

VIABILITY OF USING A ONE-DIMENSIONAL LOVIBOND SCALE FOR COLOUR MEASUREMENT OF PALM OIL AND PALM OIL PRODUCTS

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ABSTRACT

A one-dimensional Lovibond scale for measuring colour required fixing the yellow numeral in some functional relation to the red. The colour of a sufficiently large number of palm oil and palm oil products was measured using the conventional Lovibond scale. The colour of the same samples was then measured by fixing the yellow units at pre-determined values but varying the red values to get the closest colour match. The fixed yellow unit for each type of palm oil and palm oil products was determined by reading the colour of the oils at fixed yellow units of 25, 30, 35 and 40 with independent variation of the red units. Four analysts from two different laboratories were involved in the colour measurements. The readings obtained were then analysed to determine the yellow value that gave the best colour match.

Statistical evaluation showed that the colour values obtained using the one-dimensional Lovibond colour scale did not differ from the results using the conventional scale. Nevertheless, the investigation indicated that the results of palm oil and palm olein colour obtained by analysts from different laboratories using the former method were more comparable than that of the latter. As this was not observed in measurements for palm stearin, the recommendation is therefore for the retention of the conventional method of colour measurement.

Keywords: colour measurement, Lovibond colour, one-dimensional colour scale.

Date received: 13 October 2005; **Sent for revision:** 14 October 2005; **Received in final form:** 2 March 2006; **Accepted:** 8 March 2006.

INTRODUCTION

Colour is an important indication of product composition, purity and degree of deterioration. In the case of vegetable oils, it is necessary to monitor each stage of the refining process to determine whether the correct colour has been obtained, as each type of oil will have its own colour specification in the sales contract. Conventional methods of vegetable oil colour measurement are by visual

comparative techniques. Palm oil and palm oil products are measured using the Lovibond® Tintometer and reported as Lovibond values of red and yellow, plus blue or neutral if required (ISO 15305:1998). This technique involves matching the colour of light transmitted through a specified depth of oil with the colour of light transmitted from the same light source through a set of coloured glass slides. The measurement is subjective and depends on the analyst's judgement as well on the type and model of the colorimeter used. As expected of subjective methods, laboratory-to-laboratory variation in Lovibond colour measurement is large. Notwithstanding this limitation, the Lovibond colour standards are accepted throughout much of the vegetable oil trade as a proven method for assigning colour values.

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Measurement of colour is an optical challenge where the methods used must take into consideration numerous optical problems such as light source, reflectance, absorbance, type and capability of detectors, presentation of samples, temperature, etc. A possible solution is to modify the Lovibond colour scale to a one-dimensional colour scale. To do this would require fixing the yellow numeral in some functional relation to the red. Although it is acknowledged that for the most accurate colour grading, a two- and sometimes three-dimensional Lovibond scale, with independent variation of the red, yellow and sometimes blue is required to account for the variation in pigment concentration in vegetable oils, a one-dimensional Lovibond scale may help reduce the variation in results considerably as only the red needs to be varied.

For refined palm oil and palm oil products, where the range of colour variation is narrow, it may be practical to fix one of the scale variables at a constant value or restrict the variation in some fixed relation to other dimensions, so that a one-dimensional scale is effective. The Chinese National Standard GB 5525 85 specifies a method for inspection of vegetable oils where a Lovibond Tintometer is used to measure oil colour with different fixed yellow filters. In the case of first grade peanut oil for cooking, measurement of colour is carried out with Lovibond yellow fixed at 20, while the same oil for salad is measured at fixed Lovibond yellow 15. Sun *et al.* (2001) based their image analysis method for determination of oil colour on a one-dimensional Lovibond scale according to the Chinese Standard GB 5525 85. They reported good agreement ($R^2 = 0.999$) between their digital method and visual colour measurements using the Lovibond colour scale according to the Chinese standard method.

This paper presents information on the feasibility of colour measurement using a Lovibond scale based on fixing of the yellow values. It is envisaged that a one-dimensional Lovibond Scale, by reducing the number of variables used to quote colour in palm and palm oil products, may offer a more reproducible method for colour measurement of palm oil and palm oil products.

MATERIALS AND EXPERIMENTAL PROCEDURE

Materials

Refined palm oil (RPO), refined palm olein (RPOo) and refined palm stearin (RPOs) were obtained from local refineries. The assistance of officers from MPOB Licensing and Enforcement Division was enlisted for the collection of samples from various ports, namely, Pasir Gudang, Klang, Butterworth, Kuantan and Sandakan.

Apparatus/Instruments

Usual laboratory apparatus and, in particular, the following were used: Lovibond Tintometer Model F (The Tintometer Ltd, UK) and glass cells, 5¹/₄ inch (133.4 mm).

Preparation of Test Samples

All the test samples were homogenized to clear liquids by heating them in an oven set at 60°C ± 2°C or 70°C ± 2°C for palm stearin.

Determination of Fixed Yellow Units for Colour Measurement

The fixed yellow unit to be used for each type of palm oil and palm oil products was determined by reading the colour of the oils at fixed yellow units of 25, 30, 35 and 40 with independent variation of the red units as shown in *Table 1*. Analysts from two different laboratories and using different equipment were involved in the colour measurements. The readings obtained were then analysed to determine the yellow value that gave the best colour match.

Colour Measurements

ISO Method 15305. The colour of oil samples were analysed using the Lovibond Tintometer Model F. The measurements were all carried out using 5¹/₄ inch glass cells.

TABLE 1. COLOUR MEASUREMENT OF PALM OIL AND PALM OIL PRODUCTS

Sample	Measure colour using ISO 15305	Measure red colour by fixing yellow slide at:				
		20	25	30	35	40
RPO	3	-	-	3	3	-
RPOo	3	-	-	-	3	3
RPOs	3	3	3	3	-	-

Measurement of colour at pre-determined fixed yellow. The colour of the test samples, i.e. RPO, RPOo and RPOs were then measured using the pre-determined fixed yellow units shown in *Table 1*.

Precision – Repeatability and Reproducibility of Colour Measurements

Four experienced analysts who had been screened for normal colour vision by the method of Ishihara, carried out colour measurements using the Lovibond Tintometer Model F. The repeatability and reproducibility of the measurements were compared against those calculated from data collected in the inter-laboratory collaborative trial for ISO 15305.

Comparison of the Conventional Measurement with the Proposed One-Dimensional Scale

The data obtained from measurement of the test oil samples, following the conditions set out in *Table 1*, were analysed to determine the feasibility of adopting a one-dimensional Lovibond Scale.

RESULTS AND DISCUSSION

The investigations measured the colour of palm oil and palm oil products using both the conventional method (ISO 15305) and the proposed one-dimensional method. The information obtained from this study is outlined in the following sections.

Fixed Yellow Determined for One-Dimensional Colour Measurement

As shown in *Table 1*, the colour of the oil samples were first measured using the conventional ISO Method 15305. This was then followed by colour measurement by first using the particular yellow slide as shown in *Table 1* before obtaining the red value for the best colour match. *Table 2* gives the average correlation coefficient (*R*) of the red values

TABLE 2. COMPARISON OF CORRELATION COEFFICIENTS (*R*) OF RED VALUES MEASURED BY FIXING YELLOW AND BY ISO 15305

Sample	Yellow slide fixed at				
	20Y	25Y	30Y	35Y	40Y
RPO	-	-	0.9792	0.9799	-
RPOo	-	-	-	0.9579	0.9059
RPOs	0.9782	0.9656	0.9161	-	-

obtained using ISO 15305 and by fixing yellow at the units shown.

The *R*-values in *Table 2* show that the yellow unit to be fixed for colour measurement of RPO could be at 30Y or 35Y, while that for RPOo could be at 35Y and for RPOs could be at 20Y or 25Y.

Comparison of Colour Data Obtained by Different Analysts

The results of intra- and inter-laboratory colour measurement tests by the four experienced analysts were analysed using basic statistical tools for estimating uncertainty in testing.

Data Obtained Using ISO Method 15305

Two laboratories were involved in the colour measurement. The test samples were all analysed by analysts from these two laboratories and the results summarized in *Table 3*.

Table 3 shows that, except for RPOs, poor correlations were obtained for RPO and RPOo when different analysts from different laboratories, using different equipment performed the colour measurements using the same method. However, the correlations were good for all palm and palm oil fractions when different analysts from the same laboratory, using the same equipment carried out the same test. It is noted that the best correlation was for measurement of RPOs.

TABLE 3. LEAST SQUARE LINEAR REGRESSIONS FOR RED COLOUR: $Y = \text{intercept} + \text{slope} (X)$

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer			Different analysts from different laboratories using different Lovibond Tintometers		
	RPO	RPOo	RPOs	RPO	RPOo	RPOs
Dependent variable Y	Analyst 1, Laboratory A			Analyst, Laboratory A		
Dependent variable X	Analyst 2, Laboratory A			Analyst, Laboratory B		
Sample						
n	58	83	46	58	83	46
R ²	0.9799	0.9751	0.9963	0.7538	0.4381	0.9479
Slope	1.0034	0.991	0.9947	0.9935	0.9696	0.9771

Data Obtained Using Pre-Determined Fixed Yellow

The colour measurements of RPO, RPOo and RPOs were carried out using various fixed yellow slides as shown in *Table 1*. The data evaluated for comparison were those obtained with yellow fixed at 30Y and 35Y for RPO, 35Y and 40Y for RPOo and 20Y and 25Y for RPOs. The choice of these particular yellow slides was guided by the *R*-values obtained in *Table 2*.

Results from measurement of RPO. The colour of RPO samples were determined three times, once using ISO Method 15305 and twice by fixing the yellow slide at 30Y and 35Y. *Table 4* shows the results obtained in two laboratories by different analysts.

The correlations between the results from laboratories with different Lovibond Tintometers were poor compared to those obtained from the same laboratory with the same Lovibond Tintometer. The data also indicated that the correlation coefficient for measurement using fixed yellow of 30Y did not differ significantly from those obtained at 35Y.

Results from measurement of RPOo. The colour of the RPOo samples were determined three times, once using ISO Method 15305 and twice more by fixing the yellow slide at 35Y and 40Y. *Table 5* shows the

results obtained in two laboratories by different analysts.

Like the observation for RPO, the results showed that the correlations between laboratories with different Lovibond Tintometers were poor compared to those obtained from the same laboratory with the same Lovibond Tintometer. The data also indicated that the correlation coefficient for measurement using fixed yellow of 35Y was slightly better than that obtained at 40Y.

Results from measurement of RPOs. The colour of the RPOs samples were determined four times, once using ISO Method 15305 and thrice by fixing the yellow slide at 20Y, 25Y and 30Y. *Table 6* shows the results obtained. The results for red colour with the yellow slide fixed at 30Y are not shown as the low correlation coefficients in *Table 2* indicated that 30Y was not a good value to fix the yellow unit at for measurement of RPOs.

Unlike the results obtained for RPO and RPOo, the correlations obtained between the data from laboratories with different Lovibond Tintometers were much closer to those obtained from the same laboratory with the same Lovibond Tintometer. The results also indicated that the correlation coefficients from the measurements using fixed yellow at 20Y did not differ significantly from those obtained at 25Y.

TABLE 4. LEAST SQUARE LINEAR REGRESSIONS FOR RED COLOUR: $Y = \text{intercept} + \text{slope} (X)$ for RPO

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer		Different analysts from different laboratories using different Lovibond Tintometers	
	Analyst 1, Laboratory A	Analyst 2, Laboratory A	Analyst, Laboratory A	Analyst, Laboratory B
Dependent variable Y	Analyst 1, Laboratory A		Analyst, Laboratory A	
Dependent variable X	Analyst 2, Laboratory A		Analyst, Laboratory B	
Yellow slide	30Y	35Y	30Y	35Y
n	58	58	58	58
R ²	0.9530	0.9706	0.7307	0.7156
Slope	0.9973	0.9927	1.0208	1.0087

TABLE 5. LEAST SQUARE LINEAR REGRESSIONS FOR RED COLOUR: $Y = \text{intercept} + \text{slope} (X)$ for RPOo

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer		Different analysts from different laboratories using different Lovibond Tintometers	
	Analyst 1, Laboratory A	Analyst 2, Laboratory A	Analyst, Laboratory A	Analyst, Laboratory B
Dependent variable Y	Analyst 1, Laboratory A		Analyst, Laboratory A	
Dependent variable X	Analyst 2, Laboratory A		Analyst, Laboratory B	
Yellow slide	35Y	40Y	35Y	40Y
n	83	83	83	83
R ²	0.9892	0.9811	0.6487	0.6321
Slope	1.004	0.9958	0.9865	0.9744

TABLE 6. LEAST SQUARE LINEAR REGRESSIONS FOR RED COLOUR: $Y = \text{intercept} + \text{slope} (X)$ for RPOs

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer		Different analysts from different laboratories using different Lovibond Tintometers	
	20Y	25Y	20Y	25Y
Dependent variable Y	Analyst 1, Laboratory A		Analyst, Laboratory A	
Dependent variable X	Analyst 2, Laboratory A		Analyst, Laboratory B	
Yellow slide	20Y	25Y	20Y	25Y
n	46	45	46	45
R ²	0.9952	0.9937	0.8661	0.8937
Slope	0.9942	0.9961	1.021	0.9991

Comparison of Results from ISO Method 15305 and the One-Dimensional Lovibond Method

To ascertain whether there are differences in the results obtained using the two methods, paired comparison tests were carried out on the colour data obtained from analysis of the test samples.

Results for RPO. Table 7 shows the evaluation of data for 58 samples obtained by different analysts in the same laboratory using the same Lovibond Tintometer. Statistical tests were carried out to determine the differences in colour obtained using ISO Method 15305 and the one-dimensional Lovibond scale with yellow fixed at 30Y and 35Y. Table 7 shows that the Student-t critical values obtained by both analysts from Laboratory B were below the 95% confidence level (with 57° of freedom) when the yellow was fixed at 30Y and 35Y. The computation in Table 7 also shows that the results obtained by both analysts in Laboratory A had Student-t critical values above 2.000.

Results for RPOo. Table 8 shows the evaluation of data from 83 samples of RPOo obtained by different analysts in the same laboratory using the same Lovibond Tintometer and from different analysts from different laboratories using different Lovibond Tintometers. Statistical tests were carried out to determine the differences between colour obtained using ISO Method 15305 and the one-dimensional Lovibond scale with yellow fixed at 35Y and 40Y. Student-t critical values obtained by both analysts from Laboratory A were below the 95% confidence level (with 82 degree of freedom) when the yellow was fixed at 40Y. On the other hand, the Student-t critical value for fixed yellow at 35Y was below 1.999 when measured by the analyst from Laboratory B.

Thus, it would appear that there was no significant difference in the measurement of colour using the two methods if yellow were fixed at 40Y for analysis carried out in Laboratory A. However, in Laboratory B, the two methods are only comparable if the yellow were fixed a 35Y instead. This disparity could be due to a number of factors

TABLE 7. SIGNIFICANCE TESTING FOR RPO COLOUR RESULTS ANALYSED USING TWO METHODS

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer				Analyst from a different laboratory using a different Lovibond Tintometer	
	Analyst 1, Laboratory A		Analyst 2, Laboratory A		Analyst, Laboratory B	
Yellow slide	30Y	35Y	30Y	35Y	30Y	35Y
n	58	58	58	58	58	58
Mean of difference, d	0.1121	0.0690	0.0948	0.0328	0.0190	0.0172
Standard deviation of paired differences, S_d	0.0900	0.0959	0.0963	0.1049	0.1051	0.0881
Student-t critical value	9.485	5.478	7.501	2.377	1.3751	1.4897

The value of t_{crit} (with n-1 degree of freedom) at the 95% confidence is 2.000.

TABLE 8. SIGNIFICANCE TESTING FOR RPO_o COLOUR RESULTS ANALYSED USING TWO METHODS

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer				Analyst from a different laboratory using a different Lovibond Tintometer	
	Analyst 1, Laboratory A		Analyst 2, Laboratory A		Analyst, Laboratory B	
Yellow slide	20Y	25Y	20Y	25Y	20Y	25Y
n	83	83	83	83	83	83
Mean of difference, d	0.0458	0.0277	0.0494	0.0410	0.0108	0.0386
Standard deviation of paired differences, S _d	0.1150	0.2002	0.1213	0.1988	0.1473	0.1591
Student-t critical value	3.624	1.261	3.709	1.877	0.670	2.207

The value of t_{crit} (with n-1 degree of freedom) at the 95% confidence is 1.999.

such as the Lovibond Tintometer used, sampling of test sample, laboratory conditions e.g. lightings, ambient temperature, etc.

Results for RPOs. Table 9 shows the evaluation of data for 46 samples of RPOs obtained by two analysts in the same laboratory using the same Lovibond Tintometer, and from a different analyst from a different laboratory using different Lovibond Tintometers. Statistical tests were carried out to determine the difference between colour measurements carried using ISO Method 15305 and the one-dimensional Lovibond scale with yellow fixed at 20Y and 25Y. Table 9 shows that the Student critical value obtained by both analysts from Laboratory A was below the critical value (with n-1 degree of freedom) at the 95% confidence level when the yellow was fixed at 25Y. On the other hand, the results showed that the Student-t critical value for

fixed yellow of 20Y was below the critical value of 2.015 when colour was measured by the analyst from Laboratory B.

One could conclude that there was no difference in the measurement of colour using the two methods if yellow were fixed at 25Y for analysis carried out in Laboratory A. In the case of Laboratory B, the two methods were only comparable if the yellow were fixed at 20Y instead. Again, this disagreement could be attributed to the equipment used; sampling of test material, laboratory conditions e.g. lighting conditions, ambient temperature, etc.

Reproducibility and Repeatability of Colour Measurements using ISO Method 15305

FOSFA International organized an international collaborative test for measurement of colour using ISO Method 15305. The study involved nine

TABLE 9. RESULTS OF SIGNIFICANCE TESTING OF RPO_s COLOUR ANALYSED USING TWO METHODS

Conditions for colour measurement	Different analysts from the same laboratory using the same Lovibond Tintometer				Analyst from a different laboratory using a different Lovibond Tintometer	
	Analyst 1, Laboratory A		Analyst 2, Laboratory A		Analyst, Laboratory B	
Yellow slide	20Y	25Y	20Y	25Y	20Y	25Y
n	46	46	46	46	46	46
Mean of difference, d	0.1152	0.0565	0.1065	0.0565	0.1689	0.0387
Standard deviation of paired differences, S _d	0.2413	0.2296	0.2620	0.2518	0.9177	0.1061
Student-t critical value	3.239	1.670	2.758	1.522	1.248	2.445

The value of t_{crit} (with n-1 degree of freedom) at the 95% confidence is 2.015.

TABLE 10. RESULTS OF INTERLABORATORY TRIAL

Sample	Palm Oil (RPO)		Crude palm kernel oil (CPKO)	
	Red	Yellow	Red	Yellow
Number of laboratories	9	9	9	9
Number of accepted results	9	9	9	9
Mean value, in Lovibond units	2.3	21.6	5.0	47.7
Repeatability standard deviation (S_p) in Lovibond units	0.07	1.22	0.25	2.35
Repeatability limit (r), in Lovibond units	0.20	3.42	0.71	6.58
Reproducibility standard deviation (S_R), in Lovibond units	0.29	1.80	0.76	4.34
Reproducibility limit (R), in Lovibond units	0.81	5.04	2.12	12.26

laboratories analysing two samples of oil, the results of which were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data in *Table 10*. MPOB also participated in this trial.

ISO 15305 states that the values obtained from the inter-laboratory trial may not be applicable to colour ranges and matrices other than those given in *Table 10*. The following precision data were extracted from the ISO 15305 document:

Repeatability: The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same two operators using the same equipment within a short interval of time, will, in not more than 5% of the cases, exceed the repeatability limit (r) given in *Table 11*.

Reproducibility: The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will, in not more than 5% of the cases, exceed the reproducibility limit (R) given in *Table 11*. The results of colour measurement using ISO 15305 carried out in this project were within the required precision.

Reproducibility and Repeatability of Colour Measurements Using the One-Dimensional Method

Only two laboratories were involved in the inter-laboratory colour measurements using the one-dimensional Lovibond scale method. As such, there was insufficient data for statistical evaluation of precision for the method.

TABLE 11. REPEATABILITY AND REPRODUCIBILITY LIMITS

Colour scale	Level	r	R
Red: 133.4 mm cell (5 1/4 inch)	2	0.2	0.8
	5	0.7	2
Yellow: 133.4 mm cell (5 1/4 inch)	20	3	5
	50	6	12

CONCLUSION

Statistical evaluation of the results of colour measurement using the conventional colour measurement method ISO 15305 and the proposed one-dimensional Lovibond scale indicated the following:

- initial evaluation of the correlation coefficients (R) showed that the yellow unit to be fixed for colour measurement of RPO could be at 30Y or 35Y while that for RPOo could be at 35Y and for RPOs at 20Y or 25Y.
- like for the conventional method, poor correlations were obtained for palm oil and palm olein when different analysts from different laboratories, using different equipment, performed the colour measurements using the same method. However, the correlations for palm stearin were quite acceptable ($R > 0.866$).
- correlations were good for all palm oil fractions when different analysts from the same laboratory, using the same equipment carried out the same test.
- Student-t tests on the differences between the two methods showed no difference in the results from tests carried out in Laboratory B when the measurements for RPO were made at fixed yellow of 30Y and 35Y. In the case of RPOo, the results obtained using the two methods were not different when yellow was

fixed at 40Y for analysis in Laboratory A and at 35Y for analysis in Laboratory B. For measurement of RPOs, no differences were observed when measurements were made at 25Y in Laboratory A and 20Y in Laboratory B.

The results indicated that the one-dimensional Lovibond Scale did not differ from the conventional ISO 15305 method when yellow was fixed at pre-determined values. As better reproducibility was only obtained for measurement of RPOs when the former scale was used, the recommendation is to continue using the conventional method for colour measurement but to increase the precision through the use of better-designed automatic colorimeters. The MPOB automatic palm oil colorimeter (Tan *et al.*, 2002a,b; 2004) can be further improved by re-calibration and re-programming to produce readings that match those obtained using ISO 15305. The yellow values in the first prototype were calibrated independently of the red values, *i.e.* without prior knowledge of the red values. The re-calibration of the colorimeter is easily done by considering the red units while calibrating the yellow values.

ACKNOWLEDGEMENTS

We greatly appreciate the assistance of Rozita Baharudin, Head of the Quality Enforcement Unit, and the Heads of Pasir Gudang Port Station, Azahari Baharum; Butterworth Port Station, Noor Azizul Ibrahim and Kuantan Port Station, Mohd Nozri Ibrahim; for coordinating the collection of samples for this project.

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