

DEPARTURES FROM ADDITIVITY AMONG LOVIBOND RED GLASSES IN COMBINATION WITH LOVIBOND 35 YELLOW*

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Abstract

A study of the uniformity of the nominal grades of Lovibond red glasses when used with a Lovibond 35-yellow glass has been made. A total of 2,700 glasses have been investigated, considered in groups of 1,000, 1,000, and 700. Results on the first group of glasses have already been published but are incorporated in the present paper. The second and third groups confirm not only the existence but also the magnitude of the departures from additivity found in the first group. It is shown that the same important discrepancies would still occur in grading vegetable oil if uncalibrated Lovibond red glasses are used. It may be concluded, therefore, that the need for calibrated glasses still exists for color-grading purposes in the vegetable oil trade.

I. INTRODUCTION

THE definition of the color of oil in terms of Lovibond glasses dates back to 1900 when Lovibond glasses were first introduced into this country to establish a standard for "prime summer yellow cotton seed oil." The color represented by the combination of glasses, Lovibond 35Y 7.1R¹, was first adopted by the Interstate Cotton Seed Crushers' Association; this standard was later changed to 35Y 7.6R. However, the prime-summer-yellow color is but one of several reference points on the 35-yellow, variable-red scale (35Y + NR); other reference points on the same scale are used for other kinds of oils.² In the course of trade, inconsistencies occurred in the grades assigned the oils which in 1912 led the Society of Cotton Products Analysts, now American Oil Chemists' Society, to appeal to the National Bureau of Standards for an investigation of

¹Each of these glasses is one of a series of flashed red (R) and yellow (Y) glasses which are graded by the makers according to their "depth of color." The principal coloring materials used in these glasses are gold for the red and silver for the yellow. The red glasses absorb most strongly in the green and the yellow glasses in the violet.

²For example, winter cottonseed oil, coconut oil, peanut oil, and soya bean oil are defined on the 35Y + NR scale at N = 2.5, 3.5, 5, and 9, respectively. A portion of one of the rules (Crude Oil Settlements) governing transactions between members of the National Cottonseed Products Association, Inc., successors to Interstate Cotton Seed Crushers' Association, Inc., states, "Seller shall pay buyer for off color at rate of 1/2 of 1% of contract price for each 1 point in excess of 7.6 red in the case of cottonseed oil or 5 red in the case of peanut oil."

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the uniformity of Lovibond glasses. The results of this early investigation were presented orally³ to the oil chemists in 1913. At that time were reported:

1. Discrepancies as large as 0.5 among the red glasses of the same nominal value.
2. Greater discrepancies among the red glasses than among the yellow.
3. Lack of additivity.
4. Lack of a satisfactory standard set.

It was then agreed that the National Bureau of Standards set, BS 9940, be adopted as the standard and that all glasses used in the oil trade be compared with the glasses of this standard set. Before this could be done, however, it was necessary to standardize the set itself because of the irregular color intervals present. Certain of these irregular intervals may be seen by reference to a previous paper.⁴

This standardization⁵, which resulted in the adoption in 1927 of the standard Priest-Gibson N" unit and scale, is one of several projects in the program of investigations intended to reduce or eliminate the inconsistencies occurring in the color grading of cottonseed and other vegetable oils. These investigations have dealt not only with the spec-

³I. G. Priest, Report of Proceedings of the Fourth Annual Meeting, Society of Cotton Products Analysts, Chicago, Ill., June 21, 1913.

⁴G. K. Walker, *Statistical investigation of the uniformity of grades of 1,000 Lovibond red glasses*, BS J. Research 12, 269 (1934) RP653, Fig. 5.

⁵K. S. Gibson and G. W. Haupt, *Standardization of Lovibond red glasses in combination with Lovibond 35 yellow*, J. Research NBS 13, 433 (1934) RP718; also Oil and Soap 11, 246 (1934).

trophotometric analysis and the calibration of Lovibond red and yellow glasses but also with the spectrophotometric analysis of the oils themselves.⁶

Nearly 3,500 Lovibond red glasses have now been compared with the standardized glasses⁷ and a statistical analysis of the regrade values⁸ for the first 1,000 glasses graded has been published.⁹

It is the purpose of the present paper: (1) to give the results of the investigation on the first 2,700 glasses, examined in three groups, 1,000, 1,000, and 700, and (2) to compare conclusions obtained from a statistical analysis of each of these three groups.

II. ERRATIC DEPARTURES FROM ADDITIVITY IN THE LOVIBOND SCALE

1. Variations Among Glasses of Identical Nominal Grade

The variations in the regrade values, N", which were found among glasses of identical nominal grade, N, may be seen from the histograms in figure 1, which show the frequency distributions of the regrade numerals. The first group of 1,000 are represented by black areas, the second group of 1,000 by striped areas, and the third group of 700 by white areas. The ordinate indicates the number of glasses having the

⁶K. S. Gibson, F. K. Harris, and I. G. Priest, *The Lovibond color system—A spectrophotometric analysis of the Lovibond glasses*, BS Sci. Pap. 22, (1927-28) S547. D. B. Judd and G. K. Walker, *A study of 129 Lovibond red glasses with respect to the reliability of their nominal grades*, Oil and Fat Industries 5, 16 (1928). I. G. Priest, *Tests of color sense of A.O.C.S. members and data on sensibility to change in Lovibond red*, Oil and Fat Industries 5, 63 (1928). D. B. Judd, *Effect of temperature change on the color of red and yellow Lovibond glasses*, BS J. Research 1, 859 (1928) RP31. I. G. Priest, D. B. Judd, K. S. Gibson, and G. K. Walker, *Calibration of sixty-five 35-yellow Lovibond glasses*, BS J. Research 2, 793 (1929) RP58. H. J. McNicholas, *Color and spectral transmittance of vegetable oils*, J. Research NBS 15, 99 (1935) RP815; also Oil and Soap 12, 167 (1935). K. S. Gibson, *Note on the spectrophotometric grading of vegetable oils on the N" Lovibond scale*, Oil and Soap 14, 286 (1937).

⁷The method used in the selection of the standard glasses is described in J. Research NBS 13, 441-442 (1934) RP718 and the particular glasses selected are listed in BS J. Research 12, 271 (1934) RP653.

⁸By regrade values are meant values assigned according to the standard Priest-Gibson N" scale.

⁹BS J. Research 12, 269 (1934) RP653.

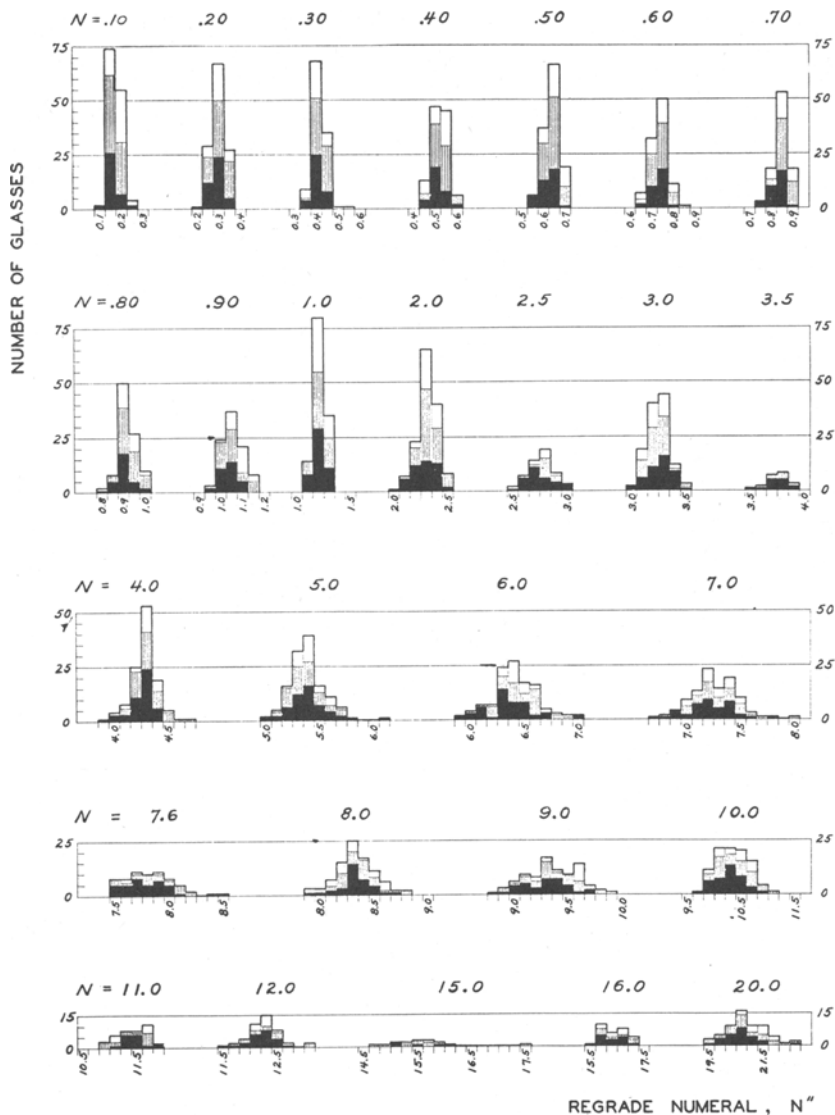


Figure 1.—Frequency distributions of the regrade numerals, N'' , among glasses of identical nominal grade, N , for the three groups of glasses investigated. Black areas represent distribution among the first 1,000 glasses; white areas, second 1,000 glasses; striped areas, last 700.

regrade numeral shown on the abscissa. In the figure there are 27 histograms, which have N values ranging from .10 to 20.0, and each histogram presents data on glasses having identical maker's numerals engraved upon them; none shown contains fewer than 16 glasses. In their preparation class-intervals were used, the sizes and positions of which were arbitrarily selected and varied according to the magnitude of N as follows:

Size of Class-Interval	Magnitude of N
$N'' = 0.05$.10 $\leq N < 1.0$
$N'' = 0.1$	1.0 $\leq N < 10.0$
$N'' = 0.2$	10.0 $\leq N < 16.0$
$N'' = 0.4$	16.0 $\leq N < 20.0$

As an example, let us consider $N = 2.0$, which contains the largest

number of glasses, 144. The size of its class-interval is 0.1 N'' unit and the class-interval centering at 2.3 contains 65 glasses, 14 of which were among the first 1,000 graded, 33 among the second 1,000, and 18 among the third 700. However, the regrade numerals of these 65 glasses as calibrated and reported were not all of this one value 2.3, but ranged in N'' units from 2.25 to 2.34¹⁰, the limits of the class-interval designated as 2.3.

It is apparent from any of the 3 groups shown in figure 1 that the variations among glasses of identical nominal grade may well have been one of the major causes of commercial disputes. As previously shown for the first group of 1,000 glasses, variations among the tenth glasses are not very large (.14 to

.25), but from $N = 5.0$ to $N = 12.0$ the average range is greater than one whole unit (1.15). Among the higher grades the variations are still larger.

The ranges exceed the tolerances in every case. From $N = 1.0$ to $N = 10.0$, the ranges exceed the tolerance of 0.1 by factors from 2.5 to 13.0. Over a quarter of the total number graded have regrade numerals which differ from the respective means by more than our tolerances. These means of the regrade numerals, N'' , for each of the nominal grades from $N = .10$ to $N = 20.0$ are given in column 3 of table 1, where also will be found in column 6 the respective magnitudes of the ranges of the regrade numerals. The ranges in some cases are so large that glasses of similar chromaticity were found having nominal grades differing by a whole unit. This overlapping by a whole unit (columns 4 and 5) occurred in 68 out of the 2,700 glasses.

The average and maximum deviations from the mean regrade numerals, columns 7 and 8, are also given, for they contribute information about the magnitude of these variations among glasses of identical grade. It may be noted from figure 1 that the range of variation is somewhat less for the third group of glasses than for the first and second groups. Figure 2 shows the average deviation from the mean regrade numeral in N'' units for each of the nominal grades from .10 to 20.0 listed in table 1. In this figure group I refers to the first group of 1,000, group II to the second group of 1,000, and group III to the third group of 700. Nominal grades below 1.0 are shown on the enlarged inset. These average deviations for groups I, II, and III are seen to increase on the average with the increase of Lovibond numeral, N ; above $N = 5.0$ they are on the whole greater than the tolerances. However, it may be seen that the average deviations for group III are on the average smaller than

¹⁰The regrade numerals, N'' , of these glasses when reported under test, have been given to 0.01 unit. This has been done in order to give the most probable regrade numeral for each glass, because the numerals are added when the glasses are used. However, this should not be taken to imply that the observations were accurate to 0.01 unit. Two glasses having grades of 2.25 and 2.34 are relatively different by about the amount indicated but each is not presumed to be accurate to 0.01 unit. The regrade numerals are certified to be correct within the following tolerances established in 1928:

Tolerance	Range of Nominal Grade
$N'' = 0.1$.01 $\leq N \leq 10.0$
$N'' = .2$	10.0 $< N \leq 16.0$
$N'' = .4$	16.0 $< N \leq 20.0$

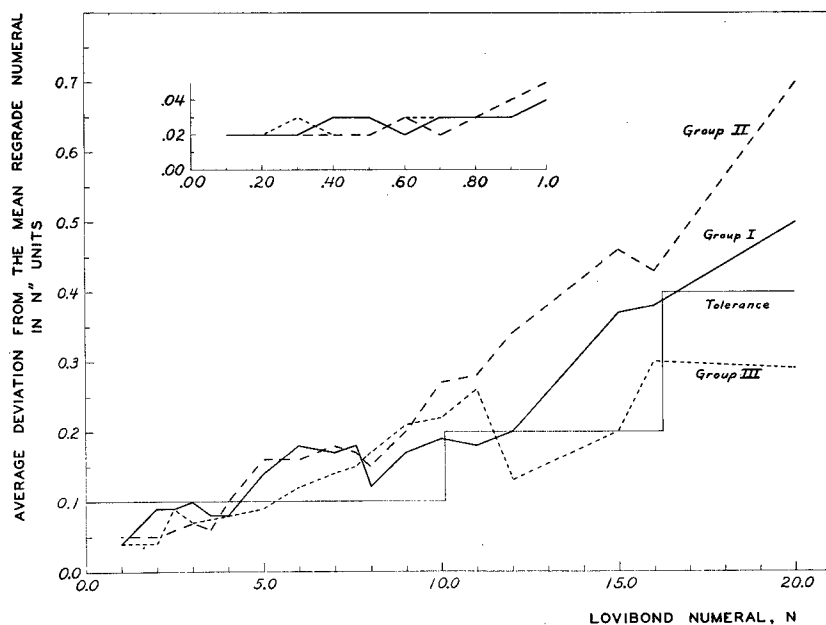


Figure 2.—Trend of the average variation among glasses of identical nominal grade for each of groups I, II, and III.

The average deviations from the mean regrade numerals in N'' units are shown for each of the nominal grades from .10 to 20.0 listed in Table 1.

those of either group II or group I. Thus in group III individual glasses of the same nominal grade tend to have more nearly the same chromaticity.

2. Variations In the Size of the Intervals

It has previously been shown¹¹ that among the glasses in group I there are failures of additivity resulting from the two types of variations, both erratic. One type, found in groups II and III also, has just been described in the previous section—namely, the variations among

glasses of identical nominal grade. The second type will now be described—namely, the variations in the size of the intervals between the average N'' numerals for successive Lovibond tenth and unit glasses. This second type of erratic variation may be seen in figure 3, which shows for each of the groups, I, II, and III, the position on the N'' scale of each of the mean regrade numerals from $N = .10$ to $N = 20.0$ ¹². If each successive tenth interval from $N = 0$ to $N = 1.0$ and each successive unit interval from $N = 0$ to $N = 20.0$ were of uniform size, there would be no failure of additiv-

ity due to this second type of erratic variation. However, it is evident from the figure that in each of groups I, II, and III such erratic variations exist both among the tenth and among the unit intervals.

From a comparison of the sizes of the tenth intervals for the three groups it may be noticed that the smallest spacing occurs between the same two nominal grades, namely, a spacing of about 0.07 between $N = .70$ and $N = .80$. Similar irregu-

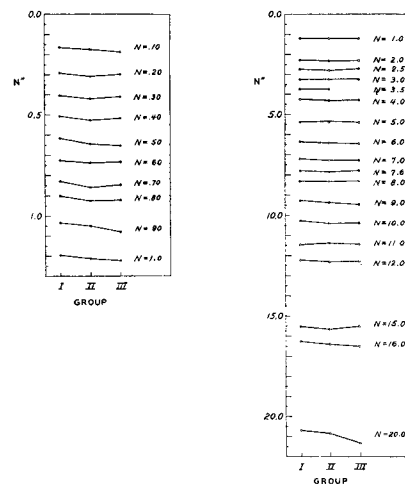


Figure 3.—Erratic variations in the size of the intervals.

Changes in the mean regrade numerals are indicated by the slopes of the lines joining them.

larities may be seen among the nominal unit intervals by comparing for the three groups the consistently small sizes of the intervals from 2.0 to 3.0, 6.0 to 7.0, and 11.0 to 12.0 with the larger sizes of the intervals from 1.0 to 2.0, 7.0 to 8.0, and 10.0 to 11.0.

This is further illustrated in figure 4, which shows for each of the three groups the deviations in N'' units from the average interval of the respective group for both the nominal tenth and the nominal unit intervals. The erratic variation of these intervals in each of the three groups is apparent from the extreme zigzag nature of the broken line connecting the points, which are plotted to represent both the magnitude and direction of the deviations from the average interval. It is seen that all three groups tend to show the same irregularities.

Attention should also be called in figure 3 to the lines connecting the mean regrade numerals for a particular nominal grade. Changes in the mean regrade numerals are in-

¹¹BS J. Research 12, 269 (1934) RP653.

TABLE 1
Variation among glasses of identical nominal grade

Nominal Grade, N	Number of Glasses	Regrade Numerals, N''				Deviations from Mean in Column 3	
		Mean	Minimum	Maximum	Range	Average	Maximum
.10	135	0.18	0.11	0.25	0.14	0.02	0.07
.20	124	.30	.22	.37	.15	.02	.08
.30	114	.41	.34	.53	.19	.02	.12
.40	111	.52	.45	.59	.14	.03	.07
.50	128	.64	.55	.70	.15	.03	.09
.60	101	.73	.66	.83	.17	.03	.10
.70	92	.85	.76	.92	.16	.03	.09
.80	97	.92	.79	1.00	.21	.03	.13
.90	93	1.05	.94	1.15	.21	.04	.11
1.0	129	1.21	1.08	1.33	.25	.04	.13
2.0	144	2.31	2.02	2.51	.49	.07	.29
2.5	53	2.76	2.44	2.97	.53	.08	.32
3.0	120	3.25	2.97	3.54	.57	.08	.29
3.5	21	3.74	3.51	3.89	.38	.08	.23
4.0	117	4.28	3.93	4.66	.73	.09	.38
5.0	129	5.38	4.99	6.07	1.08	.13	.69
6.0	116	6.41	5.93	6.98	1.05	.16	.67
7.0	101	7.25	6.74	8.01	1.27	.17	.76
7.6	65	7.82	7.46	8.52	1.06	.17	.70
8.0	91	8.32	7.90	8.80	.90	.14	.48
9.0	85	9.34	8.80	9.91	1.11	.20	.57
10.0	94	10.34	9.77	11.07	1.30	.23	.73
11.0	38	11.43	10.92	11.92	1.00	.23	.51
12.0	45	12.26	11.53	13.07	1.54	.22	.81
15.0	16	15.58	14.78	17.40	2.62	.40	1.82
16.0	29	16.36	15.61	17.06	1.45	.38	.75
20.0	59	20.87	19.51	22.87	3.36	.59	2.00

¹²All nominal grades are shown in which there were sufficient glasses to warrant the taking of a mean.

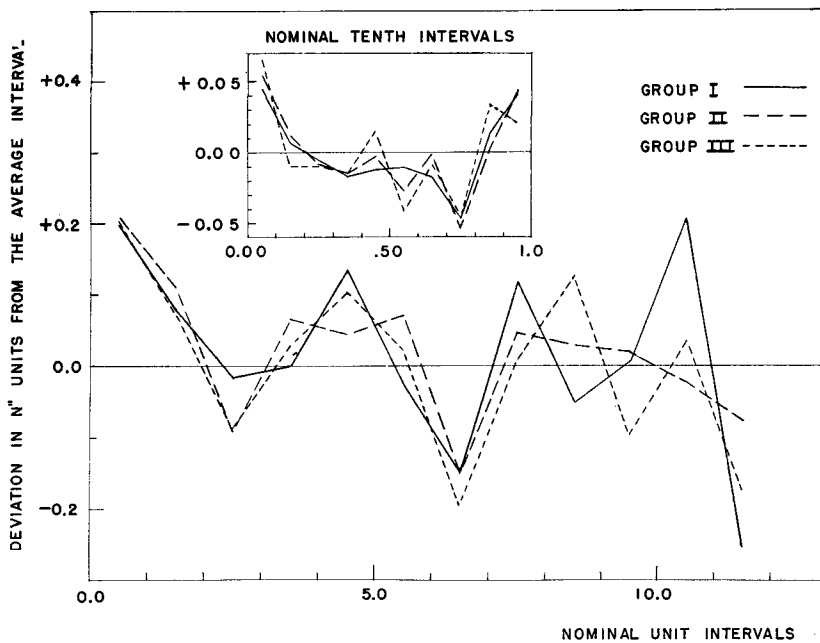


Figure 4.—Deviations in N'' units from the average interval for both the nominal tenth and the nominal unit intervals in each of groups I, II, and III. From the extreme zigzag nature of the broken line connecting the points for each group, it is seen that the intervals vary erratically and that all groups tend to show the same irregularities.

indicated by the slopes of the lines joining them. From inspection it is seen that there is little evidence of a consistent change in one direction from group I, to II, to III, such as would suggest wear in our standard glasses.¹³ This lack of change in the standards is also apparent from the tendency among the tenth glasses (6 cases out of 10) for the mean regrade numerals in group II to be higher on the N'' scale than those in either groups I or III. Among the grades above unity there is also a similar tendency, although this is not apparent from the figure because of the less open scale. The reality of this tendency was tested by weighting the deviations of the mean regrade numerals from their respective nominal grades by the number of glasses and taking an average. It was found that for the glasses up to and including $N = 1.0$, this average for group II is higher than that for groups I and III by about 40 and 50 per cent, respectively, and that for the glasses above $N = 1.0$, group II is higher by about 10 and 50 per cent, respectively. Since there is no consistent progressive variation in mean regrade numeral, these data fail to yield evidence of wear in our standard glasses.

In summary, it may be said (1) that the results of the investigation on all 2,700 glasses confirm the ex-

¹³It may be recalled that the coloring material in these glasses is concentrated in a very thin layer.

istence of the two previously found important erratic departures from additivity, namely, the variations among glasses of identical nominal grade (shown in figure 1 and table 1) and the variations in the size of the nominal tenth and unit intervals (just shown in figures 3 and 4); (2) that some improvement among the more recent glasses has been found in the size of the variations among glasses of identical nominal grade (shown in figure 2); and (3) that no improvement has been found in uniformity of the tenth and unit intervals in either of groups II and III over group I.

III. REGULAR DEPARTURES FROM ADDITIVITY IN THE LOVIBOND SCALE

It was demonstrated in the previous publication on group I that there are two types of regular¹⁴ departures from additivity in addition to the erratic departures just described. Linear equations in N'' and N , obtained by least squares adjustment of constants, demonstrated these two types.

One equation, which covers the entire range of scale from $N = .10$ to $N = 20.0$, demonstrates the first type only—that due to the existence of an intercept. The equation is, for group I:

$$N'' = 1.02 N + 0.14 \quad (1)_I$$

Even if there had been no erratic variations such as shown in figures 1, 2, 3, and 4 the existence of the

¹⁴BS J. Research 12, 281 (1934) RP653.

intercept shows a failure of additivity.

But because two straight-lines were found to describe the data better than one, two equations in N'' and N were formed for the upper and lower sections of the scale respectively. The equation up to and including $N = 1.0$ is, for group I,

$$N'' = 1.11 N + 0.06 \quad (2)_I$$

The equation above $N = 1.0$ is, for this group,

$$N'' = 1.01 N + 0.20 \quad (3)_I$$

The intercepts in both equations are again evidence of the existence of departures from additivity of the first type but the greater slope (1.11 compared to 1.01) of equation (2)_I, indicating a larger N unit for the lower section of the scale than for the upper, demonstrates the existence of the second type of departure from additivity.

To determine whether or not groups II and III also exhibit these two types of regular departures from additivity, the method of least squares has again been applied. The equations for groups II and III which cover the entire range of scale from $N = .10$ to $N = 20.0$ and which correspond to equation (1)_I are, respectively:

$$N'' = 1.03 N + 0.14 \quad (1)_{II}$$

$$N'' = 1.04 N + 0.12 \quad (1)_{III}$$

The equations for the lower and upper sections of the scale for groups II and III which correspond to equation (2)_I and (3)_I, respectively, are given below. The equations up to and including $N = 1.0$ are:

$$N'' = 1.11 N + 0.08 \quad (2)_{II}$$

$$N'' = 1.12 N + 0.07 \quad (2)_{III}$$

The equations above $N = 1.0$ are:

$$N'' = 1.02 N + 0.21 \quad (3)_{II}$$

$$N'' = 1.03 N + 0.14 \quad (3)_{III}$$

It must be concluded, therefore, from these equations that both groups II and III exhibit the two types of regular failure of additivity reported for group I. In addition, it may be noted that the constants found for groups II and III are of the same order of magnitude as those found for group I.

However, a comparison of equations (1)_I, (1)_{II}, and (1)_{III} shows an increase in slope of about one per cent between groups I and II and between groups II and III, although for group III the effect on N'' of this increased slope for any given value of N is counteracted somewhat by the decrease in intercept from 0.14 to 0.12. This same regularity of change in slope is apparent for the upper section of the scale (N greater than 1.0, equations

(3)_I, (3)_{II}, and (3)_{III}, but not for the lower section (N less than or equal to 1.0, equations (2)_I, (2)_{II}, and (2)_{III}).

In table 2 are shown the computed values of N" obtained by substituting values of N listed in column 1 in each of the foregoing equations for each of groups I, II, and III. The differences in the computed N" values for the lower section of the scale are so small that the statement may be made without qualification that the "tenths" section of the scale has remained constant from group to group. For

ered. It was shown that the changes were definitely not those of progression in such a direction as could be accounted for by changes in our standards. The permanence of our standards is the subject of another investigation, in which a study of thirteen of our standard glasses ranging in N from .86 to 20.00 has so far been completed. Three and in most cases four independent spectrophotometric determinations of these glasses, taken over a ten-year period, show no evidence of any such consistent changes. It is, therefore, concluded that there has

from N = .10 to N = 20.0, the equation is:

$$N'' = 1.03 N + 0.14 \quad (1)$$

Up to and including N = 1.0, the equation is:

$$N'' = 1.11 N + 0.07 \quad (2)$$

Above N = 1.0, the equation is:

$$N'' = 1.02 N + 0.20 \quad (3)$$

It happens that equation (1) and (1)_{II} are the same to the second decimal and that equations (2) and (2)_{II} and equations (3) and (3)_{II} are the same to within one in the second decimal.

This larger number of glasses is

TABLE 2

Values of N" derived from the indicated equations for specified values of N to show the magnitude of the changes in N" that have occurred among groups I, II, and III.

N	N"								
	Equation 1			Equation 2			Equation 3		
	Group I	Group II	Group III	Group I	Group II	Group III	Group I	Group II	Group III
1	2	3	4	5	6	7	8	9	10
0.0	0.14	0.14	0.12	0.06	0.08	0.07
.1	.24	.24	.22	.17	.19	.18
.5	.65	.66	.64	.62	.64	.63
1.0	1.16	1.17	1.16	1.17	1.19	1.19
2.0	2.18	2.20	2.20	2.22	2.25	2.20
5.0	5.24	5.29	5.32	5.25	5.31	5.29
8.0	8.30	8.33	8.44	8.28	8.37	8.33
10.0	10.34	10.44	10.52	10.30	10.41	10.44
13.0	13.40	13.53	13.64	13.33	13.47	13.53
16.0	16.46	16.62	16.76	16.36	16.53	16.62
20.0	20.54	20.74	20.92	20.40	20.61	20.74

N = 8.0 and above, however, the differences are not small enough to be considered negligible; they are of the order of magnitude of our tolerances.

The uncertainties¹⁵ in the constants for two of these equations, namely:

$$N'' = 1.02 N + 0.14 \quad (1)_I$$

$$\text{and } N'' = 1.04 N + 0.12 \quad (1)_{III}$$

have been computed and were found for (1)_I to be 0.002 and 0.013, respectively, and for (1)_{III} to be 0.003 and 0.016, respectively. The uncertainties in the constants are, therefore, not large enough to account for the total amount of the differences between the columns in the preceding table. On the other hand these differences in the constants are small. If they are to be considered as real, they might be accounted for either by a decrease in the size of the N" unit because of erosion of our standards in use, or by an increase in the size of the N unit. Each of these interpretations will be considered briefly.

Possible erosion of the standards has already been mentioned in the previous section, where changes in the mean regrade numerals from group to group have been consid-

been no change in the size of the N" unit caused by change in the standard glasses.

The possibility that these small differences in the constants could be due to an increase in the size of the N unit also brings in the element of time. If it were known that all the glasses in group III had been graded and sent out by the Lovibond establishment since the glasses in group II, and those of group II in turn since those of group I, the conclusion might justifiably be drawn that this increase in slope is indicative of an increase in the size of the N unit. There is a chance, of course, that many of the glasses of group III have been graded more recently by the makers than the glasses of group II, and group II than group I, but since there is no definite information to this effect, such an interpretation of the data, while possible, cannot be considered as certainly demonstrated.

Since groups II and III have independently confirmed the existence of two types of regular departures from additivity which are closely the same in magnitude as that found for group I, it is of interest to derive similar linear equations by least squares reduction of the data for the larger group of 2,700 glasses. For the entire range of the scale

a much more representative group from which to draw conclusions than was the first 1,000 glasses. Group I may have been considered to be not an especially representative group in view of the fact that 1,000 is a very small number compared to the many thousands of red glasses which have been supplied to this country. However, it is now found that two other groups of 1,000 and 700 glasses each, regraded over a period of about six years, confirm the two types of regular departures from additivity reported for the first group of 1,000 glasses. They not only confirm the existence of the slope and intercept in the equation previously reported but also confirm the magnitudes of each. Small significant differences between the groups have been found which might possibly indicate changes in the standards or methods of grading of the Lovibond establishment, but which perhaps indicate factors of obscure origin tending to make slightly non-representative the sampling afforded by each of the three groups of glasses.

IV. SUMMARY

The present paper gives the results of the investigation of the uniformity of the nominal grades, N, of 2,700 Lovibond red glasses in terms of the standard Priest-Gibson N" scale. The glasses have been

¹⁵Huge error, equal to 4.9 times the probable error, M. Merriman, Method of Least Squares, John Wiley & Sons, Inc., 1911.

examined in three groups of 1,000, 1,000, and 700, and the results obtained from a statistical analysis of each of the three groups are compared. The important differences in chromaticity existing among glasses of identical nominal grade are illustrated and it is found that there is a slight improvement among the glasses of the third group. The degree to which the Lovibond scale, as embodied in these 2,700 red glasses combined with 35Y, fails to be additive is shown. The second and third groups confirm not only the existence but also the magnitude of the departures from additivity found in the first group. Both reg-

ular and erratic departures are described.

It is demonstrated that the same two types of regular departures from additivity exist in each of the three groups. This is shown by means of linear equations which express the relation between the N' and N scales. Certain small differences from group to group in the slopes of the equations defining the glasses above $N=1.0$ appear significant in that they are greater than the uncertainties involved in their determination, but no adequate explanation of these differences is apparent.

Of even greater importance than the regular departures from additivity in the N scale are the erratic departures, of which there are also two types. It is shown that important discrepancies could still occur in grading oil both because of the variations among glasses of identical nominal grade and because of the variations in size of the average intervals between successive Lovibond tenth and unit glasses. Furthermore, these intervals show the same irregularities throughout all three of the groups. It may be concluded, therefore, that the need for uniformly graded red glasses still exists in the vegetable oil trade.

APPARATUS FOR CONTINUOUS EXTRACTION: ITS APPLICATION TO THE DETERMINATION OF UNSAPONIFIABLE MATTER IN FATS AND OF TOTAL FATTY ACIDS IN SOAPSTOCK*

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Abstract

The apparatus consists of a pipette-like glass tube (solvent distributor) into the side-wall and inside of which is sealed a somewhat smaller tube for carrying solvent vapor. This device is supported in a test tube, the sidearm of which is connected to a distillation flask containing the solvent. The solvent distills from the flask through the tube, is condensed in a condenser, runs down the inside walls of the distributor and accumulates in the test tube until its level reaches the sidearm, where a portion overflows back into the solvent reservoir.

Data are given comparing results obtained by official methods with those obtained using the apparatus described.

THE present official method for the determination of unsaponifiable matter, as described in the methods of the Fat Analysis Committee of the American Chemical Society and the American Oil Chemists' Society,¹ has been in use for many years and has yielded very satisfactory results.

The official method consists briefly, of (1) saponification of a small sample of the fat with an alcoholic potassium hydroxide solution, (2) quantitative transfer of the resulting solution to an extraction cylinder and dilution with alcohol and water to a specified concentration, and (3) subsequent extraction with seven successive portions of petroleum ether (AOCS). The individual extracts are separated from

the soap solution by means of a glass syphon, and the unsaponifiable matter recovered by evaporation of the solvent.

This method is, obviously, somewhat long and tedious. Many analysts have been disturbed by the large number of extractions and the length of time and close attention required, it being necessary to shake the seven extractions vigorously and long to be certain that the extraction of the unsaponifiable matter is complete.

The apparatus described in this paper was developed with the thought of reducing the time required for this determination as much as possible and at the same time improving the extraction efficiency and general manipulative ease of the method. The extraction unit is identical in principle with that described by I. E. Knapp², who proposed his apparatus for use in the ethyl ether extraction of unsaponifiable matter from rosin. It differs only in the manner in which the solvent vapor is carried to the condenser and in the manner of delivery of the extracting solvent to the bottom of the extraction tube. Since the completion of this work Ashley and Murray³ have also proposed a similar apparatus for the removal of ferric chloride from solution.

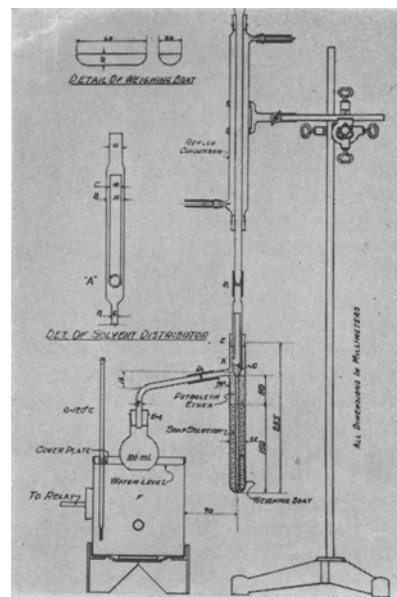


FIGURE I

Apparatus:

Figure I illustrates the principle of the unit. The solvent vapor is carried from the 100 ml. flask through the side arm of the extraction tube and the glass tube (C), sealed into the solvent distributor (B), to the condenser. The condensed solvent runs down the inside wall of (B) and accumulates in (B) until the pressure head of the liquid solvent is sufficient to force the

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