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An International Round Trial Comparing Certified Tile Calibrations

to Standard Wool Top Calibrations for Greasy Wool

By

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1. SUMMARY

The results of an international round trial comparing colour calibration using Tiles and a Reference Wool for greasy wool are reported. It is shown that wool colour measuring instruments can be successfully calibrated using a Certified Tile Calibration and Illuminant D65 with the 10° Observer.

Table A: Comparison of 95 % Confidence Limits for a Single Test in One Laboratory

	Tile Calibration (D65/10°).	Wool Calibration (D65/10°)	Wool Calibration (C/2°)	Draft Standard* DTM - 56
X	2.0	2.1	2.0	2.2
Y	2.1	2.0	1.8	2.3
Z	2.2	2.4	2.5	3.0
Y-Z	0.9	1.0	1.1	1.0 (1.5)

* Waring Blender Limits. Carding limits for Y-Z only in Brackets.

The trial results indicate that there is no difference in precision when comparing Wool and Tile calibrations with the current requirements of the draft standard.

The results suggest the industry can immediately adopt the tile calibration system. Baremes can be provided which relate the new colour values obtained in D65/10° colour space to those obtained in the C/2° colour space.

2. INTRODUCTION

This paper reports the results of an international colour round trial designed to compare two calibration systems for the measurement of wool colour on samples of greasy cores that have been cleaned prior to measurement. The paper is a continuation of the top trial reported at the last IWTO Conference in Nice.

During the late 1970's, the Wool Research Organisation of New Zealand (WRONZ) developed a Reference Wool to calibrate instruments used in measuring wool colour. This was done in response to unacceptable variation between colour measuring instruments and resulted in better agreement between such instruments[1]. However, such a calibration material does not measure colour in directly traceable CIE units due to the absorption of light by the glass of the WRONZ Wool Cell. CIE (Commission Internationale de l'Eclairge) is the organisation responsible for colour measurement standards. Furthermore, it has disadvantages such as being subject to soiling, and photobleaching by fluorescent light. While the calibration based on Reference Wool is transferred to a ceramic tile, the Reference Wool itself can only be used once. Thus, it would be desirable to have a more durable, certified, and traceable calibration standard available to calibrate colour measuring instruments.

Recent work[2] demonstrated that a Certified Tile Calibration can produce results that are linearly related to a Standard Wool Calibration when samples of top are measured. All laboratories that participated in the trial had their instruments calibrated with the same set of certified tiles. A more recent round trial[3] using tops reported a small increase in the precision limits for X, Y and Z Tristimulus values and no change in the precision of yellowness (Y-Z) for wool tops when each laboratory used its own calibration tiles.

Changing to a Certified Tile Calibration will cause an increase of several units in the certified results[4,5]. Furthermore, D65/10° produces slightly different results to C/2° for any given calibration. Hence, they should be adopted at the same time to minimise disruption to the industry. A bareme between the Wool-C/2° Calibration the Tile-D65/10° Calibration has also been established. This will assist in understanding the transition from the old to the new system. It should be noted that the bareme is indicative of what one would expect, but is not necessarily appropriate for each individual situation. However, it is still the best guide available for such comparisons.

This study reports the precision limits for X, Y, Z and (Y-Z) for cores using Illuminant C and the 2° Observer (C/2°) on a Standard Wool Calibration, and using Illuminant D65 and the 10° Observer (D65/10°) for the Certified Tile Calibration. The move to D65/10° is prompted by other industries such as the dyeing industry which use this colour space. It is also recognised as being the standard illuminant and viewing angle throughout the colour industry.

3. MATERIALS AND METHODS

3.1 Laboratories and Instruments

Five laboratories took part in the trial, using several different types of instruments. The laboratories were:

1. SGS Wool Testing Services - New Zealand
2. New Zealand Wool Testing Authority Ltd - Napier (NZWTA)
3. Australian Wool Testing Authority Ltd - Fremantle
4. Australian Wool Testing Authority Ltd - Melbourne
5. Australian Wool Testing Authority Ltd - Sydney

Note: 1. The numbers above are arbitrary and are not related to the laboratory numbers described later in the report.

2. NZWTA used both preparation methods, viz Shirley Analyser (SA) and Waring Blender (WB). For analysis purposes they were considered as two separate laboratories.

In an earlier trial[2], an instrument with a 0°/45° geometry could not be successfully calibrated with a Certified Tile. As such, all the instruments in this trial were of the 45°/0° geometry.

The participating laboratories all used spectrocolorimeters; each using one of the following types:

BYK-Gardner TCM
Hunterlab ColorQuest

3.2 Calibration Materials

Two calibration materials were used in order to make a comparison between the current situation involving WRONZ Reference Wool and that of a proposed Certified Tile.

3.2.1 Standard Wool

This was prepared to emulate the traditional Reference Wool Calibration. A BYK-Gardner TCM spectrocolorimeter was calibrated with the existing Reference Wool Calibration. Approximately 200 grams of commercially produced top was cut and Shirley Analysed. A 15 gram subsample was then taken for each laboratory and measured using an AWTA Ltd designed wool cell. Each sample was assigned its own set of tristimulus and reflectance values, as would occur with Reference Wool.

Each laboratory also received an uncertified ceramic 'B4' tile, to which the Standard Wool Calibration was transferred. This was done to enable the transfer of a Reference Wool Calibration to a tile as described in the Draft Test Method.

3.2.2 Certified Tiles

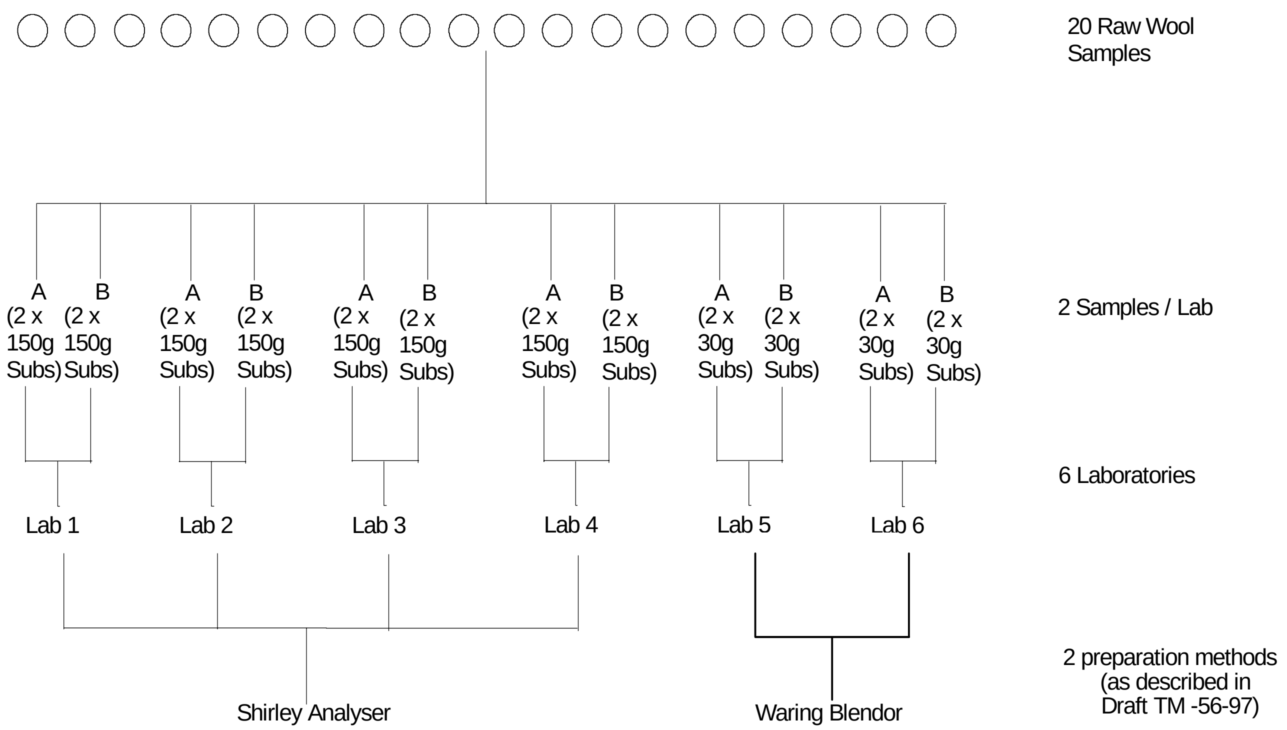
CERAM of the United Kingdom has developed certified colour tiles that can be successfully used to calibrate colour instruments[2]. Each participating laboratory purchased a set of these tiles. The black and white tiles were used to calibrate each instrument to CIE colour space. In addition, all the tiles in the set were used to ascertain the glass correction factors.

The calibration so generated was transferred to the same B4 tile that was used for the Standard Wool Calibration, with the transfer being carried out against the instrument port. This tile was then used (along with the black tile) to calibrate behind glass. Appropriate equations (as described in DRAFT TM-56-97[7]) were then used to correct for the effects of the glass.

3.3 Raw Wool Samples

Twenty different raw wool samples were blended and then weighed into 16 x 150 grams sub-samples for Shirley Analyser preparation and 8 x 30 grams sub-samples for Waring Blendor preparation. Two sub-samples were selected at random and packaged together to represent a sample in Figure 1. To comply with IWTO-0 each laboratory received a blind duplicate sample (denoted A and B in Figure 1) for each of the 20 wools(i.e. 40 individual subsamples). The wool samples were all sourced in New Zealand and covered a range in Yellowness (Y-Z) from -2 to 14.

Figure 1: Schematic of trial design



4. LABORATORY PROCEDURES

4.1 Instrument Check

To ensure the instruments to be used in the trial could measure satisfactorily, an instrument check was carried out prior to commencement of the trial. The instrument was calibrated with the CERAM white tile against the instrument port and all the CERAM tiles were then measured. If the tiles could not return a value within ±1.0 unit of the certified value, the instrument was deemed to be incapable of measuring in CIE colour space and was not to be used in the trial. All the instruments passed this test.

4.2 Transfer of Calibrations

In order to mimic a normal laboratory situation, each of the participants transferred both the Standard Wool Calibration and Certified Tile Calibration to the B4 tile supplied by AWTA Ltd.

4.3 Glass Correction

When measuring samples on an instrument that has been calibrated using a Certified Tile, it is necessary to apply corrections to take account of the glass in order to retain traceability[2,7]. Participants followed the procedure outlined in DRAFT TM-56-97[6] to calculate these laboratory/instrument-specific glass correction equations.

4.4 Preparation of Material

Each laboratory prepared the samples prior to measurement according to DRAFT TM-56-97[6].

4.5 Measurement

Wool Calibration, Illuminant C/2°

The colour instrument was set to the C/2° configuration and calibrated behind glass with the B4 tile. This was done using the calibration that had been transferred from the Standard Wool. All samples were then measured according to DRAFT TM-56-97[6].

Tile Calibration, Illuminant D65/10°

The instrument was then changed to the D65/10° configuration and again calibrated behind glass with the B4 tile. This time, the calibration that had been transferred from the CERAM white tile was used. All samples were again measured according to DRAFT TM-56-97[6]. Although the range limits would probably be affected by a change in the magnitude of the result, the current range limits were used in order to simplify the procedure. The corrections for glass were then applied to the results on the Certified Tile calibration.

4.6 Terminology for Different Calibrations

The results obtained by the two methods were compared and analysed. Due to the need to make valid comparisons between the two calibration techniques, it was necessary to convert the measured values to the same colour space using a suitable relationship. In order to minimise confusion, the following terminology will be used:

- Wool C/2° — results obtained from calibrating with the Standard Wool
- Tile D65/10° — results obtained from calibrating with the Certified Tile (including glass correction)
- Wool D65/10° — results from wool calibration, converted to the Certified Tile D65/10° colour space using the appropriate equation (see 5.2).
- Tile C/2° — results from tile calibration (including glass correction), converted to the Standard Wool C/2° colour space using the appropriate equation (see 5.2).

5. RESULTS AND DISCUSSION

5.1 Core Measurements

A summary of the results obtained on the two calibrations is shown in Table 1. It can be seen that the Certified Tile Calibration results are higher by several units than the results obtained using the Standard Wool Calibration. This is the result of two changes in the calibration. It has been shown previously[4,5] that a Certified Tile Calibration causes an increase in the magnitude of colour values as it is measuring in a traceable CIE colour space, whereas a Standard Wool Calibration includes a loss of energy through absorption by the glass of the WRONZ wool cell in the Zeiss PMQ3 primary instrument[9]. Furthermore, the change from C/2° to D65/10° will also have an impact on the measured results due to the different colour space in which the samples were measured.

Table 1: Laboratory Averages for Standard Wool calibration and Certified Tile calibration

Laboratory	Certified Tile Calibration (D65/10°)				Standard Wool calibration (C/2°)			
	X	Y	Z	Y-Z	X	Y	Z	Y-Z
1	59.7	62.6	47.6	14.8	54.0	55.3	47.1	8.1
2	59.2	61.9	47.3	14.7	54.4	55.7	47.7	8.1
3	57.7	60.4	45.7	14.8	53.3	54.6	46.6	8.0
4	58.7	61.5	46.1	15.3	53.8	55.0	46.5	8.6
5	58.9	61.4	46.0	15.4	55.6	56.5	49.2	7.3
6	59.9	62.6	47.5	15.0	55.1	56.2	47.9	8.3
Range	2.2	2.2	1.9	0.7	2.3	1.9	2.7	1.0

The between-laboratories range for the Certified Tile Calibration is lower for Z (1.9) than the Standard Wool Calibration (2.7) and the ranges for tile and wool calibration are similiar for X, Y and Y-Z.

5.2 Comparisons Between Calibrations (ie Wool C/2° versus Tile D65/10°)

Previous research[4,5] has shown that there is a linear relationship between a Certified Tile calibration and a Reference Wool calibration. Furthermore, analysis of the data from the current trial demonstrates there is a linear relationship between a Standard Wool calibration using C/2° and a Certified Tile calibration using D65/10°. Figure 2 illustrates the relationship for Y. Table 2 shows that X and Z also exhibited a linear response.

Figure 2: Relationship between Wool and Tile Calibrations for Y (based on grand means)

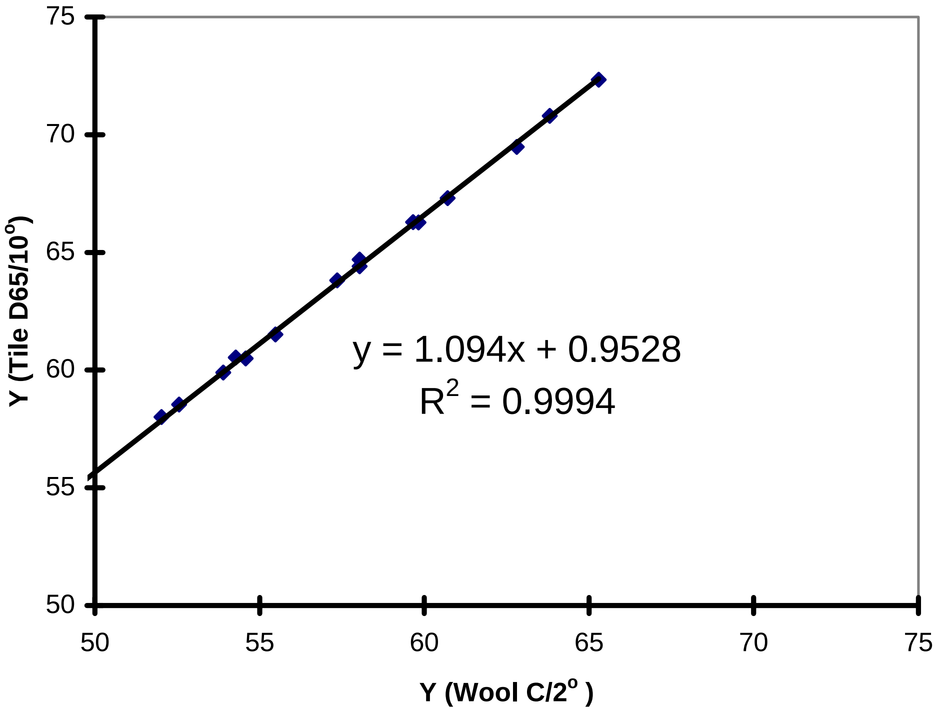


Table 2 gives the transformation equations for converting between the two calibration types for each of X, Y and Z. These have been generated from the grand mean for each sample. The high correlations and low standard errors indicate the robustness of the relationships in all three instances.

Table 2: Transformation equations for converting values generated using a Standard Wool calibration (C/2°) to a Certified Tile calibration (D65/10°) colour space

Equation (standard errors in parentheses)	R^2	SE
X_T = 1.026 (±0.006) X_W + 3.226 (±0.339)	0.9993	0.0198
Y_T = 1.094 (±0.006) Y_W + 0.953 (±0.361)	0.9994	0.0239
Z_T = 0.971 (±0.005) Z_W + 0.596 (±0.225)	0.9996	0.0336

Such relationships can be used to convert from a Standard Wool Calibration to a Certified Tile Calibration and *vice versa*. During the change from C/2° to D65/10°, it may be desirable to report colour results in both the new and old colour spaces for an interim period. This will allow all sectors of the industry to adapt to the new values over a period of time.

The equations reported in this paper differ slightly form those reported in the the top paper[3]. As the top paper included more laboratories and hence more data it is reccommended that the equations developed in the earlier paper be used in the standard.

Table 3 gives an indication of the change in magnitude to be expected for Y and Z when converting from a Standard Wool Calibration to a Certified Tile Calibration. These values have been generated using the equations in Table 2.

Table 3: A comparison of Y and Z values for a wool calibration (C/2°) and a tile calibration (D65/10°)

Y			Z		
Calibration			Calibration		
Wool C/2°	Tile D65/10°	Difference	Wool C/2°	Tile D65/10°	Difference
30.0	33.8	3.8	30.0	29.7	-0.3
35.0	39.2	4.2	35.0	34.6	-0.4
40.0	44.7	4.7	40.0	39.4	-0.6
45.0	50.2	5.2	45.0	44.3	-0.7
50.0	55.7	5.7	50.0	49.2	-0.8
55.0	61.1	6.1	55.0	54.0	-1.0
60.0	66.6	6.6	60.0	58.9	-1.1
65.0	72.1	7.1	65.0	63.7	-1.3
70.0	77.5	7.5	70.0	68.6	-1.4

The typical change expected for Y-Z is presented in Table 4. The results were calculated using the relevant equation in Table 2. This bareme was only established to give an indication of the likely differences between the two types of calibration for Y-Z. The typical change for Y-Z is presented in Table 4.

Table 4: A comparison of Y- Z values for a wool calibration (C/2°) and a tile calibration (D65/10°)

Calibration		
Wool C/2°	Tile D65/10°	Difference
-2.0	7.1	9.1
-1.0	7.9	8.9
0.0	8.7	8.7
1.0	9.5	8.5
2.0	10.3	8.3
3.0	11.0	8.0
4.0	11.8	7.8
5.0	12.6	7.6
6.0	13.4	7.4
7.0	14.2	7.2
8.0	15.0	7.0
9.0	15.8	6.8
10.0	16.5	6.5
11.0	17.3	6.3
12.0	18.1	6.1
13.0	18.9	5.9
14.0	19.7	5.7

By using the equations in Table 2, the results obtained on the wool calibration can be converted to give results in the same colour space as the Certified Tile calibration results.

Figures 3 and 4 show the difference between these sets of results for Y. The axes on these graphs have been chosen to highlight differences between the laboratories. It should be noted that research has shown that "observers could not distinguish any differences less than about 1.8 in Y or 1.1 in Y-Z" [9]. Comparing Figures 3 and 4 indicates that there is no difference between the two calibration techniques. The range between laboratory results and the grand mean is of the order of 2 units.

Figure 3: Differences from Grand Mean for Y on Tile Calibration - D65/10°

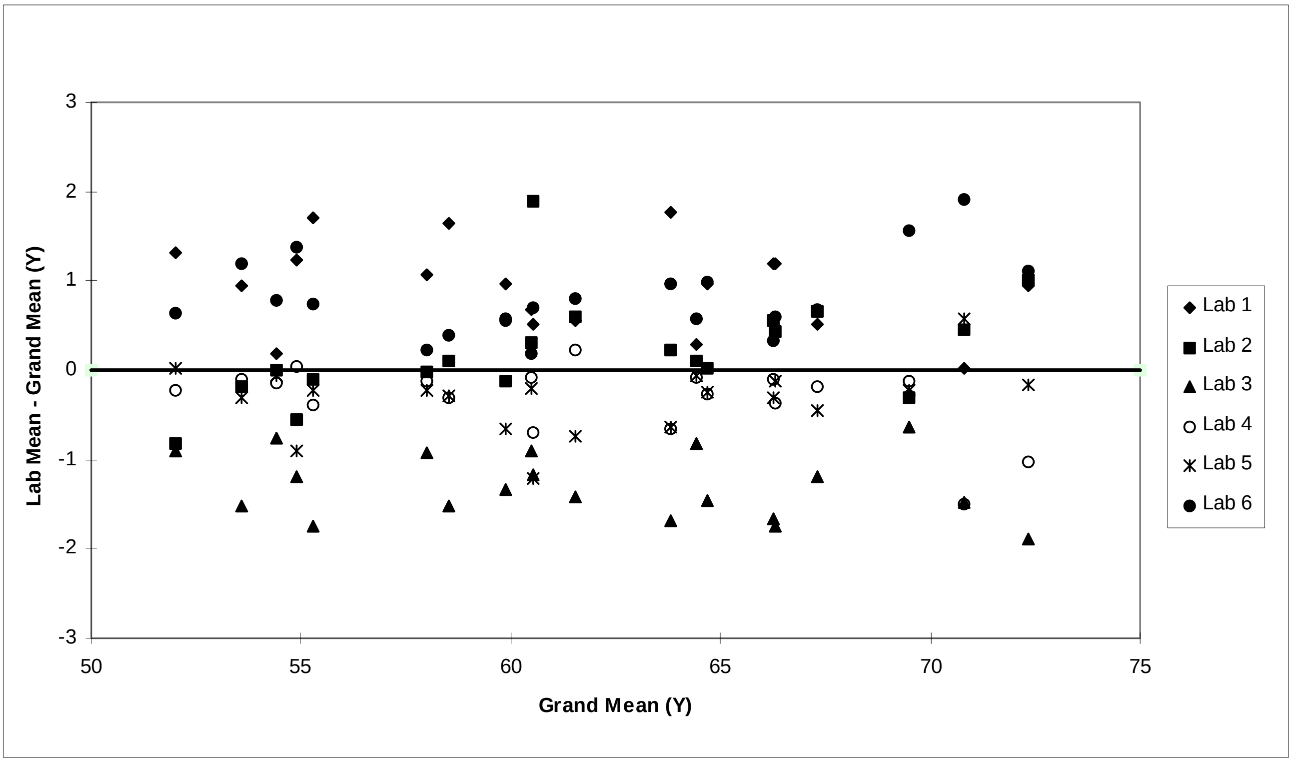
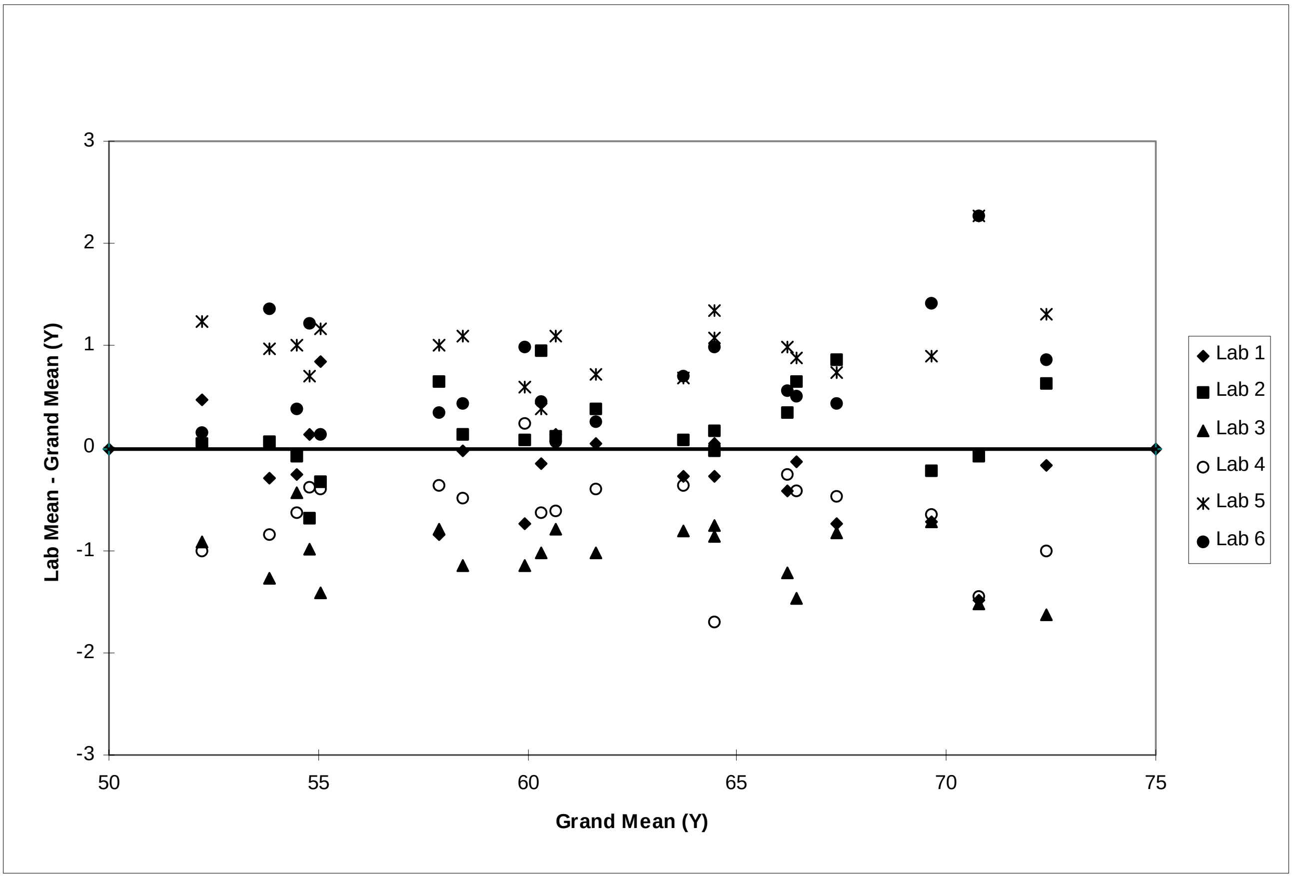


Figure 4: Differences from Grand Mean for Y on Wool Calibration - D65/10°



An analysis of the within-laboratory and between-laboratories components of variance are presented in Table 5 for the Tile D65/10°, Wool D65/10° and Wool C/2° calibrations

Table 5: Within- (σ^2_W) and Between- (σ^2_B) Laboratory Components of Variance for X, Y, Z and Y-Z on both the Certified Tile and Standard Wool Calibrations

	Tile Calibration (D65/10°)		Wool Calibration (D65/10°)		Wool Calibration (C/2°)	
	σ^2_W	σ^2_B	σ^2_W	σ^2_B	σ^2_W	σ^2_B
X	0.46	0.63	0.47	0.70	0.45	0.66
Y	0.53	0.66	0.58	0.54	0.48	0.45
Z	0.67	0.75	0.72	0.90	0.76	0.95
Y-Z	0.08	0.13	0.08	0.17	0.10	0.21

Both the within-laboratory and between-laboraties variances are similar for the two calibration techniques for X, Y, Z and Y-Z. This concurs with earlier work[2] which found the components of variance were similar for both types of calibration in C/2° colour space; however, that work was based on one operator and one set of tiles. The data in this report is for greasy wool cores that have been scoured and cleaned of contamination by each particpating laboratory using its own staff and set of Certified Tiles.

Table 6: Comparison of 95 % Confidence Limits for a Single Test in One Laboratory

	Tile Calibration (D65/10°).	Wool Calibration (D65/10°)	Wool Calibration (C/2°)	Draft Standard* DTM - 56
X	2.00	2.06	2.01	2.2
Y	2.08	2.00	1.83	2.3
Z	2.24	2.44	2.51	3.0
Y-Z	0.87	0.97	1.08	1.0 (1.5)

* Waring Blender Limits. Carding limits for Y-Z only in Brackets.

The results for the precision for a single test in one laboratory are presented in Table 6 above. They are reported to two decimal places in order to highlight any differences. In the Standard, it would be appropriate to report to only one decimal place. The results for Tile Calibration using both preparation techniques (viz Waring Blender and Shirley Analyser) agree with the confidence limits in the draft standard.

Figures 5 and 6 show the laboratory differences for Y-Z for Tile D65/10° and Wool D65/10° respectively. It can be seen that tiles provide an equally precise result to wool for Y-Z. It should be noted that in the case of Figure 6 the Wool D65/10° results were obtained by transforming the Y and Z results, then calculating the Y-Z results by difference.

Figure 5: Differences from Grand Mean for Y-Z on Tile Calibration - D65/10°

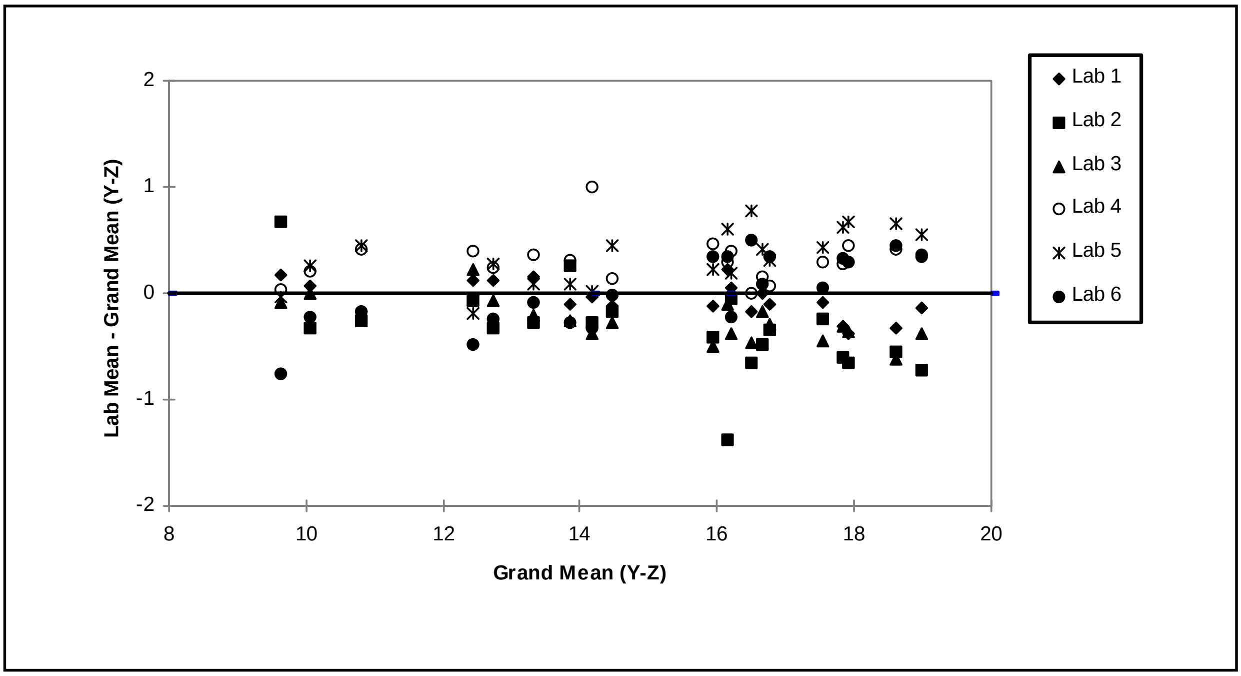
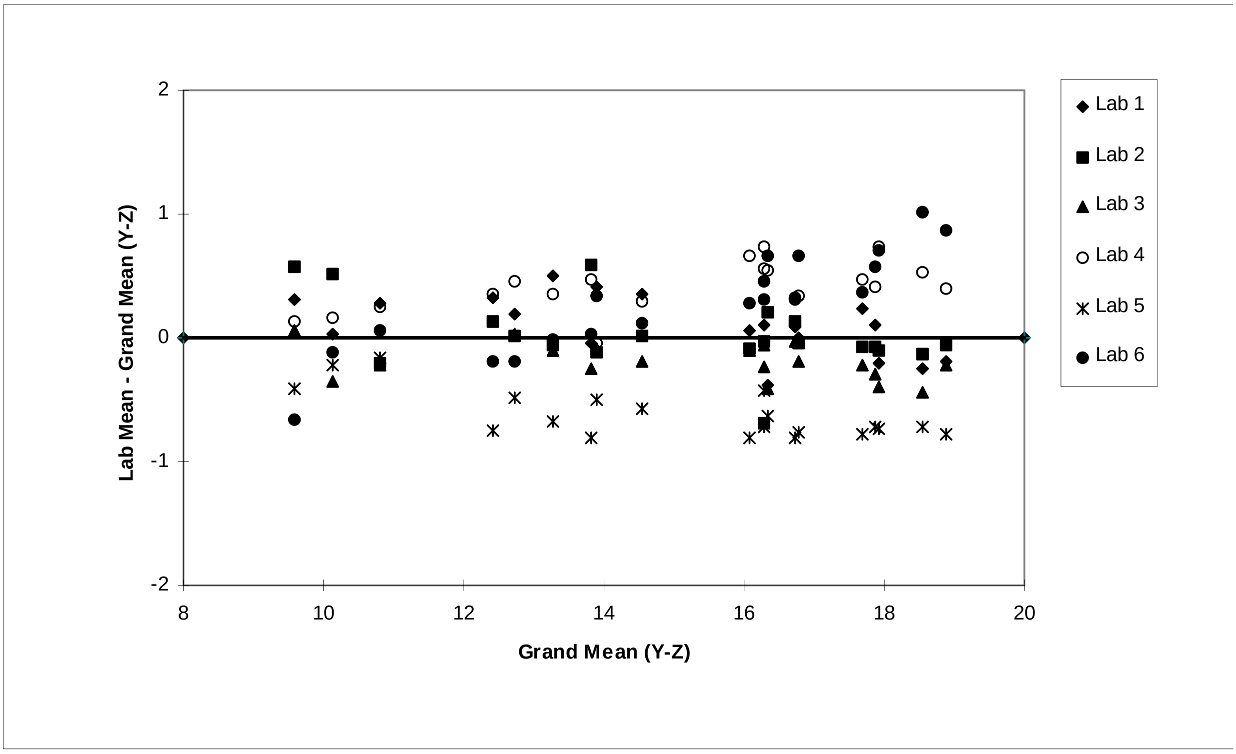


Figure 6: Differences from Grand Mean for Y-Z on Wool Calibration - D65/10°



Tables 7 and 8 in the Appendix contain the vaiance and precision data for Y and Y-Z for each wool sample.

Figures 7 and 8 show the relationship between 95% Confidence Limit and the Mean Y for both Wool Calibration (D65/10^{o}) and Tile Calibration (D65/10^{o}) respectively.

Figures 9 and 10 show the relationship between 95% Confidence Limit and the Mean Y-Z for both Wool Calibration (D65/10^{o}) and Tile Calibration (D65/10^{o}) respectively. There is some suggestion that the Confidence limit for Mean Y-Z on the Wool Calibration (D65/10^{o}) is level dependent.

6. CONCLUSIONS

Certified Ceramic tiles can be successfully used to calibrate colour measuring instruments for measuring wool samples. There is no difference in the Confidence Limits (for raw wool measurement) for each of X, Y, Z and Y-Z.

The baremes established from this trial can be used to transfer results obtained with a traditional Reference Wool calibration to that of a Certified Tile Calibration and *vice versa*.. These transformation equations will provide the means for reporting two sets of results for an interim period. The relationships reported can also be used to update any industry database to the new measurement scale. This will assist the industry in adapting to measurements reported in the new colour space.

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8. ACKNOWLEDGMENTS

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Further thanks go to Mr. Bob Couchman of the Woolmark Company for his willingness to act as an independent party in collecting the results.

9. APPENDIX

Table 7: Precision Estimates for Y

	Tiles (D65/10°)				Wool (D65/10°)			
Sample Number	Mean	Within Lab Variance	Between Labs Variance	95% Confidence Limit	Mean	Within Lab Variance	Between Labs Variance	95% Confidence Limit
1	64.7	0.1066	0.7795	1.85	64.4	0.8174	0.8854	2.56
2	63.8	0.2551	1.4442	2.56	63.7	0.2217	0.2565	1.36
3	66.3	0..2296	0.9066	2.09	66.2	0.0579	0.5956	1.58
4	67.3	0.0913	0.5219	1.53	67.4	0.1043	0.5394	1.57
5	58.5	0.3901	0.8884	2.21	58.5	0.3812	0.4075	1.74
6	53.6	0.3541	0.7762	2.08	53.8	0.4764	0.8032	2.22
7	61.5	0.4423	0.5640	1.97	61.7	0.2343	0.2741	1.40
8	52.0	0.5092	0.4788	1.94	52.2	0.2442	0.5997	1.80
9	59.9	0.4637	0.5290	1.95	59.9	0.8692	0.2155	2.04
10	70.8	0.1374	1.6659	2.63	70.8	0.2817	3.2523	3.68
11	60.5	0.2996	0.1375	1.30	60.7	0.2393	0.3272	1.48
12	66.5	0.8932	0.1724	2.02	69.7	0.2767	0.7309	1.97
13	66.3	0.2483	0.8154	2.02	66.4	0.1926	0.6691	1.82
14	72.3	0.2863	1.4249	2.56	72.4	0.5647	1.0202	2.47
15	54.9	1.1941	0.5968	2.62	54.8	2.0156	-0.2887	2.58
16	55.3	0.5491	1.0722	2.50	55.1	0.1867	0.7770	1.92
17	54.4	0.0891	0.2020	1.06	54.5	0.3742	0.1747	1.45
18	58.0	0.2546	0.2963	1.45	57.9	0.6416	0.2876	1.89
19	64.4	1.9131	-0.7359	2.13	64.5	2.3079	-0.7882	2.41
20	60.5	1.8130	0.6185	3.06	60.3.	1.0827	0.0061	2.05
Average	**61.7**	**0.53**	**0.66**	**2.08**	**61.7**	**0.58**	**0.54**	**2.00**

Table 8: Precision Estimates for Y-Z

	Tiles (D65/10°)				Wool (D65/10°)			
Sample Number	Mean	Within Lab Variance	Between Labs Variance	95% Confidence Limit	Mean	Within Lab Variance	Between Labs Variance	95% Confidence Limit
1	14.2	0.1452	0.1937	1/14	13.9	0.2334	-0.0026	0.94
2	14.5	0.1007	0.0178	0.67	14.6	0.0388	0.0987	0.73
3	12.7	0.0311	0.0482	0.55	12.7	0.0157	0.0957	0.65
4	12.5	0.0431	0.0758	0.68	12.4	0.0553	0.1446	0.88
5	16.7	0.1265	0.0282	0.77	16.7	0.1764	0.0857	1.00
6	18.6	0.0507	0.2933	1.15	18.5	0.0937	0.3754	1.34
7	15.9	0.1219	0.1034	0.93	16.1	0.0567	0.2122	1.02
8	19.0	0.0696	0.2143	1.04	18.9	0.0330	0.3030	1.14
9	16.2	0.0476	0.0548	0.63	16.3	0.0381	0.1799	0.91
10	10.0	0.1227	-0.0073	0.67	10.1	0.0799	0.0547	0.72
11	16.8	0.0410	0.0666	0.64	16.8	0.0296	0.2192	0.98
12	10.8	0.0830	0.0725	0.77	10.8	0.0263	0.0384	0.50
13	13.3	0.0304	0.0423	0.53	13.3	0.0492	0.1479	0.87
14	9.6	0.0196	0.2034	0.93	9.6	0.0258	0.1967	0.92
15	17.9	0.1833	0.2015	1.22	17.9	0.0504	0.3331	1.21
16	16.5	0.0402	0.2846	1.12	16.3	0.0284	0.2835	1.09
17	17.8	0.0262	0.2074	0.95	17.9	0.0804	0.1851	1.01
18	17.5	0.0950	0.0592	0.77	17.7	0.1096	0.1611	1.02
19	13.9	0.1033	0.0123	0.67	13.8	0.2338	0.1398	1.20
20	16.1	0.1795	0.4230	1.52	16.3	0.1539	0.2038	1.17
Average	**15.0**	**0.08**	**0.13**	**0.87**	**15.0**	**0.08**	**0.17**	**0.97**

Figure 7: Relationship between 95% Confidence Limit and Mean Y Value on Wool Calibration (D65/10°)

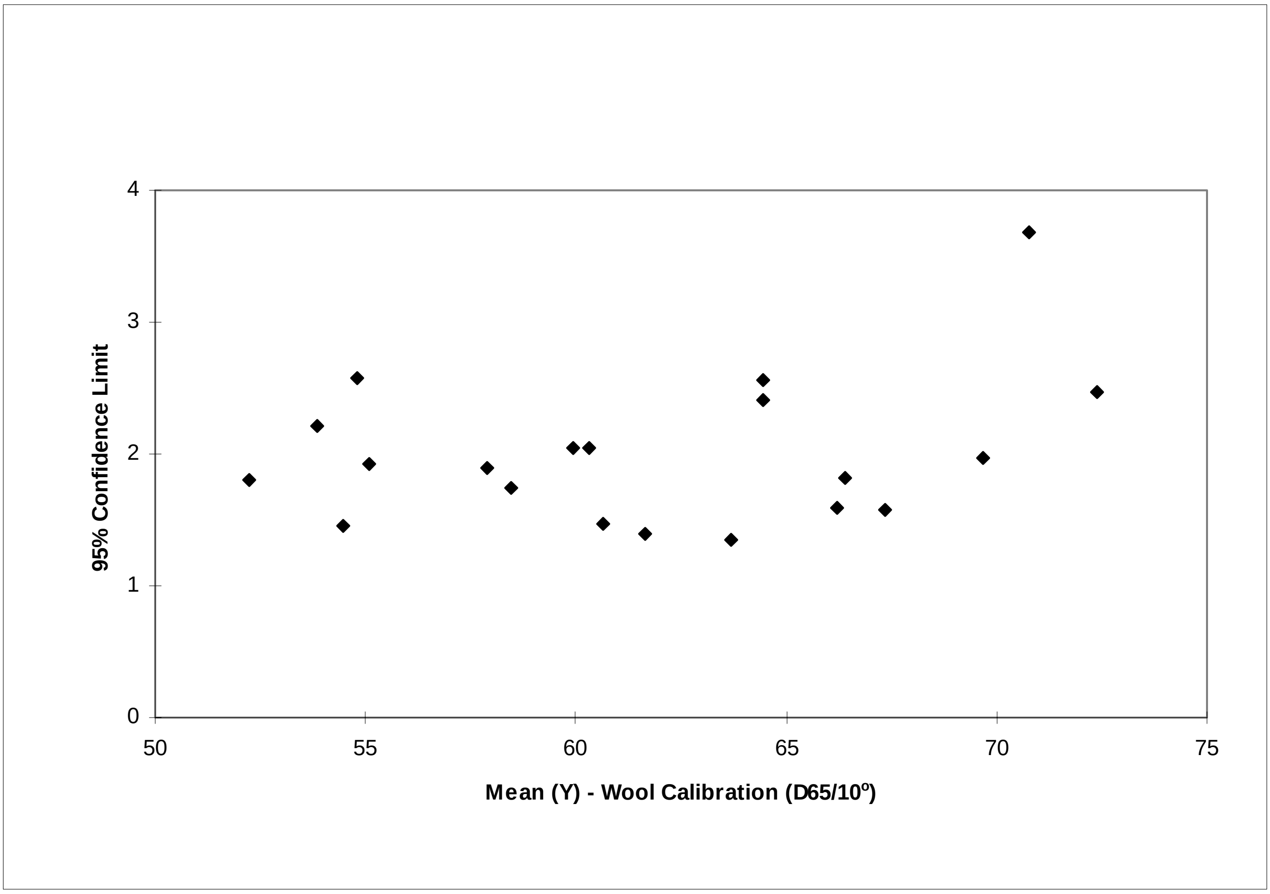

Figure 8: Relationship between 95% Confidence Limit and Mean Y Value on Tile Calibration (D65/10°)

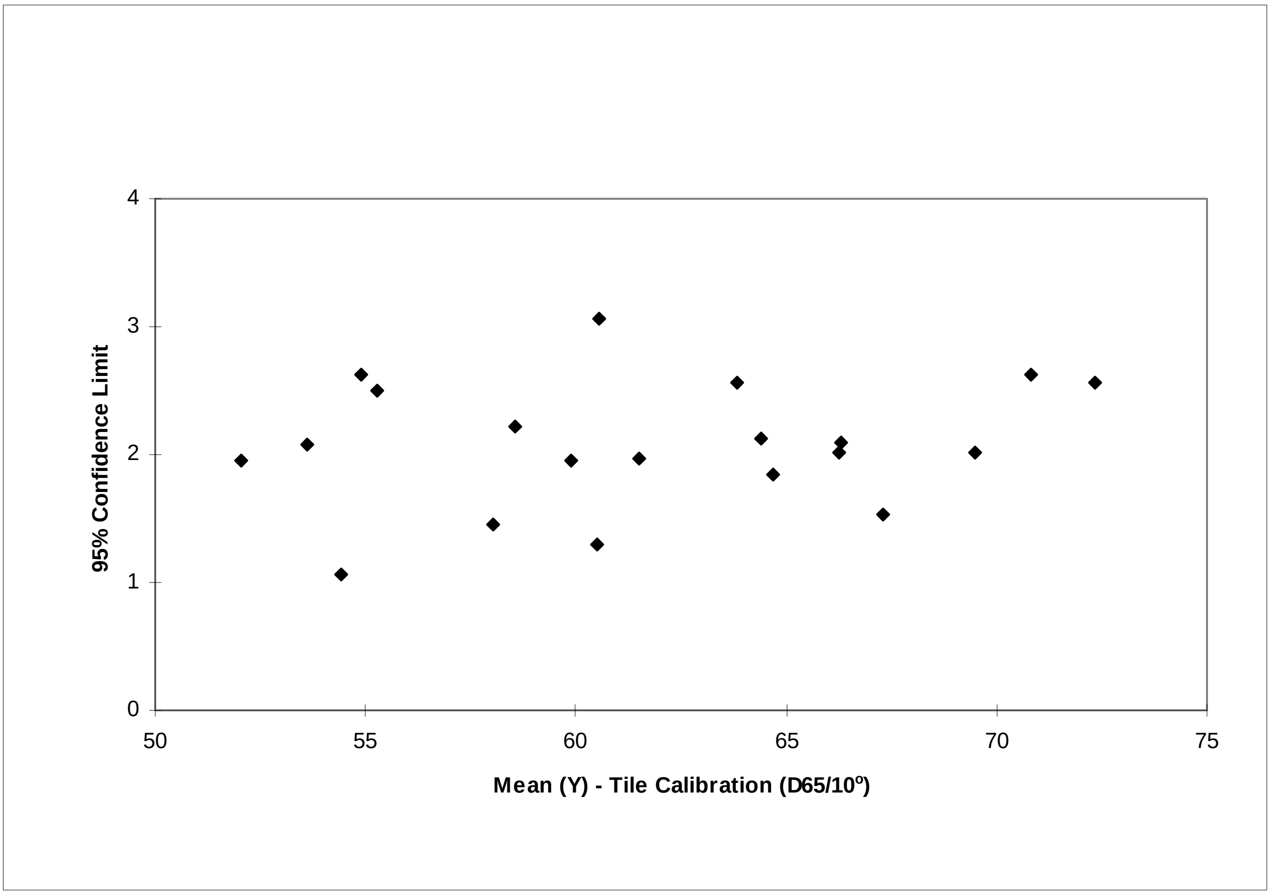

Figure 9: Relationship between 95% Confidence Limit and Mean Y-Z Value on Wool Calibration (D65/10°)

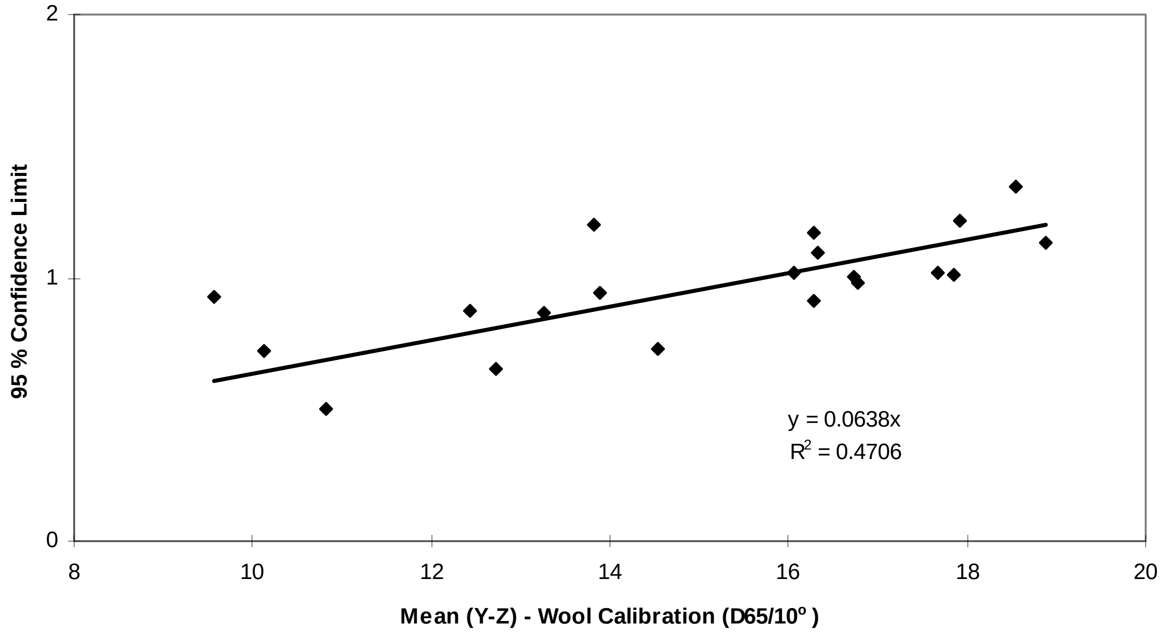

Figure 10: Relationship between 95% Confidence Limit and Mean Y-Z Value on Tile Calibration (D65/10°)