Study of Color of Quaternary Mixtures of Wines by Means of the Scheffé Design

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Standardization of the color of each non-vintage is an important duty, but color specification is difficult. In this article we investigate empirical models using Scheffé polynomials to describe color response surface for four Rioja wine mixtures. The accuracy of color response prediction is shown as color differences in CIELAB units. The color response surfaces are visualized by preparing contour plots that depict color variation over a compositional region.

KEY WORDS: blending, color, Scheffé design

According to Amerine and Joslyn (2), "Blending is used to produce standardized wine of a certain type, to accentuate a special flavor, or to balance the wine." and "The color of a wine is one of its most important attributes. Standardization of the color of each non-vintage is an important duty of the careful winemaker. Unfortunately, color specification is not simple."

The reference method for wine color (10) involves measuring the transmission at four wavelengths, 625, 550, 495, and 445 nm, followed by calculation of tristimulus values X, Y, Z, according to the Commission Internationale de l'Eclairage (3), first published in 1931. This method also indicates that tristimulus values of a mixture of wines can be obtained as an arithmetic mean of tristimulus values of blended wines weighted by the respective concentrations.

In spite of the indication of O.I.V methods regarding the color of a mixture of wines, several publications [Piracci and Spera (11), Negueruela *et al.* (9)] do not agree. To obtain the desirable properties, the usual procedure is to prepare, at random, more or less complicated mixtures of several wines and to select the most suitable empirically.

One method of solving mixture problems is the application of statistical designs and models in experimentation [Mathieu *et al.* (6); Snee (13,14); Marcos *et al.* (4,5); Alman and Pfeifer (1)]. We used the classical Scheffé Simplex experimental design (12) to calculate the color coordinates L*, a*, b* of mixtures of three red wines of Rioja, and satisfactory results were obtained (9). In this way, we added a fourth wine, which had color clearly different from the first three, and we applied the design for four components, increasing the number of experiments and the complexity of calculus.

This work shows the results obtained from this design intended to determine the color for two types of blends of four wines of Rioja: three red wines and one rosé wine, aged under one year (young wines), and three red wines and one rosé wine, aged over three years (aged wines). The results show that the Scheffé design can aid in resolving the problem of standardization of the color of wine, although within constrained proportions of the components.

To determine the color of wines, we selected the CIELAB system of color specification because the cylindrical coordinates L*, C*, H* are qualitatively related to the psychological attributes of color: lightness, chroma, and hue, respectively. Moreover, the color differences can be easily computed from

$$\Delta E^*_{a,b} = [\,(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\,]1/2 \qquad \text{Eq. 1}$$

in CIELAB units, using the rectangular coordinates a* (redness) and b* (yellowness). CIE source C and 1931 CIE standard observer data are used according to the O.I.V. method.

The color coordinates, both CIExy and CIELAB systems (3), are calculated from tristimulus values, although the calculation of these tristimulus by the CIE methods involves 40 measurements of transmitance for a 1-cm path length over the range 380 nm to 780 nm every 10 nm.

Scheffé design: Mixture experiments in general share the characteristic that the response (Y) is a function of the proportions of the q components (X_i) in the mixtures

$$0 \leq X_i \leq 1\,; \qquad X_1 + X_2 + \ldots + X_q = 1 \qquad \text{Eq. 2}$$

and not of the total amount of the mixture. These constraints define a (q - 1) dimensional simplex.

The empirical mathematical models, also called equations of response surfaces, are polynomials and correspond to the development of Taylor serial functions.

The Scheffé design covers the complete Simplex, and the algorithm is applied sequentially, starting with the most simple models, which require the least number of experiments, and continuing with the more complicated models if satisfactory results are not obtained. The evaluation of each model used is carried out by test points.

In our previous paper (9), we observed that the

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special cubic model gave satisfactory results, and we have used it for the mixtures of four components. With this model, only 14 experiments are required to obtain the coefficients of the equation and 12 additional experiments to give test points, but we added 18 experiments to obtain a better check of the model because we superimpose three designs, one for each color coordinate, and the errors are accumulative.

The equation of the special cubic model is:

$$\eta = \Sigma \beta_i X_i + \Sigma \beta_{ij} X_i X_j + \Sigma \beta_{ijk} X_i X_j X_k$$

where X is the concentration of each wine, and the coefficients can be estimated using the formulas:

$$\beta_i = Y_i$$

$$\beta_{ij} = 4Y_{ij} - 2(Y_i + Y_j)$$

$$\beta_{ijk} = 27Y_{ijk} - 12(Y_{ij} + Y_{ik} + Y_{jk}) + 3(Y_i + Y_j + Y_k)$$

where Y is the response for experiments 1-14, the L*, a*, b* coordinates in this case.

Materials and Methods

The mixtures were made by means of a repetitive syringe dispenser (accuracy < ±1%; reproducibility < ±1%) to optimize the proportions of components.

The absorbance spectrum of each experimental mixture was measured by a HP 8451A UV Diode Array Spectrophotometer, using cells plano-parallel with a path length of 1 cm, and these absorbances were used to compute the tristimulus values by the previously described C.I.E. method of 40 measurements.

We replicated each mixture of the design for 10 runs, and the error obtained at each color coordinate L*, a*, b*, is smaller than the last significant figure. Because this, the lack of fit can not be estimated by the current methods (13).

For each mixture the L*, a*, b* color coordinates were measured; the results are shown in Table 1 (young wines) and in Table 2 (aged wines), respectively.

Results

Young wines: According to the experimental results, the polynomials for color coordinates of blends of young wines are:

$$L^* = 25.3 X_1 + 14.9 X_2 + 36.0 X_3 + 73.2 X_4 + 26.0 X_1X_2 - 13.0 X_1X_3 - 79.0 X_1X_4 - 14.2 X_2X_3 - 61.0 X_2X_4 - 78.0 X_3X_4 - 158.4 X_1X_2X_3 + 39.6 X_1X_2X_4 + 344.4 X_1X_3X_4 + 158.1 X_2X_3X_4$$

$$a^* = 49.01 X_1 + 45.64 X_2 + 52.14 X_3 + 31.60 X_4 + 1.98 X_1X_2 - 8.78 X_1X_3 + 12.02 X_1X_4 + 3.84 X_2X_3 + 67.28 X_2X_4 + 43.68 X_3X_4 - 82.92 X_1X_2X_3 + 37.41 X_1X_2X_4 + 106.56 X_1X_3X_4 - 40.02 X_2X_3X_4$$

$$b^* = 33.96 X_1 + 24.00 X_2 + 28.37 X_3 + 18.38 X_4 + 6.80 X_1X_2 - 0.98 X_1X_3 + 15.20 X_1X_4 + 14.66 X_2X_3 + 44.16 X_2X_4 + 21.78 X_3X_4 - 101.04 X_1X_2X_3 - 8.31 X_1X_2X_4 - 33.84 X_1X_3X_4 - 63.42 X_2X_3X_4$$

where X_1, X_2, and X_3 are the concentrations of red wines and X_4 is the concentration of rosé wine.

Table 1. Color coordinates of mixtures of young wines experimentally measured. Exp. = number of experiment; X_1, X_2, X_3 = concentrations of red wines; X_4 = concentration of rosé wine.

Exp.	X_1	X_2	X_3	X_4	L*	a*	b*
1	1	0	0	0	25.3	49.01	33.96
2	0	1	0	0	14.9	45.64	24.00
3	0	0	1	0	36.0	52.14	28.37
4	0	0	0	1	73.2	31.60	18.38
5	1/2	1/2	0	0	26.6	47.82	30.68
6	1/2	0	1/2	0	27.4	48.38	30.92
7	1/2	0	0	1/2	29.5	43.31	29.97
8	0	1/2	1/2	0	21.9	49.85	29.85
9	0	1/2	0	1/2	28.8	55.44	32.23
10	0	0	1/2	1/2	35.1	52.79	28.82
11	1/3	1/3	1/3	0	19.4	45.53	27.31
12	1/3	1/3	0	1/3	26.6	52.50	32.49
13	1/3	0	1/3	1/3	38.7	53.41	29.65
14	0	1/3	1/3	1/3	30.2	54.40	30.19
15	1/3	2/3	0	0	14.4	43.37	22.87
16	2/3	1/3	0	0	14.6	40.72	22.29
17	1/3	0	2/3	0	17.8	43.25	25.11
18	2/3	0	1/3	0	25.3	47.42	31.75
19	1/3	0	0	2/3	47.6	47.60	31.95
20	2/3	0	0	1/3	29.9	46.88	34.26
21	0	1/3	2/3	0	21.1	45.52	25.86
22	0	2/3	1/3	0	17.6	46.75	26.53
23	0	1/3	0	2/3	34.5	51.62	30.77
24	0	2/3	0	1/3	19.0	46.71	28.32
25	0	0	1/3	2/3	50.3	48.82	27.77
26	0	0	2/3	1/3	38.4	53.41	29.06
27	1/2	1/6	1/6	1/6	25.9	49.97	32.40
28	1/6	1/2	1/6	1/6	22.0	50.10	31.02
29	1/6	1/6	1/2	1/6	30.7	52.48	30.01
30	1/6	1/6	1/6	1/2	35.8	53.54	28.78
31	1/4	1/4	1/4	1/4	26.0	49.50	31.05
32	3/10	3/10	3/10	1/10	23.2	48.05	27.30
33	0	1/2	2/5	1/10	22.3	49.63	29.32
34	1/2	0	2/5	1/10	30.2	48.38	30.47
35	1/2	2/5	0	1/10	26.8	47.54	29.86
36	1/5	1/5	2/5	1/5	28.5	51.13	28.41
37	1/5	2/5	1/5	1/5	23.0	50.07	29.26
38	2/5	1/5	1/5	1/5	27.0	49.98	29.00
39	0	1/2	1/4	1/4	23.0	52.03	28.35
40	1/5	1/5	1/5	2/5	32.7	49.77	26.20
41	0	1/5	1/5	3/5	40.0	49.12	24.00
42	5/18	5/18	5/18	1/6	25.8	50.42	29.59
43	4/15	4/15	4/15	1/5	27.3	50.79	29.58
44	7/30	7/30	7/30	3/10	30.5	51.02	27.50

Table 2. Color coordinates of mixtures of aged wines experimentally measured. Exp. = number of experiment; X_1, X_2, X_3 = concentrations of red wines; X_4 = concentration of rosé wine.

Exp.	X_1	X_2	X_3	X_4	L*	a*	b*
1	1	0	0	0	33.2	56.87	41.94
2	0	1	0	0	41.2	48.83	46.30
3	0	0	1	0	24.3	51.78	38.01
4	0	0	0	1	86.3	11.21	18.90
5	1/2	1/2	0	0	35.4	52.79	42.85
6	1/2	0	1/2	0	27.4	53.81	40.50
7	1/2	0	0	1/2	48.7	46.25	33.27
8	0	1/2	1/2	0	29.2	51.14	41.65
9	0	1/2	0	1/2	55.8	36.18	37.12
10	0	0	1/2	1/2	42.4	50.25	35.59
11	1/3	1/3	1/3	0	29.4	52.81	42.05
12	1/3	1/3	0	1/3	42.9	46.71	38.06
13	1/3	0	1/3	1/3	35.7	50.44	39.42
14	0	1/3	1/3	1/3	38.7	48.67	39.06
15	1/3	2/3	0	0	36.3	51.46	43.23
16	2/3	1/3	0	0	34.1	54.15	42.52
17	1/3	0	2/3	0	26.0	53.08	39.50
18	2/3	0	1/3	0	26.9	52.98	39.63
19	1/3	0	0	2/3	55.9	40.42	30.30
20	2/3	0	0	1/3	37.2	51.91	38.95
21	0	1/3	2/3	0	27.0	51.98	40.66
22	0	2/3	1/3	0	30.8	50.37	42.83
23	0	1/3	0	2/3	61.6	31.97	34.67
24	0	2/3	0	1/3	42.3	42.39	41.89
25	0	0	1/3	2/3	52.6	41.72	40.51
26	0	0	2/3	1/3	31.7	53.53	40.28
27	1/2	1/6	1/6	1/6	32.4	51.42	39.59
28	1/6	1/2	1/6	1/6	34.4	48.49	40.02
29	1/6	1/6	1/2	1/6	29.4	51.59	40.22
30	1/6	1/6	1/6	1/2	49.2	48.42	31.18
31	1/4	1/4	1/4	1/4	34.7	50.12	40.24
32	3/10	3/10	3/10	1/10	30.9	52.10	40.60
33	0	1/2	2/5	1/10	31.6	50.50	41.96
34	1/2	0	2/5	1/10	29.0	52.73	40.72
35	1/2	2/5	0	1/10	35.9	53.21	40.11
36	1/5	1/5	2/5	1/5	31.4	50.70	41.84
37	1/5	2/5	1/5	1/5	35.7	50.25	39.95
38	2/5	1/5	1/5	1/5	35.2	51.13	40.15
39	0	1/2	1/4	1/4	38.6	48.00	41.02
40	1/5	1/5	1/5	2/5	41.2	47.66	37.13
41	0	1/5	1/5	3/5	51.1	37.28	32.46
42	5/18	5/18	5/18	1/6	32.3	51.99	41.16
43	4/15	4/15	4/15	1/5	33.2	51.69	41.15
44	7/30	7/30	7/30	3/10	36.7	49.89	39.93

Table 3 shows the color coordinates predicted from models, and the respective color differences at the check points.

It can be observed that the color differences vary in the range 1 to 18; but the mean is 5.2 and the median is 3.5, values which are close to the limit of human color discrimination. But if we constrain the mixtures to concentrations of rosé wine in the range $0 < X_4 \leq 1/3$, color differences are between 1.6 and 3.8; the mean and the median are 2.5, closer to the limit of human color discrimination.

To give a clearer idea about the model, a graphic representation of the results, easily obtained using a computer, is much more effective. Figures 1, 2, and 3 show the isoresponse curves of the mixtures for the L*, a*, b* color coordinates, respectively, in the surface of tetrahedron.

Quaternary mixtures corresponding to the inside of the tetrahedron can be studied easily by making regular sections parallel to one of the surfaces. In this particular case, the regular arrangement of the isoresponse curves on the surface of the tetrahedron provides fairly exact information about their distribution inside. We give only the interior section at the concentration $X_4 = 1/6$ for rosé wine, superimposing the isoresponse curves for the three color coordinates in Figure 4.

The best results from the model are in the inner volume in which the color coordinates are in respective ranges:

$$25 \leq L^* \leq 30$$

$$50 \leq a^* \leq 52$$

$$29 \leq b^* \leq 30$$

The application of Beer's law to spectral absorbances to obtain the theoretical color coordinates of mixtures gives poor results, namely for the constraint volume, where the range of color differences is 1.6 to 8.1; the mean and the median are 3.7 and 2.9, respectively.

Aged wines: The resulting polynomials for color coordinates of mixtures of aged wines are:

$$L^* = 33.2\,X_1 + 41.2\,X_2 + 24.3\,X_3 + 86.3\,X_4 - 7.2\,X_1X_2 - 5.4\,X_1X_3 - 44.2\,X_1X_4 - 14.2\,X_2X_3 - 31.8\,X_2X_4 - 51.6\,X_3X_4 - 14.1\,X_1X_2X_3 - 38.4\,X_1X_2X_4 - 26.7\,X_1X_3X_4 - 28.5\,X_2X_3X_4$$

$$a^* = 56.87\,X_1 + 48.83\,X_2 + 51.78\,X_3 + 11.21\,X_4 - 0.24\,X_1X_2 - 2.06\,X_1X_3 + 48.84\,X_1X_4 + 3.34\,X_2X_3 + 24.68\,X_2X_4 + 75.02\,X_3X_4 + 5.43\,X_1X_2X_3 - 10.74\,X_1X_2X_4 - 82.26\,X_1X_3X_4 - 1.29\,X_2X_3X_4$$

$$b^* = 41.94\,X_1 + 46.30\,X_2 + 38.01\,X_3 + 18.90\,X_4 - 5.08\,X_1X_2 + 2.10\,X_1X_3 + 11.4\,X_1X_4 - 2.02\,X_2X_3 + 18.08\,X_2X_4 + 68.54\,X_3X_4 + 14.1\,X_1X_2X_3 - 9.84\,X_1X_2X_4 - 71.43\,X_1X_3X_4 - 128.07\,X_2X_3X_4$$

Like Table 3, Table 4 shows the predicted color coordinates and the respective color differences at the check points.

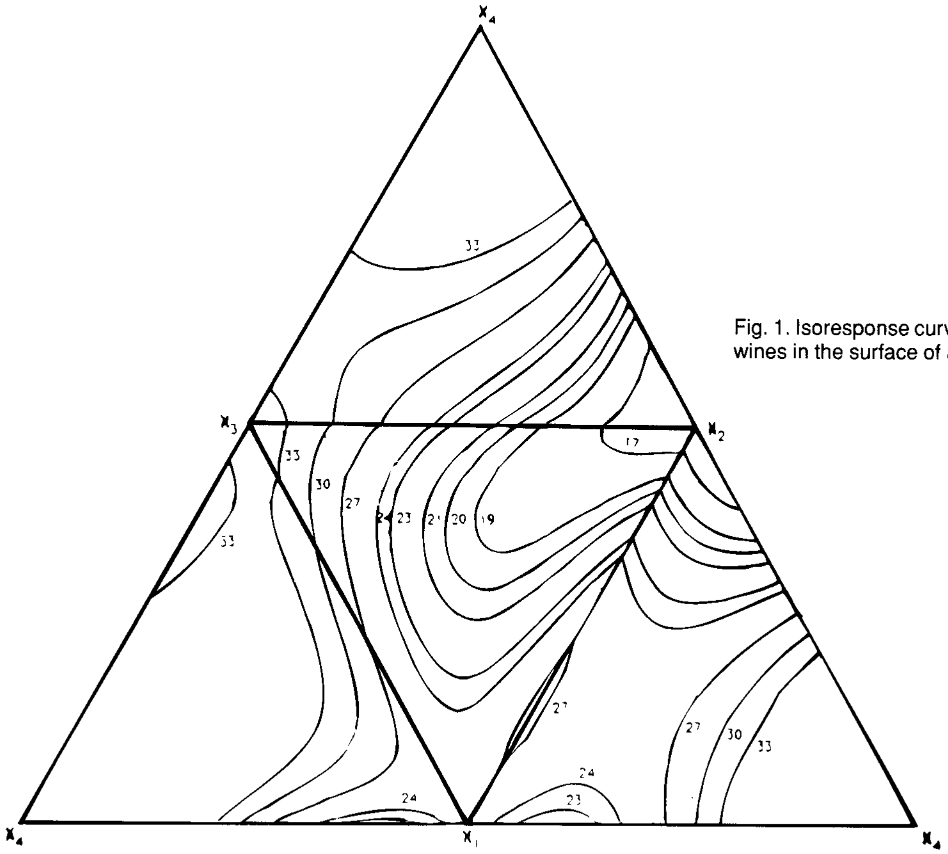
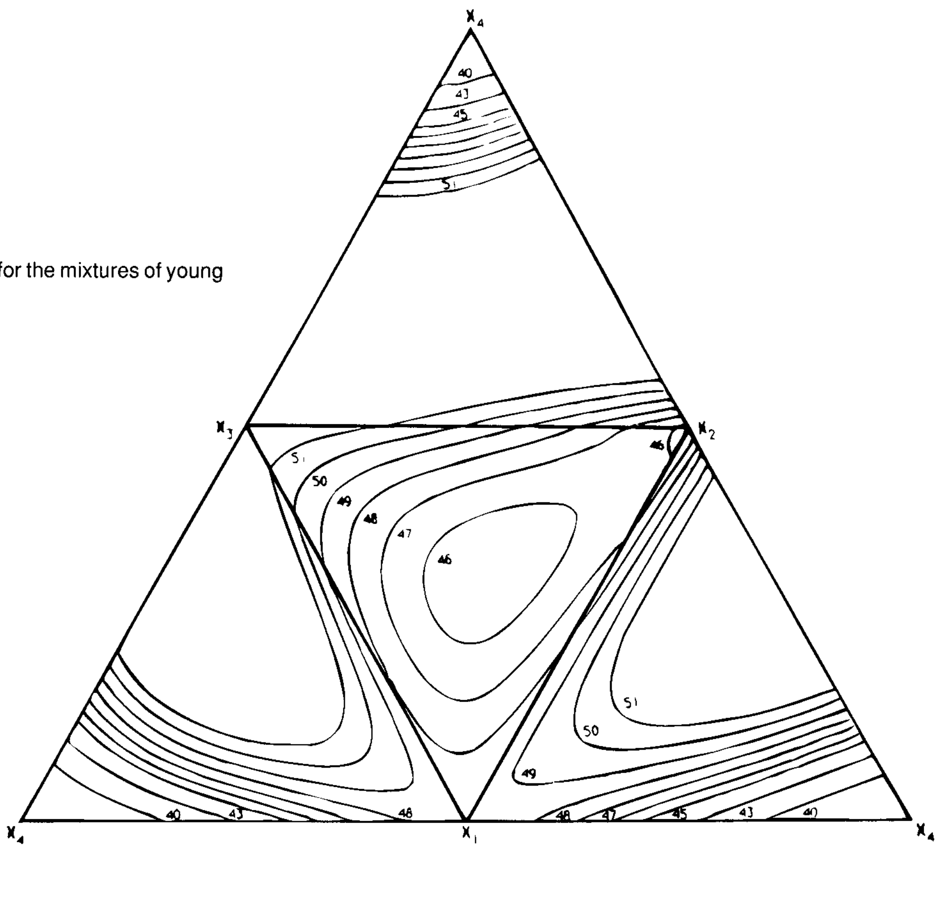


Fig. 1. Isoresponse curves of color coordinate L* for the mixtures of young wines in the surface of a tetrahedron.

Fig. 2. Isoresponse curves of color coordinate a* for the mixtures of young wines in the surface of a tetrahedron.



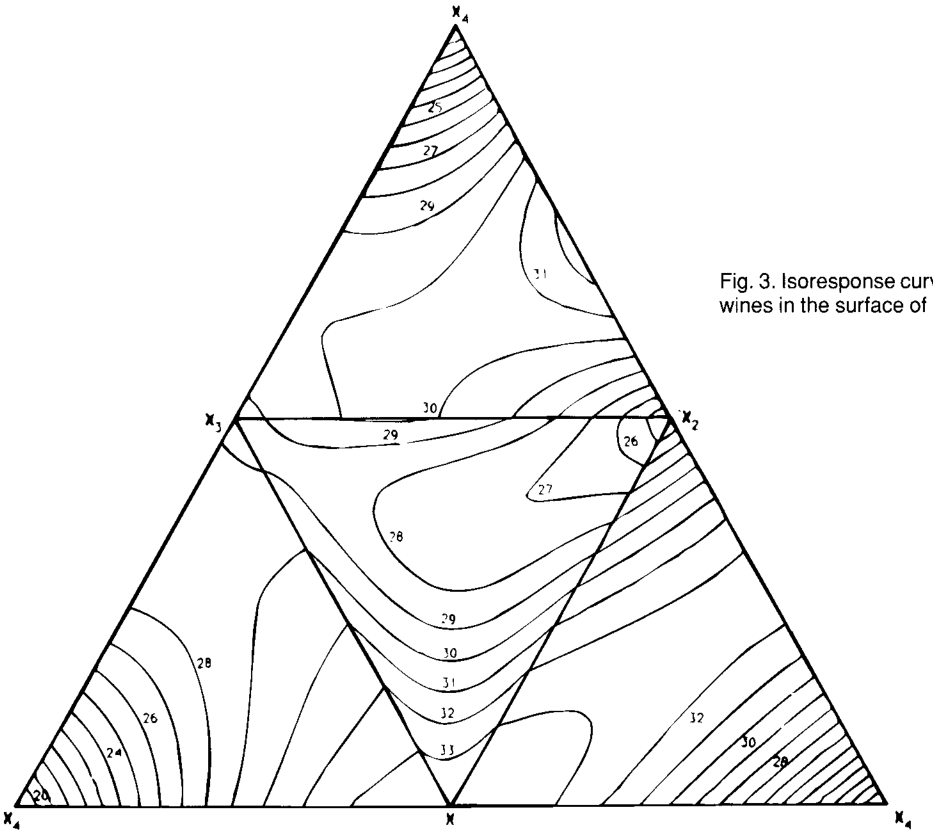


Fig. 3. Isoresponse curves of color coordinate b* for the mixtures of young wines in the surface of a tetrahedron.

Fig. 4. Isoresponse curves of color coordinates L*, a*, and b*; section parallel to X_1 - X_2 - X_3 surface, where X_4 = 1/6. Young wines.

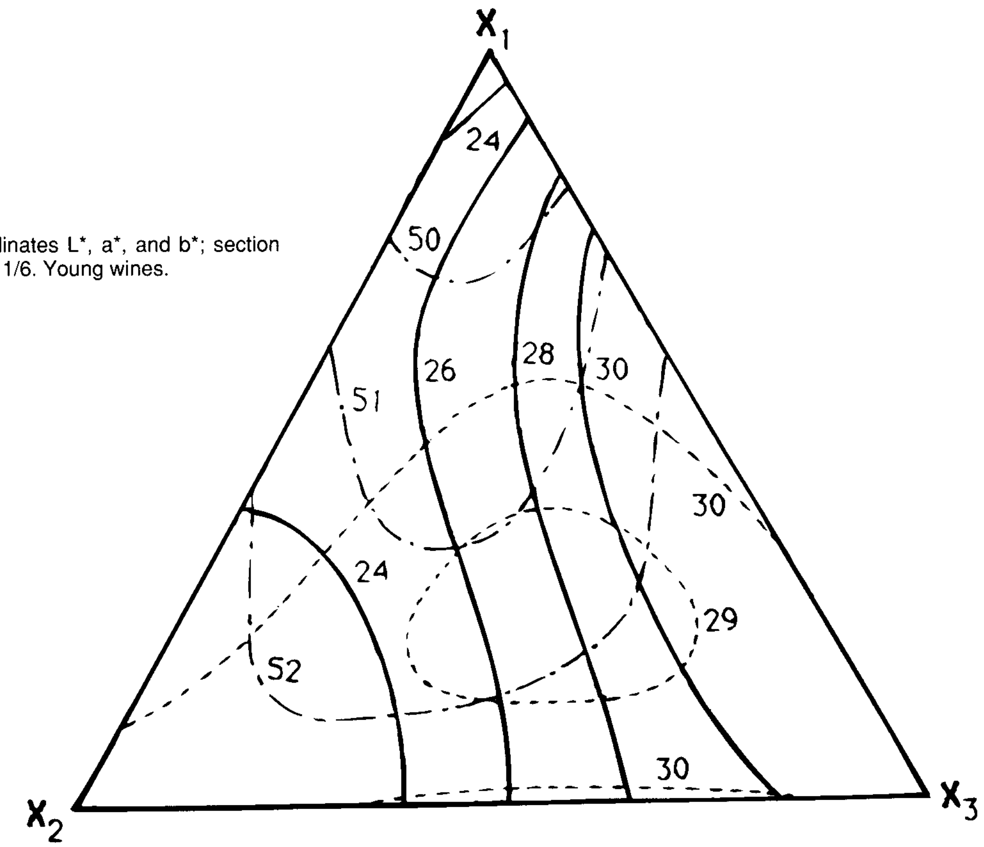


Table 3. Color coordinates of mixtures of young wines at check points from polynomials and color differences with experimental results in CIELAB units. Exp. = number of experiment in Table 1.

Exp.	L*des	a*des	b* des	Diff.
15	24.1	47.20	28.83	12.0
16	27.6	48.32	32.15	18.0
17	29.5	49.14	30.01	14.0
18	30.0	48.09	31.88	4.7
19	39.7	40.07	26.95	10.1
20	23.7	45.87	32.14	6.6
21	25.8	50.82	30.17	8.3
22	18.8	48.65	28.72	3.1
23	40.2	51.22	30.06	5.8
24	20.8	55.90	31.94	8.2
25	43.5	48.15	26.55	7.0
26	31.1	54.99	29.88	7.5
27	27.4	50.34	30.47	2.5
28	23.2	51.63	29.36	2.5
29	29.2	52.53	28.90	1.3
30	37.9	51.47	28.97	2.9
31	29.6	52.43	29.30	5.0
32	24.1	49.32	28.34	1.9
33	23.3	51.91	29.93	2.6
34	31.6	51.24	30.92	3.2
35	25.5	50.36	32.14	3.8

In this case, the color differences vary in the range 0.2 to 7.5. The mean is 2.2, and the median is 1.4. It can be observed that color differences at several points are under 1, the limit of human color discrimination in CIELAB system. If we constrain the mixtures to concentrations of rosé wine under 1/3, the range of color differences diminishes to 0.2 to 4.2, and the mean and the median are 1.4 and 1.2, respectively, very close to the limit of human color discrimination.

Figures 5, 6, and 7 show the isoresponse curves for the color coordinates in the surface of a tetrahedron, with a regular arrangement which provide fairly exact information about their distribution inside, like in the mixtures of young wines. We give only one interior section, at the concentration $X_4 = 0.3$ for rosé wine, superimposing the curves for the three color coordinates in Figure 8.

The best response is in the inner volume in which the color coordinate are in the ranges:

$$36 \leq L^* \leq 40$$

$$48 \leq a^* \leq 51$$

$$38 \leq b^* \leq 40$$

The application of Beer's law to obtain the theoretical color coordinates of mixtures, also gives poor results. The color differences for the constraint volume are between 1.6 and 6.8, and the mean and the median are 3.8.

Conclusions

The use of the Scheffé algorithm to study the color of mixtures of wines gives good results in the study of quaternary mixtures of young wines, but these results can be considered excellent in the study of quaternary mixtures of aged wines (> 3 years). In both cases, the best results are obtained when the concentration of rosé wine is constrained under 1/3.

The color differences between the experimental and the predicted colors are slightly higher than the limit of human color discrimination for the young wines, but for the aged wines the color differences are, at several check points, under this limit.

From these results, the winemaker can use the design to determine whether or not the mixture of three or four red wines will have a certain color. If the response is positive, the respective proportions of each wine are also obtained from the design, and the standardization of color may be easier.

Table 4. Color coordinates of mixtures of aged wines at check points from polynomials and color differences with experimental results in CIELAB units. Exp. = number of experiment in Table 2.

Exp.	L*des	a*des	b* des	Diff.
15	36.9	51.45	43.71	0.8
16	34.3	54.13	42.26	0.3
17	26.1	53.01	39.78	0.3
18	29.0	54.71	41.09	3.1
19	58.8	37.28	29.11	4.4
20	41.1	52.50	36.79	4.5
21	26.8	51.53	40.32	0.6
22	32.4	50.55	43.08	1.6
23	64.2	29.22	32.05	4.6
24	49.2	41.76	41.18	6.9
25	54.2	41.40	40.49	1.6
26	33.5	54.92	46.86	7.0
27	33.2	52.58	39.69	1.4
28	36.1	49.60	40.76	2.1
29	29.5	52.62	40.63	1.1
30	46.8	42.96	35.67	7.5
31	34.9	50.13	39.05	1.2
32	30.8	52.21	40.75	0.2
33	31.9	51.12	40.90	1.2
34	29.1	53.65	40.37	1.0
35	36.0	52.25	41.46	1.3
36	31.4	51.79	40.05	2.1
37	35.4	49.83	39.90	0.5
38	33.7	51.54	39.38	1.7
39	38.4	48.31	39.67	1.4
40	41.4	46.64	37.21	1.2
41	53.6	38.91	35.44	4.2
42	32.4	51.86	40.25	0.9
43	33.4	51.59	40.02	1.1
44	36.7	48.45	38.06	2.4

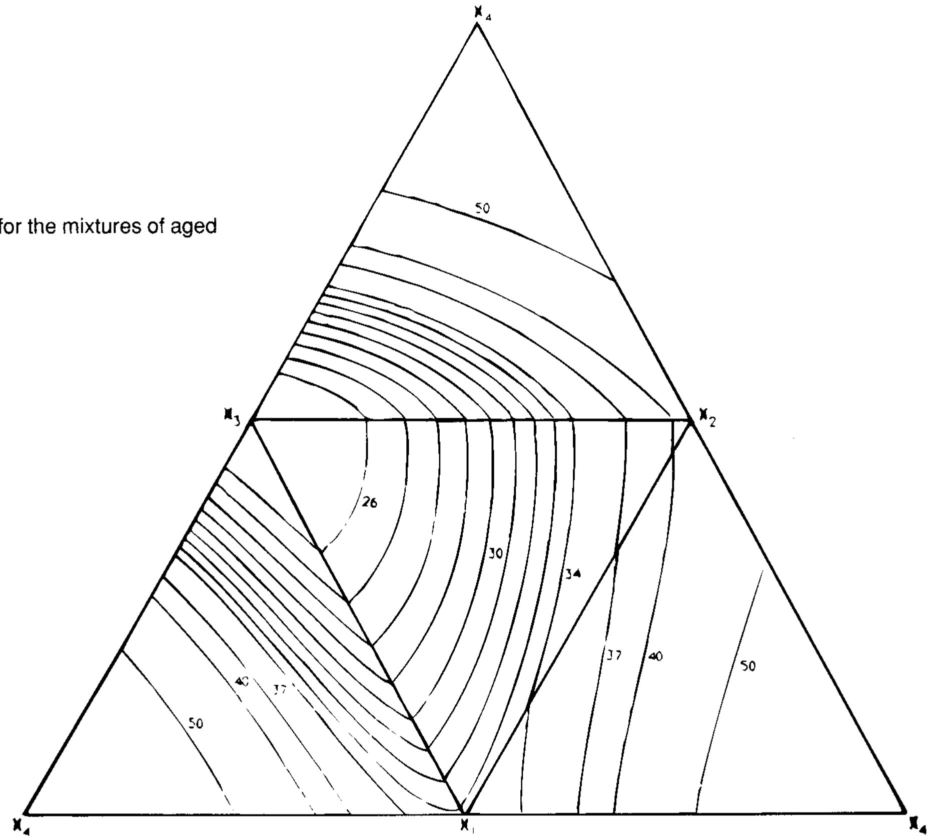


Fig. 5. Isoresponse curves of color coordinate L* for the mixtures of aged wines in the surface of a tetrahedron.

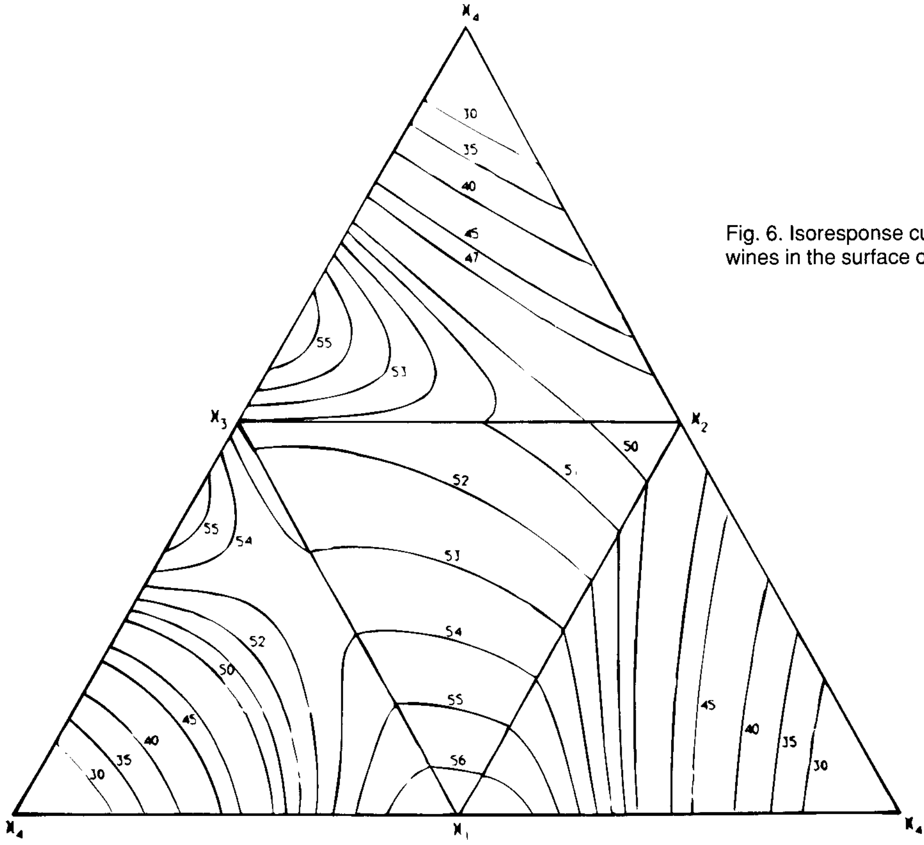


Fig. 6. Isoresponse curves of color coordinate a* for the mixtures of aged wines in the surface of a tetrahedron.

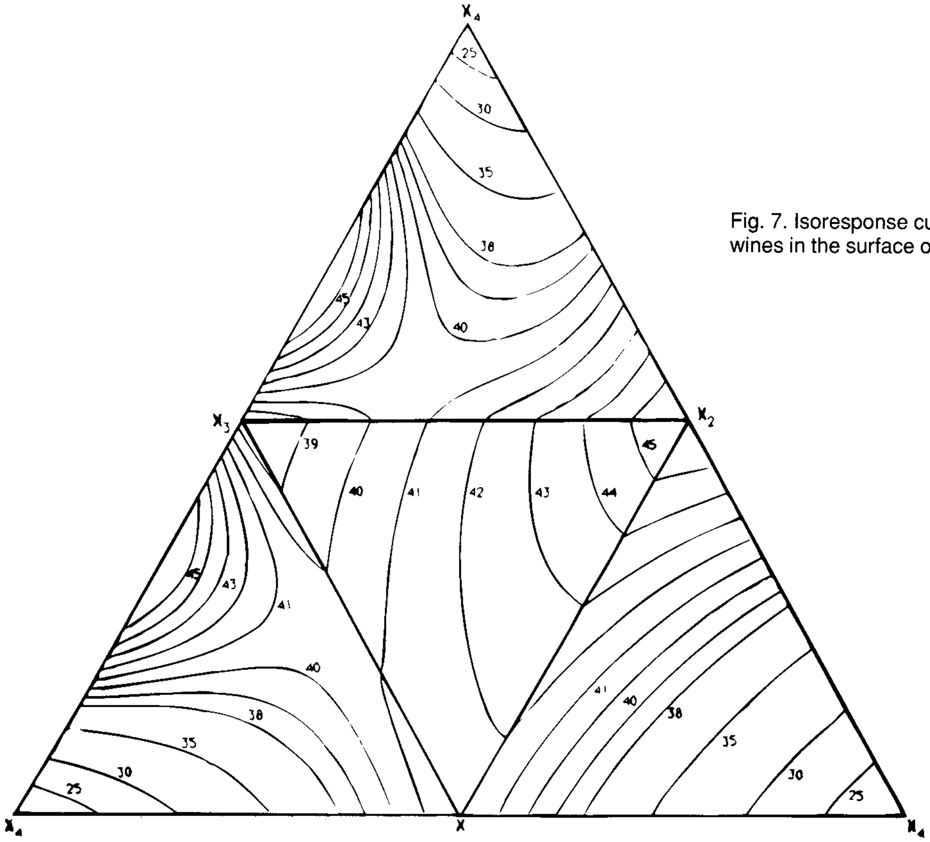
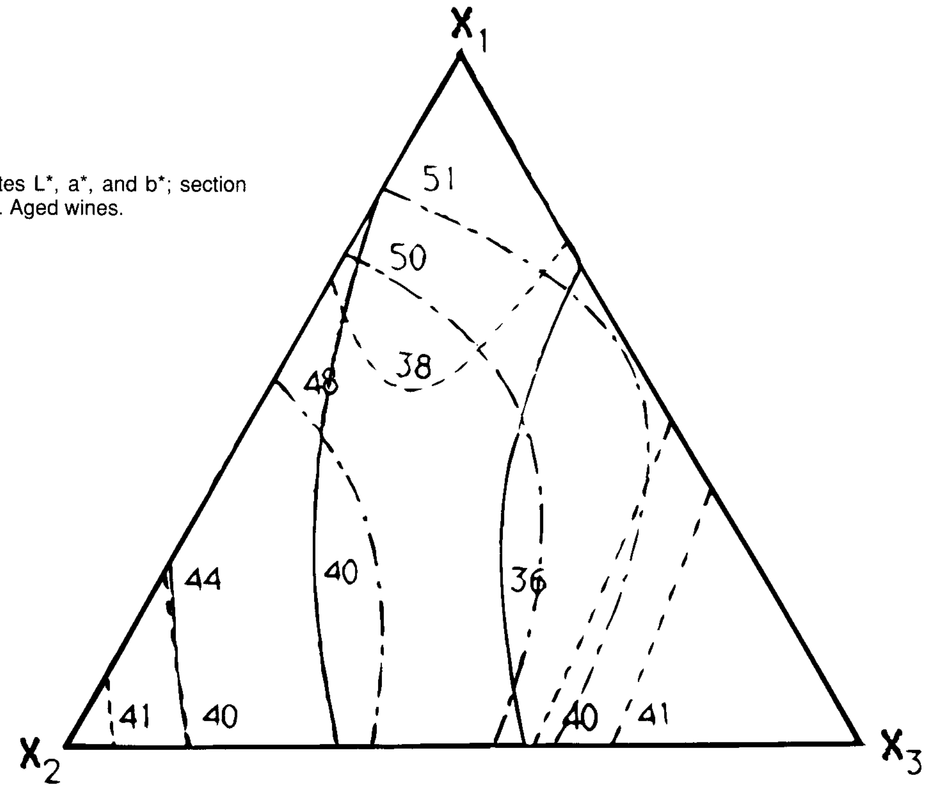


Fig. 7. Isoresponse curves of color coordinate b* for the mixtures of aged wines in the surface of a tetrahedron.

Fig. 8. Isoresponse curves of color coordinates L*, a*, and b*; section parallel to X_1 - X_2 - X_3 surface, where X_4 = 0.3. Aged wines.



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