

Boron, sulphate, chloride and phosphate contents in musts and wines of the Tacoronte-Acentejo D.O.C. region (Canary Islands)

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The concentrations of sulphates, chlorides, boron and phosphorus in musts and wines from the Tacoronte-Acentejo region in Tenerife are studied. The concentrations of these anions in must and wine vary according to the winemaking method and storage vessels and other factors. Concentrations in Tenerife wines fell within the following intervals ($\bar{x} \pm s$) from mean value – standard deviation to mean value + standard deviation: 15.9–30.7 mg litre⁻¹ H₃BO₃; 443–827 mg litre⁻¹ K₂SO₄; 230–436 mg litre⁻¹ NaCl; and 261–451 mg litre⁻¹ P₂O₅. The mean values were, respectively, 23.3 mg litre⁻¹ H₃BO₃; 635 mg litre⁻¹ K₂SO₄; 333 mg litre⁻¹ NaCl; and 356 mg litre⁻¹ P₂O₅. In the case of boron, no statistically significant differences were observed between one year and another or between young and old wines. Sulphate levels were found to be well below the legal limits, although the storage vessel was seen to play a major role, with levels higher in the wines made in cement receptacles and stored in wooden casks. Chloride concentrations were in line with those reported in the literature. As regards phosphates, there was a direct correlation between sugars and must density, indicating that concentration is largely dependent on the degree of grape maturity. © 1997 Elsevier Science Ltd

INTRODUCTION

Data for boron are always interesting for two reasons. First, anything which helps increase our knowledge of the make-up of a foodstuff is important and, second, the toxic nature of boron is uncertain. Furthermore, the presence of boron in wines can increase if boron compounds are added to prevent tartrate precipitation or if used as an antiseptic (Ribereau-Gayon & Peynaud, 1962), even where this is illegal under wine legislation (Spanish Food Legislation, 1970).

The normal level of boron in wines rarely reaches 100 mg litre⁻¹, expressed as boric acid (Mareca Cortés, 1983; Ribereau-Gayon & Peynaud, 1962). The level recommended by the OIV (1990) (*Office International de la Vigne et du Vin*) is 80 mg litre⁻¹. In wines treated with sodium borate, sodium borotartrate or fluoborate (as antiseptics), concentrations are much higher, reaching 200–500 mg litre⁻¹ (Ribereau-Gayon & Peynaud, 1962). Generally speaking, illegal treatment with boron derivatives is suspected if levels exceed 100 mg litre⁻¹.

It is not clear whether boron is an essential trace element for the human organism. In any case, in high

concentrations it is harmful. Germany prohibits all medicaments containing boric acid. Boron taken through food and drinks is estimated in the range of 1–40 mg day⁻¹, and wine can be one of the main sources of intake if levels exceed 20 mg litre⁻¹ (Lutz, 1991).

Normal musts contain small amounts of sulphates, which vary according to vine type; generally speaking, the amounts lie in the 100–400 mg litre⁻¹ range, expressed as K₂SO₄ (Ribereau-Gayon *et al.*, 1980).

Higher amounts may exist in wines given that, in addition to the sulphates present in the musts, there are also those derived from the oxidation of sulphur dioxide and calcium sulphate during 'plastering' (Ribeiro de Lima, 1992). With the exception of some top quality wines—where the levels are higher (Mareca Cortés, 1983)—this practice is authorised to a maximum level of 2 g litre⁻¹ expressed as K₂SO₄ in the final product (Spanish Food Legislation, 1970).

Moreover, the fraudulent addition of H₂SO₄ to wine leads to a clearly-detectable increase in sulphate content.

Martín Plaza (1962) cites amounts of 300 and 550 mg litre⁻¹ in Tierra Medina wines; Villa (1985) gives a figure of 282 mg litre⁻¹ for white Pinot wines in

Abruzzo. Other levels reported in the literature are of approximately 1 g litre⁻¹ K₂SO₄ (Di Stefano, 1985) for Italian virgin Marsala, a wine aged over a long time which has received various sulphites and may also contain variable proportions of mixtures of old Marsala. Other authors cite concentrations of below 2 g litre⁻¹, according to a semi-quantitative method (Álvarez Seoane *et al.*, 1988; Casais *et al.*, 1984; Casais Martínez, 1984).

Chloride, as a constituent macroelement of wine ash, is present in greater or lesser amounts depending on the same factors that affect ash content—degree of grape maturity, climatic conditions, the nature of soil, cépage, vinification conditions, must fermentation, storage and technological factors (Cordonnier, 1965). However, the most important reason why this ion varies in concentration is that levels in must and wine vary with the distance of the vines from the coast. It is important also to consider the growing interest in sodium and chloride content as a result of the use of anion-exchangers to stabilise tartrate, the possible use of chlorated additives and the legal restrictions on chloride content.

Chlorides can also be used in conjunction with egg albumin-type natural proteins as flocculating agents in wine clarification. It is also used for organoleptic correction, to enhance 'body', the tactile sensation of consistency, and to add a certain element of taste. High concentrations may be due to incorrect treatment of wine with exchange resins to reduce high potassium and calcium content (Mareca Cortés, 1983).

Chloride content in wines is extremely variable but in most cases does not exceed 500 mg litre⁻¹ (expressed as sodium chloride). Peynaud (1977) gives a weighted value of 46 mg litre⁻¹ in a study of red wine ash; Amerine & Ough (1976) give a table of chloride content ranging between 50 and 500 mg litre⁻¹ in wines from different countries (excluding Spain). For Spanish wines, the range is 175–982 mg litre⁻¹. Occasionally, some wines harvested near the coast can contain up to 1 g litre⁻¹. Spanish law permits up to 1 g litre⁻¹, expressed as sodium chloride.

Both phosphorus and nitrogen are closely related to plant growth processes (Pérez-Bryan López, 1967). Although phosphorus fertilisation is not the most important for the vine compared to other nutrients, it should not be abandoned altogether because the acidity of the soils of the zone studied may cause the formation of iron and aluminium phosphates, thus considerably reducing the phosphorus available (Trujillo Jacinto del Castillo *et al.*, 1987).

Phosphates in wines are usually related to levels in their musts. Generally, red wines have higher levels than whites. The belief that wines that are high in phosphate content are of better quality does not seem to have been confirmed to date. Phosphate content in musts varies and is due chiefly to differences in the amounts of soil phosphates. Increased levels can result in alteration of the physico-chemical clarity, called 'phosphate, white or

ferric cloudiness' of white wines, caused by the insolubilisation of iron phosphate.

Phosphate content can be increased by adding ammonium phosphate to the must; this is an authorised product used as a yeast activator. In reality, musts have sufficient amounts of phosphates to meet the needs of the microorganisms present in musts and wines. The ammonium ion sometimes specifically favours microbe development. Other authorised products for yeast activation are ammonia glycerophosphate and calcium phosphate.

According to Ribereau-Gayon *et al.* (1980), white wines have concentrations of between 100 and 750 mg litre⁻¹ P₂O₅, while reds have between 225 and 1500 mg litre⁻¹; Vogt *et al.* (1986) report that the level in German wines is 225–575 mg litre⁻¹; Amerine & Ough (1976) report that the level is 75–1350 mg litre⁻¹, and that the level in white wines is somewhat lower than in reds. For Spanish wines they give an average figure of 323 mg litre⁻¹ and an interval of 109–787 mg litre⁻¹.

MATERIALS AND METHODS

Samples

The present study was undertaken using musts and wines from the Tacoronte-Acentejo denomination area. Forty-two must samples from 25 representative wineries in the area were sampled. Seventy-five samples of wine fermented from the musts were also analysed, of which 40 were young wine samples, taken in January, February, March and April, and 35 were samples of the same wines aged in wood for an average period of 106 days and taken in June and July.

Within a particular winery a wine does not originate exclusively from a specific must, or an aged wine does not originate from a specific young wine, because of craftsmanship and production conditions. Therefore, when the statistical regression analyses between the different groups of samples—must, young and aged wines—are made for a specific parameter, the number of pairs of data correlated are less than the total number of samples.

Methodology

The method used for boron was that recommended by the AOAC's Official Methods of Analysis (AOAC, 1990) for determining boron in vegetables, based on the formation of the blue complex of boroquinizarine in a sulphuric medium, with the modifications introduced by Moreno Martín *et al.* (1971).

Procedure

5 ml of Na₂CO₃ 0,32 N were added to 10 ml of sample. This was then dried, burned on a hot plate and in an oven at 550°C. The ashes were dissolved in 20 ml of

H₂SO₄ (0.36 N) and filtered. 15 ml of quinalizarine in concentrated H₂SO₄ were added to 2 ml of the filtered liquid, cooled to room temperature and absorption was measured at 620 nm. Lastly, the absorption was compared with the data from a calibration curve.

To determine sulphate content, the procedure recommended by Spain's Ministry of Health and Consumer Affairs (1985) was used based on precipitation of the SO₄²⁻ with Ba²⁺; the Volhard method was used for the chlorides, having first mineralised the wine through nitropermanganic oxidation (Ribereau-Gayon *et al.*, 1980); for phosphates, precipitation as ammonium phosphomolybdate, the method recommended by the Office International de la Vigne et de Vin (OIV 1990), was used.

RESULTS AND DISCUSSION

Boron

Table 1 gives the mean results obtained for the must, young wine and aged wine samples of the harvests studied. As can be seen, the variation coefficient of the musts varies significantly.

The *B* content in the wines is well below the limits set in the Regulation (EEC, 1987) of the Common Organisation of the market for wine, and generally lower than those cited in the literature (Moreno Martín *et al.*, 1971, 1972, 1973; Sanz *et al.*, 1990; Villa, 1985), thus indicating either a low level of absorbable boron in the soils of the studied vineyards, which would be at odds with the fact that the soil, of volcanic origin, is exceptionally rich in minerals (Rodríguez Rodríguez, 1973), or that winegrowers in the region do not use *B* compounds either in soil fertilisation or in their wine treatments.

It was noted, also, that, in the two harvests studied, the average boron content increased from musts to young wines and from these to the aged wines: 16.1, 21.4 and 23.3 mg litre⁻¹, respectively, in one harvest and 21.3, 23.6 and 25.4, respectively, in the other.

The increase in wines compared to musts is explained by high levels of boron present in skins, pips and stalks (Moreno Martín *et al.*, 1971), which are then extracted at the maceration stage.

Table 1. Boron content of musts and red wines from the Tacoronte-Acentejo area of Tenerife (mg litre⁻¹ H₃BO₃)

	Must	New wine	Aged wine	All wines
<i>N</i>	42	40	35	75
\bar{x}	18.7	22.5	24.3	23.3
<i>s</i>	10.3	7.83	6.93	7.43
Minimum	4.02	8.02	13.4	8.02
Maximum	55.2	42.8	40.3	42.8
V.C. (%)	54.9	34.8	28.5	31.8

N = number of samples.

\bar{x} = mean value.

s = standard deviation.

V.C. = variation coefficient.

In order to determine whether the differences between the standard deviations or between the means of the different groups—must, young and aged wines—were due to random fluctuations, a significance study was carried out (Blanco & Cerdà, 1988; Goulden, 1960). It was found that the musts lay in a different population group from that of the group comprising all the wines ($\alpha = 0.0060$). However, there were no significant differences between the young and aged wines ($\alpha = 0.2948$).

The correlation study of the concentration of *B* in new and aged wines gave a low correlation coefficient, 0.414, although this can be explained by the diversity of samples from a single winery. Conversely, if we consider just one of the harvests, the correlation coefficient improves substantially (0.599), the equation of the straight line being as follows:

$$\begin{aligned} (\text{mg litre}^{-1} \text{H}_3\text{BO}_3)_{\text{ageds}} &= 14.62 + 0.4091 \\ &\times (\text{mg litre}^{-1} \text{H}_3\text{BO}_3)_{\text{news}} \\ \alpha &= 0.0086 \end{aligned}$$

Climatic conditions can explain this higher coefficient; under poor conditions the grape is gathered in one single session whereas, if the weather has been good, three different harvests are taken, thereby producing different wines.

Furthermore, in the statistical inter-relationship study carried out, a significant relationship was observed between boron and phosphate content in musts. This leads to the following regression line, where *B* content increases with content of PO₄³⁻:

$$\begin{aligned} (\text{mg litre}^{-1} \text{H}_3\text{BO}_3)_{\text{musts}} &= 0.1272 + 0.06667 \\ &\times (\text{mg llitre}^{-1} \text{P}_2\text{O}_5)_{\text{musts}} \\ \text{c.c.} &= 0.517 \\ \alpha &= 0.00045 \end{aligned}$$

Lastly, none of the wine samples exceeded 50 mg litre⁻¹ H₃BO₃. The content of any given sample has a 68% chance of falling within the 15.9–30.7 mg litre⁻¹ range, which means that the contents are well within recommended limits (Council Regulation EEC, 1987; Lutz, 1991; OIV, 1990). The histogram (Fig. 1) shows

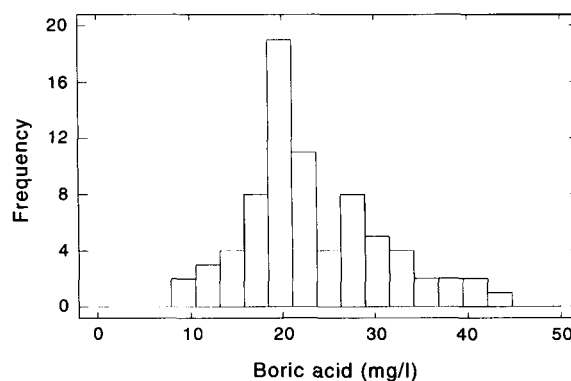


Fig. 1. Frequency histogram of the results of boron content (mg litre⁻¹ H₃BO₃) in the red wines studied.

Table 2. Sulphate content of red wines from the Tacoronte-Acentejo area of Tenerife (mg litre⁻¹ K₂SO₄)

	Must	New wine	Aged wine	All wines
<i>N</i>	42	40	35	75
\bar{x}	339	571	709	635
<i>s</i>	123	152	207	192
Minimum	148	225	398	225
Maximum	595	891	1343	1343
V.C. (%)	36.3	26.6	29.2	30.1

that there exists a relatively homogeneous Gaussian distribution, with a variation coefficient of 31.8%.

Sulphates

No calcium sulphate was added to the wines studied, nor was acidification using sulphuric acid or cask-washing with the same acid known to have taken place. The sulphate levels found in the musts (Table 2) coincide with the natural content in the area, which is slightly higher than the figure mentioned by Ribereau-Gayon *et al.* (1980). This might be explained by the type of receptacle used in the Canaries for traditional red wine maceration; during grape 'pressing' sulphates present in the cement lining of the maceration container may become dissolved by the corrosive action of the juice.

A high sulphate content in the wine can also be expected as a result of the lightly oxidising environment represented by the wooden (mainly chestnut) cask used for making and keeping traditional wine in the Canaries. The results show levels of 571 mg litre⁻¹ K₂SO₄ in the young wines, which were kept for approximately four months in the wooden casks, and 709 mg litre⁻¹ in aged wines, stored for around seven months. These concentration levels are higher than those cited in the literature (Martín Plaza, 1962; Villa, 1985).

By way of summary, sulphate content of red wines from the Tacoronte-Acentejo area (Table 2) are well below the legal limits established by the European Community (Council Regulation EEC, 1987).

Samples 10, 15 and 24 are examples of how the vessel can influence sulphate concentration. Wines of the 1987 harvest were made in a cement receptacle for the maceration-fermentation and then kept in wooden casks. That year, sulphate levels rose considerably

Table 3. Influence of the storage vessel on the sulphate content of red wines (mg litre⁻¹ K₂SO₄)

Sample no.	1987 harvest			1988 harvest		
	Must	New wine	Aged wine	Must	New wine	Aged wine
10	256 ^c	560 ^w	885 ^w	297 ^c	572 ^s	549 ^s
15	331 ^c		415 ^w	430 ^s	480 ^s	398 ^s
24	399 ^c	729 ^w	961 ^w	595 ^c	787 ^s	686 ^s

^cCement, ^wwood, ^sstainless steel.

Table 4. Chloride content of musts and wines from the Tacoronte-Acentejo area of Tenerife (mg litre⁻¹ NaCl)

	Must	New wine	Aged wine	All wines
<i>N</i>	42	37	32	69
\bar{x}	198	337	327	333
<i>s</i>	85.3	104	104	103
Minimum	49.7	157	146	146
Maximum	472	570	560	570
V.C. (%)	43.1	30.9	31.8	31.1

(Table 3), except sample no. 15, which was made in a taller cement receptacle but with a lower surface area than the traditional one, which greatly reduced the oxidising environment. The wine was later transferred to an oak cask, which is much more airtight than the chestnut version.

As for the 1988 harvest, samples 10 and 24 were made in cement receptacles and then transferred to a stainless steel vessel, whereas no. 15 was macerated-fermented and kept in a steel vessel. In the case of samples 10 and 24, it appears that the increased sulphate level seen in the young wines may be due to oxidation of the must in the receptacle, which was small and with a large surface area. With sample 15, the (high) level was maintained because it was kept in an environment which was essentially oxygen-free due to the air-tightness of the stainless steel.

Table 3 shows that the SO₄²⁻ content of samples 10, 15 and 24 (1988) did not increase, and even decreased, when kept in stainless steel, whereas it increased significantly in the samples kept in wooden casks.

Chlorides

Chloride levels in wines depend largely on their levels in musts; a good linear statistical correlation exists between the two (*c.c.* = 0.602) with high significance ($\alpha = 0.0000$).

It is important to stress that the Cl⁻ ion concentration in musts depends largely on the stage of fermentation. So, when correlation analysis of this ion with some parameters of the vinification is carried out (such as density or the extraction of polyphenols from skins) it is observed that, during the fermentation of the musts studied, the statistical correlation of the Cl⁻ concentration with the density is negative, with significance level

Table 5. Phosphate levels found in musts and red wines of the Tacoronte-Acentejo area of Tenerife (mg litre⁻¹ P₂O₅)

	Must	New wine	Aged wine	All wines
<i>N</i>	42	40	35	75
\bar{x}	279	343	371	356
<i>s</i>	79.7	88.9	101	95.1
Minimum	139	196	214	196
Maximum	490	541	582	582
V.C. (%)	28.6	25.9	27.2	26.7

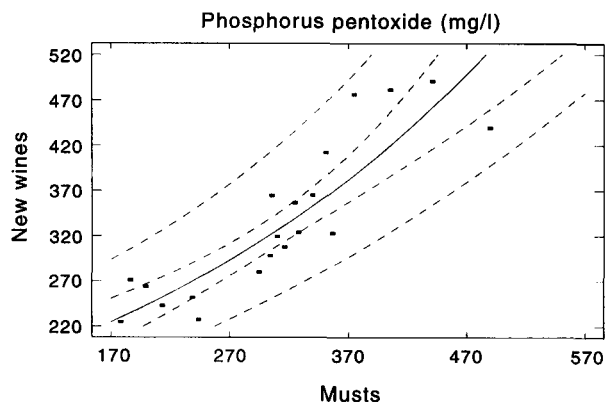


Fig. 2. Analysis of the exponential correlation with respect to the phosphorus concentration ($\text{mg litre}^{-1} \text{P}_2\text{O}_5$) between new wines and the musts from which they are made (20 pairs of data).

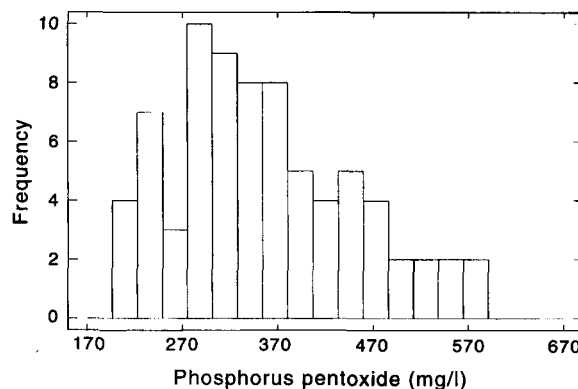


Fig. 4. Frequency histogram of the levels of phosphorus ($\text{mg litre}^{-1} \text{P}_2\text{O}_5$) in the red wines studied.

of 0.0002 (González Hernández, 1994), whereas with the permanganate index is positive, $\alpha = 0.0000$ (González Hernández, 1994).

According to the literature (OIV, 1990; Primo Yúfera, 1979), Na^+ and Cl^- ions are always linked in terms of their equivalent concentrations, except in treatments with exchange resins. In effect, in the musts studied (González Hernández, 1994), these two ions are statistically correlated, with a positive coefficient of 0.754 and $\alpha < 0.0001$. Table 4 gives the statistical description of the results obtained.

The results show that average levels of chloride lie within the general interval cited in the literature consulted, even though the area studied is a coastal one. Similarly, of the causes listed in the literature (Cordonnier, 1965; Mareca Cortés, 1983) as to why chloride levels might be artificially elevated in wines, none is possible in the traditionally-made wines studied. However, there is an age-old custom practised by some winegrowers which significantly raises chloride levels in wines but not musts: cask-washing with common salt or sea water. Sodium chloride may even be added to the wine to influence its organoleptic characteristics.

The levels found here are well below the maximum levels set by Spain's technical and health regulations,

1 g litre^{-1} (even in the wines to which sodium chloride was added). The literature reflects a wide chloride content range (Di Stefano, 1985; González Hernández *et al.*, 1986; Villa, 1985).

Phosphates

Among mineral elements, it should be noted that concentrations of some, such as phosphates, potassium or calcium, can vary considerably depending on the amount and type of fertiliser used on the vines, which thus rules out a direct relation between soil type and wine mineral content (García-Jares *et al.*, 1990). Table 5 shows the phosphate levels found in the musts and wines studied; the analysis results do not present great differences from the mean value (Di Stefano, 1985), a feature being the low variation coefficients. From collaborating wine-makers it has been possible to verify the non-use of fermentation activators, or at least none based on PO_4^{3-} or phosphorus in general.

Pérez-Ruiz *et al.* (1985) and Villa (1985) reported lower concentrations ($105 \text{ mg litre}^{-1}$), and point out that these are adequate, considering the possible formation of subsequent iron precipitations. Coll Font *et al.* (1986) also reported levels which are slightly lower

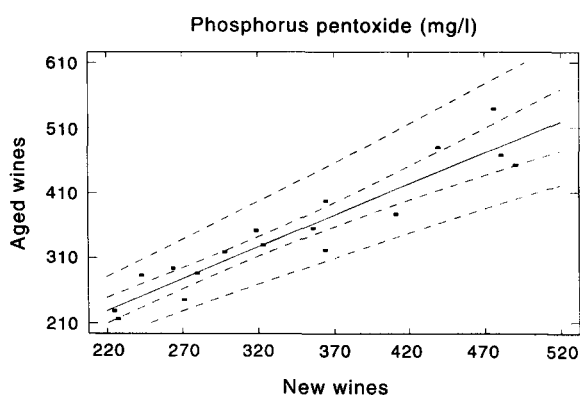


Fig. 3. Statistical correlation of the phosphorus concentration ($\text{mg litre}^{-1} \text{P}_2\text{O}_5$), multiplicative model, between old and new wines (17 pairs of data).

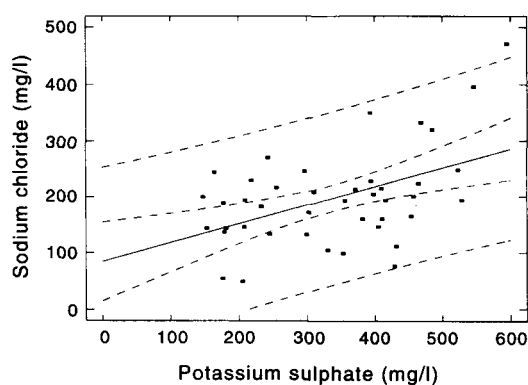


Fig. 5. Statistical linear correlation between chlorides ($\text{mg litre}^{-1} \text{NaCl}$) and sulphates ($\text{mg litre}^{-1} \text{K}_2\text{SO}_4$) in the musts studied (42 pairs of data).

than those found by us, while Di Stefano (1985) reports slightly higher figures—of the order of 375 mg litre⁻¹ P₂O₅. Even higher are the phosphorus levels in wine given by Maynar *et al.* (1980).

A comparison of average results shows that concentrations are lower in young wines than older ones. That could be because of the environmental conditions, since the new wine samples and the aged ones were collected in winter and summer, respectively, with an average difference of temperature of 5 or 10°C. The temperature conditions are very important in that they could modify the solubility of the iron phosphate; ferric cloudiness is a problem which tends to occur in winter (Peypaud, 1977).

Under normal winegrowing conditions, phosphates are relatively constant for the different sample groups. Phosphate levels in musts correlate with the new wines made from them (Fig. 2) in accordance with the following exponential regression:

$$(mg\ litre^{-1}\ P_2O_5)_{new\ w.} = EXP[4.965 + 2.648 \times 10^{-3} \\ \times (mg\ litre^{-1}\ P_2O_5)_{must}]$$

$$c.c. = 0.890$$

$$\alpha = 0.0000$$

New wines and older ones derived from these are related by a correlation coefficient of 0.947, $Y = aX^b$ (Fig. 3):

$$(mg\ litre^{-1}\ P_2O_5)_{aged\ w.} = 1.317 \times (mg\ litre^{-1}\ P_2O_5)_{new\ w.}^{0.9556} \\ \alpha = 0.0000$$

Figure 4 shows the frequency histogram of phosphate content in the studied wines. The concentrations are well below the levels needed for phosphatoferric 'cloudiness' to occur (Ribereau-Gayon *et al.*, 1980); moreover, this only occurs with a pH of less than 3.5, which is why it is not found in some wine regions, among them the area studied here (Peypaud, 1977).

Since the mineral content has a direct correlation with the maturity of the grape, *P* also shows a positive statistical correlation (*c.c.* = 0.485 and 0.464, respectively) with sugar concentration and must density (González Hernández, 1994). The α values are 0.0011 and 0.0020, respectively.

If we carry out multiple correlations between the three anions studied, using any two as independent variables and the third as the dependent one, we find that there is no significant correlation between the three parameters. However, there is a linear correlation between chlorides and sulphates in musts (Fig. 5), with a correlation coefficient of 0.482 and with a certain degree of statistical significance ($\alpha = 0.0012$), indicating that concentration of the two ions in musts is chiefly dependent on the mineral content of the grape and

increases or decreases in parallel. The equation of the corresponding curve, determined by least-squares regression, was found to be:

$$(mg\ litre^{-1}\ NaCl)_{must} = 84.42 + 0.3347$$

$$\times (mg\ litre^{-1}\ K_2SO_4)_{must}$$

CONCLUSIONS

In the case of boron, no statistically significant differences are observed between harvests or between young and old wines.

Sulphate levels are well below the legal limit, although the storage vessel is seen to play a major role in content, which is higher in wines made in cement receptacles and stored in wooden casks. Chloride levels are in line with the figures cited in the literature, while phosphates show a direct correlation with sugars and must density, indicating that concentration depends largely on the maturity of the grape.

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