INTERNATIONAL WOOL TEXTILE ORGANISATION

TECHNOLOGY & STANDARDS COMMITTEE — **CHRISTCHURCH MEETING**

Commercial Technology Forum — **April/May 2000**

Chairman: A.G.DE BOOS (Italy)
Technical Coordinator: G. MERCIER(France)

Report No: CTF 06

The Influence of Preparation Techniques on the Measurement of Fibre Curvature.

By

V.E. Fish, T.J. Mahar* and B.J. Crook

Department of Animal Science — The University of New England, Armidale, NSW, 2351, Australia.
*Australian Wool Testing Authority Ltd — PO Box 190, Guildford, NSW, 2161, Australia.

SUMMARY

Earlier published reports have indicated that the procedures for preparing samples have an effect on the measured Fibre Curvature (FC) by LASERSCAN and OFDA. Four areas of preparation have been examined. The results, which show a number of small but significant effects, can be summarised as follows:

- an increase in the mass of the subsample to be minicored from 5g to 10g increased the FC (by 2 deg/mm);

- repeatedly minicoring the subsample increased the FC (by 5 deg/mm after 15 repeats);

- the normal wear of the minicore tubes from sharp to blunt decreased the FC (by 2 deg/mm); and

- prolonged conditioning beyond the normal procedures for Mean Fibre Diameter measurement had no effect on FC.

Introduction

The current method for measuring Fibre Curvature (FC) is based on the sampling, preparation and measurement procedures that were developed for the measurement of Mean Fibre Diameter (MFD) using either LASERSCAN (IWTO-12-95) or OFDA (IWTO-47-98). A review by the current authors (Fish *et al.*, 1999) highlighted the potential impact of the preparation procedures/techniques outlined in IWTO-12-95 and IWTO-47-98 on FC measurements. The Standards Australia/Standards New Zealand Committee draft (Ranford,1999) acknowledged that sample preparation requires investigation and subsequent standardisation before a reliable and repeatable FC measurement can be made available to the wool industry.

This report investigates those preparation procedures that were considered to have the greatest effects on FC measurement (Fish *et al.*, 1999), viz. conditioning time; number of minicorings; mass of pre-coring sample; and sharp (new) and blunt (used) minicore tubes. Both OFDA and LASERSCAN measurements of FC were performed.

Methods and Materials

Independent trials were conducted to assess the effect on measured Fibre Curvature of:

- sample conditioning;
- repeat minicoring of the conditioned sample;
- the sharpness of the minicore tubes; and,
- the mass of subsample to be minicored.

Table 1 summarises the wool form and characteristics used for each trial.

Table 1 : List of Wools Used through Described Trials. For more details, see Appendix.

Trial	Form of Wool	MFD Range (µm)	Curvature Range (deg/mm)
Conditioning Time	Scoured, cored greasy lots	19 to 31	80 to 116
Repeated Minicoring, Definition of Blunt Minicore Tubes	Scoured, cored greasy lots	15 to 42	58 to 125
Sharp and Blunt Minicore Tubes	• 7 tops • 7 scoured tops • 6 scoured, Shirley Analysed top • 10 scoured sale lot cores • 10 scoured, Shirley Analysed sale lot cores	16 to 37	69 to 120
Subsample Mass	10 Sale lot cores	18 to 32	67 to 123

The specific procedures used for each of the four trials are detailed below.

A. Conditioning Time

Six tops of varying diameters and curvatures, as described in Table 1, were selected. Two x 60g samples were removed from each top and cut into 20mm sections. One 60g sample was scoured and dried. The other 60g sample was scoured, dried and Shirley Analysed. Each sample was then broken into six subsamples of 10g and conditioned as per IWTO-52 for different times between 4 hours and 7 days, that is 12 samples were tested at each of the 6 times. Subsamples (10g) were tested after 4, 6, 24, 48 hours, and 6 and 7 days of conditioning. All subsamples were measured using both OFDA and LASERSCAN.

B. Sharpness of Minicore Tubes

(a). Sharp and Blunt Minicore Tube Tips

A set of 40 wools from different origins was assembled for this trial. As described in Table 1, these wools consisted of 7 tops, 7 scoured tops, 6 scoured Shirley Analysed tops, 10 scoured sale lot cores and 10 scoured, Shirley Analysed sale lot cores. Wools were conditioned according to IWTO-52, minicored using the “blunt” tubes, and tested in a LASERSCAN using 4x1000 snippets for each sample, on replicates of 10g. Each 10g replicate was minicored 4 times to provide the 1000 snippets. “Sharp” minicore tubes were then fitted to the corer and the testing was repeated on a replicate set of 10g subsamples.

In this trial, new (“sharp”) tubes were those that had never been used in a minicorer before. Used (“blunt”) tubes were those already in place on the corer and had been in regular use for at least the 3 months prior to this investigation. A visual difference could be seen between sharp and blunt tubes, the blunt being more rounded at the cutting edge. There were no further assessments on the “sharpness” of the tubes at this time.

(b). Definition of Blunt Minicore Tube Tips

Two 10g wool samples were taken from each of 23 wools ranging in diameter from 16µm to 40µm. The first set of 23 samples was individually weighed before and after minicoring with "blunt" tubes. The resulting minicored snippets from each sample were also weighed. "Sharp" tubes were then fitted to the minicorer, and the process was repeated for the remaining set of 23 wools.

C. Repeated Minicoring

Samples of approximately 10g were taken from 23 wools ranging in diameter from 16 to 40 µm, as shown in Table 1. Samples were conditioned according to IWTO-52-96. Each sample was minicored, measured using a LASERSCAN, then re-minicored until a total of 15 minicorings and measurements had been taken for each sample. Samples were ejected and repacked between each minicoring.

D. Subsample Mass

A selection of 10 wools of various diameters, as shown in Table 1, was scoured. Four sub-samples of 10g and four sub-samples of 5g were randomly selected from each sample. All samples were allowed to condition overnight. Each sample was minicored once and measured on LASERSCAN for 2000 accepted counts.

Results and Discussion

A. Conditioning Time

The results in Figure 1 are the average of conditioning on six (6) tops, and illustrate that there is no obvious trend in the variation of Fibre Curvature, as measured by LASERSCAN, with the period of conditioning. Similar results were found for OFDA measurement.

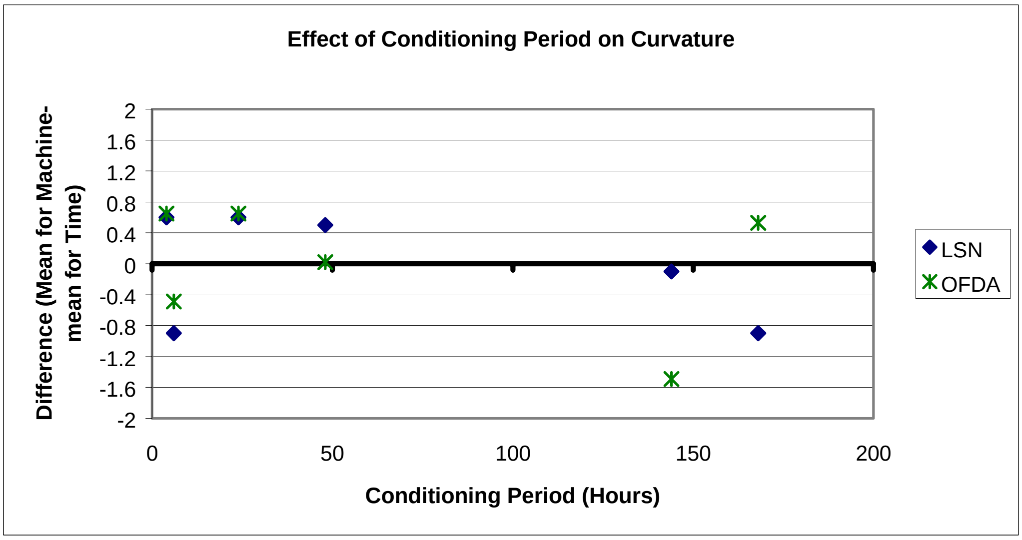

Figure 1: The effect of conditioning period on six tops over a period of one week. The difference is between the FC and the average FC at the nominated time.

An analysis of variance was conducted on the data sets for OFDA and LASERSCAN, and the P-values were found to be 0.998 for LASERSCAN and 0.999 for OFDA. Such high P values indicate that there is no effect on FC of conditioning time, and any difference that is seen is due to random chance.

Provided the sample is adequately conditioned, the FC of a sample was independent of conditioning time for conditioning periods from 4 hours to one week. It should be noted that the differences in question are very small (less than 2 deg/mm), and that the Confidence Intervals for OFDA were approximately ±9.1, while the Confidence Intervals for LASERSCAN were approximately ±6.5.

B. Minicore Tube Tips

(a). Sharp and Blunt Minicore Tube Tips

The hypothesis for this trial was that a "blunt" tube would exert more pressure on the sample than a sharper tube, therefore bending the snippets further, resulting in a higher measured FC. It is certainly the case that, when hand coring farm bales, a blunt tip on the core tube requires more coring force, thus exerting more pressure on the wool at the core tip, than this same tip after it has been sharpened. Minicore tubes have their cutting edges polished ("sharpened") on a regular basis depending on usage.

Figure 2 shows the difference in FC between the used "blunt" and the new "sharp" minicore tubes. The "sharp" minicore tubes result in a higher FC than the "blunt" minicoring tubes, (ie. negative values of the curvature difference between "blunt" and "sharp" tubes). Analysis using the procedures outlined in IWTO-0 indicated an average difference of 1.5 deg/mm (significant at p= 0.0037). The two sets of data were highly correlated, as would be expected.

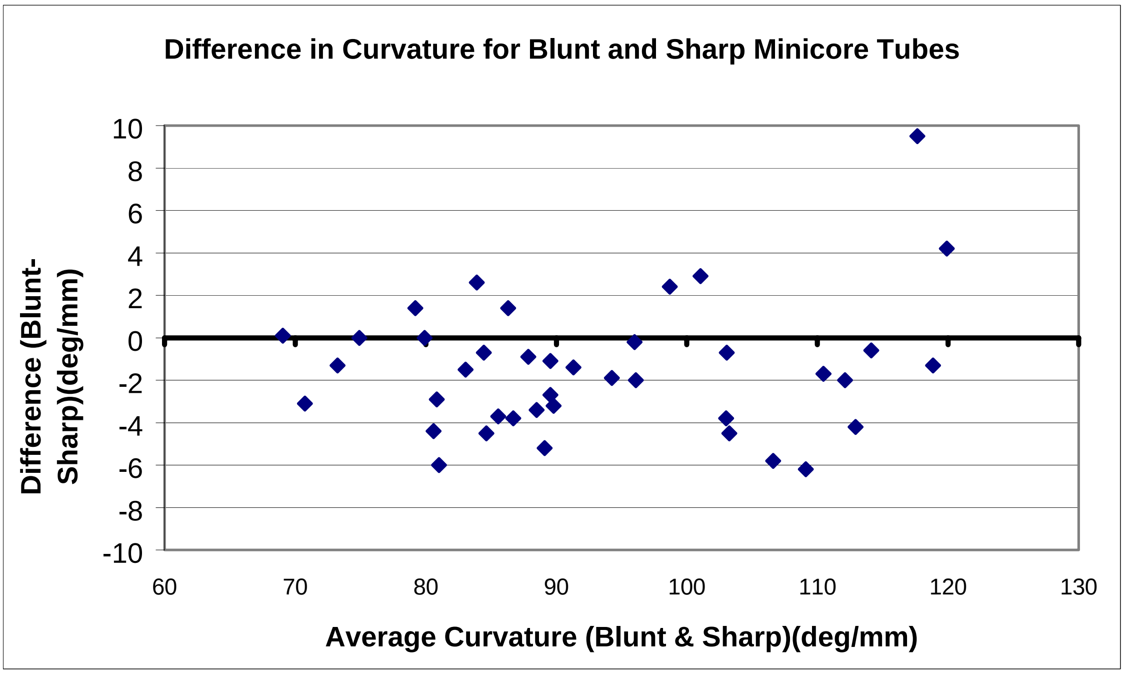

Figure 2: The effect of Minicore Tube sharpness on Fibre Curvature measurement.

This trial established that sharp minicore tubes gave higher FC than blunt minicore tubes. This result was contrary to the original hypothesis. At this time we can offer no explanation as to why "sharp" minicore tubes result in higher FC than "blunt" tubes.

In this trial, the tubes were designated "blunt" or "sharp" in relation to each other; there was no pre-determined description of the terms "blunt" or "sharp" prior to this investigation. Presumably, tubes become progressively "blunt" through minicoring of successive samples. It is a problem to establish the point at which the tubes were no longer "sharp" and were classed as "blunt". The trial described in the next section is an attempt to define the "sharpness" of the coring tube with a simple laboratory check.

(b). Defining Sharp and Blunt Minicore Tubes

It was assumed that the sharper coring tubes would core more material under the same operation of the instrument than the blunt tubes. However, results for this trial were in-conclusive. Statistical analysis shows there was no difference between the weights of material cored by sharp tubes and by blunt tubes. The results are illustrated in Figure 3.

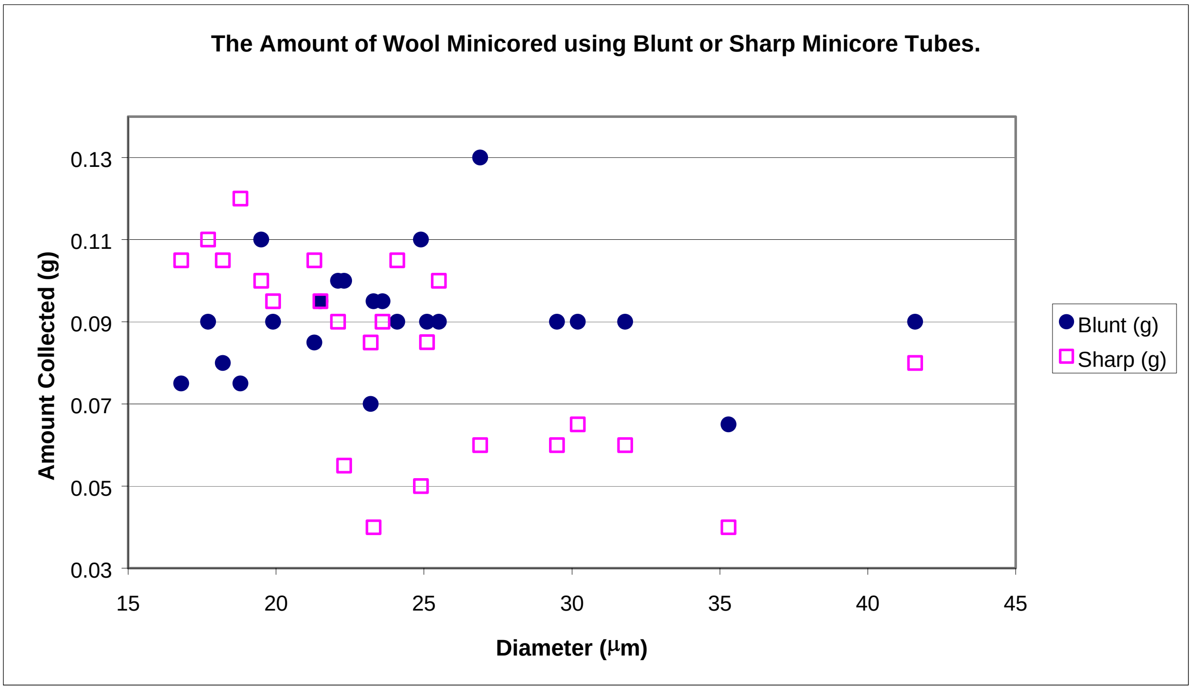

Figure 3: Weight of snippets extruded from minicore tubes designated as either "blunt" or "sharp".

The technique of weighing minicored material shows little promise as a means of assessing the degree of sharpness of a set of minicore tubes for a number of reasons.

Firstly, minicoring tubes are assumed to wear evenly, but observation shows that this is not the case. Examination of minicore tube tips under a low-powered microscope shows some of the tips are pitted and grooved, indicating some tubes appear more "blunt" than others despite having been used for the same length of time. This technique considers the coring tubes as a set. It may be more appropriate to treat each tube individually.

Secondly, there is no clear point when a set of tubes becomes "blunt". The point at which tube sharpness affects FC measurement needs to be established. Until the effect of minicore tube sharpness is more accurately determined, care needs to be taken in the interpretation of FC measurements made on woolsamples with different minicorers.

C. Repeated Minicoring

The hypothesis for this trial was that Fibre Curvature would reduce with each minicoring. Barry (1999) found that repeatedly minicoring a sample caused a small increase in the MFD of the sample. Since there is an inverse relationship between FC and MFD (Fish *et al*, 1999), one might expect a gradual decline in the FC with this increase in fibre diameter.

The process of minicoring a subsample involves placing the 10g sample into a cylindrical chamber where it is compressed by a piston, then coring tubes of 1.8mm diameter are driven through the sample. The piston is released and the tubes are withdrawn. Snippets that have been collected in each tube are then forced out of the tubes, and collected for measurement. These processes exert compressive loads on the fibre mass that would bend individual fibres.

Each point in Figure 4 represents the average of 23 samples. Initial examination found a significant difference in FC (p= 0.001) between the first and second minicorings, but there was no corresponding significant difference for MFD between the first two minicorings. Repeated minicoring of a sample increased the Fibre Curvature with each successive coring, as illustrated in Figure 4. There was a small increase in the measured Mean Fibre Diameter with successive minicorings, but not to the same extent.

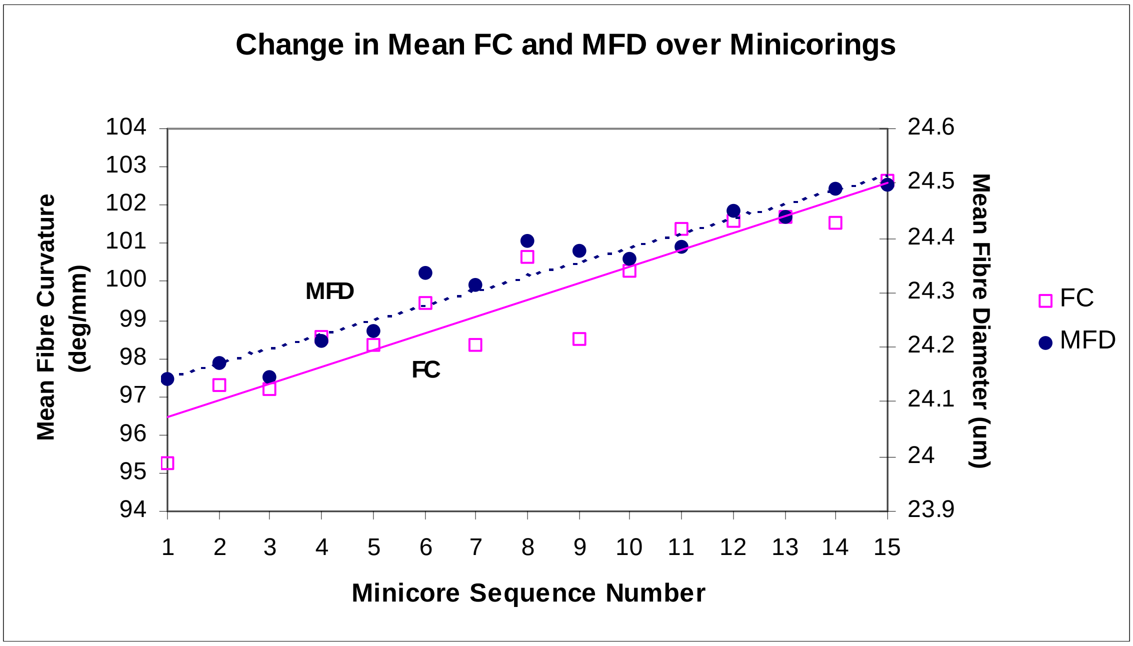

Figure 4: The average FC repeatedly minicored and measured for FC.
Samples are aqueously scoured sale lot core samples.

If the increase in FC with successive minicorings were related to preferential sampling of fibres by the minicore tubes then one would expect a corresponding decrease in Fibre Diameter. Since this is clearly not the case, the increase in FC is not directly related to an increase in MFD of the sample. Rather the increase in FC may to be related to the mechanical forces exerted on the sample by the minicoring. Repeated compressions of the sample in the minicore cylinder may result in an increase in the bending strain of each fibre. Some of these fibres may not have fully recovered to their unstrained (un-bent) state at the time of measurement.

D. Mass of Subsample

A difference in measured FC on LASERSCAN was found when 5g and 10g samples were minicored. The average difference was small (2 deg/mm) but statistically significant (p=0.015).

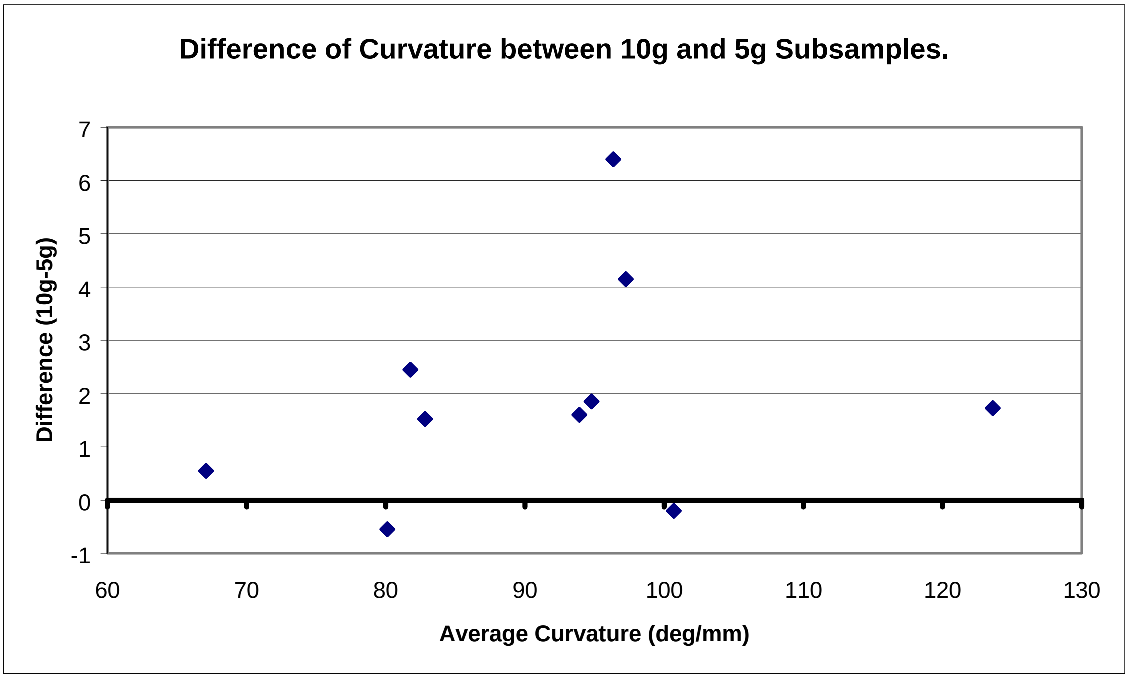

Figure 5: The effect of subsample mass on measured Fibre Curvature.

In general, the FC of a 5g subsample was lower than the FC on a corresponding 10g subsample of the same wool (Figure 5). Similar results have been generated by Crowe (2000), who also found the effect was exacerbated by repeatedly minicoring the sub-samples.

Figure 6 indicates that there was also a small diameter bias such that 10g subsamples were 0.1μm coarser, on average, than those sampled from a 5g subsample.

Changing the Committee Draft for FC from two 10g subsamples minicored twice each for each cored sale lot tested to four 5g subsamples minicored once each would benefit FC measurement, but the number of subsamples required would be doubled. This is achieved by halving the material required in the subsample from 10g to 5g, while still ensuring that all fibres have a chance to be cored.

This trial needs to be extended to include more wools to determine the consistency of the effect of subsample mass on both FC and MFD. Indications from the trial are that subsample mass effects FC, and that it may also have a small effect on MFD.

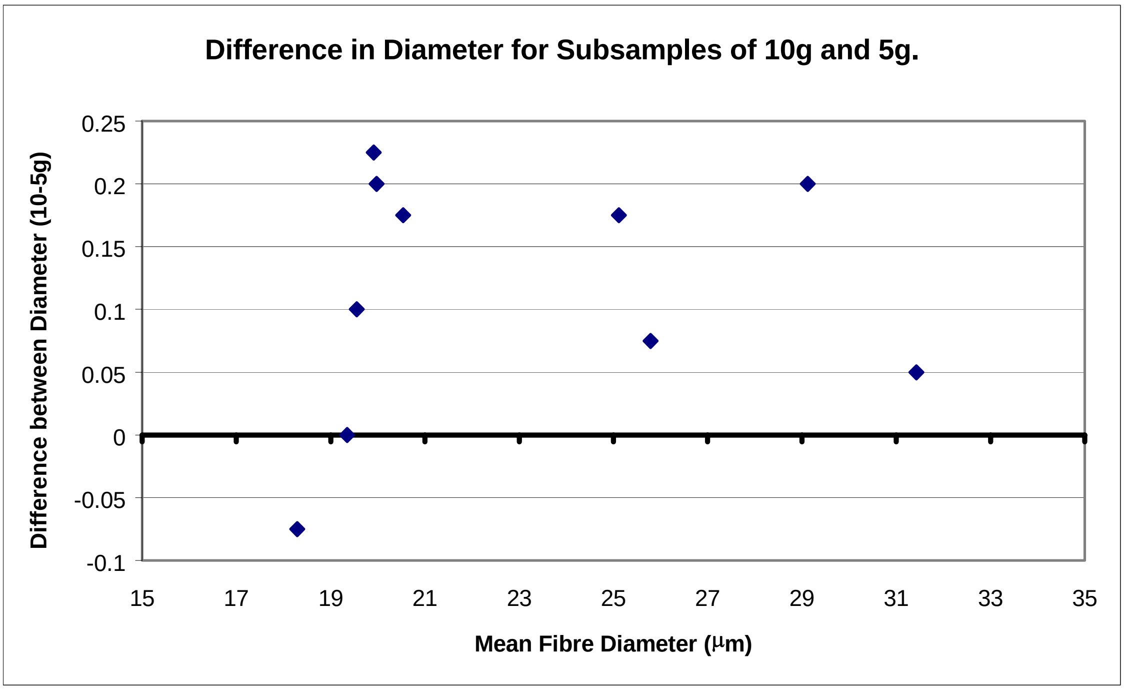

Figure 6: The effect of subsample mean on the measured mean fibre diameter.

Conclusion

The current provisions within the Committee Draft for Fibre Curvature Measurement permit small, but significant effects on FC measurement through variation in sample preparation as indicated by the trial results. Three (3) areas of preparation for measurement within the draft have been found to affect FC measurement, viz. variable subsample mass, repeated minicoring and the sharpness of minicore tube tips.

The Committee Draft for Fibre Curvature has been based on IWTO-12-95 and IWTO-47-98, which were developed for Fibre Diameter measurement using LASERSCAN and OFDA respectively. The draft for Fibre Curvature aims to reflect as closely as possible the techniques used for the measurement of MFD in IWTO-12-95 and IWTO-47-98. It was envisaged that the MFD and FC measurements will be made concurrently, but the trials reported here indicate that the current standards for MFD are not stringent enough for repeatable measurement of FC. As a result some of the preparation techniques permitted within the MFD standards are responsible for increased variation in FC results.

Several recommendations will be made to the Standards Australia/Standards New Zealand Working Group to change the draft for FC. Further recommendations may also be made to IWTO for changes to IWTO-12-95 and IWTO-47-98 where FC measurements are to be reported. These are:

- (i) change IWTO-12-95 to allow 2 x 5g subsamples to be used in all tests,
- (ii) restrict curvature measurements to aqueously scoured samples only,
- (iii) restrict curvature measurements to samples which have not been Shirley Analysed, and
- (iv) limit the number of minicores to one per subsample.

As no effect of conditioning time on the measurement of FC has been found, there is no recommendation to change the existing standards.

Acknowledgments

The funding for this project is part of a postgraduate scholarship awarded to V.E. Fish. The AWTA Ltd. Wool Education Trust is gratefully acknowledged for its financial support and the provision of this scholarship.

References

Barry, R.G. (1999) Personal Communication.

Crowe, D.W. (2000) Personal Communication

Fish, V.E., Mahar, T.J. and Crook, B.J. (1999) Fibre Curvature Morphometry and Measurement. IWTO Tech. & Stand. Committee: Nice, 1999. Report No: CTF 01.

IWTO-12-95. Measurement of the Mean and Distribution of Fibre Diameter Using the Sirolan-Laserscan Fibre Diameter Analyser.

IWTO-47-98. Measurement of the Mean and Distribution of Fibre Diameter of Wool Using an Optical Fibre Diameter Analyser (OFDA).

IWTO-52-96. Conditioning Procedures for Testing Textiles.

Ranford, S.L. (1999) Standards Australia/Standards New Zealand Working Committee Draft Test Method: Measurement of Fibre Curvature (Third Submission). IWTO Raw Wool Group Meeting: Florence, 1999. Appendix 01.

Appendix

Details of Wools Used in Trials

Trial A

No.	Trial	Form of Wool	Shirley?	MFD	FC
1	Trial A : Conditioning Time	Cored Sale Lot	Yes & No	30.6	79.4
2				24.8	87.8
3				24.9	88.6
4				18.9	110.4
5				24.6	87.3
6				30.6	79.2

Trial B (b) & Trial C

No.	Trial	Form of Wool	Shirley?	MFD	FC
7	Trial B (b) : Definition of Sharpness, & Trial C : Repeated Minicoring	Cored Sale Lot	No	41.6	56.6
8				22.4	101.1
9				19.6	101.0
10				21.6	96.7
11				22.6	94.2
12				18.9	122.9
13				25.2	81.5
14				25.0	92.4
15				29.7	82.1
16				31.7	78.0
17				15.7	121.7
18				30.4	82.4
19				23.6	92.7
20				21.3	108.0
21				23.4	95.8
22				27.0	82.0
23				17.8	114.7
24				24.3	90.7
25				35.2	73.4
26				18.2	119.9
27				23.7	86.3
28				20.1	95.9
29				16.9	121.1

Trial B (a)

No	Trial	Form of Wool	Shirley?	MFD	FC
30	Trial B (a) : Sharp & Blunt Minicore Tubes	Top	No	30.3	73.3
31				24.4	81.0
32				24.2	84.7
33				18.4	96.0
34				24.2	79.2
35				30.4	74.9
36				19.0	101.1
37		Scoured Top		30.2	80.6
38				24.7	88.5
39				24.6	89.8
40				18.7	114.1
41				24.5	89.1
42				30.5	80.9
43				19.1	112.9
44			Yes	30.7	83.1
45				24.8	89.6
46				24.6	89.6
47				18.9	119.9
48				24.8	91.3
49				30.6	79.9
50		Cored Sale Lot	No	26.5	83.9
51				21.7	98.7
52				19.8	103.3
53				18.7	103.1
54				18.0	110.5
55				16.8	118.9
56				36.8	69.1
57				29.8	85.6
58				25.3	87.9
59				22.6	94.3
60			Yes	26.2	84.5
61				21.5	103.0
62				19.5	106.6
63				18.7	109.1
64				18.1	112.1
65				16.6	117.7
66				35.7	70.8
67				29.0	86.3
68				25.3	86.7
69				22.5	96.1

Trial D

No	Trial	Form of Wool	Shirley?	MFD	FC
70	Trial D : Subsample Mass	Cored Sale Lot	No	18.3	100.8
71				25.8	81.8
72				31.4	67.1
73				19.4	97.3
74				25.1	82.8
75				29.1	80.1
76				19.6	123.6
77				20.0	93.9
78				19.9	94.8
79				20.5	96.4