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The Standardisation of Mean Fibre Curvature Using the Series 14 IH Calibration Tops as a Reference - Calibration Derived for Minicored Scoured-Wool

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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 OFDA100 instruments for the measurement of Mean Fibre Curvature. This report is the second in a series of 2 reports to investigate the potential of using the IH Series 14 Calibration Tops as a standard reference for calibrating Laserscan and OFDA100 instruments for the measurement of Mean Fibre Curvature.

The Interwoollabs IH Series 14 Calibration Tops were used as the standard reference material in this trial because there is no other form of calibration standard available for MFC. However, previous research has shown that it is the sample preparation procedures that require calibration rather than the instruments (Fish, 2002). This is because the processes fibres undergo prior to measurement of MFC affect wool MFC. As a result, any calibration of the measurement process for MFC should follow the same procedure, and use the same process that is used for the measurement of greasy wool.

Measurements generated on 10 Laserscan and 8 OFDA100 instruments showed that the application of a Mean Fibre Curvature Calibration to individual instruments showed potential for reducing the between-instrument variance for scoured top. An improvement in the between instrument SD of 4.6 deg/mm was also noted when the calibration was applied to raw wool samples, demonstrating that the application of a preparation procedure would provide an opportunity to better harmonise the MFC of instruments across the raw wool testing industry.

MATERIALS AND METHODS

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.

Minicored series 14 IH Calibration Tops

The Series 14 IH calibration tops were prepared following the normal preparation procedure used for greasy wool core calibration samples. Approximately 50g of each top (sufficient for two instruments) was cut into lengths of 20mm, then scoured, dried and conditioned in accordance with IWTO-52-96. Samples of scoured, dried and minicored top will be referred to as scoured top hereafter. 10g sub-samples of scoured top were sampled for measurement on each instrument type.

For each Laserscan, ten test specimens of 1000 snippets were measured from each of the eight scoured tops. The results of the 10 test specimens were pooled to generate MFC values for individual instruments.

For each OFDA100, a new slide was prepared for each minicored sub-sample. Resulting snippets were dispersed on an OFDA100 slide using the slide-preparer allocated to individual instruments. In accordance with IWTO-47-02, 4 slides were measured for scoured calibration tops 1 to 4, 6 slides for scoured tops 5 and 6, and 8 slides were measured for scoured top 7 and 8. A maximum of 4 minicores were generated from each 10g sample. For each scoured top measured on each instrument, the individual top measurements were pooled to generate MFC values for individual instruments.

The materials and method used to determine the "assigned" values for guillotined top, herein referred to as Assigned, have previously been reported (Fish, 2003).

Greasy Wool

Each of the 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 raw wool samples were sourced from Australia, with the remaining 6 being sourced from South Africa. Samples were selected to be high yielding, containing less than 5% Vegetable Matter Base.

Approximately 84g of each of the 42 greasy core samples (hereafter referred to as "raw wool") were dispatched to each of the laboratories. The raw wool samples were scoured, and dried using the normal commercial procedures of each laboratory. Two 20g subsamples were sampled 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 OFDA100 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-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 raw wool samples.

For OFDA100'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 raw wool samples.

RESULTS AND DISCUSSION

One of the laboratories involved in this trial has been discarded from the analysis of results due to potential problems with the integrity of their data, for both Laserscan and OFDA100. The results from this laboratory will not feature anywhere in this report.

MFC Relationship between scoured top and assigned MFC values of the series 14 IH top.

Figure 1 shows the typical relationship between the scoured top and the respective Assigned MFC value for 1 instrument, as determined by Fish (2003). It can be seen from Figure 1 that the scoured top samples displayed a higher MFC than the Assigned top value. This is an artefact of the derivation of the Assigned values, which were derived using the MFC values of the guillotined top using the same method Interwoollabs use to derive the Assigned values of MFD for the IH calibration tops (Fish, 2003). As a result, any calibration generated using the guillotined top MFC values would ultimately lead to a calibration that will effectively lower the MFC of the final calibrated value. This approach is necessary because the only source of reference material is Interwoollabs; Interwoollabs only collect data for the measurement of guillotined top. Note that the relationship demonstrated in Figure 1 is typical of all instruments, however the slope of the relationship differs across all instruments investigated.

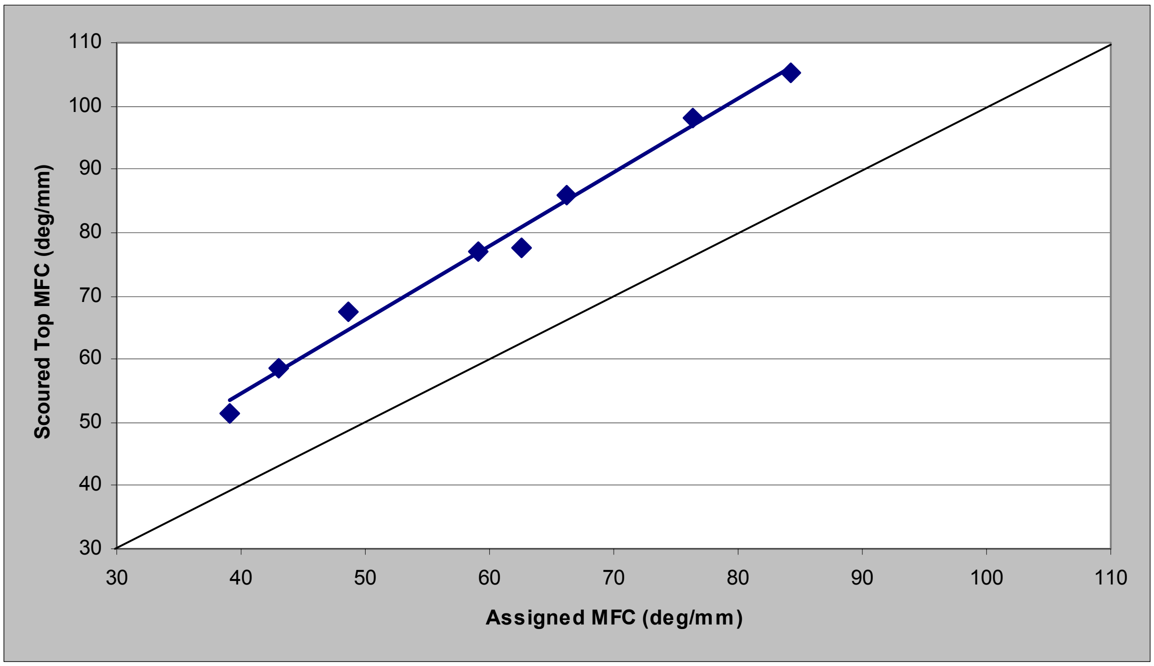

Figure 1: Relationship between the MFC of the Assigned MFC values of the Series 14 IH calibration top, and Scoured Series 14 IH calibration Top for Laserscan instrument 1.

The relationship between the Assigned MFC values and the MFC value for scoured Series 14 IH calibration top was determined for each instrument using the geometric mean (GM) regression for each instrument. The relationship between the Assigned and scoured top can be described by the equation:

$$y = ax + b,$$

where "*x*" is the guillotined top value and "*y*" is the minicored top value; "*a*" is the slope of the relationship and "*b*" is the offset.

Table 1 shows the slope (*a)* and intercept (*b*) generated for the relationship between the MFC values of scoured top and the assigned MFC values of top for individual instruments.

Table 1: Slope (a) and Intercept (b) values generated for the relationship between the scoured top and Assigned MFC values of top for individual instruments

Instrument	Slope (*a*)	Intercept (*b*)
L1	1.170	7.625
L3	1.128	9.345
L4	1.238	-0.128
L5	1.147	20.877
L6	1.256	4.097
L7	1.137	8.693
L8	1.322	-1.769
L9	1.201	10.029
L11	1.104	8.762
L12	1.081	25.700
O1	1.377	-14.763
O4	1.399	-7.793
O5	1.338	-8.126
O6	1.384	-7.686
O7	1.428	-10.088
O8	1.405	-9.917
O9	1.384	-13.588
O10	1.378	-8.267

Application of the mfc calibration to SCOURED series 14 ih calibration top

The slope and intercept values in Table 1 were applied to the scoured top values. The MFC calibrations were applied to the scoured top results. Figures 2 and 3 compare the MFC values for scoured top for individual instruments prior to the calibration process.

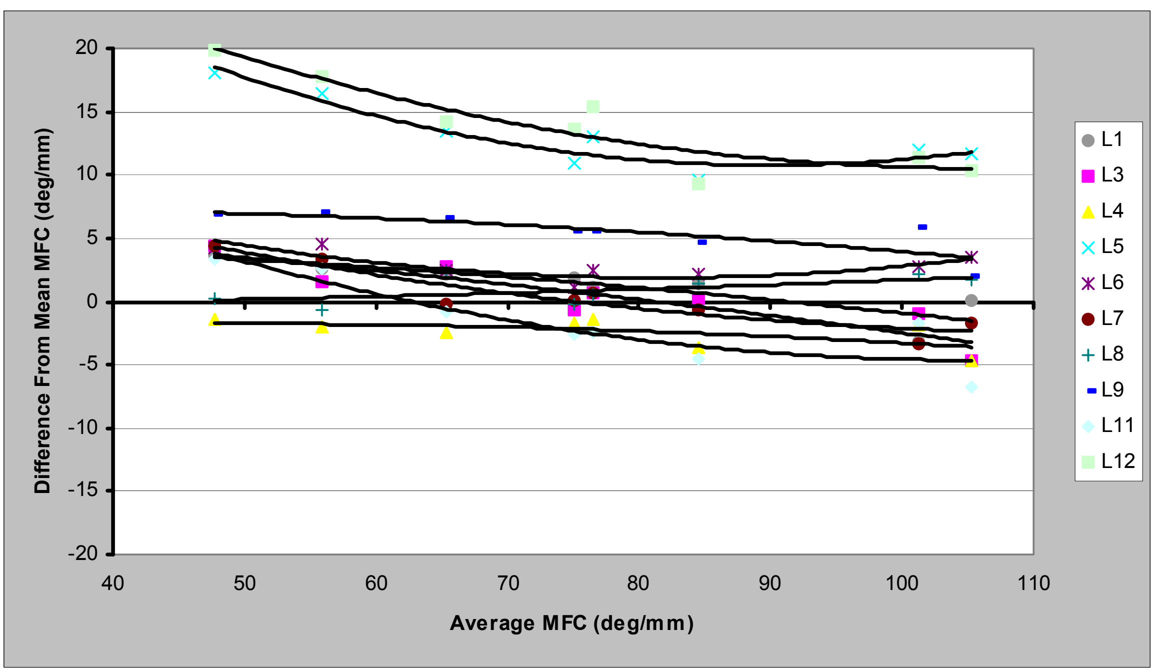

Figure 2: Difference of the scoured top results from the average MFC value, for individual Laserscan instruments.

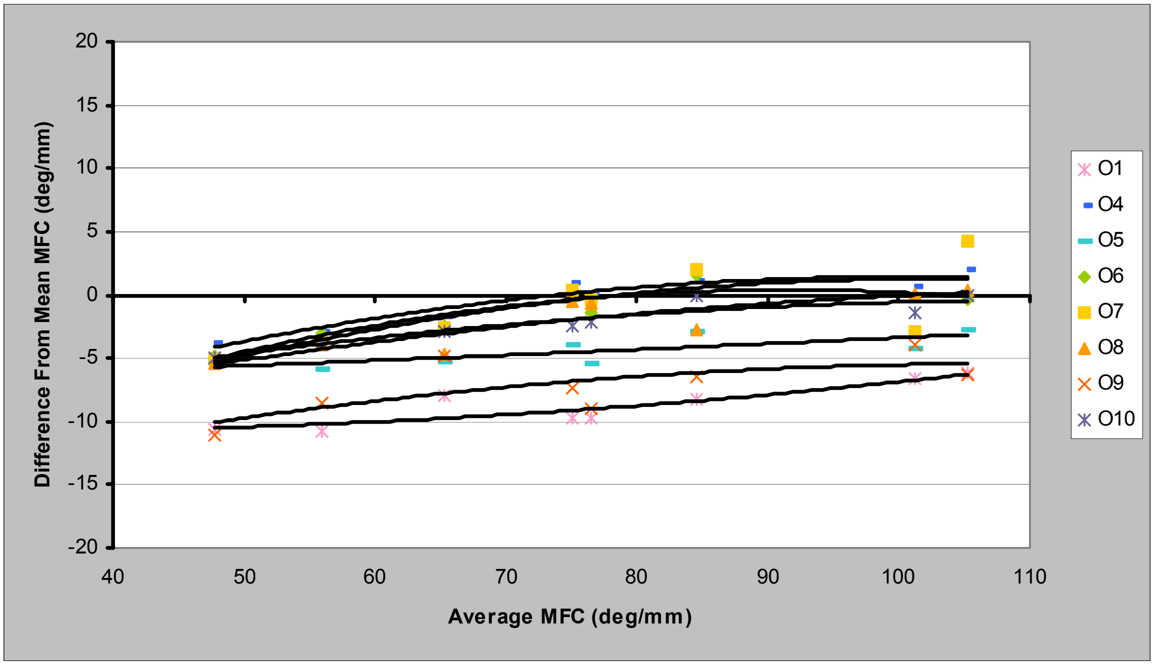

Figure 3: Difference of the scoured top MFC from the average MFC value, for individual OFDA100 instruments.

Table 2 shows the decrease in the average differences for the scoured top results when the calibration correction equation was applied to the data. However, Table 2 also indicates that while the calibration process reduced the average difference of the mean for individual instruments, the within instrument variance for 9 of the 18 instruments was increased.

Table 2: Comparison of the effect of a calibration to the scoured Series 14 Calibration top with the pre-calibration values.

Type	Instrument	Before Calibration		After Calibration	
		Average Difference (°/mm)	Variance of Difference (°/mm)^2	Average Difference# (°/mm)	Variance of Difference (°/mm)^2
Laserscan	L1	1.2	4.3	0.0	2.7
	L3	0.4	7.6	0.0	7.2
	L4	-2.4	1.4	0.0	5.1
	L5	13.1	8.0	0.0	6.2
	L6	2.9	1.3	0.0	4.5
	L7	0.3	6.2	0.0	2.1
	L8	0.9	1.2	0.0	5.7
	L9	5.5	2.6	0.0	7.2
	L11	-1.6	11.3	0.0	9.6
	L12	14.0	13.6	0.0	4.9
Laserscan Average		3.4	5.7	0.0	5.5
OFDA100	O1	-8.8	3.0	0.0	5.7
	O4	-0.4	4.4	0.0	3.4
	O5	-4.4	1.5	0.0	3.6
	O6	-1.3	4.2	0.0	5.1
	O7	-1.0	9.8	0.0	2.5
	O8	-2.2	5.2	0.0	3.4
	O9	-7.2	5.4	0.0	10.1
	O10	-2.2	2.9	0.0	3.9
OFDA100 Average		-3.4	4.5	0.0	4.7
Grand Average		0.0	5.1	0.0	5.1

#The process of calibration will set the average difference to zero, this value has been provided for consistency of reporting.

Comparisons of the scoured top values for individual instruments after calibration are presented in Figures 4 and 5. Both figures demonstrate that the calibration has reduced the differences between instruments.

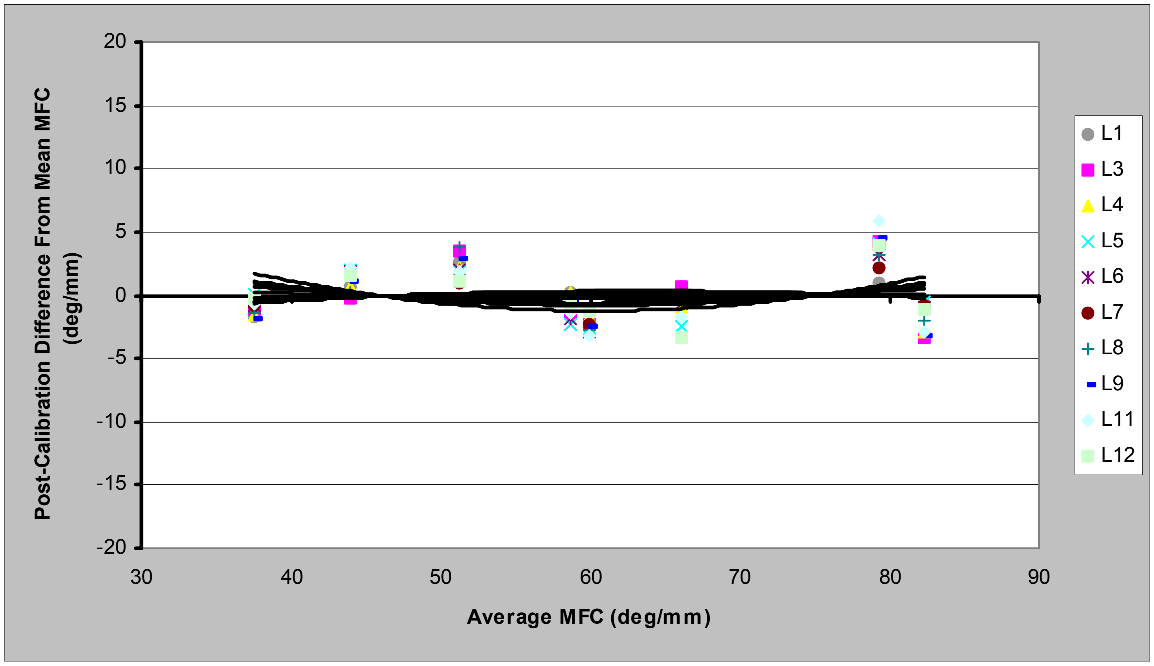

Figure 4: Difference from the Mean MFC value for scoured top measured using Laserscan, after a calibration correction has been applied.

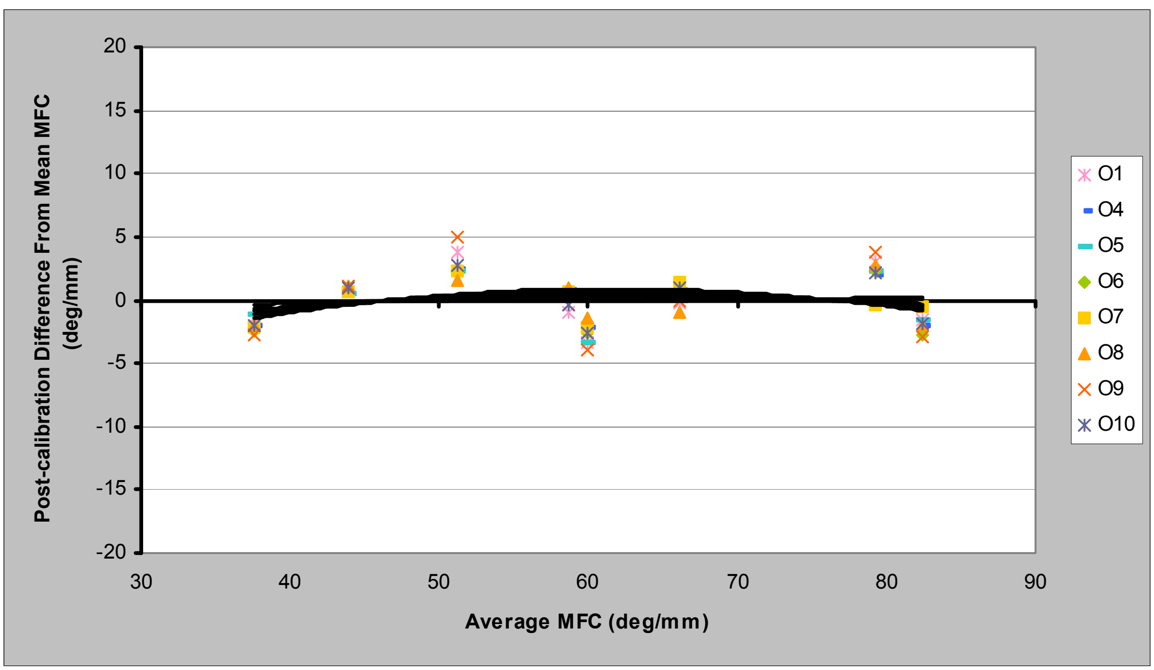

Figure 5: Difference from the Mean MFC value for scoured top measured using OFDA100, after a calibration correction has been applied.

Application of the mfc calibration to raw wool

Figures 6 and 7 show the relationship between the Mean MFC value of the 42 raw wool samples, and the raw wool measurements for individual instruments prior to the calibration process.

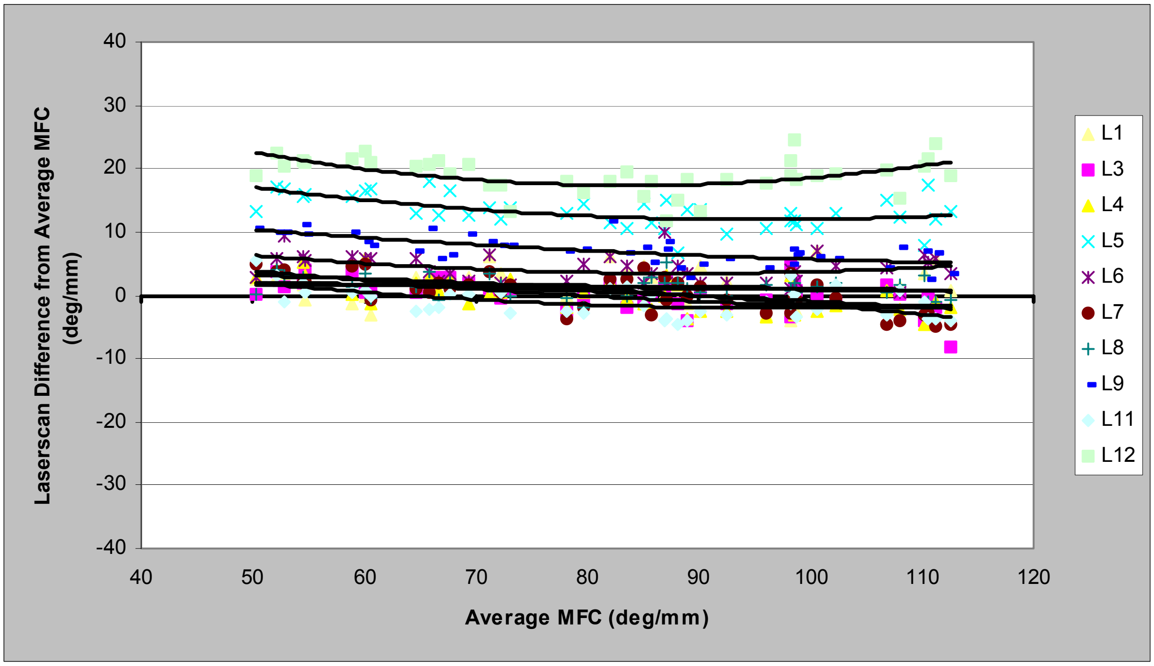

Figure 6: Relationship between the un-calibrated MFC values for raw wool, measured on individual Laserscan instruments.

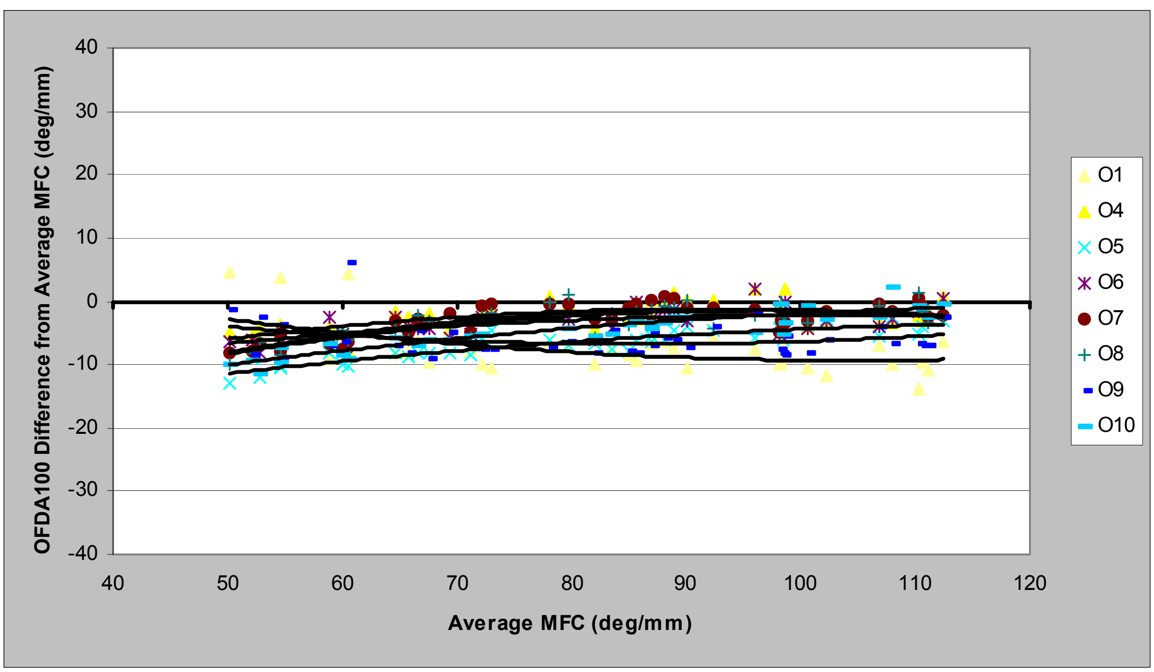

Figure 7: Relationship between the un-calibrated MFC values for raw wool, measured on individual OFDA100 instruments.

Figure 6 shows that there was a difference of approximately 20 deg/mm between instruments for Laserscan instruments. Figure 7 indicates that the difference between OFDA100 instruments is approximately 10°/mm. This indicates that there is a potential range of 30 deg/mm between the results of the same sample when measured using different instruments.

Table 3 shows the effect of the calibration process on the results of individual instruments. Prior to calibration there was a mean difference of 4.6 deg/mm between Laserscan instruments, and –4.6 deg/mm between OFDA100 instruments. The calibration process reduced the average between instrument difference to 0 deg/mm for each instrument type. Also, the between instrument variation was reduced from 6.2 deg/mm^2 to 4.5 deg/mm^2 for Laserscan, and from 7.7 deg/mm^2 to 3.7 deg/mm^2 for OFDA100. The average variance across all instruments was reduced from 6.9 deg/mm^2 to 4.1 deg/mm^2 for all instruments.

Table 3: Relationship between the average difference and variance of the difference from the mean MFC value of raw wool, before and after the MFC calibration was implemented, for individual instruments.

Type	Instrument	Before Calibration		After Calibration	
		Average Difference (°/mm)	Variance of Difference (°/mm)^2	Average Difference (°/mm)	Variance of Difference (°/mm)^2
Laserscan	L1	1.4	8.0	0.4	7.0
	L3	0.3	6.2	0.4	4.1
	L4	0.1	6.9	2.0	3.3
	L5	13.2	6.7	0.5	4.1
	L6	4.2	4.6	1.0	2.6
	L7	0.4	8.3	0.5	3.3
	L8	1.2	2.5	0.0	1.9
	L9	7.2	5.1	1.5	1.9
	L11	-1.0	5.5	1.2	5.3
	L12	19.0	7.8	5.3	11.9
Laserscan Average		4.6	6.2	1.3	4.5
OFDA100	O1	-7.5	14.5	0.5	11.8
	O4	-2.4	5.2	-1.9	2.5
	O5	-6.6	6.7	-1.9	1.9
	O6	-3.4	5.1	-2.0	1.9
	O7	-2.9	6.4	-1.9	2.1
	O8	-3.2	6.7	-1.3	1.9
	O9	-6.0	7.6	0.4	5.9
	O10	-4.4	9.2	-2.1	1.9
OFDA100 Average		-4.6	7.7	-1.3	3.7
Grand Average		0.0	6.9	0.0	4.1

Figures 8 and 9 show the MFC values of the raw wool after the calibration process was applied. A majority of the Laserscan raw wool MFC values are parallel to the mean, except for instrument 12. This instrument displayed the largest difference from the mean in Figure 6, therefore it is not surprising that this instrument does not correct as well as the other instruments. If Laserscan 12 is excluded, then there is an average difference of 0.8deg/mm between Laserscan instruments.

The relationship displayed in Figure 9 shows that the calibration process was also successful for OFDA100. However, it appears that the calibration process introduced a slight slope into the relationship, which is possibly an artefact of combining the Laserscan and OFDA100 values to generate a mean MFC value. There was a notable improvement in the relationship between OFDA100 instruments, which is supported by the results in Table 4.

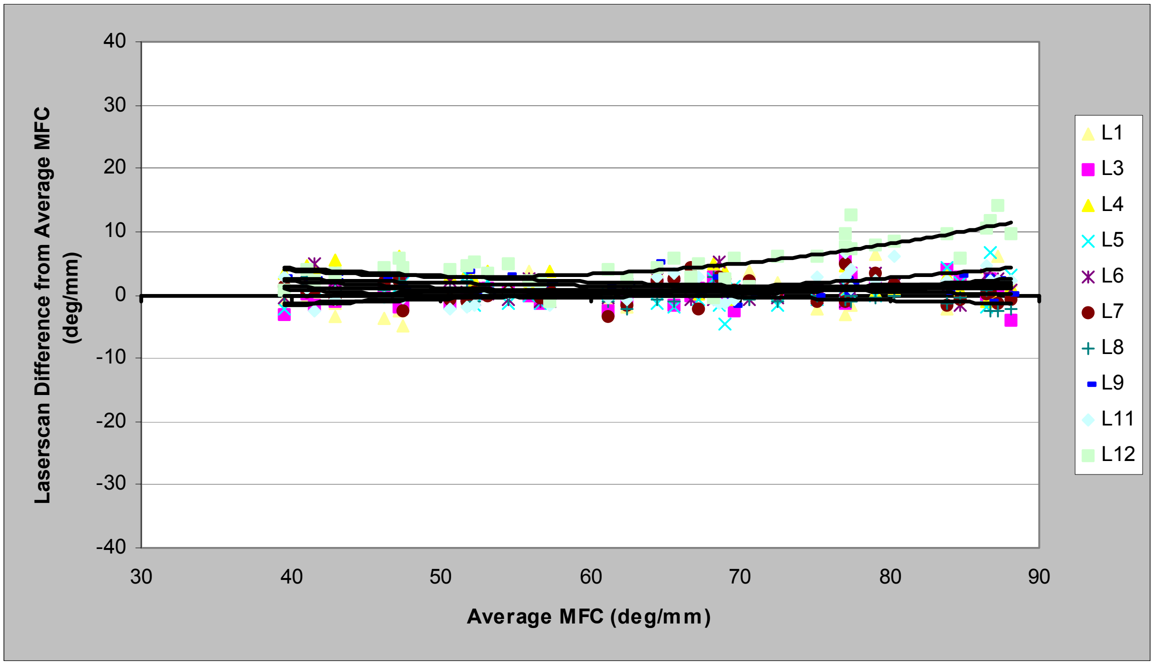

Figure 8: Relationship between the calibrated MFC values for raw wool, measured on individual Laserscan instruments.

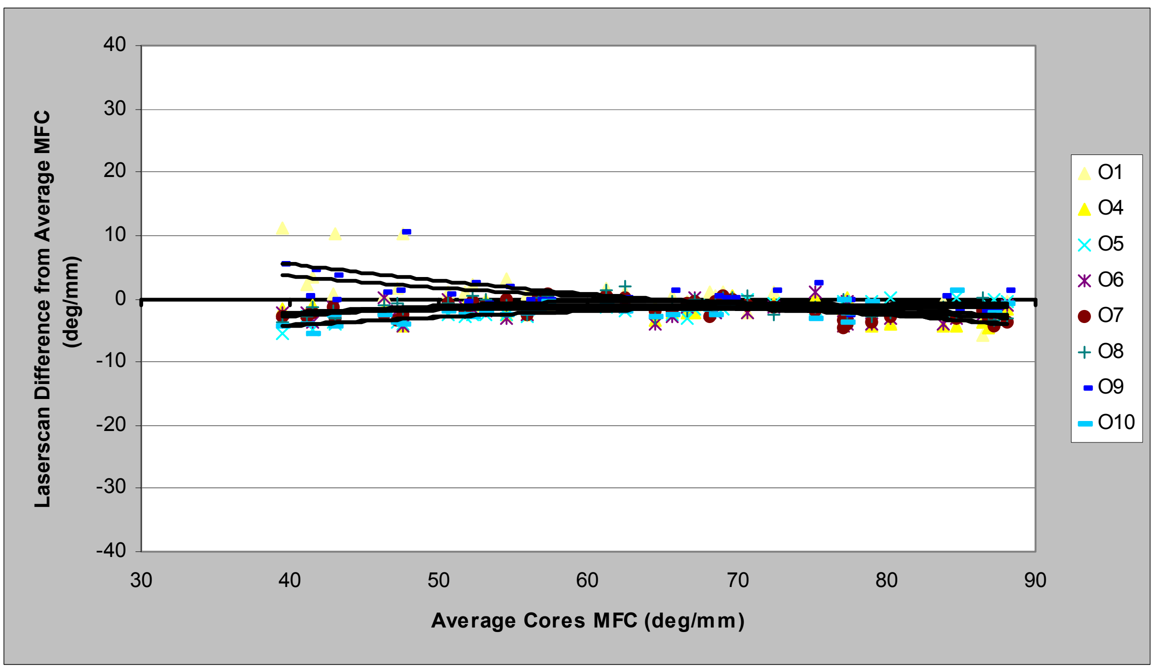

Figure 9: Relationship between the calibrated MFC values for raw wool, measured on individual OFDA100 instruments.

Table 4 shows the between instrument and between instrument-type SD before and after the calibration process was applied to the raw wool results. Calibration had the effect of decreasing the between instrument confidence interval from ±14.1 deg/mm to ±5.1 deg/mm.

Table 4: The between instrument SD and confidence interval before and after a calibration correction equation was applied to the raw wool data.

	Before Calibration (deg/mm)	**After Calibration (deg/mm)**
Between Laserscan SD	6.9	2.4
Between OFDA100 SD	3.0	1.7
Between Instrument SD	7.2	2.6
Average Confidence Limit for Instruments	±14.1	±5.1

CONCLUSION

Application of the calibration procedure had the effect of reducing the between Laserscan SD of MFC from 6.9deg/mm to 2.4deg/mm. Likewise, the calibration process reduced the between OFDA100 SD of MFC from 3.0deg/mm to 1.7deg/mm. The overall between instrument SD was reduced from 7.2 deg/mm to 2.6 deg/mm, resulting in a Confidence Interval of ±5.1 deg/mm between instruments after a calibration process has been applied to the raw wool data.

The results presented show that the use of Interwoollabs IH Calibration top to calibrate the preparation of MFC measurement provides an opportunity for the industry to harmonise the process of MFC measurement within and between individual laboratories.

The Interwoollabs IH Calibration top was used as the basis for the calibration because it is the material currently utilised for the calibration of MFD. With industry support, it would be possible to collect MFC values, along with the MFD values, during the Interwoollabs round-trails on guillotined tops. Using the collected data, MFC values would then be assigned to the relevant IH calibration tops. This method assigns values in the same method that was used by Interwoollabs for the development of calibration standards for Airflow.

Further work will be required to determine the between laboratory and within laboratory between instruments confidence limits for MFC, after the calibration process has been applied.

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