

PRODUCTION WINE ANALYSIS

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PRODUCTION WINE ANALYSIS

Preface

This text is designed to acquaint the reader with the commonly used procedures of juice and wine analysis as they are generally practiced in the industry, and as they are taught in the Department of Enology at California State University, Fresno. It is assumed that the reader has a basic preparation in the fields of chemistry and microbiology.

In developing material for this text, the authors have emphasized analyses as they would be carried out in a production laboratory. Realizing that different laboratories have different analytical capabilities, personnel as well as equipment, we have in many instances provided several different approaches to the same analysis. Throughout this book we have attempted to give special attention to practical considerations and the importance of these analyses in the total spectrum of winery operations. We hope the book's format will satisfy the interests of laboratory personnel as well as winemakers.

The process of making wine involves a series of concerns for the winemaker and staff of a winery. The first concerns are viticultural. Upon arrival of the fruit, its quality is assessed, grapes are processed and fermentation is begun. Almost immediately, and in many instances simultaneously, chemical and microbiological stability of the young and/or aging wine become important. Finally, problems do occur on occasion, and a number of what may be considered remedial techniques can be employed to produce an acceptable product.

We have used these general areas of concern as a loose framework in dividing the book into its major sections. There is, of course, much overlap between the sections; a given analytical test may be run for several reasons during the production of a wine. However, the major chapter divisions should guide the reader through the important areas of concern and provide some idea of when certain laboratory tests will be run. There is occasionally some difference in how terms are technically defined and how they are used in current practice in the industry, and we have attempted to make allowances for this. The reader will, therefore, find temperature values specified in degrees Celsius in some places and degrees Fahrenheit in others. Concentrations of low-level constituents are usually calculated in milligrams per liter (mg/L) although we are aware that much of the industry refers to these concentrations in parts-per-million (technically, mg/Kg)

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units. To make conversions between these temperature and concentration units, we have provided simple mathematical relationships in the Reagent Preparation section (Appendix II).

Each chapter is divided into introductory considerations of theory and application, intended to give the reader necessary background information, followed by a laboratory procedures section that outlines each analysis in detail. Each laboratory procedure concludes with a consideration of special precautions for the analysis in question. Appendixes covering chromatographic theory, reagent preparation, stains, and media preparation also are included.

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Section I

Sampling, Fermentation, and Production Analyses

This section begins with definitions of fruit maturity and quality starting in the vineyard, and describes measurement techniques for these factors. During initial processing and fermentation, analyses related to the production of ethanol, other alcohols, and structural considerations (including extract) are presented. This is also an appropriate place to deal with concepts of acidity, and corresponding measurements of pH, individual acid levels, and titratable and volatile acidity. Analyses for residual sugar levels found in fermented products, as well as the important phenolic compounds, are discussed in the context of their relationship to wine balance, body, astringency, aroma, flavor and color. Finally, levels of dissolved gases such as oxygen and carbon dioxide, are discussed, as well as the important concept of oxidation in juice and wine.

Chapter 1

Fruit Quality and Soluble Solids

MATURITY SAMPLING

Grape maturity assessment as well as fruit quality is critical to the successful production of palatable wines. Part of the grower's payment for the fruit often is based upon delivery of grapes at agreed-upon parameters; so care must be taken in the maturity monitoring process. Traditionally, degrees Brix (°B, weight percent sugar in the juice) and titratable acidity (g/L of acid in the juice) have been selected as the harvest parameters most important in monitoring maturity. Because of the importance of fruit maturity to ultimate wine palatability, field sampling of fruit must be performed in an objective and statistically acceptable manner.

Amerine and Roessler (1958 a, b) compared sample collection techniques and reported that berry sampling can provide an accurate and economical sampling technique. Theoretically, to be within plus or minus 1° B with a probability level of 0.05 (95 out of 100 samples), two lots of 100 berries each should be examined. In cases where one wishes to be within plus or minus 0.5° B 95% of the time, five lots of 100 berries should be collected.

Berry sampling is difficult because of the time required and variations in fruit chemistry. The chemical composition of grape berries differs with their position on the rachis, the location of the cluster on the vine, and the location of the vine in the vineyard. However, properly performed berry sampling can provide an accurate picture of the overall vineyard fruit chemistry. Jordan and Croser (1983) recommend collection of berries from the top, middle, and bottom of the cluster. Terminal berries on the rachis may be less mature than other berries. Berry sampling in various locations on the cluster may be significant in the case of larger clusters; but many persons, sample by selecting berries only from the middle of the rachis, a technique that may be acceptable in the case of varieties with small clusters. Additionally, in a berry sampling procedure the side of the cluster from which the berries are taken must be randomized. One should avoid selection of fruit from ends of rows or from isolated vines or those with obvious physiological or morphological differences. Vine (1981) suggested an alternating-row method of sample selection. Significant variations in soil type,

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shading, and so on, may play an important role in vine growth and therefore fruit maturity. Ideally, sampling should be designed to reflect differences in soil type, topography, and vine growth. For consistency, some recommend that selected vines within each block be targeted for sampling.

Samples should be collected at approximately the same time period each day. Jordan and Croser (1983) have made the following sampling recommendations: (1) edge rows and the first two vines in a row should not be sampled; (2) samples should be collected from both sides of the vine; (3) for each row, estimate the proportion of shaded bunches and sample according to that proportion. This proportion may vary with the side of the row sampled because of variation in leaf cover. Smart et al. (1977) demonstrated that white and red grapes exposed to direct sunlight may be as much as 8°C and 15°C, respectively, warmer than the ambient air temperature. Such exposure may have a dramatic effect on fruit composition. Kliewer (1985) summarizes these differences in Table 1-1. As the table shows, a sampling technique that does not consider the effect of exposure to the sun may not be accurate.

There is a natural tendency when one is picking individual berries to select those with the most eye appeal. These are often the more mature berries. Such bias in a vineyard sampling technique may result in Brix readings that are as much as 2° B higher than that measured by the winery after crushing the entire vineyard load (Kasimatis 1984).

Most growers begin sampling fruit several weeks prior to harvest. Initially, samples are taken once weekly, with a shift to more frequent intervals as harvest nears. Brix and titratable acidity are the traditional harvest parameters most important in monitoring maturity. Recently, the importance of pH, aroma, organic acid, etc., has been established.

Measurements of °B, titratable acidity, and pH, by themselves, may not be specific indicators of physiological maturity or potential wine character and pal-

Table 1-1 Comparison of shaded and control Cabernet Sauvignon fruit.

| Parameter measured | Control | Shade | Significance level |
|--------------------------|----------|---------|-----------------------|
| Harvest date | 27 Sept. | 19 Oct. | |
| °Brix | 23.3 | 21.1 | + |
| T.A.(g/L) | 5.5 | 6.4 | + |
| pH | 3.44 | 3.74 | ++ |
| K ⁺ | 2325 | 2510 | +++ |
| Malate (g/L) | 1.65 | 2.84 | ++ |
| Tartrate (g/L) | 0.86 | 0.83 | ns |
| Anthocyanins (g/g fruit) | 0.98 | 0.56 | +++ |

¹Control treatment was a bilateral cordon 3-wire "T" trellis. Shaded treatment consisted of bunching the foliage around the fruit using bird netting.

SOURCE: Kliewer and Benz 1985.

atability. These parameters will vary considerably, depending upon the season, crop load, soil moisture, and so on. Maturity is clearly a multidimensional phenomenon, and should be viewed on a relative rather than an absolute basis.

There is considerable variation in the definition of optimum maturity for a particular wine grape variety. If maturity is defined as that point in time when the berry possesses the maximum varietal character consistent with the type and style of wine desired, then the assessment of varietal character is a logical adjunct and extension of conventional definitions of maturity. Further, the optimal maturity may vary somewhat with the style of wine to be produced. Long (1984a) lists the parameters she considers important in affecting fruit development and maturity assessment, which are summarized in Table 1-2. The relative importance of each parameter is predicated upon the type and style of wine to be produced as well as the ability of the vine to continue to mature the crop. Several of these parameters are described in more detail below.

Contribution of Juice Aroma

Aroma and flavor in wine arise from several sources, including the grape and an array of chemical changes that may occur during juice processing, fermentation, and aging of wine. As the fruit matures, the aroma develops from "green" underripe tones to the more complex characteristics of the particular cultivar.

The floral, citrus, honey, and melon aromas of Chardonnay as well as the black currant, blackberry, spinach, and bell pepper of Cabernet sauvignon are

Table 1-2. Factors to consider in maturity assessment and picking decisions.

| Factors | Importance | Chapter Discussed in |
|-------------------------|--|-------------------------|
| °Brix | Final alcohol level | 1 |
| Sugar/berry ratio | Maturation/dehydration- rehydration | I |
| Aroma assessment | Varietal aroma/aroma intensity-maturity | 1 |
| Titratable acidity | Balance, style | 4 |
| pH | Balance, stability, etc. | 4 |
| Malic acid | Balance + malolactic involvements | 4, 12 |
| Vine condition | Potential for further ripening | 4 |
| Grape condition | Maturity, potential for further ripening | 1 |
| Color and phenol levels | Maturity, color, etc. | 7 |

SOURCE: Long 1984.

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properties that, although modified by processing and aging, have originated in the grape to a significant degree. Naturally the more neutral the grape, the more difficult it is to use aroma as a maturity adjunct. There is little doubt about the importance of grape aroma components to the palatability of the finished product. If grapes are harvested when varietal character is lacking or deficient, varietal character in the finished wine subsequently will be absent or reduced. Aroma evaluations are based upon subjective descriptors, such as those utilized in the aroma wheel developed by Noble et al. (1984, 1987), as well as estimations of aroma intensity.

Aroma evaluation of the juice can be used as a stylistic tool to note potential varietal character and intensity (Cootes, et al. 1981). If more aroma is required, assuming that the potential for more aroma development exists, harvest may be delayed. In California, Simi Winery developed an evaluation system using a 0 to 10 Intensity/Quality (I/Q) Scale (Table 1-3). A methodology for grape aroma evaluation is presented at the end of this chapter (see Procedure 1-1).

Procedures for rapid and easy chemical evaluations of certain aroma components (monoterpenes) have been developed (Dimitriadis and Bruer 1984; Dimitriadis and Williams 1984). Such methods may aid in reducing the subjectivity of aroma evaluation of certain cultivars such as Rieslings, Muscats, and Gewurtztraminer.

Color and Phenols (see also Chapter 7)

According to Peynaud (1984), vinification is the art of fractional extraction of the grape. Because of the importance of phenolic compounds to color, this is of particular significance in red wines. There is a sound basis for using the level of total anthocyanins and total phenols in the fruit as an additional harvest criterion. However, anthocyanin levels appear to vary more widely than any other commonly measured compounds in grapes in response to environmental influences. Thus, it remains difficult to measure these levels on a routine basis, and to interpret the results in relation to winemaking.

Table 1-3. Rating scale for evaluation of grape maturity. Scores will be recorded as I/Q (Intensity /Quality), each on a scale of 1 to 10.

| I: | No aroma at all | 0 pts |
|----|---|----------|
| | Neutral—aroma of neutral juice | 1-2 pts |
| | Slightly varietal aroma | 3-5 pts |
| | Distinctive varietal aroma | 6-8 pts |
| | Very distinctive varietal aroma | 9-10 pts |
| Q: | Spoiled or off quality | 0 pts |
| | Broad, uninteresting, unpleasant aroma | 1-2 pts |
| | Some pleasing varietal character | 3-5 pts |
| | Distinctive varietal aroma | 6-8 pts |
| | Very distinctive varietal characteristics | 9-10 pts |

SOURCE: Simi 1983.

Grower Input

Grower input as it relates maturity decisions refers to the intimate relationships between vineyard management, fruit chemistry, and ultimately, wine palatability. Such factors as vine population, training, trellis systems, hedging, crop load, spray schedule, fertilization, irrigation, and so on, will influence fruit ripening and maturity decisions. For example, overcropping delays maturity and can result in reductions in titratable acidity, malic acid, phenols, the levels of volatile components, and color, as well as causing an elevation in berry pH.

Sugar per Berry

Simple measurement of Brix, as described in Procedures 1-3 and 1-4, although a useful tool in wine production, has certain drawbacks when used by the grower and winery to evaluate crop maturation. First, Brix is defined as sucrose per 100 g juice. It is a measure of the grams of all soluble solids per 100 g juice, and thus includes pigments, acids, glycerol, and so on, as well as sugar. Because the fermentable sugar content of grape must accounts for around 95% of the total soluble solids, determination of °B provides a reasonable approximate measurement of sugar levels. However, this measurement involves a ratio (wt/wt) of sugar to water that may change because of the physiological condition of the fruit. Another problem one encounters when using standard methods of °B determination is that no consideration is given to the weight of fruit used in the sample preparation. Thus, it is entirely possible that over a period of days, °B may show no change, but in fact there may be major changes in the weight of fruit (either increases or decreases) comprising that sample. Obviously this information would be of value to both the grower and the vintner in selecting harvest dates to optimize fruit maturity.

Several wineries have evaluated alternatives to traditional interpretation of soluble solids measurements (°B). As compared with °B, which measures soluble solids (in g/100 g juice) of a collected sample, the concept of reporting sugar per berry utilizes the same initial Brix measurement but extends it such that the final value takes into account the weight of a berry sample.

For example, the data of Table 1-4 were collected from randomly selected 100-berry samples. The samples were taken from the same vineyard at five-day intervals. Upon sample preparation, the soluble solids content (°B) by refractometry of both samples was measured to be 22° B. The grower's conclusion was that there had been no change in the maturity of the fruit. However, sugar

Table 1-4. Sample harvest data for 1985 Barbara grapes.

| | Sept. 5 | Sept. 10 |
|---------------|-----------|----------|
| °B | 22 | 22 |
| Sample weight | 22 112 | 118 |
| (grams) | | |