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The Calibration of Colour Instruments Using a Certified Tile and a Standard Wool Top

By

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1. Summary

An international round trial involving five laboratories was conducted to evaluate the potential of reporting wool colour measurements in a traceable CIE colour space. Two calibrations were compared: a ‘certified tile’ calibration and a ‘standard wool’ calibration. It was found that for the tile calibration one of the laboratories gave results that differed from the other participants. When this laboratory was removed from the analysis the components of variance and 95% confidence limits were similar to those obtained using wool as a calibration standard. Further investigations revealed the cause of the difference for the one laboratory was due to the particular instrument design - the most likely cause being the geometry(ie $0^0/45^0$).

2. Introduction

A report presented to the International Wool Textile Organisation in 1996[1] introduced the concept of measuring wool colour in a traceable CIE colour space. The CIE (Commission Internationale de l’Eclairge) is the International Organisation responsible for colour measurement standards, and traceability to CIE is provided by calibration with certified tiles. However, since the late 1970’s, instruments used to measure the colour of raw wool have been calibrated with Reference Wool provided by the Wool Research Organisation of New Zealand (WRONZ). The procedure to calibrate with a Reference Wool was introduced to harmonise results from different designs of instruments and wool cell holders, and early round trials confirmed the improvement in precision that was gained by this approach[2]. It has been previously shown[3,4] that there is a difference between the tristimulus results obtained using a certified tile calibration and those obtained using a Reference Wool calibration of up to 10 units.

By using a Reference Wool calibration the wool industry is not measuring colour in traceable CIE units, but rather in a colour space that includes any effects arising from the WRONZ wool cell[2]. The adoption by many companies of quality systems such as ISO 9000 has led to an effort to provide a colour measurement system which is directly traceable to international standards. Porter[5] states that “companies the world over are instituting the ISO standards and thus creating a unified language of quality control and quality assurance”. This will increase the demand for traceable colour measurements. An additional benefit of measuring in a CIE colour space is that it may bring raw wool colour measurement in line with the colour measurement procedures of later stage processors (e.g. dyers), who do not calibrate with Reference Wool and do not measure the wool in a wool cell. The calibration used by wool dyers is generally the manufacturer’s calibration, and it should be noted that this type of calibration was shown in the late 1970’s to generate unacceptable levels of between instrument variability[2]. However, with the increasing importance of traceability and quality standards these high levels of variability may be reduced.

As mentioned in an earlier report[1] on wool colour measurement in a CIE colour space, the difficulty with obtaining traceability is the requirement to measure the wool behind optical glass. However, it was also discovered that by the use of a glass correction equation, compensation could be made for the glass effects and traceable measurements, on tiles, could be obtained. This enables an object to be measured 'behind-glass', have a glass correction added, and consequently have an accurate prediction for the value of the object measured without glass.

This paper expands the research from the earlier report by applying the use of a certified tile calibration and a glass correction to the measurement of wool samples in an International Round Trial. A 'standard wool' calibration was also used for comparison purposes. If a tile calibration is to be successful, then it is important that the levels of between-instrument variability are of similar magnitude to those obtained with a Reference Wool calibration.

3. Materials and Methods

3.1. Laboratories and Instruments

Because of the different instrument types and wool cell designs it was important that the trial involved a number of laboratories. The following laboratories participated in the trial:

1. Wool Research Organisation of New Zealand.
2. New Zealand Wool Testing Authority Ltd.
3. Riverina Wool Combers Pty. Ltd.
4. G. H. Michells and Sons Pty. Ltd.
5. Australian Wool Testing Authority Ltd.

Note: These numbers have no reference to the laboratory numbers as depicted in the results section of the report.

A list of the different instrument types, along with their illumination and measurement geometry are listed in Table 1. The Hunterlab instruments use a constant volume wool cell, which requires the test specimen to be weighed so that a constant density is achieved. The remaining cells were constant pressure designs which allow a variable mass to be placed in the cell.

Table 1: List of instrument types and instrument geometry.

Instrument type	Instrument Model	Viewing Geometry
Spectrocolorimeter	BYK-Gardner TCM	$45^0/0^0$
Spectrocolorimeter	Hunterlab Labscan	$0^0/45^0$
Colorimeter	Hunterlab D25M	$45^0/0^0$

3.2. Calibration Materials

Two calibrations were used in the trial. The first calibration standard was a glossy white ceramic tile certified by the National Physical Laboratory in the United Kingdom. The second calibration was a 'standard wool'. This calibration standard was prepared by AWTA Ltd using wool top. The tristimulus values and reflectance values were also assigned by AWTA Ltd after measurement on a BYK Gardner spectrocolorimeter. The spectrocolorimeter was calibrated with the existing Reference Wool calibration, before the 'standard wool' was measured in the AWTA designed wool cell.

Instruments used to measure the colour of wool are normally calibrated with Reference Wool supplied by the Wool Research Organisation of New Zealand (WRONZ). Although the 'standard wool' in this trial was not supplied by WRONZ, the differences between a wool and a tile calibration can be accurately assessed.

3.3. Measurement Materials

3.3.1 Wool top samples

For each of the two calibrations, twenty (20) wool top samples sourced from both Australia and New Zealand were measured in duplicate. The top samples were cut into 20mm pieces before being Shirley Analysed. After conditioning the samples were bagged and sent to the laboratories. A separate set of samples was prepared for each calibration. The colour characteristics of the samples are shown in Table 2.

Table 2: Colour characteristics of measurement samples (Illuminant C, 2^0 observer).

	Brightness (Y)	**Yellowness (Y-Z)**
Maximum	68	-1
Minimum	40	14
Average	60	4

3.3.2 Ceramic tiles

The trial also involved the calculation of a glass correction. This glass correction was added to all measurements that were made using the NPL certified tile calibration. As mentioned earlier, the purpose of the glass correction is to provide traceability for wool colour measurement, since the presence of the glass reduces the reflectance values . AWTA Ltd commissioned the manufacture of a boxed set of glossy ceramic tiles from CERAM in the United Kingdom. These tiles, which cover the entire colour spectrum, can be used for two purposes. Firstly they allow a laboratory to monitor the accuracy of their colour measuring instruments. If a laboratory can measure these tiles to within 0.5 units of their certified values, then the machine can be deemed to be accurate and traceable to CIE[6]. Secondly, the tiles can be used to calculate a glass correction equation. The list of tiles that have been manufactured by CERAM, together with their nominal reflectance values are shown in Table 3.

Table 3: Ceramic tiles used to calculate glass correction.

Tile No.	**Description**	**Nominal Reflectance (%)**
1	Grey	5
2	Grey	25
3	Grey	33
4	Grey	40
5	Grey	50
6	Grey	60
7	Grey	70
8	Grey	80
9	White	90
10	Wool 1	70
11	Wool 2	60

Tiles number 10 and 11 are glossy ceramic tiles that were produced to have colour characteristics similar to those of typical wool samples. They are cream tiles with nominal Y-Z values of 0 and 8 respectively.

It should be noted that the purpose of this trial was to investigate differences between two types of calibration. It was not designed to give estimates on the precision of the issued colour result. In order to ensure that the only variable was calibration type, only one set of ceramic tiles was used in the round trial and one operator performed all the measurements. In addition, the wool measurement samples were prepared by one laboratory.

3.4. Laboratory Procedures

3.4.1 Glass correction

The first step in the trial was the calculation of the glass correction equation. The equation was derived in the following manner:

- Calibrate the instrument using the certified NPL white glossy tile against the instrument port (‘against-port’). Measure the 11 CERAM tiles ‘against-port’.
- Calibrate the instrument using the certified NPL white glossy tile behind the optical glass (‘behind-glass’). Measure the 11 CERAM tiles ‘behind-glass’.
- Calculate the difference between the ‘against-port’ and the ‘behind-glass’ measurements.

The earlier report[1] on measurement in a CIE colour space has shown that the difference between the ‘against-port’ and the ‘behind-glass’ measurements was curvilinear. With the use of regression analysis, a glass correction equation was derived. This correction equation was applied to all measurements based on the certified tile calibration.

3.4.2 Wool measurements

1. The spectrocolorimeter or colorimeter was calibrated with the NPL glossy white tile ‘behind-glass’. The first set of 20 duplicate wool samples was then measured, using the existing wool cell in each laboratory. The glass correction, as determined above, was then applied to the measurement results.
2. The spectrocolorimeter or colorimeter was calibrated using the ‘standard wool’ supplied by AWTA Ltd ‘behind-glass’. The second set of 20 duplicate wool samples was then measured.

The differences between the measurement results for the tile calibration (with the glass correction) and those for the wool calibration were then compared.

4. Results and Discussion

4.1. Glass correction

The glass correction curves for Y for each of the five laboratories can be seen in Figure 1. It is evident from this graph that the correction curves are very similar for each laboratory. There is one curve that is slightly lower than the other, however this only corresponds to a difference of approximately 0.2-0.3 units. The correction curves for X and Z follow similar patterns to that depicted in Figure 1.

The actual glass correction equations for X, Y and Z, along with their R^2 and standard error (SE) values, are shown in Table 4. The high R^2 and low SE values of the regressions show the effectiveness of the CERAM tiles in providing an accurate glass correction.

Figure 1. Glass Correction Curve for Y

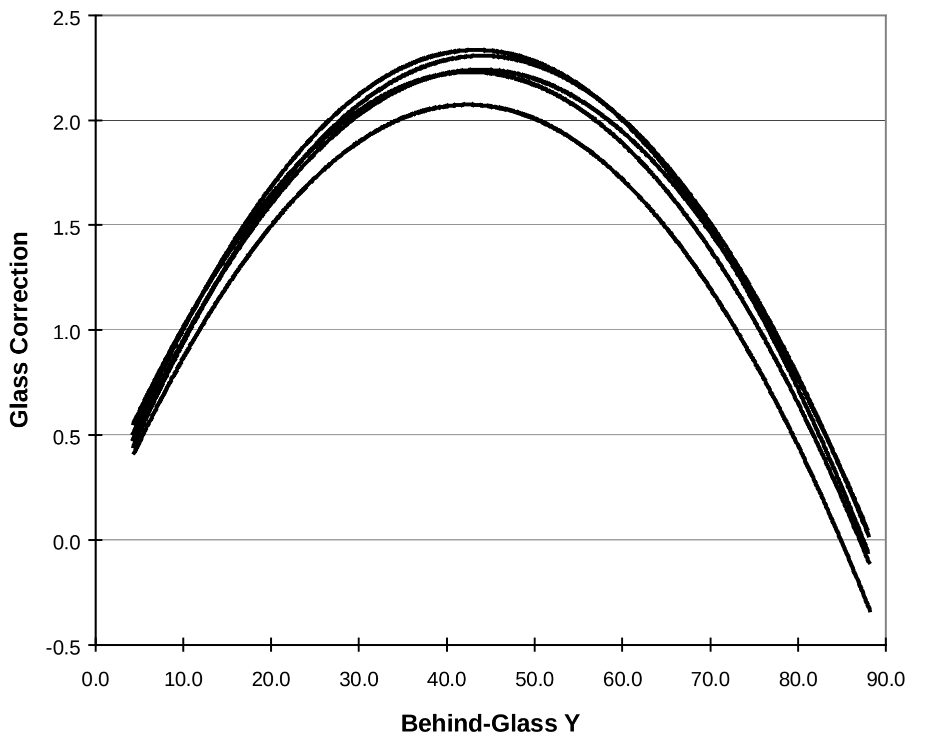

Table 4: Glass correction equations (C) that enable 'behind-glass' measurements (x) to be converted to predicted 'against-port' values.

	X	Y	Z
Laboratory 1			
Correction Equation	$C = -0.00105(x^2-86.24x)$	$C = -0.00103(x^2-88.20x)$	$C = -0.00087(x^2-101.96x)$
R^2	0.956	0.962	0.962
SE	0.162	0.155	0.188
Laboratory 2			
Correction Equation	$C = -0.00116(x^2-86.24x)$	$C = -0.00114(x^2-88.20x)$	$C = -0.00098(x^2-101.96x)$
R^2	0.974	0.980	0.986
SE	0.123	0.111	0.114
Laboratory 3			
Correction Equation	$C = -0.00121(x^2-86.24x)$	$C = -0.00119(x^2-88.20x)$	$C = -0.00102(x^2-101.96x)$
R^2	0.989	0.991	0.992
SE	0.083	0.077	0.085
Laboratory 4			
Correction Equation	$C = -0.00117(x^2-86.24x)$	$C = -0.00116(x^2-88.20x)$	$C = -0.00099(x^2-101.96x)$
R^2	0.995	0.997	0.998
SE	0.052	0.042	0.038
Laboratory 5			
Correction Equation	$C = -0.00119(x^2-86.24x)$	$C = -0.00119(x^2-88.20x)$	$C = -0.00099(x^2-101.96x)$
R^2	0.993	0.997	0.994
SE	0.062	0.046	0.069

4.2. Wool measurements

A comparison between the mean measurement results for the certified tile calibration (plus glass correction) and the ‘standard wool’ calibration is shown in Table 5.

Table 5: Average measurement results over 20 duplicate samples for the certified tile and ‘standard wool’ calibration.

	NPL Tile Calibration			‘Standard Wool’ Calibration		
Laboratory	X	Y	Z	X	Y	Z
1	66.1	67.8	61.5	58.7	60.1	55.8
2	65.9	67.5	61.3	58.6	60.1	55.1
3	67.3	69.0	62.8	58.8	60.4	55.6
4	66.3	67.8	61.4	58.6	60.0	55.2
5	66.5	68.2	61.8	58.6	59.9	55.6
Range (all data)	1.4	1.5	1.5	0.2	0.5	0.7
Range (lab 3 omitted)	0.6	0.7	0.5	0.1	0.2	0.7

The data presented shows that Laboratory 3 gives different measurement results for the tile calibration. However, for the ‘standard wool’ calibration with all five laboratories, measurement results are in close agreement to each other. The range in X, Y and Z for the tile calibration was 1.4, 1.5 and 1.5 respectively. If, however, Laboratory 3 was excluded from the analysis this range drops down to 0.6, 0.7, and 0.5 units and compares more favourably to the ‘standard wool’ calibration.

While the results confirm the Reference Wool calibration approach for these five laboratories, it is clear that four of the five laboratories were able to produce calibrations based on certified tiles that were as good as the ‘standard wool’ calibrations. The reason for the divergence exhibited by Laboratory 3 needs to be resolved.

The cause of the differences for Laboratory 3 may be due to the different wool cell design for this spectrocolorimeter, or in addition it may be due to the geometry of the machine. The laboratory used a Hunterlab Labscan spectrocolorimeter which has a $0^0/45^0$ geometry. The remaining instruments in the trial are of the $45^0/0^0$ design.

Table 6 shows the variance and precision limits for the measurements when all the laboratories are included in the analysis, and also when Laboratory 3 is removed. The between-laboratories component of variance is very high for the tile calibration when all five laboratories are included in the analysis. However, when the outlying laboratory is removed the components of variance and the confidence limits for the tile calibration are comparable to those for the wool calibration.

It should also be noted that measurement results for the tile calibration are approximately 8 units higher than those for the wool calibration. This will inherently increase the confidence limits for the tile calibration by approximately 5-7%.

The ‘standard wool’ calibration included a between-subsample variance, since a separate wool standard was used for each laboratory. For the tile calibration, the use of one certified ceramic standard removed this source of variance. Despite the fact that separate wool standards were used, each of these standards was assigned its own set of tristimulus values in an attempt to minimise the effect of any between-subsample variance.

Table 6: Between-Laboratories (σ_b^2) and Within-Laboratory (σ_w^2) components of variance and 95% Confidence Limits for the certified tile and the ‘standard wool calibration.

	Certified Tile Calibration			'Standard Wool' Calibration		
5 labs	Variance			Variance		
	σ_w^2	σ_B^2	95% CL	σ_w^2	σ_B^2	95% CL
Y	0.04	0.34	1.2	0.05	0.02	0.5
Z	0.08	0.37	1.3	0.09	0.11	0.9
Y-Z	0.03	0.01	0.4	0.02	0.12	0.7
4 labs						
Y	0.05	0.07	0.7	0.05	0.004	0.5
Z	0.09	0.03	0.7	0.09	0.13	0.9
Y-Z	0.03	0.01	0.4	0.02	0.15	0.8

Apart from the benefit of traceability, there are additional advantages in using a certified tile calibration over a Reference Wool calibration. Firstly, the certified values on a tile are valid for ten years, showing the versatility and durability of a tile calibration. This is compared to a wool calibration which is subject to soiling and can only be used once. For a calibration standard that has a single use, the cost of Reference Wool (approximately $3000 AUD) is high.

4.2.1 Additional investigations

In an attempt to find a solution to the divergence of Laboratory 3 from the remaining participants in the trial, subsequent work was performed. The same wool samples measured on the Hunterlab Labscan were measured on a different spectrocolorimeter (ACS CS3 Chroma Sensor). This instrument was the same design as that used by the remaining 4 laboratories who participated in the trial. (i.e. $45^0/0^0$ geometry). If, for Laboratory 3, the data from the ACS instrument is used instead of that for the Labscan instrument, good agreement is reached between the laboratories for both calibrations (Table 7).

Table 7: Average measurement results over 20 duplicate samples for the certified tile and 'standard wool' calibration.

	NPL Tile Calibration			'Standard Wool' Calibration		
Laboratory	X	Y	Z	X	Y	Z
1	66.1	67.8	61.5	58.7	60.1	55.8
2	65.9	67.5	61.3	58.6	60.1	55.1
3 (ACS)	66.4	68.0	61.9	58.6	60.1	55.7
4	66.3	67.8	61.4	58.6	60.0	55.2
5	66.5	68.2	61.8	58.6	59.9	55.6
Range (all data)	0.6	0.7	0.6	0.1	0.2	0.7

The wool samples were compressed in the same constant-volume wool cell as that used for the Labscan measurements. The results show that using this type of wool cell on a $45^0/0^0$ geometry instrument, equivalent results to the remaining 4 laboratories (which also used $45^0/0^0$ instruments) can be obtained. This suggests that the divergence of Laboratory 3 reported in Table 5 and Table 6 is due to machine design (possibly the $45^0/0^0$ geometry).

The instruments used to produce test certificates for raw wool colour measurement are all of the $45^0/0^0$ design. If the use of $0^0/45^0$ instruments for raw wool colour testing are removed from IWTO DRAFT-TM-56, then wool colour measurements in a traceable CIE colour space are possible.

4.3. Relationship Between Wool Calibrations and Tile Calibrations

It has been shown previously[3,4, 7] that the relationship between a certified tile calibration and a Reference Wool calibration is linear. This is also evident in the results from this trial, and Figure 2 shows an example of this linearity for one of the laboratories. The graph featured in Figure 2 is typical of the relationships between wool and tile calibrations in each laboratory. A list of the regression statistics comparing the wool and the tile calibrations for each laboratory is presented in Table 7.

Figure 2. Comparison of Tile and Wool Calibrations (Y readings for Laboratory 5)

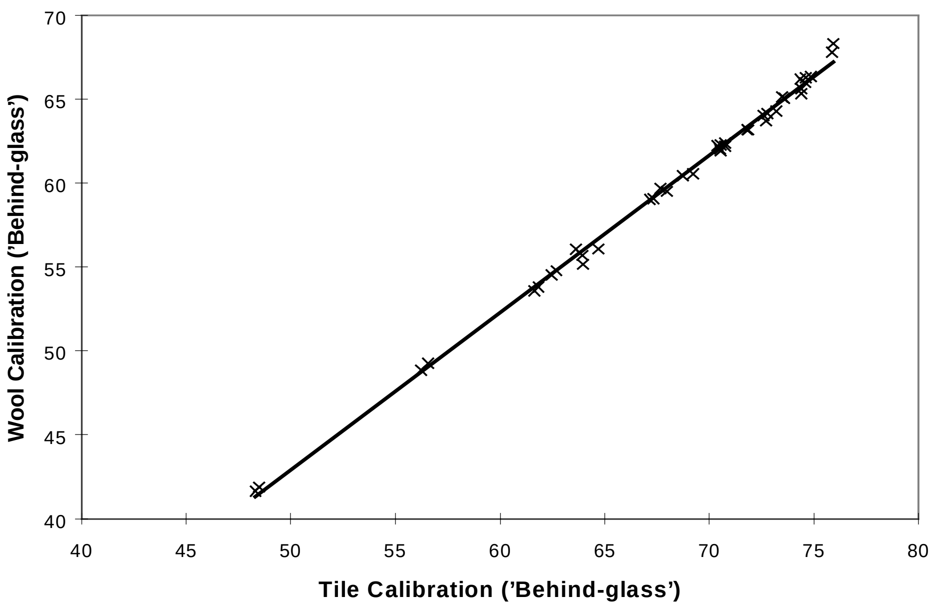

Table 7: Regression equations relating a 'standard wool' calibration (y) to a certified tile calibration (x) for each laboratory.

	X	Y	Z
Laboratory 1			
Equation	y = 0.946x - 3.881	y = 0.940x - 3.662	y = 0.947x - 2.402
R^2	0.9976	0.9977	0.9980
SE	0.300	0.310	0.462
Laboratory 2			
Equation	y = 0.927x - 2.438	y = 0.924x - 2.338	y = 0.924x - 1.627
R^2	0.9969	0.9969	0.9984
SE	0.351	0.367	0.410
Laboratory 3			
Equation	y = 0.935x - 4.041	y = 0.934x - 4.118	y = 0.919x - 2.120
R^2	0.9984	0.9985	0.9987
SE	0.244	0.249	0.370
Laboratory 4			
Equation	y = 0.932x - 3.208	y = 0.933x - 3.289	y = 0.922x - 1.426
R^2	0.9982	0.9983	0.9985
SE	0.263	0.273	0.393
Laboratory 5			
Equation	y = 0.941 - 4.018	y = 0.937x - 3.977	y = 0.945x - 2.724
R^2	0.9970	0.9970	0.9974
SE	0.340	0.357	0.528

The standard errors for the slopes and intercepts in Table 7 were of the order of ±0.006 and ±0.5 respectively.

A change to CIE colour space (i.e. a certified tile calibration or a Reference Wool calibration with cell effects removed) will result in a change to the reported tristimulus values. The best estimate of the expected changes are summarised in Table 8 for Y and Z and in Table 9 for Y-Z. These estimates have been derived by using an average of the regression equations presented in Table 7.

Table 8: Comparison between Y and Z values for wool and tile calibrations.

Y			Z		
Calibration		Difference	Calibration		Difference
Wool	Tile	(Tile-Wool)	Wool	Tile	(Tile-Wool)
40.0	46.5	6.5	40.0	44.8	4.8
42.0	48.6	6.6	42.0	46.9	4.9
44.0	50.7	6.7	44.0	49.0	5.0
46.0	52.8	6.8	46.0	51.1	5.1
48.0	55.0	7.0	48.0	53.3	5.3
50.0	57.1	7.1	50.0	55.4	5.4
52.0	59.2	7.2	52.0	57.5	5.5
54.0	61.3	7.3	54.0	59.6	5.6
56.0	63.5	7.5	56.0	61.7	5.7
58.0	65.6	7.6	58.0	63.8	5.8
60.0	67.7	7.7	60.0	65.9	5.9
62.0	69.8	7.8	62.0	68.0	6.0
64.0	71.9	7.9	64.0	70.1	6.1
66.0	74.1	8.1	66.0	72.2	6.2
68.0	76.2	8.2	68.0	74.3	6.3
70.0	78.3	8.3	70.0	76.4	6.4

Table 9: Comparison between Y - Z values for wool and tile calibrations.

Y-Z		
Calibration		Difference
Wool	Tile	(Tile-Wool)
-2	-0.2	1.8
-1	0.8	1.8
0	1.9	1.9
1	2.9	1.9
2	3.9	1.9
3	5.0	2.0
4	6.0	2.0
5	7.1	2.1
6	8.1	2.1
7	9.1	2.1
8	10.2	2.2
9	11.2	2.2
10	12.3	2.3
11	13.3	2.3
12	14.3	2.3

The industry needs to decide how to manage the change. It is recommended that starting from some point in time (between seasons would be best for grower countries) that both results be displayed for the two colour spaces. This would remain in force for a fixed period of time to be decided by the Commercial Regulations and Contracts Committee of IWTO.

5. Conclusions

The results have demonstrated that for four out of the five laboratories were able to provide harmonious results using a certified tile calibration. The reason for one laboratory being divergent needs to be resolved. The use of a ‘standard wool’ sample to calibrate colour instruments reduced significantly the divergence of one laboratory from the others.

The benefits from the use of certified tiles over a wool calibration are such to justify further research to resolve the reported differences.

The commercial sector of the industry will need to play a leading role through any change over to CIE colour space measurements.

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