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The Standardisation of Mean Fibre Curvature Using the Series 14 Calibration Top as a Reference.

By

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SUMMARY

An international round trial was conducted across 6 core-test laboratories to investigate the potential of using the IH Series 14 Calibration Tops as a standard reference for calibrating Laserscan and OFDA instruments for the measurement of Mean Fibre Curvature.

Measurements were generated on 12 Laserscan and 10 OFDA instruments. Calibration equations based on the Geometric Mean Regression were used to calibrate raw data measurements for Mean Fibre Curvature to a standard basis.

The application of a Mean Fibre Curvature calibration to individual instruments showed potential for reducing the between-instrument variance in the measurement of Mean Fibre Curvature for guillotined sliver, but it did not provide the same improvement when samples prepared from greasy wool were measured using the calibration derived for tops.

The critical issue appears to be the difference in the linear relationships between uncalibrated data for top samples and the grand mean compared to the same relationship for samples prepared from greasy wool.

Further research is needed to explain the differences between the two relationships.

INTRODUCTION

Since the introduction of Fibre Curvature measurement in the 1990s, between-instrument differences (both within-type and between-type) have been a feature of the measurement. The measured result has been shown to be sensitive to the sample preparation procedures (Fish, 2000). The two instruments (Laserscan and OFDA) commonly used for Fibre Curvature measurements have different preparation requirements. For example, OFDA requires the use of a fibre spreader whereas Laserscan does not.

Baxter (2002) examined the use of graticules as a means of calibrating OFDA instruments. While further refinement of this method shows promise as a method of comparing between OFDA instruments, it does not offer a method of comparison between the two methods currently in commercial use.

The method of calibration examined in this report investigates the potential to use the Interwoollabs IH Series 14 Calibration tops as a reference. Interwoollabs already provides a service to the International Wool Textile Industry by providing Fibre Diameter calibration standards. The reference values assigned to the calibration standards are derived from International Round Trials that are conducted twice per year. It is feasible that Fibre Curvature measurements could be collected during these round trials. If a procedure using assigned Mean Fibre Curvature (MFC) values to calibrate both Laserscan and OFDA 100 instruments was demonstrated to reduce the between-instruments variance, Interwoollabs would be the preferred industry organisation to provide Mean Fibre Curvature Reference material to the wool industry.

MATERIALS AND METHODS

LABORATORIES

Six raw wool core-testing laboratories were involved in the trial, they were:

- AWTA Ltd Fremantle,
- AWTA Ltd Melbourne,
- AWTA Ltd Sydney,
- NZWTA Ltd Napier,
- SGS New Zealand Wool Testing Services, and
- WTB S.A. South Africa.

IH Calibration Tops

Each laboratory prepared samples of IH Series 14 calibration top from their own supplies. Two sets were prepared: one for measurement by the normal tops procedures using the guillotine method and the second set was prepared by cutting the top into lengths of approximately 20mm followed by the normal preparation procedures used for greasy wool core samples. The scoured top calibration will be addressed in a future IWTO paper.

For the guillotine method, approximately 1m of each of the 8 IH Series 14 calibration tops was dried and conditioned in accordance with IWTO-52-96.

For each Laserscan instrument, ten test specimens of 1000 snippets each were measured from each of the eight tops. Each test specimen was generated from a new guillotined sample. For each top for each instrument, the 10 measurements were pooled to generate a MFC value.

For each OFDA 100, each slide was prepared from a new guillotine cut of a section of each top, and the resulting snippets dispersed on an OFDA slide using the slide-preparer allocated to each individual instrument. In accordance with IWTO-47-02, 4 slides were measured for calibration tops 1 to 4, 6 slides were measured for calibration tops 5 and 6, and 8 slides were measured for calibration tops 7 and 8. For each top for each instrument, the individual top measurements were pooled to generate MFC values.

Greasy Wool

Each of the six participating laboratories was supplied with 42 samples of greasy wool cores ranging in MFC values from approximately 40 to 120 deg/mm. Thirty-six of the unknown samples were sourced from Australia, with the remaining 6 being sourced from South Africa. Sub-samples were selected to be high yielding, containing less than 5% Vegetable Matter Base.

Approximately 84g of each of the 42 greasy core samples was dispatched to each of the laboratories. The greasy core samples were scoured, and dried using the normal commercial procedures of each laboratory. Two 20g subsamples were separated from the scoured mass and conditioned in the Standard IWTO atmosphere for a minimum of 24 hours prior to measurement.

The sub-samples were not Shirley Analysed prior to measurement on either instrument type.

One of the sub-samples was allocated for Laserscan measurement and the other for OFDA 100 measurement.

For the Laserscans, each 20g sub-sample was halved to create 2 x 10g sub-sub-samples. The 2 sub-sub-samples were introduced into the minicore apparatus of each Laserscan. The sub-sample was minicored for the first test specimen of 1000 snippets, and on completion of measurement, was minicored a second time for the second test specimen. The sub-sub-samples were then swapped between the 2 instruments and the procedures repeated to provide test specimens 3 and 4. The four MFC values (2 from the first 10g sub-sub-samples and 2 from the second 10g sub-sub-sample) resulting from each instrument were pooled to give an individual instrument estimate of MFC for each of the 42 unknown samples.

For OFDA 100's, each 20g sub-sample was halved to create 2 x 10g sub-sub-samples. The first 10g sub-sub-sample was introduced into the minicore/slide preparation apparatus and minicored for the first test specimen (slide 1), and minicored a second time for the second test specimen (slide 2). The sub-sub-samples were then swapped between the 2 instruments and the procedure repeated for test specimens 3 and 4. The four MFC values resulting from each instrument (2 from first 10g sub-sub-sample and 2 from second 10g sub-sub-sample) were pooled to give an individual instrument estimate of MFC for each of the 42 unknown samples.

RESULTS AND DISCUSSION

Attempts to harmonise instruments have to be performed at the individual instrument level. For this reason the results for individual instruments are used as the basis for reporting both before and after calibration.

The results for the tops measurements are summarised in Table 1 and presented graphically in Figures 1 and 2.

Table 1: Tops Measurements Prior to Calibration - Comparisons between Instrument Averages and the Differences for Each Instrument

Type	Instrument	Top average (°/mm)	Difference from Pooled Instrument Mean (°/mm)
Laserscan	L1	62.9	0.3
	L2	62.3	-0.3
	L3	60.0	-2.7
	L4*	54.6	-8.1
	L5*	68.2	5.6
	L6	61.7	-1.0
	L7	63.4	0.8
	L8	62.8	0.2
	L9	62.7	0.1
	L10	64.4	1.8
	L11	63.5	0.9
	L12*	76.4	13.7
Laserscan Average#		62.6	
OFDA100	O1	55.3	-1.8
	O2	57.5	0.3
	O3	57.6	0.5
	O4	57.8	0.6
	O5	56.9	-0.2
	O6	57.1	-0.1
	O7*	65.3	8.1
	O8*	64.2	7.0
	O9	56.0	-1.2
	O10	59.0	1.8
OFDA Average#		57.2	
Grand Average#		59.9	

Note: * indicates the results are considered to be outliers within the group
# Indicates the outlier results have been excluded from the calculation of the averages.

From Table 1 it is clear that, based on the overall instrument means, there were a number of divergent instruments for both Laserscan (3) and OFDA 100 (2) instruments. This will be confirmed in the tables and figures presented later in the report. It is normal practice to exclude any outlier results from the calculation of any average value that is to be used as an "Assigned" value in any future calibration. Interwoollabs follows this practice for assigning values to calibration tops for MFD. For the purpose of this report the instruments flagged as outliers have been included in all the tables and figures in the report, but they have not been included in the calculation of any overall performance statistics.

Table 1 also shows that there was an average difference of 5.4 °/mm between the Laserscan and the OFDA100 instruments, with the average MFC values for Laserscan and OFDA being 62.6°/mm and 57.2°/mm respectively.

ASSIGNING MFC VALUES TO IH SERIES 14 CALIBRATION TOPS

The MFC values generated by 22 instruments (12 Laserscans and 10 OFDAs) were combined (excluding outlier instruments) to form a pooled (assigned) MFC value for each of the 8 IH Series 14 calibration tops, such that the grand mean for each top was calculated as:

$$Top\ MFC = \frac{(Mean\ \ for\ OFDA\ Instruments) + (Mean\ \ for\ Laserscan\ Instruments)}{2}$$

The method above ensured that each instrument-type contributed equally to the formation of the assigned value for each top, regardless of the number of instruments within each instrument type. Table 2 details the assigned MFC value for each of the IH Series 14 calibration tops.

Table 2: Mean Fibre Curvature Values generated for IH Series 14 Calibration Tops.

Top	Laserscan (°/mm)	OFDA100 (°/mm)	Assigned MFC (°/mm)
1	83.1	85.4	84.2
2	77.6	75.1	76.2
3	67.8	64.8	66.3
4	61.4	56.7	59.1
5	64.9	60.1	62.5
6	52.9	44.5	48.7
7	48.3	37.7	43.0
8	45.2	33.0	39.1

COMPARISONS BETWEEN THE MEASURED MFC VALUES AND THE ASSIGNED VALUES FOR TOPS PRIOR TO CALIBRATION.

Table 3 presents the individual instrument average differences from the Assigned MFC values for each top. It is recognised that the method of calculation is incestuous in that the same data has been used to derive the Assigned values as was used to validate the calibration. However, it was considered advantageous to present the data in this format to enable easy comparisons with later tables and figures.

Table 3: Comparison between the average difference from the assigned value for individual instruments before calibration, and the variance of the differences from the assigned, generated for top.

Type	Instrument	Average Difference (°/mm)	Variance of the Differences $(°/mm)^2$
Laserscan	L1	3.0	3.7
	L2	2.4	3.6
	L3	0.1	2.8
	L4*	-5.3	9.3
	L5*	8.3	20.1
	L6	1.8	5.7
	L7	3.5	16.1
	L8	2.9	7.0
	L9	2.8	6.7
	L10	4.5	7.1
	L11	3.6	8.3
	L12*	16.5	20.0
Laserscan Average#		2.7	6.8
OFDA100	O1	-4.6	3.7
	O2	-2.4	8.5
	O3	-2.2	8.1
	O4	-2.1	7.8
	O5	-3.0	3.7
	O6	-2.8	6.3
	O7*	5.4	21.1
	O8*	4.3	13.0
	O9	-3.9	5.7
	O10	-0.9	7.0
OFDA Average#		-2.7	6.3
Grand Average#		**0.0**	**6.6**

Note: * indicates the results are considered to be outliers within the group
# Indicates the outlier results have been excluded from the calculation of the averages.

The positive average differences for Laserscan and negative average differences for OFDA100 are a direct consequence of using the mean of the Laserscan and OFDA100 values as the “Assigned” value. The overall zero Grand Average is also a consequence of the calculation procedures.

The variance of the differences from the “Assigned” values is an indication of the within-instrument variance. Once the outliers have been removed the pooled averages were 6.8 $(°/mm)^2$ for Laserscan and 6.3 $(°/mm)^2$ for OFDA100.

Figures 1 (Laserscan) and 2 (OFDA100) show the individual instrument differences from the “Assigned” value for each top. The instruments identified as outliers from the instrument average are, once again, clearly seen as divergent in these Figures. Quadratic trend lines have also been included for each instrument. The individual instrument slopes are a consequence of using the mean of the Laserscan and OFDA100 values as the “Assigned” value. Therefore at low MFC values the Laserscans exhibit a positive difference and the OFDA100s negative differences. The average differences between the two should be zero. The process of calibration is intended to reduce both the between-instrument type differences and the variation exhibited between instruments of the same type.

Figure 1: Difference between the measured MFC values for individual Laserscan instruments and the "Assigned" values prior to Calibration.

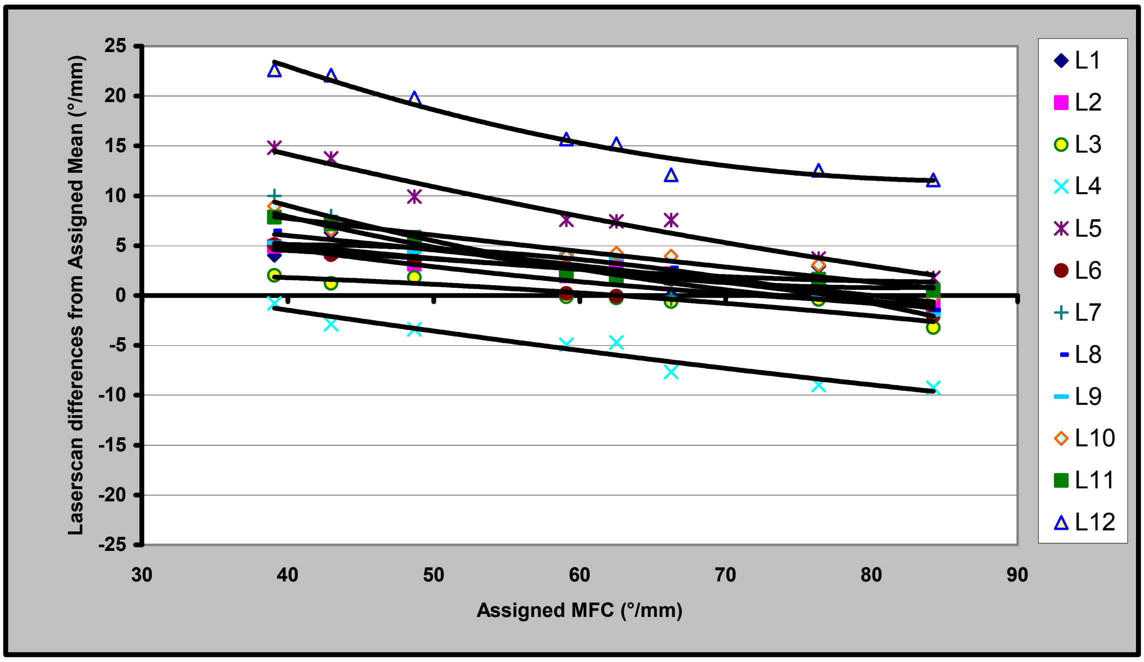
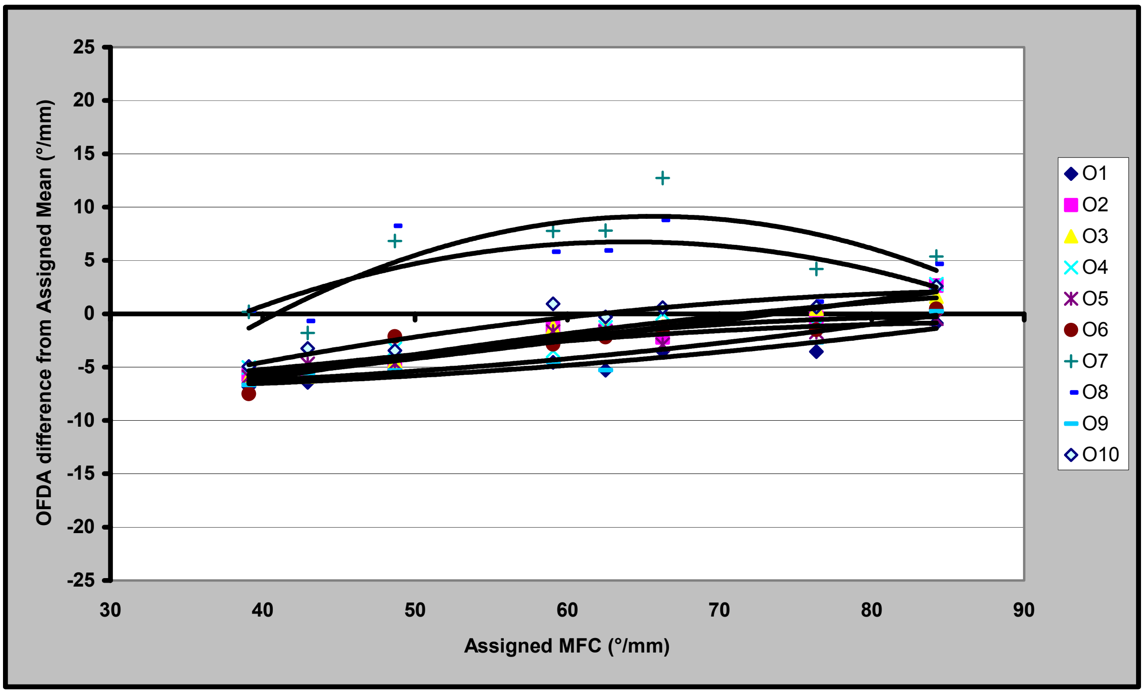


Figure 2: Difference between the measured MFC values for individual OFDA 100 instruments and the "Assigned" values prior to Calibration.



MEAN FIBRE CURVATURE CALIBRATION PROCEDURES

MFC calibration equations were developed for each instrument from the measured MFC values prior to calibration and the Assigned top values. A linear regression equation was generated for each instrument such that the relationship between each instrument's measured values and the Assigned values were described as:

$$y = ax + b,$$

where: "*x*" is the measured MFC value on an individual instrument prior to calibration; "*a*" is the slope of the line in relation to the "Assigned" value; and "*b*" is the offset of the relationship.

This equation is inverted such that "*x*" can be solved for "*y*" as follows:

$$x = \frac{y - b}{a}$$

The slope (*a*) and intercept (*b*) were determined for each instrument using a Geometric Mean Linear Regression analysis (IWTO-0-00). The results are summarised in (Table 4).

Table 4: The Slope (*a*) and Intercept (*b*) values generated for the relationship between Assigned top values and top values generated by individual instruments.

Instrument	Slope (a)	Intercept (b)
L1	0.913	8.090
L2	0.886	9.286
L3	0.903	5.864
L4	0.816	5.716
L5	0.725	24.799
L6	0.873	9.361
L7	0.768	17.412
L8	0.837	12.655
L9	0.848	11.955
L10	0.843	13.923
L11	0.833	13.619
L12	0.735	32.340
O1a	1.114	-11.402
O2a	1.178	-13.070
O3a	1.177	-12.854
O4a	1.165	-12.015
O5a	1.112	-9.3642
O6a	1.142	-11.299
O7a	1.168	-4.672
O8a	1.086	-0.850
O9a	1.136	-12.056
O10a	1.156	-10.272

The MFC calibration equations were applied to the top values used to generate the equation for each instrument. The variance of the calibrated top values from the Assigned MFC values was then compared with the variance of the uncalibrated top values from the Assigned MFC values.

Table 5: Comparison between the average difference from the Assigned value for individual instruments after calibration, and the variance from the Assigned value, generated for top.

Type	Instrument	Average Difference (°/mm)	Variance of Difference $(°/mm)^2$
Laserscan	L1	0.0	2.1
	L2	0.0	0.4
	L3	0.0	0.5
	L4*	0.0	0.9
	L5*	0.0	1.5
	L6	0.0	1.9
	L7	0.0	3.4
	L8	0.0	0.4
	L9	0.0	1.0
	L10	0.0	1.1
	L11	0.0	1.6
	L12*	0.0	3.4
Laserscan Average#		0.0	1.4
OFDA100	O1	0.0	0.3
	O2	0.0	0.5
	O3	0.0	0.2
	O4	0.0	0.8
	O5	0.0	0.5
	O6	0.0	1.1
	O7*	0.0	12.0
	O8*	0.0	10.3
	O9	0.0	0.9
	O10	0.0	0.7
OFDA Average#		0.0	0.6
Grand Average#		**0.0**	**1.0**

Note: * indicates the results are considered to be outliers within the group.
# indicates the outlier results have been excluded from the calculation of the averages.

Table 5 shows that the implementation of the MFC calibration equations for tops decreased the within-instrument variance from an average of 6.6°/mm^2 (Table 3) to 1.0°/mm^2. This indicates that the implementation of a top based calibration equation can successfully improve the within-instrument variance when measuring top for both Laserscan (6.8°/mm^2 to 1.4°/mm^2) and OFDA100 (6.3°/mm^2 to 0.6°/mm^2).

Figures 3 and 4 graphically compare the calibrated MFC data with the Assigned values.

Figure 3: Comparison between the difference of the calibrated MFC values from the Assigned values for individual Laserscan instruments.

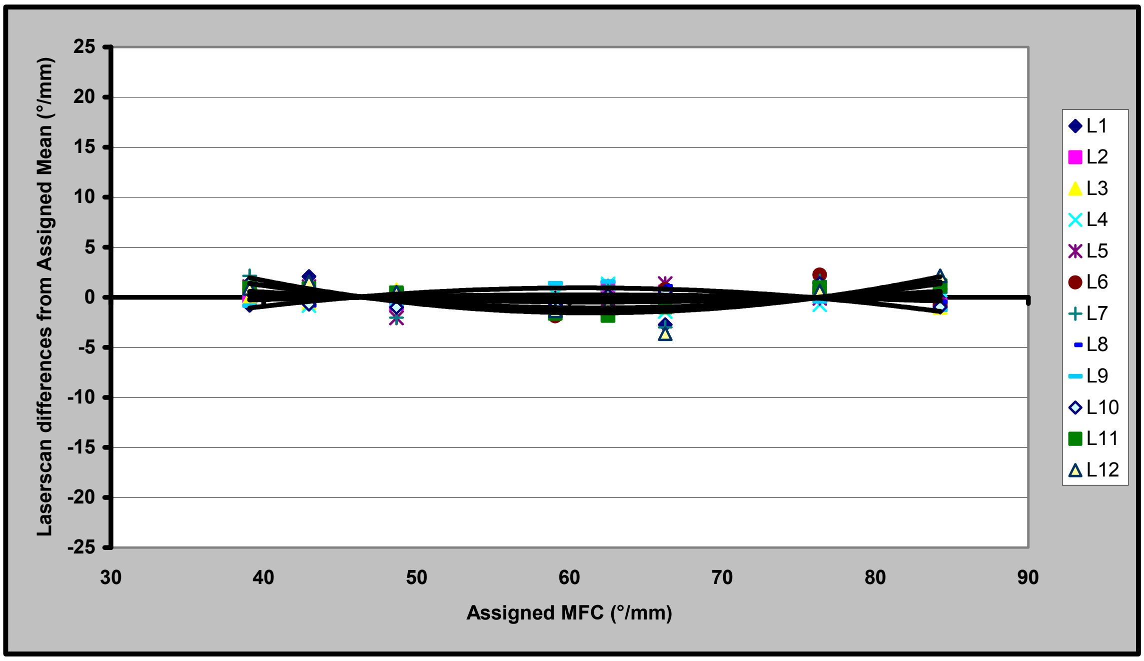
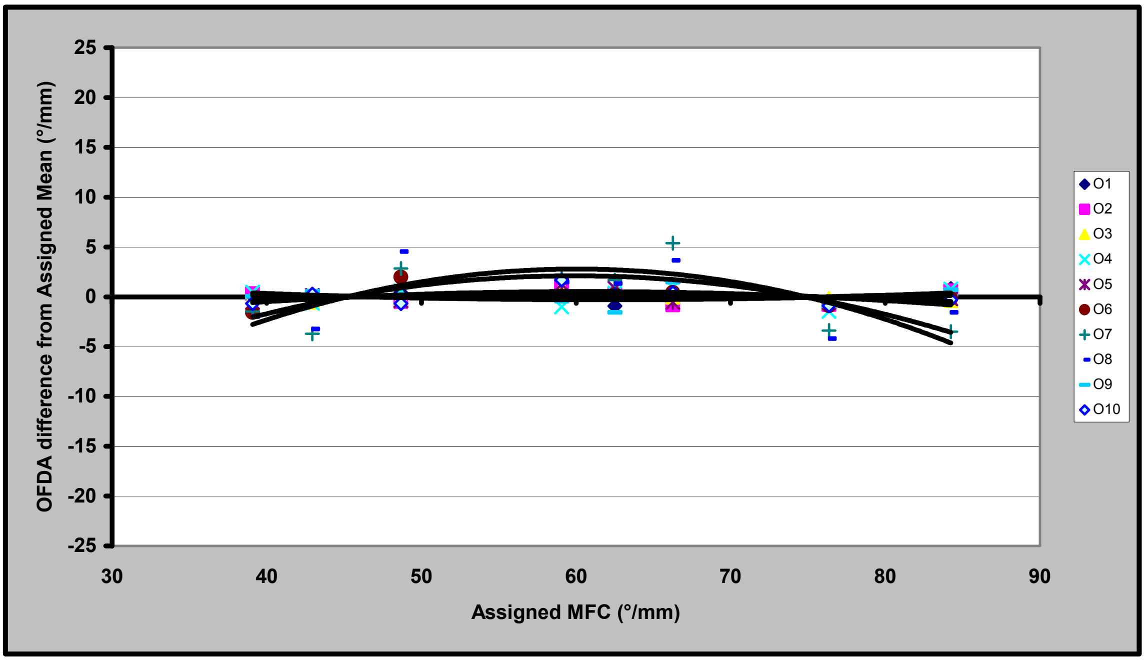


Figure 4: Comparison between the difference of the calibrated MFC values from the Assigned values for individual OFD100 instruments.



Although the data in Figures 3 and 4 is the calibration data refitted on itself, there is a strong indication that the calibration process has resulted in an improvement in instrument harmonisation. Furthermore, where a particular instrument has been adjudged an outlier prior to calibration, it has been harmonised with the other instruments after calibration. In addition, the differences that were evident between the Laserscan and OFDA instruments have been successfully removed.

To verify these results, another set of Independent tops could be used, however this has not been addressed in this trial.

COMPARISONS BETWEEN THE MEASURED MFC VALUES AND THE AVERAGE VALUES FOR GREASY WOOL BEFORE CALIBRATION.

In the case of measurements made on samples that have been prepared from greasy wool there is no intention to have an Assigned value because it is not intended to produce greasy wool calibration standards. For the purpose of comparing data the average for each instrument type and the Grand Average have been calculated in the same way as was used for the Top samples. Table 6 summarises the average results for the 42 greasy samples.

Table 6: Greasy Wool Measurements Prior to Calibration - Comparisons between Instrument Averages and the Differences for Each Instrument

Type	Instrument	Greasy Wool Average (°/mm)	Difference from Instrument Mean
Laserscan	L1	83.7	-2.3
	L2	91.6	5.6
	L3	82.6	-3.4
	L4*	82.4	-3.6
	L5*	95.5	9.6
	L6	86.5	0.6
	L7	82.7	-3.2
	L8	83.5	-2.4
	L9	89.5	3.5
	L10	92.2	6.2
	L11	81.3	-4.6
	L12*	101.2	15.2
Laserscan Average#		85.9	
OFDA100	O1	74.8	-3.5
	O2	81.7	3.4
	O3	81.5	3.2
	O4	79.9	1.6
	O5	75.7	-2.6
	O6	78.9	0.6
	O7*	79.4	1.0
	O8*	79.1	0.7
	O9	76.3	-2.0
	O10	77.9	-0.5
OFDA Average#		78.3	
Grand Average#		82.1	

Note: * indicates the results, <u>based on measurements of tops</u>, are considered to be outliers within the group.
# indicates the outlier results, <u>based on measurements of tops</u>, have been excluded from the calculation of the averages.

From Table 6 it appears that the instruments that were considered to be divergent based on top measurements are not necessarily divergent when measuring samples prepared from greasy wool. In addition, a different set of instruments could now be considered to be divergent. For example, L4 and O8 were considered divergent based on tops measurements but appeared to be not so on the basis of measurements made on samples prepared from greasy wool. The difference between Laserscan and OFDA was 5.4°/mm for the tops and this increased to 7.6°/mm for greasy wool samples.

Table 7: Comparison of Instrument average difference for greasy wool values from the overall average value for Laserscan and OFDA100 before calibration.

Type	Instrument	Average Difference	Variance Differences
Laserscan	L1	1.5	7.2
	L2	9.4	10.1
	L3	0.5	6.9
	L4*	0.3	7.5
	L5*	13.4	8.2
	L6	4.4	4.4
	L7	0.6	8.0
	L8	1.4	2.7
	L9	7.3	5.5
	L10	9.7	8.7
	L11	-0.8	6.0
	L12*	19.2	8.5
Laserscan Average#		3.8	6.6
OFDA	O1	-7.3	15.9
	O2	-0.4	3.8
	O3	-0.6	4.7
	O4	-2.2	5.2
	O5	-6.4	6.2
	O6	-3.2	4.9
	O7*	-2.7	6.3
	O8*	-3.0	7.0
	O9	-5.8	8.3
	O10	-4.3	8.6
OFDA Average#		-3.8	7.2
Grand Average#		0.0	6.9

Note: * indicates the results, based on measurements of tops, are considered to be outliers within the group.
# indicates the outlier results, based on measurements of tops, have been excluded from the calculation of the averages.

Table 7 also questions the consistancy of instrument divergence between measurements made on tops and measurements made of samples prepared from greasy wool. It appears that Laserscan instrument L2 would be considered more divergent from the group than L6, which is different from the top measurements. Similar comparisons can be drawn for the OFDA100 instruments O1 and O7.

Figures 5 and 6 graphically represents the individual instrument differences described in Table 7.

Figure 5: Comparison between the difference from the grand average for individual Laserscan instruments before calibration.

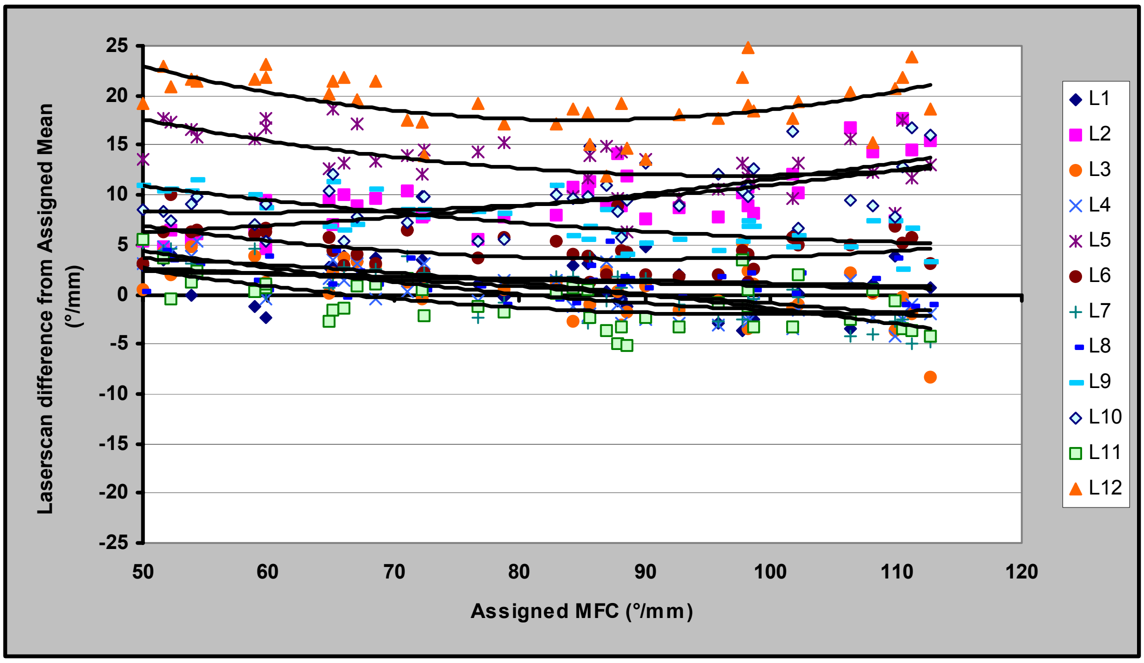
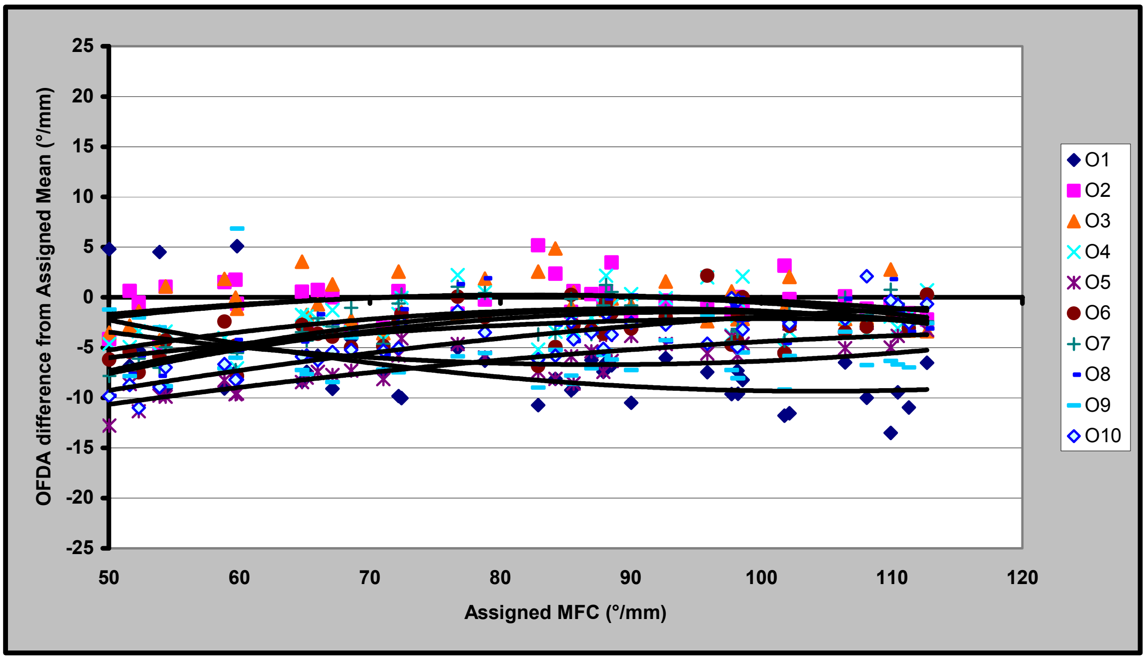


Figure 6: Comparison between the difference from the grand average for individual OFDA100 instruments before calibration.



Figures 5 and 6 demonstrate that there is more variation in the greasy wool results than was observed for the top samples (see Figures 1 and 2). This is not unexpected because the sample preparation procedures for greasy wool samples involve more processes than the preparation guillotined top samples. It has already been shown that sample preparation procedures can influence the measured MFC (Fish, 2002). Therefore, the critical question is whether calibrating each instrument can reduce the observed variation between individual instruments and between instrument types.

COMPARISONS BETWEEN THE MEASURED MFC VALUES AND THE AVERAGE VALUES FOR GREASY WOOL AFTER CALIBRATION.

Table 8 summarises the instrument differences from the Grand Average after transforming the raw measurements using the calibration that was derived from the top samples.

Table 8: Comparison of Instrument average difference for greasy wool values from the overall average value for Laserscan and OFDA100 instruments after calibration.

Type	Instrument	Average Difference (°/mm)	Variance Differences (°/mm)^2
Laserscan	L1	-0.1	8.9
	L2	10.2	28.3
	L3	2.2	5.6
	L4*	11.3	7.8
	L5*	14.9	34.5
	L6	5.7	7.9
	L7	2.4	12.7
	L8	2.0	10.5
	L9	8.7	4.3
	L10	9.8	33.3
	L11	-1.4	10.2
	L12*	11.1	49.0
Laserscan Average#		4.4	13.5
OFDA100	O1	-5.3	26.7
	O2	-2.3	12.4
	O3	-2.5	13.6
	O4	-3.8	8.7
	O5	-5.9	1.6
	O6	-3.7	5.9
	O7*	-10.7	6.7
	O8*	-9.1	4.2
	O9	-4.9	16.0
	O10	-6.5	2.4
OFDA Average#		-4.4	10.9
Grand Average#		0.0	12.2

Note: * indicates the results, based on measurements of tops, are considered to be outliers within the group.
# indicates the outlier results, based on measurements of tops, have been excluded from the calculation of the averages.

The calibration process increased the between-instrument differences both within and between instrument types. The average difference between Laserscan and OFDA100 instruments has also increased, and an increase in the variance of the differences was also noted. These results were unexpected. Calibration with assigned values for guillotined top samples has not improved the harmony between instruments when measurements are performed on samples prepared from greasy wool.

The results from Table 8 are also presented graphically in Figures 7 and 8.

Figure 7: Comparison between the difference from the grand average for individual Laserscan instruments after calibration.

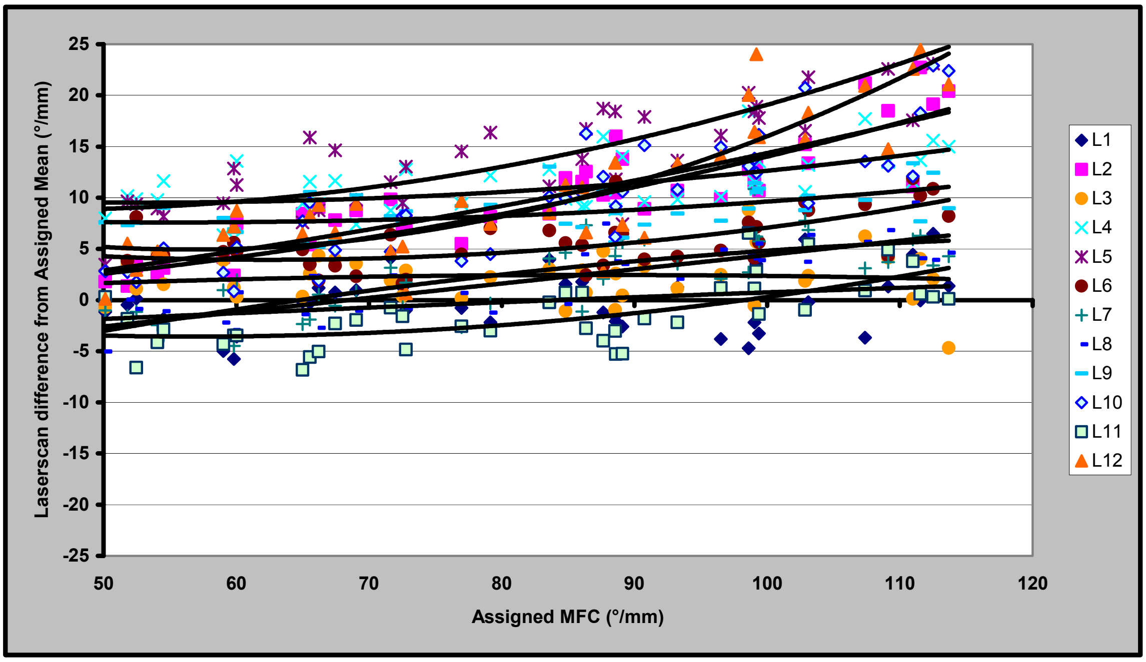
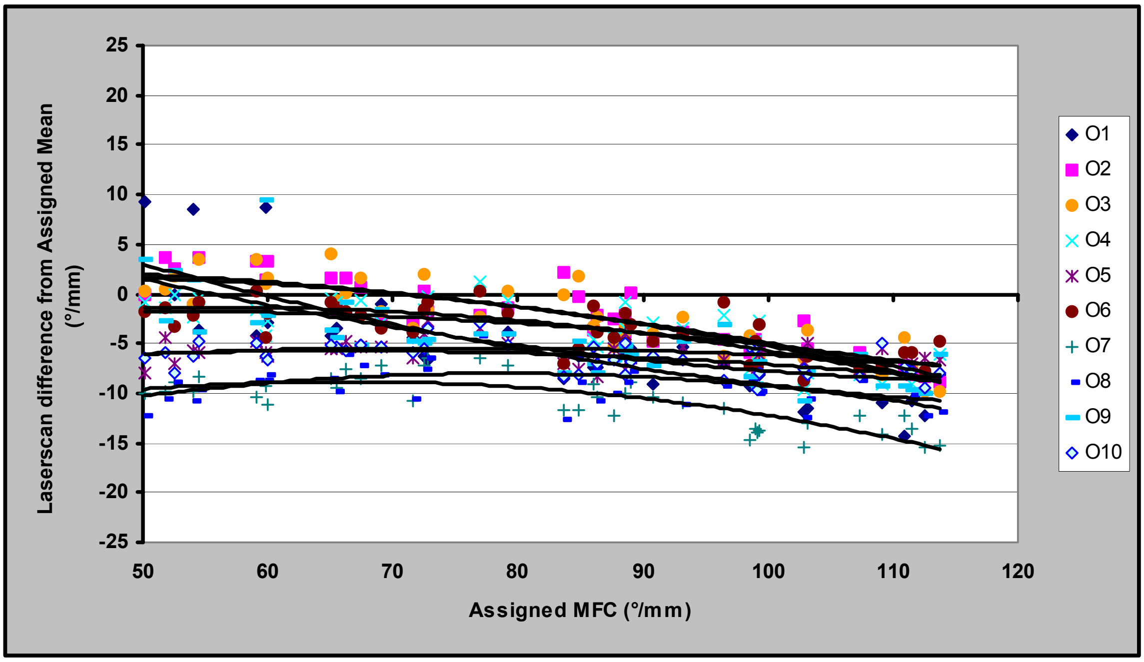


Figure 8: Comparison between the difference from the grand average for individual OFDA100 instruments after calibration.

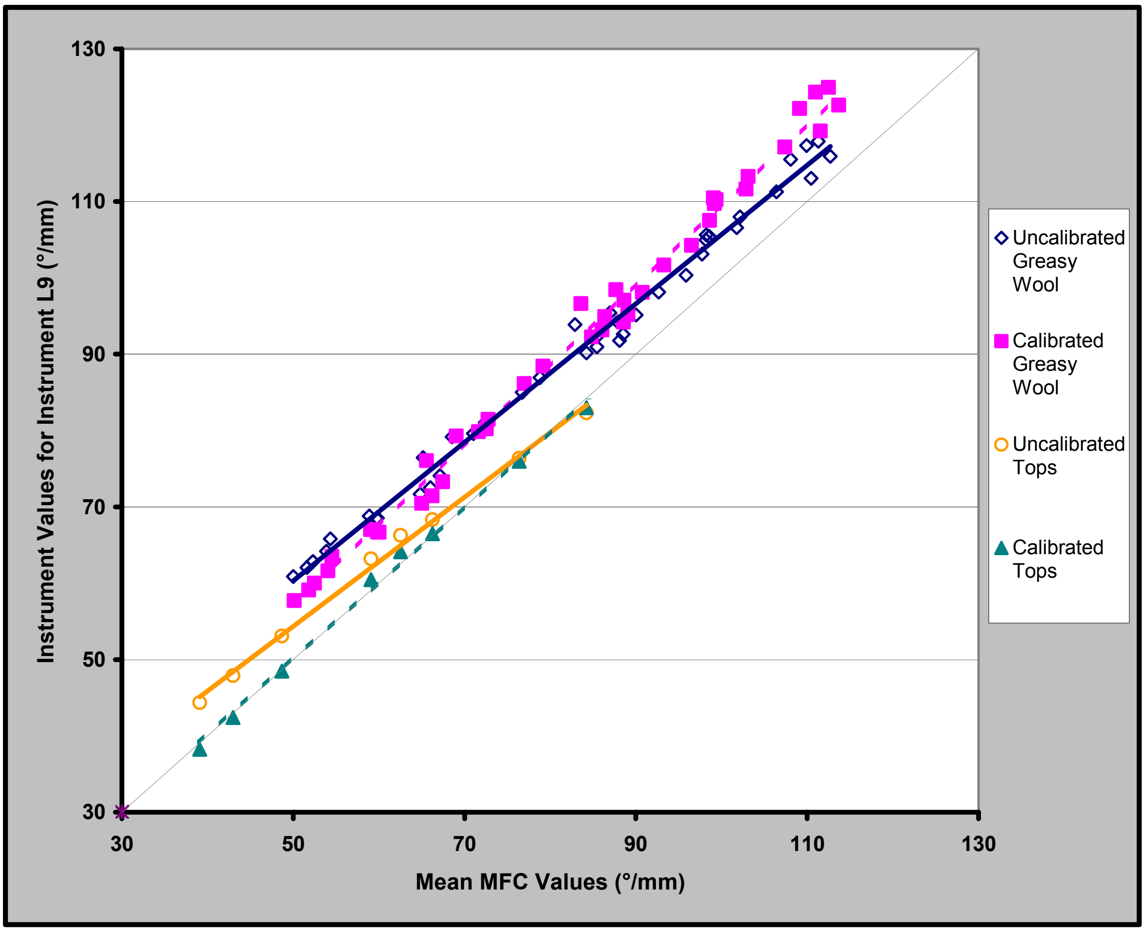


Unlike the tops, where the calibration improved the harmony between instruments, the calibration process has added variation to the measurements made on samples prepared from greasy wool.

COMPARISON OF TOPS AND "GREASY WOOL" RELATIONSHIPS

In an attempt to explain the reason for the difference in performance of the calibration process on tops and on greasy wool, the relationships between tops and greasy wool were examined for the Laserscan instrument L9 (see Figure 9).

Figure 9: The relationship between uncorrected and corrected values for Laserscan 9



There are several important points to note from Figure 9.

The first is the comparison between the Tops values prior to calibration and after calibration. The tops values prior to calibration sit above the 1:1 relationship with the mean. The application of the calibration to the data transforms it such that the relationship between the calibrated data and the mean are very close to a 1:1 relationship (denoted ▲ in Figure 9).

For the greasy wool data prior to calibration the values also sit above the 1:1 relationship but they have a steeper slope than the tops values prior to calibration. After applying the calibration the slope of the relationship becomes even steeper, moving the lower MFC samples closer to the 1:1 relationship and the higher MFC samples further away. This skewing of the relationship is the mechanism that is increasing the variation for the samples prepared from greasy wool.

It is clear that for the calibration process to work the uncalibrated data for both the tops and the samples prepared from the greasy wool must fall on the same relationship. For the Laserscan instrument L9 the relationships are clearly different.

Further research is required to understand the factors that influence the tops and greasy wool relationships. As part of this trial the participating laboratories also measured top samples that had been prepared using the same scouring, drying, minicoring procedures that were used to prepare the greasy wool samples. Analysis of these data will be the subject of a separate report to a future IWTO meeting.

CONCLUSIONS

The calibration procedure for tops was found to improve the harmonisation between instruments of the same type, and between instruments of different types. This conclusion needs to be verified with top samples that are independent of the calibration process. If Interwoollabs were able to collect Mean Fibre Curvature data from their Round Trials it may help laboratories to identify if an instrument is an outlier for MFC and therefore in need of servicing.

Calibrating instruments for measuring the Mean Fibre Curvature (MFC) of samples prepared from greasy wool using calibrations based on guillotined tops was not successful. The calibration process added further variation between instruments rather than reducing this variation. The source of the additional variation appears to be related to the relationships that exist in the data prior to calibration, between the individual instrument data and the overall grand average across all instruments.

The sample preparation procedures for greasy wool samples are different and more complex than those used for tops and this could be the critical limiting factor. Analysis of additional data collected at the time of measurement will be used to determine if a calibration process based on calibration top prepared like greasy wool samples can be used to improve the harmony between instruments.

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