transit through hot climates.

Do not confuse percentage regain with percentage of moisture content:

$$\% \text{ regain} = \frac{\text{weight of moisture present}}{\text{weight of dry wool}} \times 100$$

$$\% \text{ moisture content} = \frac{\text{weight of moisture present}}{\text{weight of dry wool} + \text{moisture}} \times 100$$

On-line systems for continuous moisture measurement of scoured wool are also available, such as the Drycom system (<http://www.streatahead.com/Pages/Drycom%20Menu.html>) from Streat Instruments. These control systems enable the required regain to be maintained without human intervention. Figure 6.16 shows the sensors at the exit of the wool dryer. These measure the electrical conductivity of the wool mat, which is related to its moisture content. This control system is described more fully in the topics on Wool Drying (Topic 9), and Control and Instrumentation (Topic 7).

Figure 6.16 Drycom sensors for detecting the moisture content of dried scoured wool.
Source: Streat Instruments.



Other tests on scoured wool

pH

Control of the pH of scoured wool is necessary for certain processes, such as carpet printing. For one hour, 2 g of scoured wool are shaken with 100 mL of distilled water. The pH of the extracted liquid and the water is measured by pH meter.

Insect resistance

Testing for this is done by either chemical analysis or by exposing the wool to a colony of beetle and moth larvae (bioassay).

Peroxide-bleached wool detection

The wool is boiled in a solution of lead acetate and glacial acetic acid. Bleached wool remains unstained, while unbleached wool stains a dark brown.

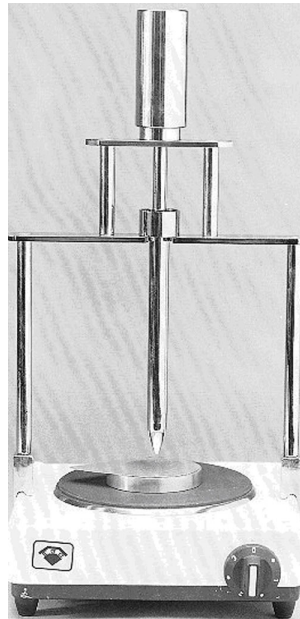
6.8 Testing in the woollscour

The following tests are those that may be regularly carried out in the woollscour in order to monitor the quality of the scoured wool product.

Alcohol extractables or rapid grease extraction

Figure 6.17 shows the rapid grease extraction apparatus.

Figure 6.17 Rapid grease extraction apparatus. Source: Wood, 2006.



The rapid grease extraction test is carried out as follows:

1. A 2g sample of dried scoured wool is placed in the metal tube and compressed by a weight
2. A solvent (dichloromethane) is slowly added to the wool to dissolve the grease – the solution drips into a weighed dish positioned on the hot plate below
3. The solvent evaporates on the heated tray, leaving the extracted grease behind as a residue
4. To obtain the weight the wool grease, reweigh the dish with the residue. Express the weight of grease as a percentage of either the conditioned weight, or the dry weight of the original sample.

Although the rapid grease test is quick, simple and uses inexpensive apparatus, it has the disadvantage that with such a small sample size, results of poor accuracy are generally obtained. The Soxhlet extraction method, which is carried out properly drawn core samples, gives more accurate results.

Regain

Because of the high output of modern scouring operations, it is necessary to often check the dried wool for regain, in order to be able to adjust the drying conditions if the wool is being over dried or under dried.

At the end of the dryer, the regain can be monitored by electronically sensing the conductivity of the wool by an automatic moisture control system. A more accurate off-line test for regain can be carried out using the sample dryer as shown in Figure 6.18.

A rapid regain test is conducted as follows:

1. Make up a hand-drawn sample of about 0.5 kg of wool from different parts of the dry scoured wool consignment
2. Place the wool in the can
3. Weigh the can of wool. If the weight of the can is already known, the weight of the scoured wool can be calculated
4. Place the can on the wool dryer for a set time at a thermostatically controlled heat
5. Reweigh the can of wool after drying, and calculate the dry weight of wool
6. Use the formula for Regain% to calculate the regain of the scoured wool.

Both the above tests are often called the *rapid tests*. The equipment required is robust and is not particularly expensive and only limited training is required. More rapid and accurate results, however, are provided by more recently introduced testing systems based on the NIRA principle.

Figure 6.18 Rapid regain unit (shown with five 100g sample cans, but a single 500g can is also available). Source: Wood, 2006.



Near infrared reflectance analysis (NIRA)

Most of the major New Zealand wool scourers operate *Near Infrared Reflectance Analysis* (NIRA) instruments. These enable measurements of regain, residual grease content, and scoured wool (or 'as-is') colour to be rapidly carried out on each bale during production. NIRA is also capable of determining mean fibre diameter, bulk and medullation content in wool samples, but these additional capabilities are not routinely used in scourers.

How does NIRA work? Water and grease molecules readily absorb radiation in the near infrared region of the electromagnetic spectrum and consequently they vibrate in response to the absorbed energy. Both of these materials have a characteristic pattern of absorption and reflection of the radiation that is readily measured by an instrument called a *spectrometer*. This instrument can be 'trained' (i.e. calibrated) to convert this complex spectral information into an estimate of the percentage of water or grease contained by a scoured wool sample. A personal computer interfaced to the spectrometer has the sophisticated software installed for carrying out the calculations.

Calibration of the NIRA spectrometer is an exacting task which involves measuring many samples of known moisture and grease levels, across the widest possible range of wool types. If an inadequate calibration set of wools is used (e.g. only crossbred wools used for calibration when finer wools may need to be measured), the estimates provided by the NIRA spectrometer are likely to be unreliable. That is, there will be poor agreement between the NIRA estimates of moisture and grease content and those provided by the accepted standard method used by test houses (e.g. residual grease measurement by DCM or ethanol extraction using the Soxhlet apparatus).

'First generation' NIRA systems which have been operating in some New Zealand wool scourers since the mid 1980s, use the InfraAlyzer instrument. A series of absorption filters, inserted in turn into the beam of light reflected from the wool sample, transmit specific bands of radiation in

the near infrared region of the spectrum and the transmitted radiation is measured by lead sulphide detectors. The set of energy results provided by the filters is used in computing the moisture and residual grease content of the wool sample. The InfraAlyzer requires the sample of scoured wool to be minicored to get a representative subsample, which is placed in a wool cell for insertion in the instrument.

More sophisticated instruments have since been introduced to New Zealand scours, culminating in the WRONZ NIRASPEC system in 2000. This instrument, which has been specifically designed for modern wool scouring operations, can accept a much larger sample than earlier NIRA spectrometers without the need for subsampling by minicoring. In the latest model the sample of scoured wool is transferred from the coring machine to the NIRASPEC instrument for measurement without human intervention (Figure 6.19).

Figure 6.19 NIRASPEC S system for in-scour testing. Source: Wood, 2006.



The latest NIRA spectrometers do not use absorption filters but instead use the principle of light diffraction by a grating. As a result, their sensitivity can extend into the visible region of the spectrum and they can also be calibrated to measure wool colour. Thus NIRA technology provides scours with a complete quality assurance system.

Wool scours generally test core samples from every bale with NIRA in order to:

- make timely and appropriate changes to the scouring conditions when moisture or grease levels deviate off-target, and
- provide the client with comprehensive data on the scouring history of their consignment, with bale-by-bale results for moisture content, residual grease content and as-is colour.

For certification purposes, the scour is still required to forward a core sample to a test house for testing. But these results may arrive too late for intervention – the wool may be already in a container on a ship bound for its destination.

NIRA instruments are relatively expensive and, as with most scientific equipment, must be protected from the demanding conditions that prevail in a wool scour. However, they are sufficiently user-friendly that staff with no scientific background can operate them successfully.

The principles and practice of NIRA testing in a wool scour are covered in more detail in the reading “*A Beginner’s Guide to NIRA*” (Wood 2004.pdf).

Testing scouring liquor and effluent

Analytical methods, some of which we have already mentioned, are beyond the scope of this topic, but Stewart (1988) provides a useful outline. The tests are used to determine the contents of scouring liquor and effluent.

The results of the tests influence (a) the efficiency of water usage, and (b) the control of effluent to the sewer.

The tests that may be carried out on various components of the liquor and effluent include:

1. Woolgrease — top (that part that is obtained by conventional centrifuging)
2. Total woolgrease
3. Suint
4. Total solids
5. Dirt and residuals
6. Insect resist treatment (mothproofing agent)
7. Detergent
8. BOD₅ (biochemical oxygen demand)
9. COD (chemical oxygen demand), and
10. SS (suspended solids).

Chemical analysis of woolgrease recovered from the scouring operation is carried out to determine its commercial usefulness. The colour of woolgrease is important when determining its commercial usefulness. Pale greases are suitable for a wider range of uses than dark greases. Pale greases normally contain fewer impurities than dark greases.

Readings



The following readings are available on CD:

1. Stewart, R G. 1988, Analysis and Testing, in: *Woolscouring and Allied Technology*, Caxton Press, Christchurch, New Zealand, ISBN 0-908699-23-9, Christchurch, New Zealand.
2. Wood, E.J. 2004, A Beginner's Guide to NIRA, Canesis Network Ltd, Christchurch, New Zealand.
3. Wood, E.J. Length after Carding Parameters, Canesis Network Ltd, Christchurch, New Zealand.
4. Wood, E.J. The Basics of Wool Colour Measurement, Canesis Network Ltd, Christchurch, New Zealand.

Activities

Multi-Choice Questions

Useful Web Links

Assignment Questions



Available on WebCT



Submit answers via WebCT



Available on WebCT



Choose ONE question from ONE of the topics as your assignment. Short answer questions appear on WebCT. Submit your answer via WebCT

Movies

The following movies are available on CD:

- Canesis Network Ltd., Almeter 04.
 - Canesis Network Ltd., Baling and Sampling
-

Summary

Wool scouring adds value to wool by removing the natural and acquired contaminants. It is essential that the scouring is thorough and consistent and that a clean, bright product of the required moisture content is delivered to the client. To achieve good scouring it is necessary to regularly check wool quality by testing samples regularly in the scour. To provide firm evidence to the client that the required specifications for quality have been achieved, certificated results are produced by a test house from the testing of a sample of scoured wool sent from the scour.

This topic looks at the full range of certificated tests that are routinely carried out on scoured wool consignments in Australia and New Zealand – yield, vegetable matter content and solvent extractables, fibre diameter, colour, regain and length after carding. In addition, the key in-scour tests for quality are included – the rapid tests for grease and regain, and the increasing use of NIRA technology.

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- Streat Instruments, Moisture Measurement and Control, product brochure, New Zealand.
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Glossary of terms

See also the Glossary provided by AWTA (see References for web address).

Alcohol extractables	Material that can be removed from wool by ethanol
Almeter	The instrument system for determining the fibre length characteristics of slivers and tops; it comprises two units (1) fibre sampling device to form a specimen, and (2) a measuring unit
As-is colour	The measured colour of wool, without any cleaning of the sample
Ash	The residue remaining when wool is burnt in a furnace, as a step in the yield test; alternatively called 'mineral' content indicating the main components of the residue

ASTM	American Society for Testing and Materials – the principal standardisation body in the USA
AWTA	Australian Wool Testing Authority – the major wool test house and supplier of related services in Australia
Barbe	The mean fibre length of a set of wool fibres where the mass of fibres in each length interval has been used to form a distribution. The Almeter is used to measure tops and slivers for this parameter
Base colour	The measured colour of a thoroughly cleaned sample of wool (produced by a standard test house procedure)
Brightness	A measure of the intensity of reflectance of a wool, in the green (Y) spectral region
Coefficient of variation	A statistical measure of the variability exhibited within a set of values. It expresses the standard deviation as a percentage of the mean value
Colorimeter	An instrument used to determine the colour of wool by measure its tristimulus values
Commercial yield	Any of several yield results quoted in wool trading, calculated from wool base and vegetable matter results
Consignment	A scoured wool delivery (or scourment)
Coring	Removal of a sample by driving a tube of circular cross-section, and with a sharpened replaceable tip, into a bale of wool
DCM	Dichloromethane (or methylene chloride), the solvent often used for extracting residual grease from scoured wool
Fibre diameter	The thickness of individual fibres; usual to quote an average value, the <u>mean</u> fibre diameter
Gilling	A mechanical process on a wool sliver that straightens and aligns the fibres and also attenuates the sliver (i.e. makes it thinner)
Hauteur	The average length of a group of wool fibres, sorted by number into length categories. The Almeter is used to measure this parameter for slivers and tops
IWTO (International Wool Textile Organisation)	The international body overseeing procedures and test methods pertaining to the international trading of wool
Laserscan	An instrument that detects shadows of wool fibre snippets passing through a laser beam, thereby determining their fibre diameters and distribution of fibre diameters
Length After Carding (LAC)	A fibre length test for scoured wool where a consignment is sampled, and the sample is then carded, gilled and measured on the Almeter
Micron (or micrometer)	The unit for measuring fibre diameter; one micron (μm) equals one millionth of a metre
NIRA	Near Infrared Reflectance Analysis – measuring the amount of radiant energy reflected off a material, in the infrared region of the spectrum; from this spectral information, data on the composition and contamination of the material can be determined
Objective measurement	A system where the properties of wool are determined by instruments rather than by subjective appraisal
OFDA (Optical Fibre Diameter Analyser)	An instrument for measuring fibre mean and distribution using an automated microscope and image analysis techniques

Regain	The mass of moisture in a mass of wool, determined under prescribed conditions and expressed as a percentage of the clean, over-dry mass of fibre
Sample	The wool drawn by appropriate methods from a lot or consignment; in order for an IWTO test certificate to be issued the sampling must be carried out in accordance with the IWTO sampling regulations
Shirley Analyser	A laboratory carding machine used mostly for removing vegetable matter from wool and blending the fibres for subsequent measurement
Snippet	A very short length of fibre, around 2 mm long; usually cut for fibre diameter measurement
Solvent extractables	Greasy contaminants on wool that are readily dissolved by organic solvents such as alcohol or dichloromethane
Specific Opening Energy (SOE)	A method of measuring the entanglement of scoured wool using the energy required to card a given mass of wool
Spectrophotometer	A instrument used to measure the reflectivity of a surface within the visible spectrum of light; can be used to measure wool colour as an alternative to a colorimeter
Test house	A laboratory which carries out tests in accordance with IWTO test methods and regulations; it must be licensed in order to issue IWTO test certificates
Tristimulus value	One of the three reflectance readings obtained when using a colorimeter, specified by the CIE as X (red), Y (green) and Z (blue)
Vegetable matter	Burrs (including hard heads), twigs, seeds, leaves and grasses present in wool
Wool base (%)	The oven-dry mass of wool fibre free from all impurities, expressed as a percentage of the mass of the original sample
Yellowness	The difference between the light reflectances of wool in the green and blue regions of the spectrum, expressed as the difference between the tristimulus values Y – Z
Yield	The amount of clean fibre, at a standard regain, that is expected to be produced when a delivery is processed. It may be expressed as a clean mass (in kg) or as a percentage of the mass of raw wool prior to processing