#### AN ABSTRACT OF THE THESIS OF

Rita Miranda-Lopez for the degree of Master of Science in Food Science and Technology presented on December 7, 1990.

Title: A Maturity Trial Study of Pinot noir Wines: Aroma

Profile by Sniffing Gas Chromatographic Effluent

Abstract	approved:		
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The quality of wine is highly influenced by the weather. Temperature, solar radiation and humidity influence the formation and concentration of aroma-active compounds and aroma-active precursors in the grapes. Pinot noir grapes require a slow ripening, under cool temperatures, in order to achieve their fullest flavor. The main objective of this study was to provide an overall aroma picture that could help, along with the chemical indices, to decide the optimum harvesting time to obtain certain distinctive aroma attributes in the wine.

This particular work represents the first stage in a broad plan aimed to understand the dependence of the wine flavor chemistry on the ripening of the grapes. This maturity trial was planned to last several years; it was expected that the outcome and techniques developed in the present study would be essential in delineating the steps to follow. The results of the aroma analyses for the 1987 and 1988 vintages are reported here. In each vintage, Pinot noir grapes were harvested at three different times, covering a range from early to late maturity.

A sniffing technique based on gas chromatographic aroma detection by a trained panel was implemented. This technique has proved to be a useful tool to measure qualities and intensities of aromas. The method was effective in detecting many of the aroma-active compounds and in identifying aroma differences between the wines studied.

The aroma profiles for the wines were found to be very different from each other within and across vintages. There were only 10 aroma peaks common to all three 1987 wines, 16 aroma peaks common to the 1988 wines, and 4 aroma peaks common to both vintages. Late maturity wines had more aroma-active peaks than the other 2 wines for both vintages. The 1988 wines had a higher number of aroma-active peaks than the 1987 wines. The percentages of aroma-active peaks not detected by the Flame Ionization Detector (FID) were 45% in the 1987 wines, and 66% in the 1988 wines. The overall climatic conditions in those years were very different. The 1987 season was characterized as hot and dry, producing an early harvest. The weather in 1988 was more of a typical season for Oregon, producing a normal to late harvest. Further study is needed to

fully understand the flavor chemistry occurring during grape ripening.

# A Maturity Trial Study of Pinot noir Wines: Aroma Profile by Sniffing Gas Chromatographic Effluent

Ву

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A THESIS

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#### **ACKNOWLEDGEMENTS**

TO BE{[IN THE RIGHT(PLACE + TIME)] + (WITH THE RIGHT PERSON)}
= TO FOLLOW GOD'S ADVICE

TO MAKE THE RIGHT DECISION IN THE RIGHT MOMENT UNDER THE RIGHT CONDITIONS = TO DO SCIENCE UNDER GOD'S ADVICE

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# A MATURITY TRIAL STUDY OF PINOT NOIR WINES: AROMA PROFILE BY SNIFFING GAS CHROMATOGRAPHIC EFFLUENT

#### INTRODUCTION

Grapes are a nearly ideal substrate for yeast fermentations (producing wines), because of their high sugar content, fruit acids, minerals and other compounds. Another grape characteristic is that the grape flavor character is not lost after fermentation. Grapes have been harvested for winemaking since prehistoric times; since then numerous investigations have been undertaken in order to improve the quality of the wine. In climates like the northwest, where every year the weather is unpredictable, the quality of wine is always in jeopardy. Each year, the winemakers simply do not know the best time to harvest the grapes. In order to balance the taste, there is always the option of harvesting early and adding sugar or harvesting late and adding acid; but, it is impossible to add aroma-active compounds to get the appropriate wine aroma.

Even though grape quality, from a winemaking viewpoint, is difficult to define, researchers have been trying to predict when the grapes reach their maximum quality. Most of these

studies have focused on the physiological maturity, including color. Only a few studies have been done in order to understand how the flavor composition is affected by grape maturity. A Pinot noir maturity trial was initiated in 1987 in the Departments of Horticulture and Food Science and Technology at Oregon State University. The purpose of this trial was to understand how the aroma of finished wines differ dependent on grape maturity, in order to facilitate development of a commercial tool to monitor changes in fruit composition and character. Although this maturity trial will be continued for several years, only the 1987 and 1988 vintage results are reported here.

#### 1.1) The weather records

The monthly weather reports described, in general, 1987 to have been hotter than 1988. The only time that 1988 was hotter than 1987 was in July. The weather for both years was almost the same from mid August to October. The monthly precipitation was higher in 1987 than in 1988 from January to March, and in July. It was lower in 1987 than in 1988 from March to June, and from August to October.

1.2) Vineyard site and description of the maturity trial

The Pinot noir experimental block is at the Woodhall III Vineyard in Alpine. It is a typical Willamette Valley, slope hillside, non-irrigated site, with clay-loam soil. When the fruit started to ripen, chemical indices were determined. The <sup>O</sup>Brix of the must (crushed grapes) determines the harvest season. Pinot noir wines have good varietal character in western Oregon when <sup>O</sup>Brix values in must range from 20 (generally the start of the harvest season) to 25 (generally the end of the harvest season); this conclusion was drawn after seven years (1976-1982) of monitoring grape maturity and wine composition (Lombard et al., 1986).

For 1987, the harvest season started on September 4 and finished on September 29. In 1988, the harvest season started on September 29 and finished on October 25. For further details of the trial refer to Watson (1988) and Watson et al. (1989).

1.3) Selection of grape maturity levels to be evaluated in each vintage

Five maturity levels from 1987 and six maturity levels from 1988 were sampled. Due to time constraints, it was impossible to study more than three harvest dates per vintage. Therefore, preliminary tastings were conducted to aid in selection of the wines to be studied. A group of researchers

familiar with Pinot noir wines selected the wines to be used in this study. They evaluated the aroma and taste of the 5 wines of the 1987 vintage and the 6 wines of the 1988 vintage. Each set of wines was evaluated at different times; the 1987 wines were evaluated in Spring 1989 and the 1988 wines were evaluated in Fall 1989. Wines were presented in tulip shaped wine glasses, covered with a watchglass containing 30 mL of sample: each researcher was seated in individual testing booths with subdued red lighting, and glasses were coded with a 3digit random number. After the wines were evaluated a discussion among the researchers took place. Three wines were selected (from each vintage) on the basis that each wine was very different from the others in aroma and taste. The three wines covered the range of an early, middle and late harvest time typical for Oregon. For the 1987 vintage, the "early" wine was harvested September 11, the "middle" wine was harvested September 16, and the "late" wine was harvested September 29. For the 1988 vintage, the "early" wine was harvested September 29, the "middle" wine was harvested October 13, and the "late" wine was harvested in October 25.

## 1.4) Pinot noir composition of must and finished wine

The must analysis at harvest and ethanol content in the finished wine for the 6 Pinot noir wines can be found in Table 1.

In 1987, the earliest Pinot noir grapes were harvested September 11 with a Brix of 20.4, a titratable acidity (TA) of 7.9 g/L and pH of 3.4; the weather during this period was hot and dry, and the increase in 'Brix was slow; the decrease in acidity and the increase in pH were abnormal for Pinot noir at this stage of harvest. By September 16 the Brix increased to 21.0, the TA dropped slightly to 7.2 g/L, but the pH had dropped from 3.4 to 3.27; this harvest date corresponded to the only time during the ripening season when there was any measurable rainfall, probably causing the decrease in some of the chemical The latest Pinot noir grapes were harvested by September 29, the <sup>O</sup>Brix increased to 24.4, the T.A. continued to decrease to 6.2, and the pH rose sharply to 3.6. Malate content was almost the same from the early to the middle harvest sample, but decreased from the middle to the late harvest sample; the opposite happened to the tartrate content as it increased from early to middle, and stayed almost the same from the middle to late harvest wine. The percentage of ethanol in the finished wine was 11.1 in the early harvest wine, 11.3 in the middle harvest wine, and 13.6 in the late harvest wine.

In 1988 the harvest season began nearly a month later than in 1987, thus ripening was considered slower in the 1988 vintage. The earliest Pinot noir grapes were harvested September 29 with a 'Brix of 21.8, a T.A. of 10.2 and pH of 3.23. "Optimal" physiological maturity by conventional standards with

respect to °Brix, TA and pH was probably reached by early to mid October. By October 13 the °Brix increased to 23, the TA declined to 8.2, and the pH increased to 3.34. For the last Pinot noir grapes, harvested October 25, the OBrix continued to increase to 24.7, the TA decreased to 7.6, and the pH increased slightly to 3.37. The rapid increase in pH after the ripening period in 1987 did not occur in 1988. Malate and tartrate contents decreased with the maturity of the grape. The percentage of ethanol in the finished wine was 11.8 in the early harvest wine, 13.6 in the middle harvest wine, and 14 in the late harvest wine.

The °Brix and TA levels in the must at harvest and the ethanol content in the finished wine were higher in 1988 than 1987, however, at the end of the harvest the sugar was almost at the same level in both years, 24.4 in 1987 and 24.7 in 1988. On the contrary the total changes in malate content of the must were remarkably similar both years despite the differences in weather during maturation. Malate decreased from about 4.7 to 3.2 g/L. Tartrate content increased with the maturity of the grapes in 1987, while in 1988 it decreased. The 1987 vintage was considered a warm and dry season, while 1988 was characterized as a "later than normal" season.

## 1.5) Winemaking procedure

The Pinot noir grapes were picked in the morning. By noon, the grapes were crushed and destemmed, and 25 ppm of SO<sub>2</sub> was added. After an hour, the must was inoculated with rehydrated yeast (Wädenswil yeast) at a ratio of 1 gr yeast/gal of must, and fermented for 8-10 days. Then, the juice was pressed from the grape pulp with the Willmes bladder press. Finally, the wine was inoculated with 20 mL/gal of malolactic bacteria (Er1a) culture, and maintained at 18°C until malic acid disappeared. After completion of malolactic fermentation, moderate tartaric acid additions were made to lower the pH and attain a proper taste balance in the different lots as necessary (Yorgey, 1990). For more details refer to Watson (1988) and Watson et al. (1989).

### 1.6) Objectives of this study

- a) To train a GC-sniffing panel. Each panelist should be capable of describing aromas and rating intensities from aroma-active peaks eluting from a GC column.
- b) To identify by GC-MS compounds present in Pinot noir wines.
- c) To confirm identification through use of standards.
- d) To determine the aroma profile of Pinot noir wine extracts, by sniffing the GC-effluent.

- e) To compare how each of these aroma profiles differ, dependent on grape maturity within the same vintage.
- f) To compare how aroma profiles of wine extracts differ across vintages.
- g) To compare how the aroma profile of harvest dates differ from one vintage to the other.
- h) To develop a "fingerprint" of the aroma profile expected in an early, middle or late maturity wine.
- i) To study individual panelist variability.

#### LITERATURE REVIEW

#### 1) GRAPE MATURITY

The quality of wine is largely dependent on the composition of the grapes which is closely related to grape maturity. The notion of grape maturity is rather vague, because it is difficult to measure. Temperature, solar radiation, and moisture received during the growth period are among the most important factors influencing the development and maturation of the fruit. One must also recognize that grape maturation is not similar every year. Sometimes technological maturity (the point at which an optimum wine will result) is difficult to obtain, because it may not occur at the same time as physiological maturity (determined by grape weight, amount of sugar, pH and total acidity) which is most often used to determine the beginning of the harvest period (Lafon-Lafourcade, 1986).

Physiological grape maturity indices have been used as indicators of wine quality for many years (Ribereau-Gayon, 1978; Gallander, 1983; Hrazdina et al., 1984). Du Plessis (1984) reviewed the relationship between optimum maturity and quality parameters in grapes. Lafon-Lafourcade (1986) developed a rapid analytical technique to estimate free and

potential monoterpenes in Muscat, which is easily applicable in the control of maturity.

Several studies have been done in order to understand how wine flavor composition is affected by grape maturity. Nelson and Acree (1978) reported that the compounds responsible for the Concord varietal character are formed during the latter stages of maturation. Williams et al. (1984) found that a higher level of flavor compounds could be established in the grapes by leaving them on the vine for extended periods. Williams et al. (1986) reviewed the role of monoterpenes in grape flavor influenced by pre- and post-harvest factors. Cabrera et al. (1988) investigated the formation of higher alcohols, esters and terpenes by five yeast strains from grapes at different degrees of ripeness. Maturity studies related to the changes of various monoterpenes have been conducted by Hardy (1970), Williams et al. (1981), Augustyn and Rapp (1982), Marais (1983), Williams et al. (1984), and Strauss et al. (1986).

### 1.1) Pinot noir grapes and wines from Oregon

Physiological maturity indices of musts and finished wines from the 1976 to the 1982 vintages, are found in Information Circular 701, Agricultural Experiment Station, Oregon State University (OSU) (Lombard et al., 1986).

Approximate chemical indices range values for Oregon Pinot

noir must are: 21.5 - 24.5 <sup>O</sup>Brix, 6 - 9 g/L TA, 3.35 - 3.8 pH, 2 - 5 g/L malate and 2.5 - 8 g/L tartrate; and wine: 11.5 - 13.5 % ethanol (Yorgey, 1990). Tanger (1984) comments that, for Pinot noir wines from California, acidity and pH are more important than sugar content in creating wines of greater aging potential related to its varietal character.

McDaniel et al. (1987) reported the aroma descriptive analysis of Pinot noir wines fermented by several strains of malolactic bacteria. Er1a (a strain of malolactic bacteria produced at OSU) produced a high aroma character in overall intensity, fruity (mainly blackberry, cherry and dried fruit), spicy (mainly black pepper), caramelized (mainly honey and butterscotch), and a low aroma character in vegetative, earthy and chemical. It is known that an early maturity Pinot noir wine will abound in floral and vegetative character and a late maturity Pinot noir wine will abound in dry fruit and caramel character. Oregon Pinot noir is characterized for its berry, herbal, and black pepper character.

In 1987, a Pinot noir maturity trial was initiated in the Departments of Horticulture and Food Science at Oregon State University. Physiological maturity indices of Pinot noir musts and wines from the 1987 and 1988 vintage are described by Watson (1988) and Watson et al. (1989). Micheals et al. (1989) reported the sensory characteristics of Pinot noir wine from the 1987 vintage by a wine industry panel. McDaniel et al.

(1989) reported the sensory evaluation of 1987 Pinot noir wines by different panels: a trained descriptive panel (for evaluating wine attribute intensity), a winemaker panel (for rating wine quality parameters), and a GC-sniffing panel (for identifying aroma-active compounds).

#### 2) FLAVOR AND WINE

Among the various factors that contribute to the enjoyment of wine, its aroma is possibly the most important. Since several hundred different compounds can take part in the formation of wine aroma it is rare that a special component could be identified as being responsible for nuances of a specific flavor; wine aroma is formed by the balance of all those aroma-active components (Rapp and Mandery, 1986). Substances contributing to the aroma comprise many different classes of organic compounds, and they are normally minor constituents of the total volatile extract; their concentrations are measured in parts per million or even less. Rapp and Mandery (1986) report the thresholds of the volatiles to be between 10-4 and 10-12 g/L, and the concentrations of individual components can range from 10-1 to 10-10 g/kg.

## 2.1) Steps in the aroma analysis

The ultimate goal of basic aroma research is to isolate and separate the volatile constituents, and to establish the identity and importance of those substances responsible for the characteristic aroma and flavor of wine.

#### 2.1.1) Extraction

The majority of the volatile constituents of wine are very hydrophilic, so an organic non-miscible and inert solvent can be used for extracting the volatiles. Since the compounds present in wine represent a wide range of physical and chemical properties, i.e. concentration, volatility, functional group, etc., no one technique allows total and definitive extraction of the wine aroma constituents (Etievant, 1987; Fehl and Marcott, 1989; Williams and Tucknott, 1973).

Two of the most common solvents used for extraction are Freon 113 (1,1,2-trichloro-1,2,2-trifluoroethane) and ethyl acetate. The Freon extraction is always done first, followed by the ethyl acetate extraction of the remaining sample (Acree, 1989; Yong et al., 1989). Each organic solvent may extract aroma-active compounds to different extents and each solvent must be presumed to distort the true balance of concentrations of flavor components to some degree (Cobb et al., 1978).

Freon 113 is a non-polar solvent which extracts primarily non-polar components from the wine. Advantages of Freon 113

are that it will not extract water, or interfering fermentation products such as methanol, ethanol, and some organic acids. Freon is easy to handle in the laboratory because of its high density, high purity, low boiling point (48°C), low toxicity, and nonflammability (Tyler et al., 1978; Nelson et al., 1976; Cobb et al., 1978).

Ethyl acetate is the least polar solvent which can still be separated from water. The utility of ethyl acetate is that it extracts some polar compounds that Freon 113 is not capable of removing, like furaneol (Acree, 1989). There are some compounds with a certain polarity that could be extracted either by Freon or by ethyl acetate, like beta-phenyl ethanol. Ethyl acetate has a boiling point of 77°C. Disadvantages for using ethyl acetate are that it is highly miscible (7.9%) with water, it extracts some pigments and high molecular weight sugars, and it may undergo hydrolysis with esters being formed as artifacts.

# 2.1.2) Separation and detection by Gas Chromatography (GC).

Food extract analysis by GC provides a reasonably clear picture of its composition with respect to complexity, relative magnitude and distribution. GC using capillary columns has been the analytical tool of choice for measuring the volatile

composition in Pinot noir wine (Brander et al., 1980; Kwan and Kowalski, 1980), in other grapes and/or wines (Augustyn and Rapp, 1982; Etievant and Bayonove, 1983; Marais, 1986), for following changes in volatiles during grape ripening (Hardy, 1970) as well as for determining chiral flavor and aroma compounds (Engel et al., 1989).

Although GC has been a tremendous asset for the characterization of volatile composition, it also has its limitations as it provides no indication of a peak's aroma activity.

# 2.1.3) Identification and quantification of aroma-active compounds by GC-Sniffing

Since the development of GC procedures, scientists have routinely sniffed the individual compounds eluting from the end of the column to evaluate both quality and intensity. In most cases, there was no formal quantification of aroma intensity. Also, usually only one person's observations were noted. Observations of this type are reported for studies on wine aroma by Nelson and Acree (1978), Noble (1978), and Augustyn and Rapp (1982).

Acree et al. (1984) developed a technique named charm that formalizes the process of sniffing GC effluent. Charm is a human bioassay to determine the odor activity of each compound

in a mixture by sniffing the GC effluent through a series of In charm analysis, the volatiles are extracted from dilutions. the sample with solvents and separated by GC. The presence or absence of odor in the effluent as a function of time is recorded by the subject using a microcomputer. The odor variable has the value of "zero" (cannot be perceived) or "one" (can be perceived) at every time during the run. The sample is then diluted and chromatographed again. The weaker odors disappear, but the stronger ones are still detected. This serial dilution is continued until no odor is detected at all. Combining these results in a particular way produces a graph called a charm response chromatogram (Acree and McLellan, 1989). Each peak is caused by the perception of a different odor-active chemical. and the area under each peak equals the amount of that chemical in the sample divided by its odor-detection threshold. The largest peaks in the graph correspond to the chemicals with the most intense odor. Applications of charm analysis have been used to determine the odor activity of compounds in wine (Nelson et al., 1978; Nelson and Acree, 1978; Acree and Cottreli, 1985), grape juice (Braell, 1986), apples (Acree and McLellan, 1989), crackers (Yong et al., 1989), and to determine variability in thresholds (Marin et al., 1988).

Schieberle and Grosch (1984, 1987 and 1988) developed a similar method called Aroma Extract Dilution Analysis (AEDA). This technique is a systematic approach to evaluate the potent

odorants of a food. A stepwise-dilution is done by fractionation using column chromatography, and sometimes followed by HPLC. Each volatile fraction is separated by GC and the odors are perceived at a sniffing port. The sensory significance of each odorant is evaluated by AEDA, in which an odor unit of a compound is defined as the ratio of its concentration in the aroma extract to its odor threshold. An odor unit is equal to 1 in the undiluted sample. An aromagram is obtained by plotting the odor unit values against the retention index. The results obtained by AEDA are directly proportional to the charm results (Schieberle et al., 1990). Applications of AEDA analysis have been used to determine the odor activity of compounds in wheat and rye bread (Schieberle and Grosch, 1984 and 1987), in cherries (Schmid and Grosch, 1986), in the autoxidation of linoleic acid (Ullrich and Grosch, 1987), in lemon oil (Schieberle and Grosch, 1988), and in cucumbers and muskmelons (Schieberle et al., 1990).

McDaniel et al. (1989) and Sanchez (1990) have developed another method, not based on thresholds, but based on aroma intensity at a single stimulus level. The panelist responds to the intensity of the stimulus by using a time-intensity device with a 15 cm scale ranging from none to extreme. At the same time verbal descriptions of the aroma are collected by the experimenter. This method is very different from the previous reported charm and AEDA studies in that each sample is

replicated four times by each of the four panelists. A consensus aromagram is made with those aroma-active peaks that are repetitive in 2 out of 4 replicates per panelist, as well as those perceived by at least 3 out of 4 panelists. A major finding by McDaniel et al. (1989) and Sanchez (1990) is that large differences in perception occur among the panelists, thus supporting the need for the development of consensus aromagrams. They have used this method to identify significant aroma-active peaks in wine (McDaniel et al., 1989) and beer (Sanchez, 1990).

#### 2.2) Flavor development in wine

#### 2.2.1) Introduction

The first studies on wine aroma were performed by Henning and Villforth (1942). Using classical chemical methods they managed to identify a few compounds. In the late fifties Bayer and Bassler (1961) were the first to apply GC in the field of wine aroma; they identified some higher alcohols and a few esters. The sixties were characterized by the expansion of GC as a method to isolate and identify aroma components in wines (Webb, 1967). The seventies were characterized by the development of GC-MS coupling; Schreier (1979) reviewed the flavor composition of grapes and wines. During the past 30

years the knowledge of the flavor of alcoholic beverages has thoroughly changed. The continued development of sophisticated instrumental methods (such as NMR, Fourier transform IR spectroscopy, etc.), together with the increased objectivity of sensory evaluation techniques have resulted in a more rigorous approach to the understanding of the complex subject of wine flavor. Much work has been devoted to investigating biochemical mechanisms which today enables researchers to describe routes by which the flavor compounds in alcoholic beverages are formed (Muller et al., 1973; Marais, 1983; Nykanen, 1986; Strauss et al. 1986; Williams, et al. 1986; and Williams et al. 1989).

### 2.2.2) Flavor compounds of interest to this study

Aroma in wine comes from compounds which originate from the grapes during ripening and from processing (action of certain enzymes during the crushing of the grapes), aroma compounds produced by fermentation, and the bouquet which results from the transformation of aroma compounds during aging. The amounts of the aroma components can be influenced by environmental factors (climate, soil), cultivar, the condition of the fruit, the conditions during fermentation (pH, temperature, juice nutrients, microflora) and various post-fermentation treatments (clarification, blending, etc.).

#### 2.2.2.1) Acids

The quantitatively dominating acids of grapes are malate, tartrate, and citrate, the first two of which account for over 90% of the total acid content of grapes. Most of the fatty acids present in wine result from the winemaking process, principally from alcoholic fermentation (Kepner et al., 1969; Schreier, 1979; Brander et al., 1980; Shinohara, 1985; Noble et al., 1987). The largest amount of volatile fatty acids of chain length up to 12 carbons consists of compounds like hexanoic, octanoic and decanoic acids (Schreier, 1979). Although those volatile acids are present in wines in only trace quantities, their very low odor thresholds and their very pungent odors undoubtedly make them important aroma contributors. Some of the acid compounds of interest to this study are: isovaleric or 3-methyl butyric acid, with a rancid, cheese, sweaty and putrid odor; hexanoic or caproic acid, with a sour, vinegar, sweaty, rancid and pungent odor; octanoic or caprylic acid, with a oily, fatty and rancid odor; and decanoic or capric acid with a fatty, unpleasant, rancid odor.

### 2.2.2.2) Alcohols

A number of aliphatic alcohols with more than two carbon atoms found in wines come from grapes or are produced by various mechanisms during alcoholic fermentation. Some of the alcohols found in grapes which are not influenced by yeast fermentation are: 2-ethyl-1-hexanol, 3-octanol, 1-octen-3-ol and 1-phenethanol (Schreier, 1979).

The following alcohols come either from the grape, or as a fermentation product: hexanol (Killian and Ough, 1979; Brander et al., 1980; Etievant and Bayonove, 1983; Noble et al., 1987), benzyl alcohol (Schreier, 1979; Brander et al., 1980; Simpson, 1980; Williams et al., 1982a; Hrazdina et al., 1984; Noble et al., 1987), and beta-phenyl alcohol (Brander et al., 1980; Etievant and Bayonove, 1983; Hrazdina et al., 1984; Nykanen, 1986; Rapp and Mandery, 1986; Noble et al., 1987). Benzyl alcohol, beta-phenyl ethanol (Williams et al., 1982a), linalool (Shimizu and Watanabe, 1981; Williams et al., 1981, 1982 and 1982b; Etievant and Bayonove, 1983; Rapp and Mandery, 1986; Noble et al., 1987) and alpha-terpineol (Williams et al., 1981, 1982 and 1982b; Rapp and Guntert, 1985; Rapp and Mandery, 1986; Noble et al., 1987; Rapp, 1987) are liberated from glycosides present in the grapes.

There are some alcohols produced from yeast fermentation, among the commonly known are 2-methyl-1-butanol, 3-methyl-1-butanol, n-propanol, isobutanol and betaphenyl ethanol (Schreier, 1979; Thoukis, 1981; Nykanen, 1986;

Rapp and Mandery, 1986), as well as 2,3-butanediol (Simpson, 1980), and cis-3-hexen-1-ol (Brander et al., 1980).

Some of the other alcohols found in Pinot noir (Meunier and Bott, 1979; Brander et al., 1980; Schreier, 1980; and Cliff, 1989) are: 3-methyl-1-butanol or isoamyl alcohol with a vegetative, pungent and chemical odor; n-pentanol with a sweet, balsamic; n-hexanol with a fragrant, woody, resinous, heavy, herbal, green odor; cis-3-hexen-1-ol with a fresh, green and grassy odor; linalool with a lavender, floral, citrus, fruity and sweet odor; 2,3-butanediol with a faint, sweet, perfume, almond, and artificial fruit odor; alpha-terpineol with a pine oil, floral, and faint fruity odor; benzyl alcohol with a sharp burning taste and faint aromatic odor; and beta-phenyl ethanol with a floral, rose, fragrant and perfumery odor.

## 2.2.2.3) Aldehydes

Most of the aldehydes present in grapes or grape juice are only detectable in wines in the initial phase of fermentation (Nykanen, 1986; Schreier, 1979; and Rapp and Mandery, 1986). Among the carbonyls, acetaldehyde is the major component and generally constitutes more than 90% of the total aldehydes present (Nykanen, 1986). Benzaldehyde is one of the aldehydes found in Pinot noir (Meunier and Bott, 1979; Brander et al.,

1980; Cliff, 1989) which has an almond, sweet and cherry fragrance.

#### 2.2.2.4) Esters

Numerically, fatty acid esters are the largest group of flavor compounds in wine. As volatile compounds which impart a pleasant smell, they are of great importance to wine aroma (Nykanen, 1986). Although small quantities of esters are present in grapes prior to fermentation, the amounts are negligible compared to those introduced enzymatically by the yeast (Ramey and Ough, 1980). The fatty acid biosynthesis is explained by Killian and Ough (1979), Brander et al., (1980), and Nykanen (1986).

Some of the esters found in Pinot noir (Meunier and Bott, 1979; Brander et al., 1980; Schreier et al., 1980a; Cliff, 1989) are: ethyl hexanoate or ethyl caproate with a fruity, apple, banana and wine aroma; ethyl octanoate or ethyl caprylate with a fruity, floral, banana, pineapple, pear and brandy aroma; ethyl decanoate or ethyl caprate with a brandy, oily, fruity and grape aroma; ethyl dodecanoate or ethyl laurate with a soapy, citrus, detergent odor; and ethyl palmitate with a soapy and waxy odor.

Ethyl lactate and diethyl succinate are not true products of fermentation (Riberau-Gayon, 1978). Rapp and Guntert (1985) report that diethyl succinate is formed in low

concentration during fermentation. These two esters are found in Pinot noir (Meunier and Bott, 1979; Brander et al., 1980; Schreier et al., 1980a; Cliff, 1989); ethyl lactate with a fruity, sweet, cherry and tropical fruit aroma, and diethyl succinate with a faint and pleasant aroma.

Phenyl ethyl acetate has been found in grapes (Schreier, 1979) and its formation in wine depends upon the concentration of phenyl ethanol during sugar fermentation (Nykanen, 1986). This ester is found in Pinot noir (Meunier and Bott, 1979; Brander et al., 1980; Schreier et al., 1980; Cliff, 1989) and has a sweet honey aroma.

# 2.2.2.5) Lactones

Among the many volatile constituents of wine, the lactones (delta- and gamma-lactones) might be responsible, to a certain extent, for the pleasant aroma of wine. Smith (1989) comments that a small change in the structure of the lactone will change the aroma from coconut to peach to celery to beef bouillon. The quantification may be difficult due to problems associated with their isolation and their low concentration in wine, as well as some analytical problems due to their thermal stability (Ryder, 1965; Muller et al., 1973; and Nakamura et al., 1988). The metabolic origin of gamma-lactones was

investigated by Muller et al. (1973), Schreier (1979), Etievant and Bayonove (1983), and Engel et al. (1989).

Some of the gamma-lactones commonly found in Pinot noir are the following; gamma-butyrolactone (Brander et al., 1980; Schreier et al., 1980) which is considered to have a faint, sweet and caramel aroma; gamma-octalactone (Meunier and Bott, 1979; Schreier et al., 1980) having a coconut like aroma; and gamma-nonalactone (Meunier and Bott, 1979; Brander et al., 1980; Nakamura et al., 1988) which has a sweet odor reminiscent of coconut, apricots, peaches, and pineapple, as well as licorice and anise. The odor threshold of gamma-nonalactone is supposed to be extremely low and, hence, very small amounts might be sufficient to affect the total flavor. Its concentration in Pinot noir is 16-27 ppm, which is close to or exceeding the threshold (Nakamura et al., 1988).

# 2.2.2.6) Phenolic substances

Phenolic substances are very important to the taste, color and odor of wines. The metabolic pathways from which phenolic compounds arise were studied by Schreier (1979), Etievant (1981), and Rapp and Mandery (1986). Acetovanillone, and methyl and ethyl vanillate are the phenolic compounds of interest to the present study. Ethyl vanillate (Tesniere et al., 1989), methyl vanillate (Williams et al., 1989), and

acetovanillone (Schreier, 1979; and Williams et al., 1989) were found to be present in grapes. Furthermore, their concentration increases during yeast fermentation (Strauss et al., 1987; Tesniere et al., 1989; and Williams et al., 1989). Williams et al. (1989) report that these phenolic compounds are derived in plants from phenylpropanoids via side-chain degradation and elongation reactions, from the shikimic acid metabolic pathway. Acetovanillone has a vanilla, molasses, caramel, and spicy odor; methyl vanillate has a vanilla, herbal and spicy odor; and ethyl vanillate has a sweet, vanilla and spicy odor.

# 2.2.2.7) Sulfur-containing substances

Methionol or 3-methylthio-1-propanol was identified as the main component of S-compounds in wine (Rapp and Guntert, 1985) and its concentration varies from 0.5 to 2 ppm (Schreier, 1979). Schreier (1979) studied methionol formation based on model fermentations. This compound has a characteristic vegetable and cabbage aroma.

# 3) ODOR PERCEPTION

# 3.1) Language development

One of the most important tasks of persons engaged in sensory evaluation of food is the useful communication of information about the volatile characteristics of the product, i.e. accurate odor identification and description.

One problem in dealing with odor quality is that it is difficult to adequately describe odor, because we lack a specific language for odor quality (Goldstein, 1989; Lawless, 1989). Engen (1987) stated that it is difficult to use other people's descriptions in naming odors and to learn new names for odors. He reported that verbal odor description tends to be personal, referring to objects with which a person has had experience. He states that people tend to categorize odors based upon the kind of object from which odors emanate, and not based upon general semantic nouns (label or descriptor name). Even though people may have difficulty in discriminating between odors, people have a remarkable ability to remember a specific odor (Amerine and Roessler, 1983). Cain and Pierce (1979) and Lawless (1984) suggest that a successful odor identification depends on: commonly encountered substances, a long-standing connection between an odor and its name, and aid in recalling the name.

The success of various training procedures for descriptive analysis of foods suggests that learning and motivation are important components in the development of ability in aroma recognition; this would include the evaluation of GC effluent

which represents a dynamic phenomenon since an aroma appears and disappears quickly.

#### 3.2) Individual differences

# 3.2.1) External causes that produce threshold variability

It is well known that olfactory thresholds are extremely variable across subjects. Odor thresholds detected for one subject vary from time to time, and the same odor sensation is described differently among people (Keverne, 1982; Amerine and Roessler, 1983; and Stevens et al., 1988). Pangborn (1981) and Stevens et al. (1988) stated that a number of psychophysical and methodological variables influence the validity of threshold measurements; including the number of panelists participating, the experience of each panelist to the stimulus and to the test procedure, the training involved, the number of replications, as well as the type of statistical analysis applied. Lawless (1989) added that panelists' fatigue, cultural factors and a specific anosmia are other factors contributing to individual differences. Marin et al. (1988) applied charm analysis to evaluate variation in odor detection thresholds. Her results show that sex does not contribute as much to charm variation thresholds as does age and subject to subject variation.

Stevens et al. (1988) reported that except for pathology and aging, olfactory sensitivity may, on the average, be relatively uniform from person to person, and the apparent large individual differences reported in the literature probably reflect fluctuations in an individual's measured threshold from one testing time to another. They tested this by conducting 20 threshold measurements for three compounds for three subjects on different testing days. They found that the variability of each subject was of comparable magnitude to that encountered among large groups of subjects tested only once. In contrast, the mean thresholds of the three subjects were much alike. Chastrette et al. (1988) found that certain aroma-active compounds present more variability than others, compounds with musty/amber or musk/animal character impart less individual variability than compounds with a fatty or floral character. Gross-Isseroff and Lancet (1988) studied 6 odorants at 3 different concentrations over 21 panelists. They concluded that some compounds induce individual variability depending on the dilution stage; benzaldehyde was found to give the highest variability of the 6 compounds that they studied.

3.2.2) Individual variabilities due to anosmia and/or hyposmia

Individuals with specific anosmias are generally defined as those who have a good sense of smell but lack the ability (partial or totally) to perceive a particular odorant. Henkin (1988) reports that hyposmia (decrease of smell function) is quite common and that anosmia (inability to detect or recognize vapors at primary and accessory areas of olfaction) is rare. Smell dysfunctions are characterized by Henkin (1987) to be of the following types: type I hyposmia as a qualitative inability to recognize vapors at the primary olfactory area (it involves the interaction between the olfactory epithelium and the olfactory nerves); type II hyposmia as a quantitative decrease in ability to detect or recognize vapors; and anosmia as the inability to detect or recognize vapors at either primary or accessary olfactory areas, (it involves an abnormality in at least one of the three major components (receptors, nerves and brain) of the olfactory systems. Another type of smell dysfunction is the so called dysosmia (Henkin, 1981) which is characterized by a distorted perception of smell, it is not a loss of smell; in general, people with this symptom describe pleasant odors as peculiar, odd, unusual, or unpleasant aromas. Henkin (1987) and Smith (1989) report that almost everyone has a hyposmia to something; hyposmias are more common than hypogeusias (taste disorders). An estimated 10 million Americans have chemosensory disorders (Smith, 1989). Several researchers have tried to explain how or why hyposmia or anosmia are

caused (Guillot, 1981; Keverne, 1982; Henkin, 1987; Smith, 1989; and O'Connell et al., 1989). Among the several causes that produce anosmia or hyposmia are: genetics, Kallman's syndrome, Alzheimer's disease, Parkinson's disease, head injuries (post-traumatic), respiratory infections, allergies, hormonal disturbances, dental problems, laryngectomies, medications, prolonged exposure to toxins (insecticides), radiation therapy (Smith, 1989), as well as deficiency of zinc in the diet (Wastney and Henkin, 1988) and deficiency of guanine nucleotide-binding protein (Weinstock et al., 1986).

In 1986, The National Geographic magazine published "The Intimate Sense of Smell" and included with it a scratch-and-sniff survey prepared in cooperation with scientists from the Monell Chemical Senses Center in Philadelphia; 1.5 million readers responded to the survey, from which 26,200 replies were randomly selected from the United States. Also 100,000 responses from abroad were selected for an international comparison. They found that women smell more acutely than men, pregnant women may have a diminished sense of smell; 2 persons in 3 have suffered a temporary loss of smell, primarily attributed to colds, flu, allergies, chemicals, pregnancy, and head injury; and 1.2 percent of the population can not smell at all (Gilbert and Wysocki, 1987).

Some of the compounds to which people are anosmic or hyposmic are: 3% of the population can't smell isovaleric acid;

33% can't smell cineole (minty odor); 6% can't smell trimethylamine (a fishy odor); 90% can't smell iodocreosol (produced by a reaction of lemon and iodine); 20% can't smell hydrogen cyanide (Smith, 1989); 0.1% can't smell butyl mercaptan (skunk odor) (Keverne, 1982); 13% of the National Geographic survey participants couldn't smell either androstenone (sweat/musky) or galoxide (musk) (Gilbert and Wysocki, 1987). Amoore (1967) studied a group of individuals anosmic to carboxylic acids. His results show that the degree of hyposmia is low in small (formic and acetic acids) or large (octanoic, nonaoic, and decanoic acids) carbon-length acids; however the degree of hyposmia increases to a plateau for butyric, valeric, hexanoic and heptanoic acids; more individuals were anosmic to isovaleric acid than to any other acid. Hyposmia and anosmia of several compounds have been studied, such as those compounds with a minty odor: 1,8-cineole, Imenthol and l-carvone (Marin et al., 1988); and compounds with urine-sweaty odor: cis-4-(4'-t-butylcyclohexyl)-4-methyl-2pentanone (pemenone) and 5-alpha-androst-16-en-3-one (androstenone) (O'Connell et al., 1989).

Individual differences exist and are the reason for using more than one panelist in a sensory evaluation. Day to day variability also exists and supports the need for replication of the experiment.

#### **MATERIALS AND METHODS**

# 1) PINOT NOIR WINES

The Pinot noir maturity trial was begun in 1987 at the Woodhall III Vineyard. In this study, the aroma profile analysis by sniffing the GC-effluent was done for the 1987 and 1988 vintages. Three wines were selected (from each vintage) on the basis that each wine was very different from the others in aroma and taste, and that those wines covered the range of an early, middle and late harvest time typical for Oregon. For the 1987 vintage, the "early" wine was harvested September 11, the "middle" wine was harvested September 16, and the "late" wine was harvested September 29. For the 1988 vintage, the "early" wine was harvested September 29, the "middle" wine was harvested in October 13, and the "late" wine was harvested in October 25. The must analysis at harvest and ethanol content in the finished wine for the 6 Pinot noir wines can be found in Table 1.

# 2) EXTRACTION METHOD

The extraction procedure is outlined in Fig 1. The Freon 113 (1,1,2-trichloro-1,2,2-trifluoroethane, obtained from

Miller-Stephenson, Los Angeles, CA, Freon PCA "Precision Cleaning Agent") extraction was applied to the 3 Pinot noir wines from each vintage, while the ethyl acetate (ethyl acetate (obtained from Omnisolv, EXO241-3) extraction was employed only in the 1988 wines.

## 2.1) Freon extraction

For each wine, 3 bottles randomly chosen were mixed; the extraction was replicated twice. A 375 mL aliquot of wine was extracted with an equal volume of Freon 113 in a 2 L flask. The mixture was flushed with nitrogen, and mixed with a magnetic stirrer for 4 hr. Minimum emulsion formation was achieved by stirring at the lowest speed possible, and by using a long magnetic stirring bar, approximately 7.5 cm long. After 10 hrs the two phases were transferred into a 2 L separatory funnel and allowed to stand for 2 hr in order for both layers to reach equilibrium before starting the separation. The Freon layer was on the bottom of the separatory funnel since it had a higher density than the aqueous layer.

The Freon layer was dried using anhydrous sodium sulphate and then concentrated in a Kuderna-Danish apparatus at 57°C to nearly 5 mL; then the three ball Snyder condenser was replaced by a smaller condenser, and the concentration continued to 1 mL. The Freon extract was then poured into a 2

mL vial with a Teflon cap, 0.5  $\mu$ L of tetradecane was added as an internal standard, and flushed with nitrogen. The Freon extract was held at -20°C until needed. Prior to the GC-MS identification, the Freon extract was concentrated an additional 10-fold with  $N_2$ .

# 2.2) Ethyl acetate extraction

The aqueous layer, arising from the Freon layer separation, was added to 250 mL of ethyl acetate in a 2 L flask. The mixture was flushed with nitrogen, and mixed under magnetic stirring for 30 min, at the lowest speed possible with a 7.5 cm stirring bar. After 2 hr the phases were transferred to a 2 L separatory funnel and allowed to stand for 2 hr in order for both layers to reach an equilibrium before starting the separation. The ethyl acetate layer was in the upper part of the separatory funnel since it had a lower density than the aqueous layer.

The ethyl acetate layer was dried using anhydrous sodium sulphate and then concentrated on a Kuderna-Danish apparatus at 80°C down to 2 mL. The ethyl acetate concentrate was then poured into a 2 mL vial with a Teflon cap, and 1  $\mu$ L of tetradecane was added to the mixture, and flushed with nitrogen. The extract was held at -20°C until needed.

# 3) GAS CHROMATOGRAPHY-SNIFFING

# 3.1) Equipment description and GC parameters

The gas chromatograph used in this study was a Hewlett Packard 5890 equipped with a fused silica column: 0.53 mm. ID and 30 M in length, it was coated with 0.25  $\mu$  of Supelcowax 10 (which is functionally equivalent to Carbowax 20M); the coated capillary was obtained from Supelco, Inc., Bellefonte, PA. Samples of 2  $\mu$ L were injected with a 16:1 split ratio. The GC injector was at 200°C, and the detector was at 250°C. The helium carrier gas flow was set at 3.3 mL/min (which gave a linear velocity of 25 cm/sec).

The GC column for the Freon extract was held initially at 80°C for 5 min, rate A was 5°C/min to 155°C; rate B was 4°C/min to 240°C and held at the final temperature for 30 min.

The GC column for the ethyl acetate extract was held initially at 80°C for 3 min; rate A was 10°C/min to 155°C; rate B was 4°C/min to 240°C and held at the final temperature for 45 min.

# 3.2) Test procedure

This method was used to assay Pinot noir extracts for aroma-active components separated by GC and detected by a human subject (Fig. 2).

The delivery system consisted of a 1 cm. ID and 60 cm length glass tube, coated with silicone (Sylon CT, Supelco, Inc., Bellefonte, PA) in order to avoid the adsorption of compounds on the walls.

When aroma data were collected, the column was moved from the Flame Ionization Detector (FID) to the sensory detector port, which is a detector housing without the flame, allowing the compounds to be directed to the panelist. The delivery system device sat on top of this detector.

The GC effluent was mixed with humidified purified air at a flow of 11 L/min. The air flow was adjusted as the sum of the linear velocity of the helium carrier gas plus makeup gas coming from the column, thus maintaining the resolution. The air, coming from a compressed air tank, was filtered through a charcoal filter and directed to a water bath at 25°C. The relative humidity of the air, measured by the difference in temperature between wet and dry bulb thermometer, was 60% at the end of the sniffer.

Panelists were instructed to breathe normally while sniffing the GC effluent. Every time that an odor was detected, the panelist responded to the intensity of the stimulus by using a time-intensity device with a 15 cm. scale ranging from 0=

"none", 7= "moderate", and 15= "extreme". The sensory intensity of the GC effluent was registered directly onto an IBM computer with data collection software developed in this laboratory. At the same time the panelist described verbally the aroma perceived; these data were gathered by the experimenter. This yielded a list of retention time ranges corresponding to regions of aroma-active peaks.

A daily standardization of subject response data was achieved by running a series of normal hydrocarbons chromatographed under the same GC conditions as the stimulus samples. Retention time of aroma-active peaks were converted to retention indices (Kovats indices) by a linear interpolation of the hydrocarbon retention data (Kovats et al.,1958). Those retention times from sensory responses were coincident with the retention times of the peaks coming through the GC column and detected by a Flame Ionization Detector (FID).

# 3.3) Panel selection

Panelists 1 and 2 participated in the evaluation of both 1987 and 1988 wines. Panelists 3 and 4 only participated in the evaluation of 1987 wines. Panelist 5 evaluated only the 1988 wines aroma determination; panelist 5 was a well trained and experienced person in aroma description of wines. Because panelist 4 was relatively insensitive or conservative when

evaluating the 1987 wines, and because of the lack of availability of subjects that were familiar with describing Pinot noir aroma, three well trained and experienced panelists (1,2 and 5) in Pinot noir aroma description were considered to be sufficient for the 1988 wine evaluation.

Panelists 1, 2, 3 and 4 participated in the evaluation of Freon extracts of early, middle and late maturity Pinot noir wines of the 1987 vintage, which took place in Spring 1989. Panelist 1, 2 and 5 participated in the assessment of Freon and ethyl acetate extracts of early, middle and late maturity Pinot noir wines of the 1988 vintage, which took place in Fall 1989.

# 3.4) Training procedure

The panelists underwent a training procedure in order to become familiar with the sensory properties of the aromaactive compounds of Pinot noir wine extracts, and to practice sniffing the GC effluent and using the time-intensity device. During training, which consisted of 10 sessions, the panelists were exposed to Pinot noir wine extracts as well as a mixture of some of the compounds present in Pinot noir.

Prior to sniffing, panelists were presented with standards in order to help them describe the aroma-active compounds present in the sample. A list of the standards used is in Table 2.

# 3.5) Data analysis

The time (min) referred to in this section is the elution time from a GC run, under the temperature program previously described. After smelling the entire GC run of the 1987 wine a couple of times, it was decided by the experimenter that the majority of the aroma-active peaks eluted between 7 and 42 min. The aroma of the first compounds eluted (up to 4 min) was overlapped with the aroma of the solvent. From 4 to 7 min, there was hardly any distinct aroma eluted. After 42 min, the column temperature was 240°C and produced a strong background odor making the smell of the GC effluent unpleasant.

Data collection was conducted from 7 to 42 min, only in the 1987 wine evaluation. During the training for the 1988 wine evaluation, panelists noticed an interesting peak directly before the point at which they would normally start recording their responses. Therefore, for the 1988 wine evaluation, runs were started at six and half minutes instead of at seven minutes.

In the testing procedure each sample was evaluated four consecutive times. Even though the conditions were exactly the same for each run, vast differences occurred between replications. As an example, Fig. 3 shows the 1<sup>St</sup> and 4<sup>th</sup> replications for the early 1987 (9/16) sample for panelist 3. In

general the number of aroma-active peaks perceived by a panelist increased through the number of replicates; peaks with low intensity and small area were the ones more likely to be lost from one replicate to the other. This points out the importance of multiple replications. Since each panelist spends as much time breathing out as breathing in, it is expected that for each run some peaks will be missed. In addition panelist's day to day sensitivity is expected to vary.

Times and intensities of peaks detected at least 50% of the time, in the four replications, for each panelist were averaged. With these new values a graph of time vs. intensity was constructed per panelist and per wine; these graphs are called "aromagrams". Examples of these aromagrams can be found in Figs. 4-6 for the 1987 wines and in Figs. 7-9 for the 1988 wines.

Then, a consensus aromagram for each sample was created. For the 1987 consensus aromagram, Fig. 10, the time and the intensity of those peaks that were detected by at least three of the four panelists were averaged. For the development of the 1988 consensus aromagram, Fig. 11, because only 3 panelists participated, different criteria were adopted for identifying significant peaks. Those peaks that were detected in 2 of the 4 replicates by all three panelists were included in the consensus aromagram. Also included were peaks detected in at least 3 of the 4 replicates by 2 of the panelists, detected

once by the third panelist and described by that panelist with descriptors similar to those used by the other two panelists.

# 4) GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS)

# 4.1) Equipment description and GC-MS parameters

The Freon extract was analyzed by GC-MS-DS using a Finnigan 1015C quadrupole mass spectrometer operated in the electron impact mode (70 eV) and interfaced by a glass jet helium separator to a Varian 1400 GC. The glass GC column was obtained from Supelco Inc.; it was 0.75 mm. ID and 60 M in length. The column had a 1  $\mu$ M bonded film of Supelcowax 10 (which is functionally equivalent to Carbowax 20 M). The Freon extract also was run on a second glass capillary of exactly the same dimensions, but coated with a 1  $\mu$ M film of Supelco SPB-1 (methyl silicone). The samples were injected (neat) without splitting. The GC injector was at 225°C, and the GC column was held initially at 80°C for 5 min, and then programmed at 2°C/min. to 200°C and held at the final temperature.

When the sample was injected, the Freon peak was excluded from the MS, after the Freon eluted, the column was connected, the MS was turned on and data collection started. By doing this Freon interferences were avoided.

Data were acquired and processed using Riber 400 software, version E0. Identification of compounds was aided by the QMATCH program (library of 25,100 compounds) and the NIST Mass Spectral Data Base (version 2.0 for PCs), containing about 50,000 compounds. The John Wiley and Sons. mass spectral database on CD ROM (1988 version) was also used.

Freon extract components were identified on the basis of their mass spectral fragmentation and Kovats index. Off-line file searching using an IBM-AT made use of a database file named KOVATS that was created under the Paradox program from Borland International. The KOVATS database contains Kovats indices for two GC liquid phases, as well as the MW and the six most abundant ions in the mass spectrum; there are now over 4000 file entries, but many are multiple entries for the same compound (Libbey and Sturtz, 1989).

# 4.2) Confirmation of compounds

A Freon mixture containing 30 of the compounds identified in the Freon extract (Table 3) was made in order to simulate the Freon wine extract; the concentration of each of those 30 compounds was calculated from the late 1987 chromatogram. Aroma quality was confirmed by the experimenter; the aroma of these 30 compounds was described in almost the same way as

the Freon samples were judged. All the compounds were identified by Kovats indices as well as aroma.

Five compounds, 3-methyl-1-pentanol, 4-methyl-2-pentanol, amyl-4-hydroxy butanoate, n-butyl acetamide and ethyl isobutyl succinate, were also tentatively identified by GC-MS in the Freon extract, but it was not possible to obtain these compounds, so their Kovats indices and aromas could not be confirmed. Another compound tentatively identified by GC-MS was 3-hydroxy butyric acid, this compound is available on the market, but its Kovats and aroma could not be confirmed due to the fact that this compound was not soluble in Freon 113.

#### **RESULTS AND DISCUSSION**

## 1) COMPOUNDS NOT PREVIOUSLY REPORTED IN PINOT NOIR

During grape ripening, 2 main types of aroma compounds are formed. The first group is related to those aroma-active compounds where chemical composition is not changed during winemaking. The second group is related to the aroma-active precursors where the chemical composition is changed into aroma-active compounds during the process of winemaking. Therefore the aroma of the wine is principally influenced by the chemical composition of the grape at the moment of harvest. Williams et al. (1989) state that the fruit transforms secondary metabolites (intermediates) into aroma-active compounds or aroma-active precursor, and also the fruit stores them.

In the present study, only 30 aroma-active compounds were identified by chemical name and aroma in a Pinot noir wine, Freon extract (Table 3). Among those 30 compounds, methionol (3-methylthio-1-propanol), methyl and ethyl vanillate, and acetovanillone were not previously reported to be in Pinot noir (Meunier and Bott, 1979; Brander et al., 1980; Schreier et al., 1980; and Cliff, 1989). None of the acids were previously reported; in this study, the following acids were identified: isovaleric (3-methyl butyric), hexanoic, octanoic, decanoic and tridecanoic acids

Methionol (3-methylthio-1-propanol) is produced during yeast fermentation (Rapp and Guntert, 1985). Methionine seems to participate in the formation of methionol (Schreier, 1979). However, the methionol metabolic pathway is still not clear (Schreier, 1979; Nykanen, 1983; Rapp and Guntert, 1985).

Ethyl vanillate (Tesniere et al., 1989), methyl vanillate (Williams et al., 1989), and acetovanillone (Schreier, 1979; and Williams et al., 1989) were found in grapes. Furthermore, their concentration increases during yeast fermentation (Strauss et al., 1987; Tesniere et al., 1989; and Williams et al., 1989). These phenolic compounds are derived from the shikimic acid pathway (Williams et al., 1989).

For ethyl vanillate R is COO-CH<sub>2</sub>-CH<sub>3</sub>, for methyl vanillate R is COO-CH<sub>3</sub>, and for acetovanillone R is CH<sub>3</sub>-C=O.

Fatty acid synthesis takes place during the winemaking process, and principally from the yeast (alcoholic) fermentation. Fatty acid synthesis requires acetyl-CoA as a starting material

The first stage of the synthesis involves the formation of malonyl-CoA

$$O$$
 O  $||$   $||$   $+ H^{+}$   $CH_{3}$ - $C$ - $S$ - $C$ o $A$  + ATP +  $CO_{2}$  +  $H_{2}$ O ==>  $COO^{-}$ - $CH_{2}$ - $C$ - $S$ - $C$ o $A$  + ADP+ Pi

An enzyme complex containing acetyl-CoA, biotin enzyme, and malonyl-CoA is formed,

the reaction proceeds to form acyl-CoA and then two carbon atoms derived from malonyl-CoA are successively added to the primer acyl-CoA:

$$CH_3$$
- $C$ -- $CH_2$ - $C$ - $S$ --Enzyme complex--HS  $\parallel$   $O$  O

$$CH_3$$
- $CH_2$ -- $CH_2$ - $CH_2$ -C-S--Enzyme complex-SH + malonyl-CoA || O

Consequently, the process yields saturated fatty acids which contain an even number of carbon units (Schreier, 1979; Shinohara, 1985; and Nykanen, 1986).

## 2) CONSENSUS AROMAGRAMS

The results presented here are only from analysis of the Freon extract. The ethyl acetate extract data will not be presented here. The standardization of the intensity of the aroma-active peaks through data collection was achieved by concentrating each wine extract to exactly 375-fold, and by injecting precisely 2 µL of wine extract into the GC. The results are presented in the following two main sections. The first section presents the consensus aromagrams which were made with aroma-active peaks detected by the majority of the panelists. This section covers the comparison of consensus aromagrams versus FID chromatograms, within the same vintage, and across vintages. The second section covers the individual aromagrams for each individual panelist which were

made by averaging aroma peaks detected in at least 2 out of the 4 replicates per wine extract. In this section individual responses are discussed, as well as individual differences across panelists.

## 2.1) Consensus aromagrams versus FID chromatograms

The consensus aromagrams for the different Pinot noir wines from the 1987 and the 1988 vintages are shown in Figs. 10 and 11 respectively, and the aroma description and intensity of each aroma-active peak is displayed in Appendix 1. Large differences occurred on the consensus aromagram across maturities within the same vintage, as well as from one vintage to the other (Table 4). In both vintages the largest number of aroma peaks was noted at the late harvest date. The number of aroma-active peaks was generally higher in the 1988 vintage than in the 1987 vintage (Table 4). In the consensus aromagram certain peaks appear, disappear, or change in aroma intensity dependent on the stage of grape harvest; thus delineating the dynamics of aroma development (Appendix 1).

In the 1987 vintage (Table 4), the number of aroma-active peaks increased with the maturity. The aroma peak intensity was highest for the middle harvest wine. Over 20% of the total number of aroma peaks for the early or middle wine were unique and did not appear in the other two wines, while 46% of the

aroma peaks of the late wine were unique. There were only 10 aroma peaks that were detected in all three 1987 wines. Moreover, in the 1988 vintage (Table 4), the number of aromaactive peaks was highest in the late wine, followed by the early and then by the middle wines. The aroma peak intensity was almost the same in the early and late wine, but a decrease was noted in the middle wine. For the early or middle wines, over 25% of the total number of peaks were unique to that specific wine, while in the late wine almost 53% of the peaks were unique. There were 16 aroma peaks common to all three 1988 wines.

Figures 12 and 13 contain the FID chromatograms for the two vintages. The minimum concentration of a compound in the wine extract that the integrator was able to report is about 1 ppm. The concentration of each peak for each wine extract is presented in Appendix 2. It is difficult to observe any pattern of change in the FID chromatograms from one maturity to the other within and across vintages. Typically, the only change that can be noticed is the variation in concentration (peak area) in the majority of the compounds. The following data are related only to those peaks whose concentration in wine extract was at least 1 ppm (Appendix 2).

In the 1987 vintage, the early and late wines had the highest number of FID peaks at 91 and 90, respectively; while the middle wine had only 76 peaks. There were 72 peaks

common to the 1987 wines. Most of the peaks in the middle 1987 wine (66%) had the highest concentration, and most of the peaks in the early 1987 wine (59%) had the lowest concentration. In the early or late 1987 wines, 8 and 7 peaks, respectively, were unique to that specific wine, while the middle 1987 wine did not have any unique peaks. This trend did not hold true for the 1988 vintage. The early wine had the highest number of peaks, 74, while the middle and late wines had 59 and 61, respectively. There were 53 peaks common to the three 1988 wines. In the 1988 wines the concentration of most of the compounds decreased with the maturity of the grape; in the early 1988 wine, 42% of the peaks had the highest concentration, while in the late wine, 41% of the peaks had the lowest concentration (Table 4). Almost 15% of the total number of peaks in the early 1988 wine were unique and detected only in this wine; and less than 7% of the total peaks in the late 1988 wine appeared were unique; once again, the middle wine did not have unique peaks.

A comparison of the FID peaks versus the consensus aromagram, Figs. 14-19, for any one wine shows that there were many areas with significant aroma character where little response is noted by the FID. The patterns of aroma responses across maturities are very different, with the FID responses reflecting poorly the significant aroma peaks. It is well known that most of the aroma-active peaks perceived in wine are

present in extremely small concentrations. The percentage of aroma-active peaks reported by the FID (with a concentration higher than 1 ppm) in the 1987 wine extracts were 54, 64 and 46%, respectively; and in the 1988 wine extracts were 35, 29 and 33%, respectively (Table 4). Therefore, approximately 45% (1987) and 66% (1988) of the aroma-active peaks were not detected by the FID. Since the integrator response was not capable of detecting most of the aroma-active peaks, this confirmed that sniffing the GC-effluent gives a more sensitive and reliable response.

## 2.2) Maturity comparison within the same vintage

The data reported in this section considers only the consensus aromagrams of the three different maturity wines from a particular vintage. This section is divided into a discussion of unique peaks, detected in only one of the wines per vintage, and a discussion of peaks common to all three wines per vintage.

Data on the character of each aroma-active peak was gathered independently for each peak (Appendix 1). Moreover, it is the blend of those aromas one experiences when smelling wine. In order to begin to develop a picture of what the blend of the independent aromas might be like, peaks were placed in aroma character groupings (Table 5). The frequency of use takes

into account each time a description was predominant for a peak. Several terms were often predominant descriptors for just one peak. Therefore 53% frequency of use for fruity on the 1988 late wine means that the term fruity was used to describe 53% of all of the aroma-active peaks for that wine.

## 2.2.1) Vintage of 1987

The number of consensus aroma-active peaks detected in the early, middle and late wines were 26, 28 and 37, respectively. The average aroma peak intensity for the three wines were 5.8, 8.4 and 7.2, respectively (Table 4). All three wines had high percentage frequencies of fruity and floral. Vegetative, spicy and woody were used with almost the same frequency in the three wines. In addtion, the early harvest (Sept 11) wine had a high frequency percentage of cotton candy (12%) (Table 5). The middle harvest (Sept 16) wine had a high percentage of stinky/dirty socks/cheese (21%). The late harvest (Sept 29) wine had high floral (84%) and earthy/musty (16%) percentages, and a low sweet (16%) percentage.

# 2.2.1.1) Unique aroma peaks per wine

Unique aroma peaks were defined as those which occur in only one of the maturities. In the early harvest (Sept 11) wine,

6 (out of 26) peaks were unique (Appendix 1), not present in the other maturities. None of the unique peaks had their identity confirmed by GC-MS. The average aroma peak intensity of these unique peaks was 4.7. They were described as: floral (67%), woody (50%), fruity (33%), vegetative (33%), sweet (33%), spicy (17%), musty/earthy (17%), and chemical (17%). None of the peaks was detected in the first 16 minutes of elution time.

In the middle harvest (Sept 16) wine, 6 (out of 28) peaks were unique (Appendix 1). The only unique aroma-active peaks confirmed by chemical name and aroma were benzaldehyde, 15.57 min, and methyl vanillate, 41.51 min. These unique peaks had an average aroma peak intensity of 8.5 and were described as: fruity (67%), floral (50%), woody (50%), sweet (33%), chemical (33%), and vegetative (17%). The elution times of most of these unique aroma-active peaks were in the first half of the experimental run, from 14 to 23 min.

In the late harvest (Sept 29) wine, 17 (out of 37) peaks were unique (Appendix 1). The only unique peaks that were confirmed by chemical name and aroma were ethyl octanoate, at 13.2 and 13.4 min, and ethyl vanillate, 41.8 min. The aroma peaks at 13.2 and at 13.4 min were produced by ethyl octanoate as confirmed by GC-MS. Panel response to this compound often took the form of two peaks, as if the aroma disappeared and then reappeared. Both aroma peaks fell within the range of ethyl octanoate on the FID chromatogram. These unique peaks

had an average intensity of 6.5 and were described as: floral (88%), fruity (59%), earthy/musty (23.5%), woody (23.5%, chemical (23.5%), sweet (18%), spicy (18%), and vegetative (18%).

## 2.2.1.2) Common peaks across wines

There were only 10 aroma-active peaks common to the 3 wines from 1987 (Appendix 1). The only common aroma peaks identified by chemical name and aroma were: linalool, 16.21 min; and beta-phenyl ethyl acetate, 23.45 min. It was of interest to examine how the concentration and aroma intensity changed across maturity levels. The average intensities of these common peaks for the early, middle and late wines were 6.7, 8.4 and 8.3 respectively. In general the aroma intensities from the early wine had the lowest values of the three wines (6 out of 10 peaks). There was no pattern followed from the middle to the late wines; however, in half of the peaks the intensity was the same for these two wines.

The peak eluted at 21.22 min did not follow a pattern common to other peaks. This peak was the only one having the same aroma intensity in the early and late wine, and an intensity decrement in the middle wine. Furthermore, this peak presented a quality difference changing from dirty socks (early wine) to toasty/earthy/grainy (middle wine), and finally to

piney/hay/baked potato (late wine). This aroma peak was also detected in the 1988 wines, but the aroma descriptors used were very different from the 1987; they were described as floral and fruity. Pure methionol elutes at 21.75 min with an additional minor peak at 21.2 min. This additional peak is an unknown methionol impurity which has a stinky/dirty socks aroma.

It is difficult to explain why this peak was described so differently across maturities. One explanation is that the character changed with a change in concentration. For the early maturity, a concentration of 59 ppm was described as dirty socks; for the middle maturity a lower concentration of 17 ppm was described as toasty/earthy/grainy; and for the late maturity, a concentration of 44 ppm was described as piney/hay/baked potato (Appendix 2). The peak at 21.2 min may not be totally tied to methionol, as methionol is highest in concentration in the middle wine. Also, concentrations calculated from integrator readings in this area of the chromatogram may be misleading because peak resolution is poor. A second explanation is that the 21.2 peak is a mixture of compounds. Berglund et al. (1973), Lawless (1986), and Olsson (1986) studied the behavior of aroma-active compounds while interacting in a mixture; they concluded that the aroma produced from a mixture of compounds could be either heterogeneous (the aroma of one compound predominating over

other aromas), or homogeneous (the aroma of each compound is combined forming a new aroma).

# 2.2.2) Vintage of 1988

The numbers of consensus aroma-active peaks, detected in each of the three wines, were 34, 28 and 55, respectively. The average aroma peak intensities for these three wines were 11.7, 8.5 and 11.3, respectively (Table 4). The early and middle wines had almost the same frequency percentage in the following attributes: fruity, floral, vegetative, and sweet. The late wine had the highest spicy (58%), vegetative (54%), fruity (53%), and sweet (33%) frequency percentage (Table 5).

# 2.2.2.1) Unique peaks per wine

The early harvest (Sept 29) wine had 9 (out of 34) unique peaks (Appendix 1). The only aroma-active peak confirmed by GC-MS was the one eluted at 25.99 min, which was produced by the tailing of beta-phenyl ethanol. The major beta-phenyl ethanol peak (at 25.79 min) was a common peak across vintages. The 9 unique peaks had an average aroma peak intensity of 9.0 and were described as: vegetative (67%), chemical (56%), floral (44%), fruity (22%), and spicy (22%).

The middle harvest (Oct 13) wine had 7 (from a total 28) unique peaks (Appendix 1). None of these peaks were identified by GC-MS. The unique peaks had an average aroma peak intensity of 8.2, and were described as: floral (71%), fruit (57%), chemical (57%), vegetative (43%), spicy (29%), and sweet (14%). These unique aroma peaks were eluted in two blocks, from 14.48 to 22.25 min and 30.4 to 31.3 min.

The late harvest (Oct 25) wine had 29 (from a total of 55) unique peaks (Appendix 1). Seven unique peaks were identified by chemical name and aroma: ethyl octanoate, 13.40 min; benzaldehyde, 15.57 min; and the first part of the beta-phenyl ethyl acetate peak at 23.37 min (the second part was a common peak across vintages); benzyl alcohol, 24.78 min; ethyl palmitate, 33.53 min; and octanoic acid, 35.66 min. The aroma peak detected at 26.24 min was identified as gamma-octalactone which in pure form has a coconut-like and sweet aroma. Here, however, it was described as fruity and floral, perhaps due to its proximity to the tailing of the beta-phenyl ethanol peak. The average aroma peak intensity of the 29 peaks was 9.0 and the descriptors used were: spicy (72%), vegetative (52%), floral (48%), fruit (45%), sweet (31%), chemical (21%), woody (17%), and earthy/musty (10%).

### 2.2.2.2) Common peaks across wines

There were 16 aroma-active peaks common to the 3 1988 wines (Appendix 1). The eight common aroma peaks identified by chemical name and odor were: 3-methyl-1-butanol, 6.45 min; ethyl hexanoate, 7.28 min; linalool, 16.21 min; methionol, 20.64 min; beta-phenyl-ethyl acetate, 23.45 min; hexanoic acid, 23.91 min; beta-phenyl ethanol, 25.79 min; and gamma-nonalactone, 28.71 min. The average intensities for the common peaks from the early, middle and late wines were 10.5, 9.3 and 11.2, respectively. Nine of the 16 peaks were highest in the late wine. In general the aroma intensity of the middle wine had the lowest value of the three wines (10 of 16 peaks).

## 2.3) Comparison across both vintages

The 1987 and 1988 vintage harvest dates were very different due to the warmer than normal conditions during the 1987 vintage. The latest harvest date in 1987, 9/29, when the grapes were at 24.4 OBrix was the same date as the earliest harvest date in 1988, when the grapes were at only 21.8 OBrix (Table 1). These climatic conditions dramatically affected the

FID response and the panel response to aroma-active peaks. There were more peaks in the FID chromatograms for the 1987 wines (91, 76 and 90, respectively) than for the 1988 wines (74, 59 and 61, respectively). However, the number of aroma-active peaks in samples from both vintages was relatively the same, except for the late 1988 wine which had almost twice the number of peaks of any other wine. The aroma peak intensities in the 1987 vintage wines were lower than in the 1988 vintage.

The data reported in this section consider all six wines. The values reported for each attribute were calculated by counting its frequency of use. This was based on the total number of aroma peaks detected per wine; for example in the early 1987 wine (Table 5 and Appendix 1), fruity was used in 15 of the 26 aroma peaks, which corresponded to 58% frequency of use.

Overall, the 1988 vintage had a much higher number of peaks described as dried fruit, vegetative, herbal and spicy (Table 5). For example, the 1987 wines had an average of only 4 peaks described as dried fruit while the 1988 wines had an average of 13 peaks. All of the individual maturity levels across vintages were higher in dried fruit for the 1988 wines, however the largest difference was found in the late wines where 6 and 21 peaks were described as dried fruit-like for the 1987 and 1988 wines, respectively. The same general pattern was followed for vegetative, herbal and spicy. Moreover, even

the earliest 1988 wine was higher in dried fruit, vegetative, herbal and spicy character than any of the 1987 wines.

There were a few aroma notes that occurred in the 1987 wines that were not perceived in the 1988 wines. Vanilla, stinky/cheese/dirty socks, and meaty were used occasionally to describe peaks in the 1987 wines. Also predominant in the 1987 early and middle wines was a very strong cotton-candy character. The same strong character was found as an impurity in commercially procured gamma-nonalactone. This impurity has not been identified. Although not as predominant as dried fruit, vegetative, herbal and spicy, several other important aroma notes occurred in the 1988 wines. Caramel and cabbage/sulfur were used an average of 10 and 12% of the time, respectively, to describe the aroma-active peaks from the 1988 wines.

## 2.3.1) Unique peaks per vintage

The 1987 vintage had a total of 37 unique aroma-active peaks across the early, middle and late wines that were not found in any of the 1988 wines (Appendix 1). Nine of the aroma peaks were common to at least two of the 1987 wines. A total of 58 aroma-active peaks were unique to the 1988 vintage, 11 of which were common to at least two of the 1988 wines. From both vintages, approximately half of these unique aroma peaks

were detected in the late wines (49 and 53%, respectively). Comparing the unique aroma peaks, the 1988 vintage average percentages were higher than the 1987 for: vegetative, spicy, earthy/musty, and chemical character.

#### 2.3.2) Common peaks across vintages

There were only 4 aroma peaks common to all six wines. They were linalool, beta-phenylethyl acetate, beta-phenyl ethanol, and the compound eluted at 21.22 min which was found to be an unknown methionol impurity. There were 29 aromaactive peaks common to both vintages (at least one wine per vintage) (Appendix 1). The identity of eight of the 29 peaks was confirmed by GC-MS. Among them, there were 2 esters (ethylhexanoate and ethyl octanoate), 1 alcohol (beta-phenyl ethanol), 1 terpene alcohol (linalool), 1 aldehyde (benzaldehyde), 1 acid (hexanoic acid), 1 acetate (beta-phenylethyl acetate), and 1 sulfur compound (methionol). Each attribute frequency was averaged per vintage for these common peaks. In the 1987 vintage the attribute frequency of use was higher than in 1988 for fruity, earthy/musty, and woody. The attribute frequency of use was higher in the 1988 vintage for vegetative, spicy, sweet, and chemical attributes. Floral was the only attribute having an equal frequency of use in both vintages.

#### 2.3.3) Harvest date comparison across vintages

Although early, middle and late harvest dates were selected for each vintage, large climatic differences existed between the vintages (section 1.2.3). Therefore, it was of interest to determine how closely the harvest dates from each vintage would relate chemically and/or perceptually (Table 4 and 5, and Appendix 1).

The early 1987 wine had a lower number of aroma-active peaks (26 vs. 34) and average aroma peak intensity (5.8 vs. 11.7) than the early 1988 wine. There were only 11 aroma peaks common to the early 1987 and 1988 wines (Appendix 1). Besides the four common compounds mentioned earlier, methionol and hexanoic acid were also identified by GC-MS. In both wines, most of the aroma-active peaks were eluted between 14 and 34 min. The early 1987 wine attribute frequency was higher than the early 1988 wine for cotton candy, woody, stinky/cheese/dirty socks, and meaty attributes. ln contrast, the attribute frequency in the early 1988 wine was higher for dried fruit, vegetative, spicy, earthy/musty, caramel, pungent, cabbage/sulfur/burnt match, and soapy/fusel oil. Floral was the only attribute whose frequency of use was almost the same in both early wines.

The middle 1987 and middle 1988 wines had the same number of aroma-active peaks (28) and aroma peak intensity

(8.5). However, there were only 10 aroma peaks common to both wines (Appendix 1). Besides the four common peaks mentioned earlier, ethyl hexanoate and methionol were also identify by GC-MS. The middle 1987 wines aroma peaks eluted throughout the entire experimental run (7 to 41.5 min), while most of the aroma peak on the middle 1988 wine eluted from 14 to 33.8 min. The middle 1987 wine attribute frequency was higher than in the middle 1988 wine in cotton candy, woody, stinky/dirty socks/cheese, and meaty. However, the attribute frequency in the middle 1988 was higher in dried fruit, vegetative, spicy, caramel, pungent, and soapy/fusel oil. The middle 1987 and 1988 wines had almost the same average percentage in floral, and earthy/musty.

The late 1987 wine had a lower number of aroma-active peaks (37 vs. 55) and aroma peak intensity (7.2 vs. 11.3) than the 1988 wine. There were 13 aroma peaks common to both wines. Besides the four common peaks mentioned earlier, ethyl hexanoate, ethyl octanoate, and hexanoic acid were also identify by GC-MS. The late 1987 wine attribute frequency was higher in floral, vanilla, stinky/dirty socks/cheese, and meaty. The 1988 late wine attribute frequency was higher in dried fruit, vegetative, sweet, caramel, smokey/toasty, chemical, pungent, and cabbage/sulfur/burnt match. The late 1987 and 1988 wines had almost the same average frequency percentage in woody, and soapy/fusel oil.

It is evident from the above results that data from the same maturity levels across vintages were more different than similar. Attempts to match other maturity levels (i.e. early 1987 to middle 1988) were no more successful.

## 3) INDIVIDUAL RESPONSES

It should to be mentioned that panelists 1-4 participated in the evaluation of the 1987 wines, and panelists 1, 2 and 5 evaluated the 1988 wines. Each wine extract was evaluated four consecutive times per panelist. Individual aromagrams were made with those aroma peaks that were detected in at least 2 out of the 4 replications.

# 3.1) Individual aromagrams

An important finding of this study was how differently each panelist responded to each sample (Table 6). There were several differences in sensitivity to the various wines among and within panelists. Most of the panelists detected the highest number of aroma peaks in the late maturity wines; but there was no pattern followed for the early or middle harvest wine.

Figures 4-6 contain an aromagram for each panelist for the early (9/11), middle (9/16), and late (9/29) Pinot noir wine

extracts from the 1987 vintage. One can observe from these figures, that compared to the other panelists, panelist 2 was not using the full range of the intensity scale and was not as sensitive to the higher molecular weight compounds; panelist 3 was more sensitive to higher molecular weight compounds, and panelist 4 was somewhat conservative (or insensitive) in responding to the GC effluent. Largest differences among panelists were found in the early 1987 wine; where the number of aroma peaks detected per panelist range from 29 to 67. In the middle and late 1987 wines, the number of peaks detected for panelists 1-3 were almost the same. Panelist 4 always detected the least number of aroma peaks per wine (Table 6).

Figures 7-9 contain an aromagram for each panelist for the early (9/29), middle (10/13), and late (10/25) Pinot noir wine extracts from the 1988 vintage. One can observe from these figures that panelist 2 now was using the full range of the intensity scale; panelist 1 was more sensitive to the higher molecular weight compounds, than the other panelists; and panelist 5, the new panelist, was not as sensitive to the higher molecular weight compounds and was somewhat conservative in scale usage. In the 1988 wines, panelist 1 detected the highest number of peaks, panelist 2 detected the lowest number of aroma peaks in the middle wine, and panelist 5 detected the lowest number of peaks in the other two wines.

A possible explanation for the individual aromagrams variations presented in the 1988 wines (Figs. 7-9), it is how the age of the panelists may have affected the sense of smell; where panelist 1 was the youngest, and panelist 5 was the oldest. Marin et al. (1988) report that variation in odor detection between younger and older subjects exists, particularly with later-eluting compounds.

#### 3.2) Individual differences

Each panelist was able to perceive compounds that none of the other panelists perceived (Table 6). Differences among individual panelists are discussed separately by vintage because of the differences in panelists evaluating each vintage. A listing (aroma descriptors) of these aroma-active peaks unique to each panelist are in Appendices 3.1-3.3 for the 1987 wines, and in Appendices 4.1-4.3 for the 1988 wines. Inspection of these data reveals variability in the use of descriptors and in the degree of sensitivity among panelists. Some of those variabilities are explained in the two following sections: the first section contains a discussion of unique aroma-active peaks detected by each panelist, and the second section contains a discussion of the variability of perception of certain aroma-active peaks from one panelist to another (Appendices 3.1-3.3 and 4.1-4.3).

# 3.2.1) Unique aroma-active peaks detected per panelist

In the 1987 wines (Table 6, and Appendices 3.1-3.3), the total number of unique peaks detected in the three wines per panelist were: 22, 35, 22, and 5, respectively. These unique peaks represented 6-20% of the total number of aroma peaks detected per panelist. The average aroma intensity for the unique peaks was from slight-moderate to moderate. In the 1988 wines (Tables 6, and Appendices 4.1-4.3), the total number of unique peaks detected in the three wines per panelist were 53, 40, and 28, respectively. These unique peaks represented 14-28% of the total number of peaks detected per panelist. The average aroma intensity for the unique peaks was from slight-moderate to extreme.

Each unique aroma peak detected per panelist was analyzed in order to see if there was any pattern followed with respect to the aroma descriptors used, as well as to sensitivity to a specific chemical group. No pattern was discovered.

3.2.2) Individual variability in perception of specific aroma-active peaks

Because there were so many peaks unique to each individual, the question of specific anosmias arises. However, due to the use of only one concentration, the term anosmia here is used cautiously. Differences may be due more to threshold level than true anosmias. Only a few situations are presented here: first, when one panelist could not detect a compound that the majority of the panelists could; and second when one panelist perceived repeatedly across several maturity levels a compound un-perceivable by the others.

Examples of the first case are the following: Panelist 1 could not detect either the aroma compound eluted at 13 min nor ethyl octanoate, 13.18 min; the rest of the panelists detected both compounds in the late 1987 wine. Panelist 3 could not detect, in the early or middle wines, the aroma compound eluted at 21.84 min. Panelist 4 could not detect 2,3-butanediol, at 16.96 min, in the middle and late wines; hexanoic acid, at 23.94 min, in the early and late wines, methyl vanillate, at 41.50 min, nor ethyl vanillate; or the unknown compounds eluting at 12.78, 15.93, and 24.50 min. Panelist 5 could not detect the unknown compound eluting at 29.90 min.

There were 3 occasions when a unique peak was detected in the 3 wines by the same panelist. Panelist 2 detected the unique aroma peak eluting at 17.86 min in all the 1987 wines; in the three 1988 wines, this panelist detected the unique aroma peak eluting at 13.04 min. Panelist 5 detected the unique aroma

peak at 16.63 min in the three 1988 wines. There were several cases where a unique aroma peak was detected in only two of the wines, or an aroma peak was unique in two of the wines, but in the third wine this aroma peak was a consensus peak.

Even though there was large variability present in the individual responses per wine, the occurrence of unique aroma peaks across all 3 maturities was rare. This kind of aroma analysis needs to be conducted with at least three subjects, and also each evaluation per subject needs to be replicated several times.

#### **FINDINGS**

#### 1) FID

- 1.1) It was difficult to observe any pattern of change in FID chromatogram peaks across maturities and vintages.
- 1.2) Of the 91, 76 and 90 peaks (where concentration was reported) for the early, middle and late 1987 wines, 72 peaks were in common. Of the 74, 59 and 61 peaks (where concentration was reported) for the early, middle and late 1988 wines, 53 peaks were in common.
- 1.3) FID peaks common to all maturities which had the highest concentration were found in the middle wine for 1987 and in the early wine for 1988.

#### 2) AROMAGRAM

- 2.1) The largest number of aroma-active peaks was noted at the late harvest date for both vintages.
- 2.2) The number of aroma-active peaks was generally higher in the 1988 vintage (climatically a normal-late vintage) than in the 1987 vintage (climatically an early-hot/dry vintage).
- 2.3) There were only 10 aroma peaks that were detected in all three 1987 wines. There were 16 aroma peaks that were detected in all three 1988 wines. There were only 4 aroma peaks common to all the 1987 and 1988 wines.

- 2.4) The early and middle wines had approximately 25% of their peaks as unique peaks while the late wine had approximately 50% of its peaks as unique peaks for both vintages. Approximately 50% of the unique peaks per vintage were detected in the late wines.
- 2.5) All three 1987 wines had a high frequency percentage of peaks described as fruity and floral. The vegetative, spicy and woody frequency percentages were almost the same for any of the 1987 wines. In addition, the early wine had a high percentage of cotton candy, the middle wine had a high percentage of stinky/dirty socks/cheese, the late wine had a high percentage of floral and earthy/musty aromas.
- 2.6) The early and middle 1988 wines had almost the same frequency percentage of peaks described as fruity, floral, vegetative and sweet. The late 1988 wine had a high percentage of spicy, vegetative, fruity and sweet odors.
- 2.7) Comparing both vintages, the 1988 vintage had a much higher number of peaks described as dried fruit, vegetative, herbal and spicy. A few aroma notes occasionally detected only in the 1987 vintage were vanilla, stinky/dirty socks/cheese, meaty, and cotton candy.

2.8) Based on common peaks, there was not a clear match between any of the maturity wines of 1987 with any of the maturity wines of 1988.

#### 3) AROMAGRAM/FID

- 3.1) From 15-24% and 14-30% of the peaks from the FID response were aroma-active for 1987 and 1988, respectively.
- 3.2) Approximately 45% of the aroma-active peaks were not detected by the FID for the 1987 vintage. Approximately 66% of the aroma-active peaks were not detected by the FID for the 1988 vintage.

## 4) INDIVIDUAL RESPONSES

- 4.1) In many cases, one panelist was able to detect twice as many compounds in the same wine as another panelist.
- 4.2) No panelist was more or less sensitive to a certain aroma character or chemical group.
- 4.3) There were several panelists who could not detect aroma in a peak where aroma was detected by all the other panelists.
- 4.4) There were a few cases where a panelist detected an aroma peak across all three maturities which was undetectable by all of the other panelists.

Fig. 1 Extraction method for Pinot noir wines

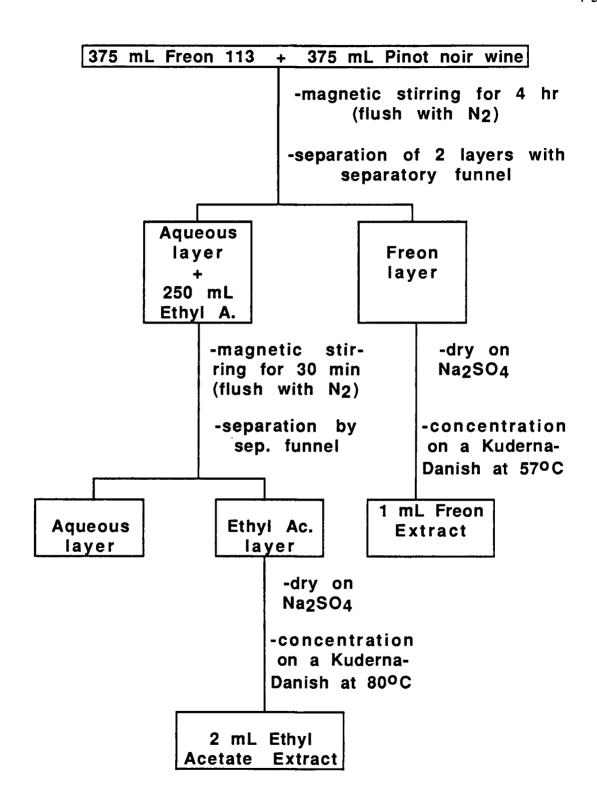
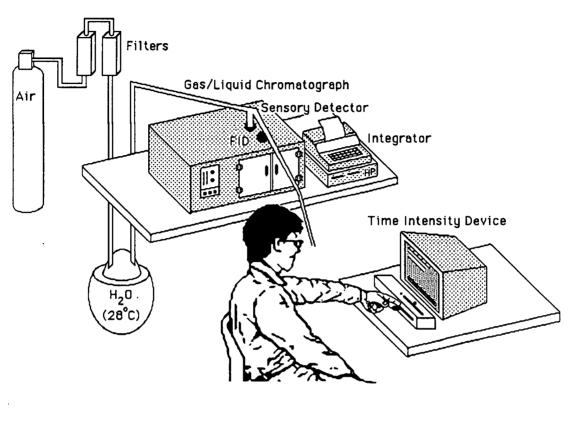


FIG. 1

Fig. 2 Equipment used to sniff and evaluate the GC effluent



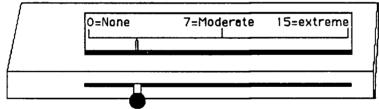


FIG. 2

Fig. 3 1st and 4th replication of middle 1987 wine, panelist 3

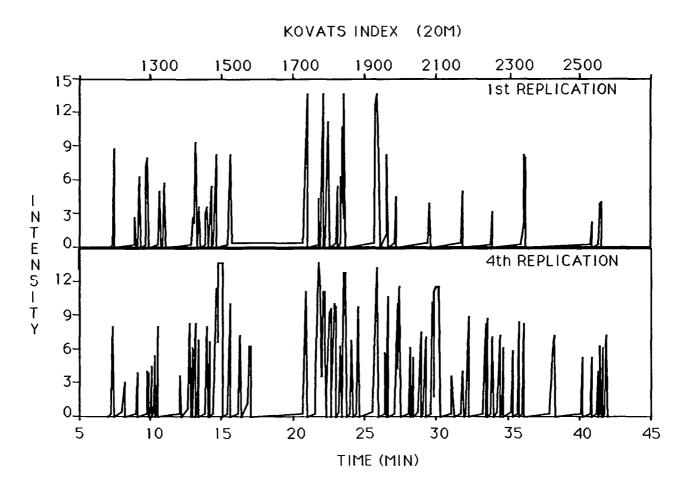


FIG. 3

Fig. 4 Aromagram of early 1987 wine, panelist 1, 2, 3 and 4

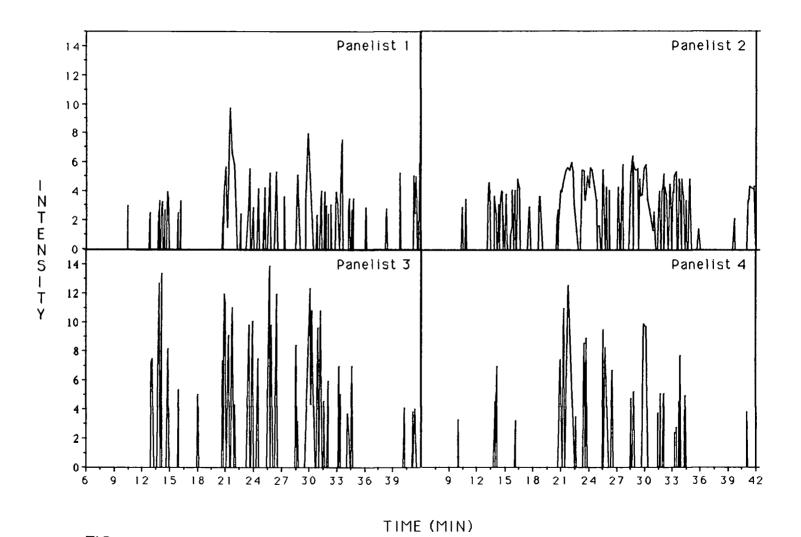


FIG. 4

Fig. 5 Aromagram of middle 1987 wine, panelist 1, 2, 3 and 4

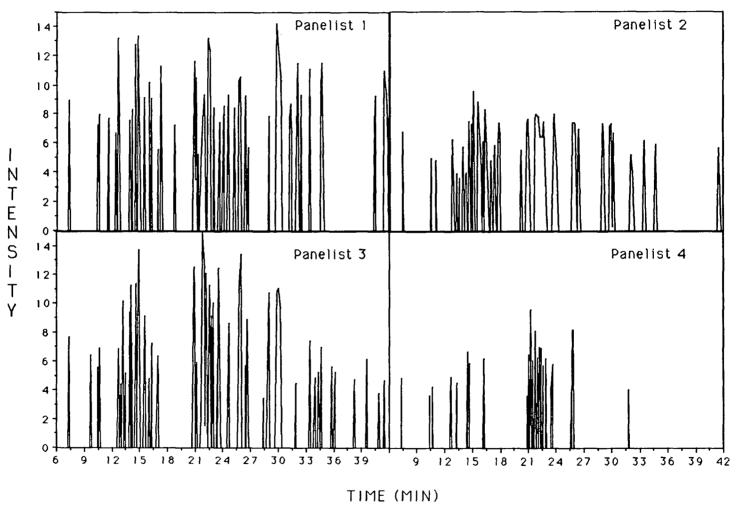


FIG. 5

Fig. 6 Aromagram of late 1987 wine, panelist 1, 2, 3 and 4

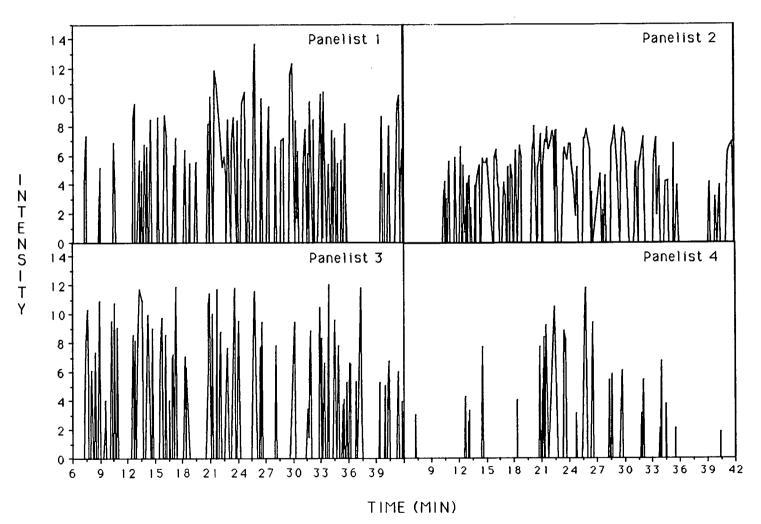


FIG. 6

Fig. 7 Aromagram of early 1988 wine, panelist 1, 2 and 5

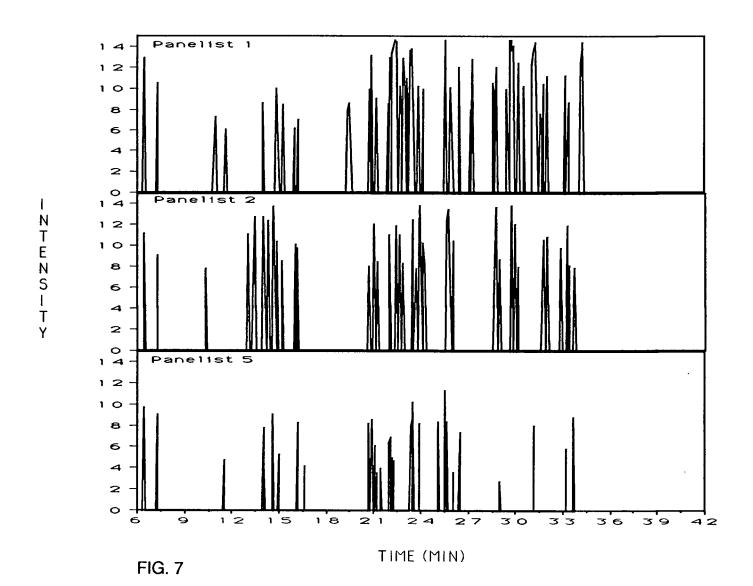


Fig. 8 Aromagram of middle 1988 wine, panelist 1, 2 and 5

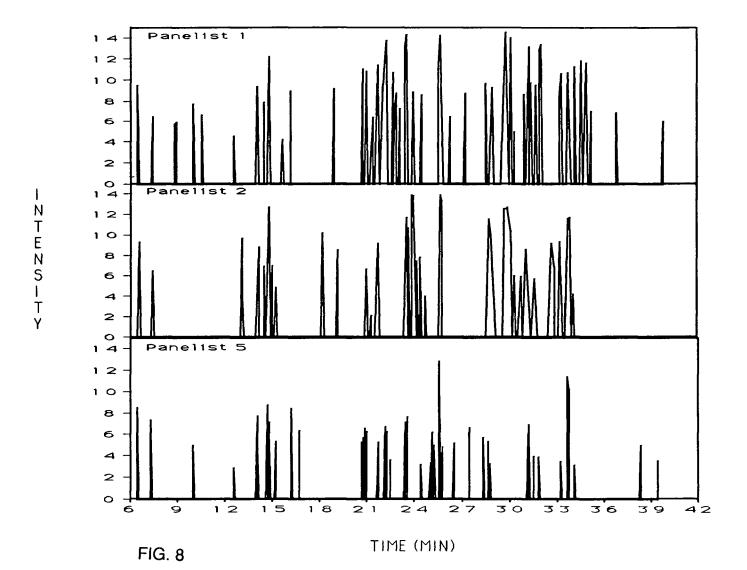


Fig. 9 Aromagram of late 1988 wine, panelist 1, 2 and 5

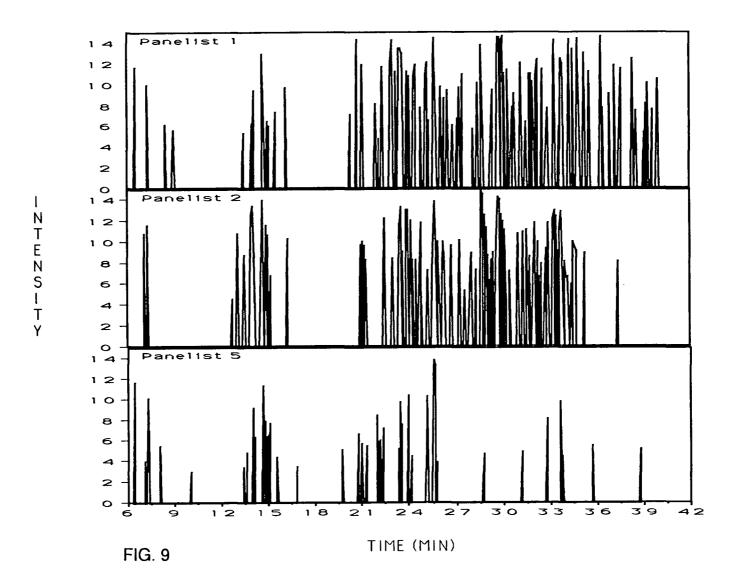


Fig. 10 Consensus aromagram of early, middle and late 1987 wines

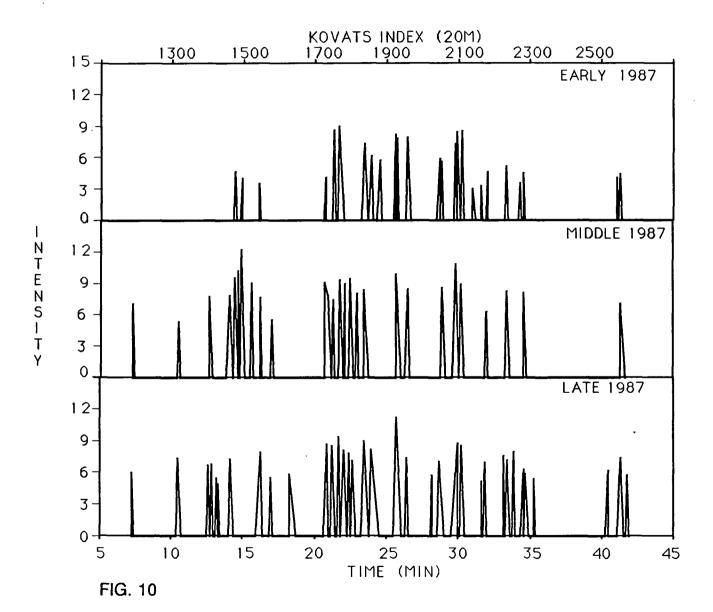


Fig. 11 Consensus aromagram of early, middle and late 1988 wines

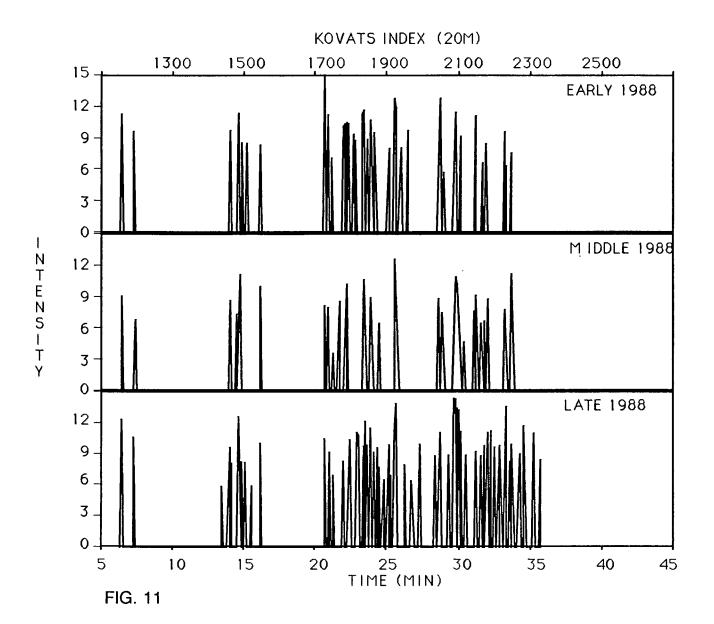


Fig. 12 FID chromatograms of early, middle and late 1987 wines. Compounds identified are listed in Table 3

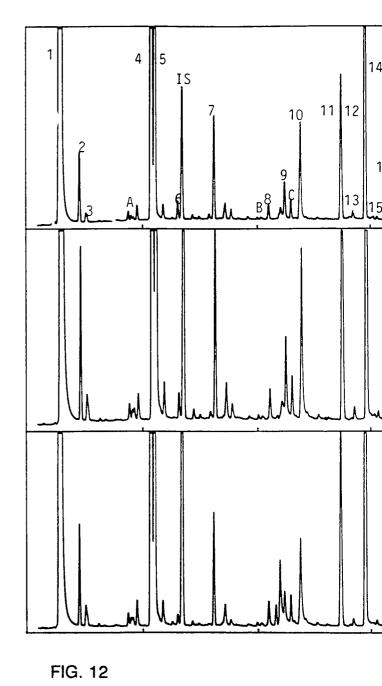


Fig. 13 FID chromatograms of early, middle and late 1988 wines. Compounds identified are listed in Table 3

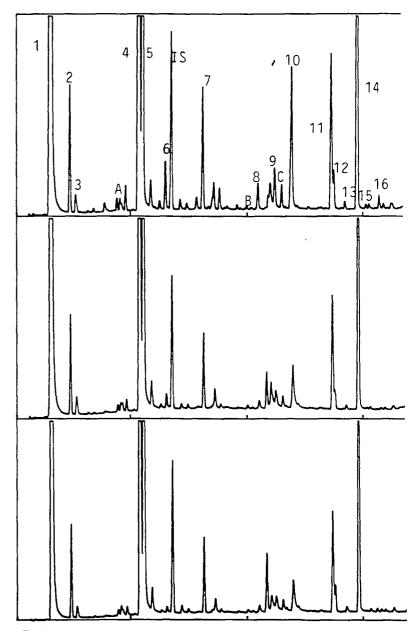


FIG. 13

Fig. 14 FID chromatogram and consensus aromagram of early 1987 wine

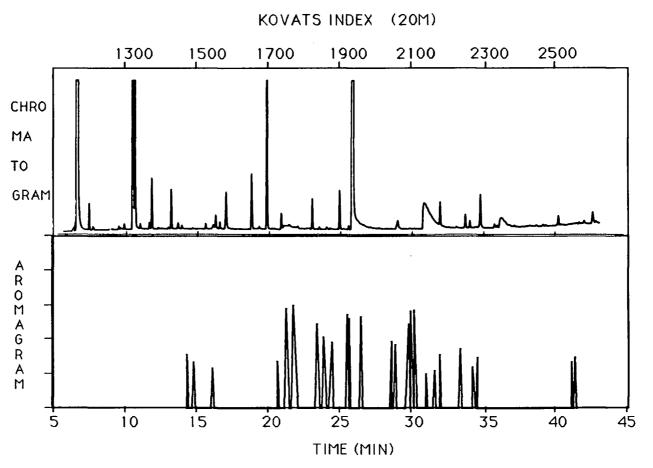


FIG. 14

Fig. 15 FID chromatogram and consensus aromagram of middle 1987 wine

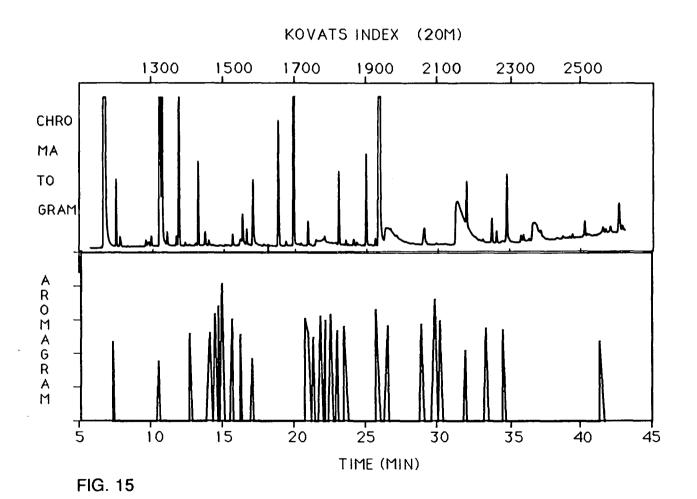


Fig. 16 FID chromatogram and consensus aromagram of late 1987 wine

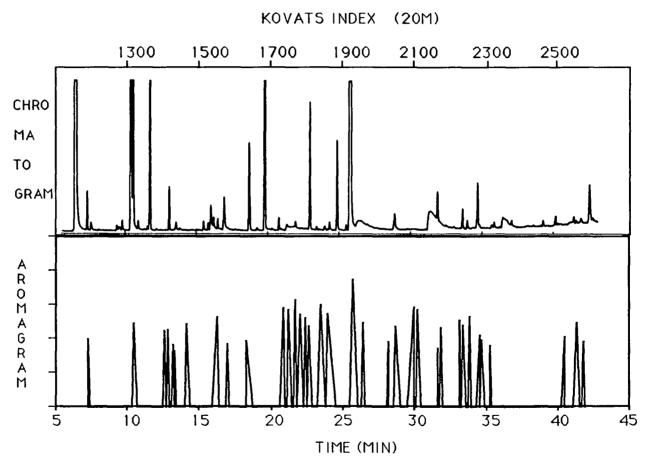


FIG. 16

Fig. 17 FID chromatogram and consensus aromagram of early 1988 wine

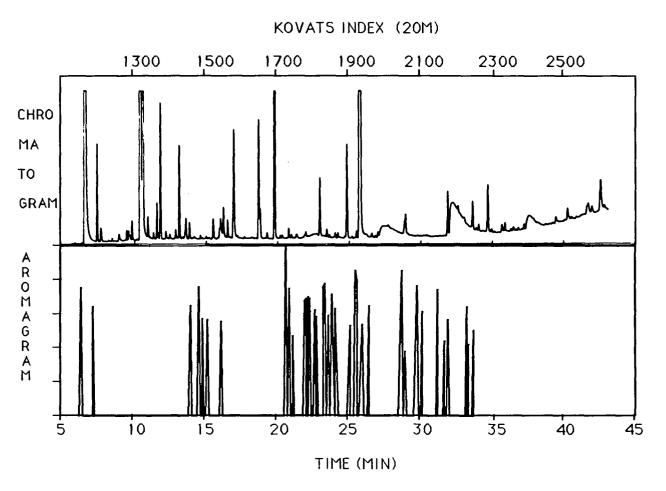


FIG. 17

Fig. 18 FID chromatogram and consensus aromagram of middle 1988 wine

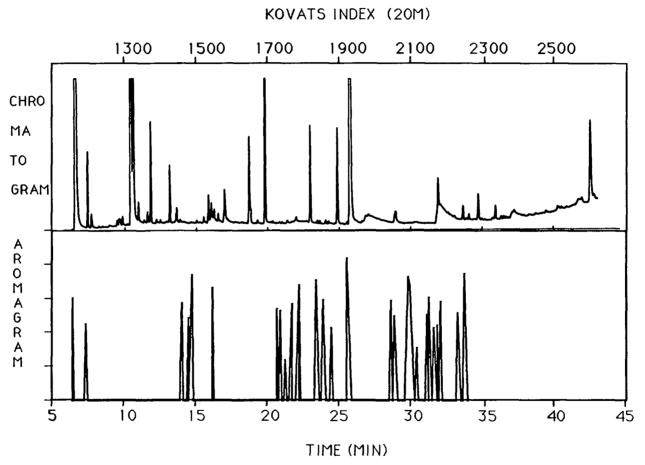


FIG. 18

Fig. 19 FID chromatogram and consensus aromagram of late 1988 wine

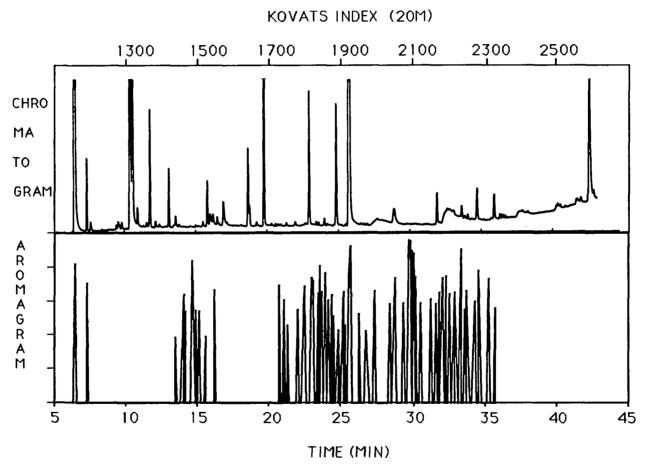


FIG. 19

Table 1 Composition of 1987 and 1988 Pinot noir musts and wines

				Must Composition						
	Maturity levels	Harvest Date	<sup>o</sup> Brix	T.A. g/L	рН	Malate g/L	Tartrate g/L	Ethanol %		
1	early	9/11	20.4	7.9	3.40	4.2	6.8	11.1		
9 8 7	middle	9/16	21.0	7.2	3.27	4.1	7.2	11.3		
	late	9/29	24.4	6.2	3.60	3.2	7.3	13.6		
1 9	early	09/29	21.8	10.2	3.23	4.7	7.6	11.8		
8	middle	10/13	23.0	8.2	3.34	3.6	6.9	13.6		
8	late	10/25	24.7	7.6	3.37	3.3	6.0	14.0		

Table 2 Standards developed for Pinot noir aroma description

Term	Standard
blackberry	Oregon, canned blackberry in heavy syrup, 50 g ;frozen blackberry,
•	50 g; blackberry jam, 50 g
raspberry	Oregon, canned raspberry in heavy syrup, 50 g; frozen raspberry, 50 g
strawberry	frozen strawberry, 50 g
grapes	red grapes, 50 g
pineapple	canned tidbits in its own juice, 50 g
peach	canned peaches in heavy syrup, 50 g
apple	fresh sliced, 50 g
cherry	Oregon, canned cherry pie filling, 50 g; 2 candies
plum	canned plum in heavy syrup, 50 g
prune	dried prunes, 50 g
fig	dried figs, 50 g
raisin	raisins, 50 g
roses	5 uL extract in 40 mL water; fragrance test filters (Orlandi, Inc.,
_	Farmingdale, NY)
lilac	5 uL extract in 40 mL water; fragrance test filters (Orlandi, Inc., Farmingdale, NY)
violet	5 uL extract in 40 mL water; fragrance test filters (Orlandi, Inc., Farmingdale, NY)
Hawaiian	5 uL extract in 40 mL water; fragrance test filters (Orlandi, Inc.,
blossoms	Farmingdale, NY)
allspice	ground allspice, 3 g
cinnamon	ground cinnamon, 3 g
black pepper	ground black pepper, 3 g
nutmeg	ground nutmeg, 3 g
anise	leaves, 3 g
licorice	red licorice and black licorice, 5 cm each
herbal	an equal mixture of rosemary, marjoram, tarragon, oregano, basil
:	leaves, thyme, 3 g
mint	leaves, 3 g
tobacco	one cigarette Camel (R.J. Reynolds Tobacco Co., Winston, Salem,NC)
molasses	molasses, 3 g
butterscotch	2 candies
vanilla	vanilla extract, 3 g
burnt_match_	one burned match (Diamond Brands, Inc., Minneapolis, MN)

All the standards were evaluated in 350 mL wine glasses covered with watch glasses.

Table 3 Compounds confirmed to be present in Pinot noir wine

Peak #	Compound Name	Ret. Time (min)	Ret. Index (I20M) <sup>3</sup>	Source	Aroma Descriptors
11	3-methyl-1-butanol (isoamyl alcohol)	6.45	1209	K & K	vegetative, chlorine, methol, glue, whiskey, pungent, chemical
2	ethyl hexanoate (ethyl caproate)	7.33	1244	Aldrich	fruity, apple, banana, wine
3	n-pentanol (amyl alcohol)	7.60	1252	Aldrich	sweet, balsamic
4	ethyl lactate	10.39	1354	Eastman	fruity, sweet, cherry, tropical fruit, buttery, butterscotch
5	n-hexanol	10.56	1360	Aldrich	fragrant, woody, green, resinous, heavy, herbal
6	cis-3-hexen-1-ol	11.54	1391	Compagnie Parento	fresh, green, grass, floral
7	ethyl octanoate (ethyl caprylate)	13.29	1454	Aldrich	fruity, floral, banana, orange, pear, pineapple, brandy
8	benzaldehyde	15.52	1534	Aldrich	almond, sweet, floral, cherry, citrus, fragrant
9	linalool	15.98	1550	Aldrich	fragrant, lemon, sweet, fruity, citrus
10	2,3-butanediol	16.94	1582	Eastman	faint, sweet, perfume, almond, artificial fruit
11	gamma-butyrolactone	18.66	1647	Aldrich	faint, sweet, caramel
12	ethyl decanoate (ethyl caprate)	18.74	1651	Eastman	brandy, oily, fruity, grape
13	isovaleric acid (3-methyl butyric acid)	19.12	1665	Aldrich	rancid, cheese, sweaty, putrid, stinky
1 4	diethyl succinate	19.76	1689	Aldrich	faint, pleasant, winey

Table 3 (cont.)

Peak #	Compound Name	Ret. Time (min)	Ret. Index (KI20M)	Source	Aroma Descriptors
15	alpha-terpineol	20.28	1708	Aldrich	fragrant, pine oil, floral, lilac, faint fruity
16	methionol	20.75	1727	IFF	vegetative, cabbage, stinky
17	beta-phenyl-ethyl- acetate	23.43	1831	Pfaltz & Bauer	sweet, honey, fruity, rose
18	hexanoic acid (caproic acid)	23.99	1853	Aldrich	sour, vinegar, cheese, sweaty, rancid, fatty, pungent
19	ethyl laurate (ethyl dodecanoate)	24.19	1861	Pfaltz & Bauer	soapy, citrus, detergent, green, fruity, floral
20	benzyl alcohol	24.84	1886	Aldrich	sharp burning taste, faint aromatic
21	beta-phenyl ethanol	25.73	1922	Aldrich	floral, roses, perfumery
22	gamma-octanolactone	26.25	1943	Pfaltz & Bauer	coconut, sweet
23	gamma-nonalactone	28.88	2049	Pfaltz & Bauer	coconut, peach, pineapple, sweet, anise, licorice
24	ethyl palmitate	33.61	2245	Pfaltz & Bauer	soapy, waxy
25	octanoic acid (caprylic acid)	35.67	2334	Aldrich	oily, fatty,rancid, soapy, sweet, faint fruity
26	decanoic acid (capric acid)	36.28	2361	Aldrich	fatty, unpleasant rancid odor, citrus
27	methyl vanillate	41.64	2603	Pfaltz & Bauer	vanilla, herbal, spicy, caramel
28	ethyl vanillate	41.96	2616	Pfaltz & Bauer	sweet, vanilla, spicy
29	acetovanillone	42.56	2640	Pfaltz & Bauer	vanilla, spicy, molasses, eugenol

Table 3 (cont.)

Peak #	Compound Name	Ret. Time (min)	Ret. Index (120M)	Source	Aroma Descriptors
30	tridecanoic acid	43.54	2678	Aldrich	fatty, citrus, unpleasant
A2	3-methyl-1-pentanol				
В	3-hydroxy butyric acid				
С	4-methyl-2-pentanol				
D	amyl-4-hydroxy butanoate				
E	n-butyl acetamide				
F	ethyl isobutyl succinate				
	Succinate				

- 1 Numbered compunds were confirmed by chemical name and aroma.
- 2 Compounds A-F in italic letters were only tentatively identified by GC-MS.
- 3 I20M means Kovats indices on Carbowax 20M phase.

Table 4 Consensus aromagram and number of FID peaks, for Pinot noir extracts

			برامم	1987	lata	ا ا	1988	lata
		number of aroma peaks	early 26	middle 28	late 37	early 34	middle 28	late 55
С	Α	number of aloma peaks	20	20	3,	54	20	33
0	R	average aroma peak	5.8	8.4	7.2	11.7	8.5	11.3
N	0	intensity						
S E	M A	unique aroma peaks:						
N	Ĝ	per vintage <sup>1</sup>	6	6	17	9	7	29
S	R	across vintages <sup>2</sup>	10	9	18	14	13	31
U	Α	aurooo viinagoo						
S	М	common aroma peaks:						
		per vintage		10	1	l	16	
		across vintages		<u>.</u>		9		
		number of peaks with	91	76	90	74	59	61
		concentration ≥1 ppm						
	^	and the second of the second						
	CROMA	peaks with the highest concentration:						
		per vintage	5	50	10	31	12	10
		across vintages	2	3	0	31	12	12
F		t to a t						
D	T 0	peaks with the lowest concentration:						
	Ğ	per vintage	54	5	11	11	12	25
	R	across vintage	41	3	9	1	2	3
	Α							
	М	unique peaks:  per vintage	8	0	7	11	0	4
		across vintages	25	15	24	7	2	5
		uoroso viinagos					_	J
		common peaks:						
		per vintage across vintages		72	1	9	53	
F	Α	across virilages			4	9		
i		aroma peaks with	14	18	17	12	8	18
D	0	concentration ≥1 ppm						
	M		4.0	0.0	<i>e</i> 4	65	74	6.7
		percentage of aroma peak not detected by	46	36	54	65	71	67
V	R	FID						
S.	Α							
	<u> M</u>	<del> </del>	<del></del>			<u> </u>		

value reported per vintage is considering the three wines harvested in the same year.
 value reported across vintage is considering the six wines from both years.

Table 5 Attribute's frequency used to describe aroma-active peaks from Pinot noir wine extracts, at three different maturity levels, 1987 and 1988 vintages

	ea	ırly		87 ddle	la	te	ea	rly		88 ddle	la	te
					1 outly impair late							
	Frequency of use											
	#1	%2	#	%	#	%	#	%	#	%	#	%
attribute		on		on		on		on		on		on
		26		28		37		34	_	28		55
fruity	15	58	19	68	20	54	14	41	13	46	29	53
dried fruit	3	12	4	14	6	16	9	26	8	29	21	38
diled iidit	3	12	. •	1 🤫	· ·	10		20	0	23	21	30
floral	14	54	14	50	31	84	19	56	16	57	25	45
vegetative	6	23	7	25	7	19	14	4 1	11	39	30	54
herbal	4	15	4	14	4	11	12	35	6	21	23	42
		_		_					_			
spicy	3	12	2	7	5	14	6	18	7	25	32	58
earthy/musty	3	12	2	7	6	16	6	18	2	7	4	7
sweet	9	35	9	32	6	16	8	24	8	29	18	33
cotton candy	3	12	2	7	0	0	1	3	0	0	1	2
caramel	0	0	2	7	1	3	5	15	5	18	6	11
woody	6	23	9	32	7	19	4	12	3	11	9	16
smokey/toasty	5	19	7	25	4	11	4	12	3	11	9	16
vanilla	0	0	2	7	1	3	0	0	0	0	0	0
chemical	5	19	7	25	6	16	13	38	8	29	13	24
dirty socks/	3	12	6	21	1	3	0	0	0	0	0	0
stinky/cheese												
meaty	1	4	1	4	1	3	0	0	0	0	0	0
sulfur/cabbage	0	0	1	4	0	0	7	21	2	7	5	9
soapy/fusel oil	0	0	0	0	2	5	3	9	4	14	2	4
pungent	1	4	0	0	1	3	4	12	4	14	7	13

<sup>1 #</sup> indicates the number of times that atribute was used to describe aroma-active peaks, one aroma peak could be described with more than one attribute.

<sup>2 %</sup> indicates the percentage of use for each attribute based on the total number of peaks detected per wine.

Table 6. Individual aroma-active peaks per panelist by treatment.

					Panelis	t	
		<del>_</del>	1	2	3	4	5
	Ε						
	Α	total number of peaks	52	67	38	29	
	R						
	L	consensus peaks	31	28	29	21	
	Y	non-consensus peaks	17	23	6	7	
		unique peaks	4	16	3	1	
	М						
1	i	total number of peaks	46	40	43	24	
9	D						
8	D	consensus peaks	31	28	29	20	
7	L	non-consensus peaks	6	6	4	2	
	<u>E</u>	unique peaks	9	6	10	2	
	L	total number of peaks	62	64	59	33	
	Α						
	Т	consensus peaks	35	38	37	25	
	Ε	non-consensus peaks	18	13	13	6	
		unique peaks	9	13	9	2	
	_		4.0				
	E	total number of peaks	46	36			29
	A		0.7				0.0
	R	consensus peaks	27	28			22
	L Y	non-consensus peaks	6	1			3
		unique peaks	13	7			4
_	M	A Anti-complete of margina	47	0.5			4.0
1	I	total number of peaks	47	35			43
9	D D		00	0.0			0.4
8 8		consensus peaks	28 4	22			24
O	L E	non-consensus peaks	4 15	2 11			2 17
	_ <u></u>	unique peaks	15	<u> </u>			17
	L	total number of peaks	80	75			41
	Ā	total Humber of peaks	00	, 5			<b>-+</b> 1
	Ť	consensus peaks	49	34			30
	Ė	non-consensus peaks	6	19			4
	_	unique peaks	25	22			7

<sup>-</sup> The total number of peaks is equal to the sum of consensus, non-consensus and unique peaks.

<sup>-</sup> Consensus peaks are those detected by the majority of the panelists.

<sup>-</sup> Non-consensus peaks are those detected by two of the panelists.

<sup>-</sup> Unique peaks are those detected only by one of the panelists.

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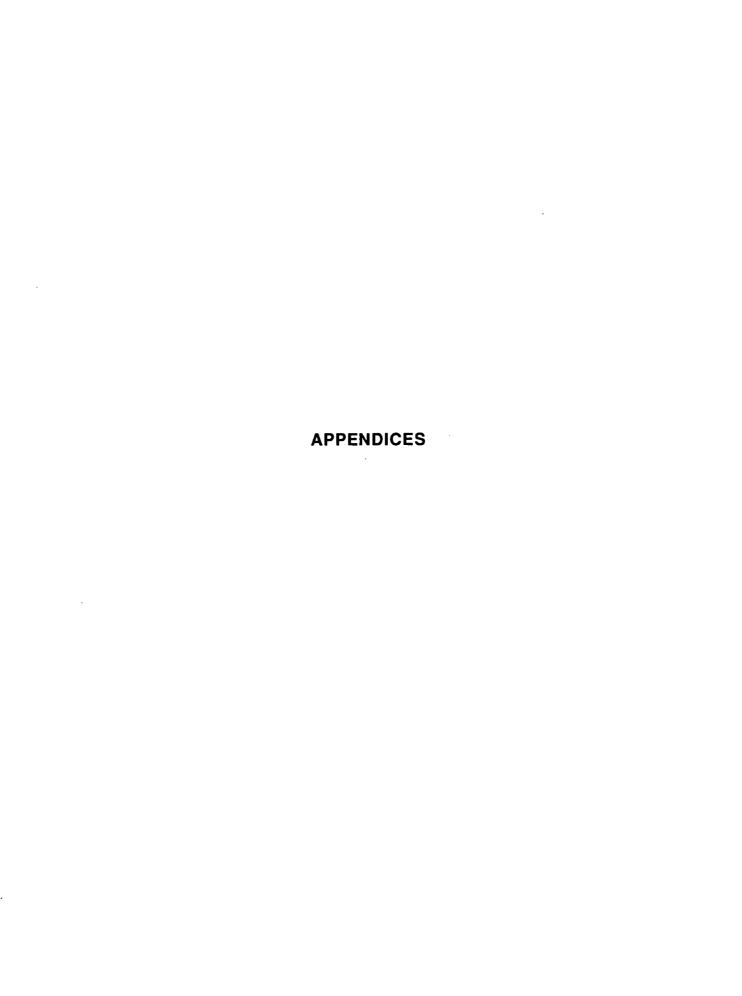
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Appendix 1 Consensus of aroma-active peaks and their intensities and character descriptors as defined by retention times (RT), Kovats indices (I20M).

RT (min)		1987			1988		Chemical name <sup>3</sup>
[I20M] <sup>1</sup>	Early	Middle	Late	Early	Middle	Late	- IIIIII
6.45 [1209]				11.2 <sup>2</sup> fusel oil, spicy, musty	9.1 smokey- nutty, herbal, spicy, fusel oil	12.3 fusel oil, fruity, smokey	3 - methyl- 1 - butanol
7.28 [1244]		7 sweet fruity, floral, cherry, banana	6 fruity, floral	9.6 floral	6.7 floral, sweet, musty, tropical fruit	10.5 floral, pungent, banana	ethyl hex- anoate
10.45 [1358]		5.3 sweet, floral, fruity, green	7.3 floral				ethyl lactate / n- hexanol
12.60 [1433]		7.8 cheese, grainy, smokey, toasty, sulfur	6.7 floral, spicy, toasty, grainy				
12.80 [1439]			6.7 floral, fruity, toasty				
13.05 [1446]			5.5 perfumy, floral, musty, microlab				

RT (min)		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
13.18 [1453]			5.5 floral, fruity, musty				ethyl oc- tanoate
13.40 [1458]			4.9 fruity, floral, musty			5.8 floral, spicy, pungent	ethyl oc- tanoate
14.00 [1478]		7.8 floral, toasty, brown sugar		9.7 floral, musty, chemical	8.6 herbal, pungent, piney, smokey, acrid	9.6 floral, pungent, herbal, caramel	
14.15 [1483]			7.3 floral, fruity			8.1 floral, herbal, citrus, spicy	
14.25 [1487]	4.6 floral, fruity	9.5 floral, fruity, sweet					
14.48 [1494]					7.4 floral, pungent, spicy		
14.66 [1500]		10.2 vegeta- tive, fresh green, smokey, herbal		11.4 pungent, sweet, herbal, musty, tobacco	11.2 herbal, smokey, winey, caramel	12.5 pungent, spicy, toasty, vegeta- tive, herbal, smokey	

RT		1987			1988		Chemical
(min)		1.501			1000		name
[120M]	Early	Middle	Late	Early	Middle	Late	
14.87 [1508]	4 floral, fruity, spicy, pungent, vegetati_ ve	12.2 toasty, cheese, stinky, smokey		8.5 ripe fruit, herbal, burnt match		8.2 toasty, pungent, herbal, smokey, nutty	
15.06 [1515]						8.1 toasty, smokey, pungent, spicy, piney	
15.24 [1522]				8.5 floral, herbal, spicy, burnt match			
15.57 [1534]		9 ripe fruit, floral				5.9 floral, smokey, spicy	ben- zalde- hyde
15.91 [1547]	3.5 floral, dry fruit, black pepper, smokey						unknown linalool impurity
16.21 [1557]	3.5 floral, linalool	7.7 heavy fruity, peach, cherry, floral, sweet	7.9 floral, linalool	8.3 linalool, floral	10 linalool, floral	10 linalool, floral, herbal	linalool

RT		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
16.95 [1583]	Lany	5.6 floral, waxy fruity	5.6 floral, fruity, herbal	Carry	Middle	Late	2,3-bu- tanediol
18.25 [1630]			5.8 floral, fruity, sweet				
20.64 [1723]	4.1 fruity, toasty, meaty, baked potato	9.1 toasty, green, meaty, baked potato		15 herbal, vegeta- tive, cabbage, floral	8.1 cabbage, soapy, burnt match	10.4 cabbage, dill, vegeta- tive	me- thionoi
20.82 [1730]			8.7 malty, floral, meaty, buttery				
20.92 [1734]				11.2 floral, caramel, fruity	8 floral, fruity	9.1 floral, prunes, sweet, herbal	
21.04 [1738]						7.4 floral, sweet, herbal, spicy, vegeta- tive	
21.22 [1746]	8.7 dirty socks	7.4 toasty, earthy, grainy	8.6 piney, hay, baked potato	7 floral, fruity, herbal, grassy	4 floral, prunes	6.9 dried fruit, butter- scotch	unknown methionol impurity

RT (min)		<u>1987</u>			<u>1988</u>		Chemical name
[120M]	Early	Middle	Late	Early	Middle	Late	l lamo
21.64 [1762]	g dirty socks, musty floral	9.3 dirty socks, floral, fruity, stinky	9.4 dirty socks, floral, fruity		8.6 floral, herbal, pungent, cabbage		unknown methionol impurity
21.95 [1773]		9.1 dirty socks, stinky, floral, fruity		10.3 floral, herbal, vegeta- tive, cabbage		8.25 floral, spicy, dry fruit	unknown methionol impurity
22.05 [1778]		9 fruity, apricot, dirty socks, sour apple	8.1 floral, fruity	10.5 herbal, tobacco, vegeta- tive, medicinal			unknown methionol impurity
22.17 [1782]				•	10.2 cabbage, dried fruit, caramel		
22.25 [1784]					10 floral, prunes		
22.32 [1787]		·		10.4 pungent, herbal, cabbage, fusel oil			unknown methionol impurity
22.41 [1791]		9.4 floral, toasty, smokey, malty	7.8 floral, musty, herbal			10.3 tobacco, herbal	

RT		1007			1000		Chaminal
(min)		<u>1987</u>		·	<u>1988</u>		Chemical
[120M]	Early	Middle	Late	Early	Middle	Late	name
22.67 [1800]			7.1 floral, herbal, earthy	9.3 herbal, cabbage, fusel oil			
22.84 [1807]				8.8 herbal, spicy, vegeta- tive, cabbage, toasty			
22.96 [1811]		8 fruity, stinky				11 floral, cabbage, pungent	
23.37 [1829]						9.7 floral, prunes, herbal, spicy	beta- phenyl- ethyl acetate
23.45 [1832]	7.3 fruity, apple, pear, sweet	8.4 fruity, apple, peach	g ripe fruit, apple, roses, berry	11.6 ripe fruit, floral	10.6 floral, ripe, apple, prunes, tobacco	12.1 dried fruit, minty, butter- scotch	beta- phenyl- ethyl acetate
23.60 [1838]				8.8 floral, caramel, earthy, grassy		9.9 dried fruit, tobacco, caramel, sweet herbal	

RT		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
23.91 [1850]	6.2 toasted bread, tobacco, fruity		8.2 floral, toasty	10.7 floral, earthy, smokey	8.9 herbal, nutty, pungent	11.5 dried fruit, tobacco, spicy	hex- anoic acid
24.16 [1860]				9.5 smokey, toasty, earthy		9.1 dried fruit, smokey, spicy, burnt match	ethyl dode- canoate
24.38 [1869]					6.5 fruity, butter- scotch	9.5 dried fruit, herbal, nutmeg	n-butyl aceta mide?
24.46 [1872]	5.8 toasty, fruity, herbal					7.7 dried fruit, spicy, herbal, caramel	
24.78 [1884]						6.5 dried fruit, spicy	benzyi alcohol
25.19 [1899]				8 ripe fruit, caramel		9.9 roses	ethyl isobutyl succinate ?
25.28 [1904]						6.9 floral, vegeta- tive	

RT		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
25.59 (1916)	8.2 floral, roses		-	11.2 floral, roses	12.5 roses	11.3 roses	beta- phenyl ethanol
25.79 [1925]	7.8 floral, roses	9.9 floral, roses, lilac, fruity	11.2 floral, roses, lilac	12.7 roses, pungent	12.5 roses	13.8 roses	beta- phenyl ethanol
25.99 [1933]				8 roses, pungent			beta- phenyl ethanol
26.24 [1943]						7.9 roses, fruity	gamma- octa- lactone
26.43 [1950]	8 roses, laurel	8.5 floral, fruity, berry	7.4 floral, roses, lilac	9.7 floral, vegeta- tive			unknown beta- phenyl ethanol impurity
26.58 [1956]			7.4 floral, roses				
26.78 [1964]						6.4 herbal, hay, earthy, allspice, sweet	
27.26 [1983]						9.9 floral, fruity, tobacco, musty	

RT		1987			1988	<del></del>	Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
28.22 [2017]			5.7 floral, ripe fruit				
28.28 [2028]						8.8 dried fruit, smokey, candy	
28.59 [2037]	5.8 cotton candy, fruity, cherry		7 black- berry, rasp- berry, peach, cherry				
28.71 [2042]				12.8 cotton candy, cherry, dried fruit	8.8 fig, caramel	11 cotton candy, butter- scotch	gamma- nona lactone
28.85 [2048]	5.6 fruity, cherry, berry, cotton candy	8.6 cotton candy, fruity					gamma- nona lactone
29.01 [2054]				5.7 fruity, floral, caramel	7.5 fig, caramel, tobacco		
29.32 [2067]						8.9 prunes, floral, butter- scotch, cinnamon	unknown gamma- octa lactone impurity

OT		1007		<u> </u>	1000		Oh a mia a l
RT (min)		<u>1987</u>			<u>1988</u>		Chemical name
[120M]	Early	Middle	Late	Early	Middle	Late	Hairio
29.77 [2085]	7.3 fruity, cherry, berry, cotton candy	10.8 cotton candy, fruity		11.4 ripe fruit, sweet, candy, perfume	10.9 caramel, fruity, floral, vegeta- tive	14.3 fig, cin- namon, nutmeg, herbal	unknown gamma- nona lactone impurity
29.96 [2092]	8.5 fruity, berry, prune, cherry		8.8 berry, cherry, jammy			13.4 dried fruit, spicy, caramel	
30.06 [2096]						13.2 dried fruit, spicy, butter- scotch	
30.18 [2101]	8.6 floral, fruity, sweet, jammy	8.9 ripe fruit, herbal	8.5 heavy fruity, sweet, floral	9.2 fruity, caramel, herbal, spicy		11.1 dried fruit, spicy, caramel	unknown gamma nona lactone impurity
30.36 [2109]					4.7 prunes. cherry, rasp- berry, herbal		
30.52 [2115]						8.8 spicy, prunes, herbal	
30.99 [2134]	3 sweet, floral						

RT (min)		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
31.09 [2139]					7.6 floral, soapy, vegeta- tive		
31.19 [2143]				11.1 dried fruit		9.2 floral, fruity, earthy, grassy, butter- scotch	
31.27 [2147]					9.1 prunes, floral, anise		
31.59 [2159]	3.3 floral, fruity		5.2 fruity, berry, peach, prune, cherry		6.5 floral, spicy	8.7 fig, peach, spicy, herbal	
31.66 [2163]				6.6 ripe fruit			
31.77 [2167]						9.8 dried fruit, herbal, cinnamon	
31.89 [2173]			7 fruity, spicy, floral	8.5 floral, spicy	6.7 floral		

RT		1987		<del></del>	1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
31.99 [2176]	4.7 floral, gardenia, herbal, sweet	6.3 fruity, herbal, cinnamon					
32.08 [2179]					8.8 spicy, waxy, soapy	11 spicy, herbal, floral	
32.28 [2188]						11.2 floral, spicy, smokey, soapy	
32.52 [2197]						9.6 spicy, musty, nutty, smokey	
32.77 [2211]						8.7 spicy, herbal, floral	
32.90 [2214]						9.7 spicy, herbal, floral, coconut	
33.20 [2227]			7.6 floral, soapy	9.6 floral, ripe fruit			
33.31 [2232]				6.4 ripe fruit, spicy, herbal	7.7 floral, spicy, vegeta- tive	13.5 floral, fruity, cinnamon	

RT (Thin)		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
33.40 [2236]	5.2 grainy, fruity	8.2 floral	7.2 floral, soapy				
33.53 [2245]						8.2 spicy, candy, hay, herbal	ethyl palmi- tate
33.75 [2251]				7.5 floral, fruity, coconut, smokey	11.2 coconut, spicy, sweet, musty	9.9 spicy, coconut, herbal, sweet	
33.84 [2255]			8 spicy, herbal, pungent				
34.21 [2270]	3.6 smokey, stinky, solvent						
34.30 [2276]						9 nutmeg, herbal, caramel	
34.53 [2284]	4.5 black pepper, fruity, herbal	8.1 black pepper, floral, fruity, herbal	6.3 floral, black pepper, green			11.7 spicy, vegeta- tive	
34.75 [2298]			5.9 floral, black pepper, plum				

RT		1987			1988		Chemical
(min) [120M]	Early	Middle	Late	Early	Middle	Late	name
35.27 [2317]						11 floral, ripe fruit, burnt match	
35.35 [2322]			5.4 floral, sweet, woody	·			
35.66 [2335]						8.4 floral, cabbage	oc- tanoic acid
40.46 [2550]			6.2 fruity, ferment- ed apple, smokey				
41.12 [2580]	4 floral, herbal, sweet, musty						
41.38 [2590]	4.4 sweet, floral	7.1 caramel, vanilla	7.4 floral, ripe fruit, caramel				
41.51 [2598]		7.1 caramel, butter- scotch, vanilla, fruity					methyl vanil- late

RT (min)	1987			1988		Chemical name
[120M] Early	Middle	Late	Early	Middle	Late	Namo
41.84 [2612]		5.8 floral,				ethyl vanil-
		fruity, sweet, vanilla				late

- 1 I20M means Kovats indices on Carbowax 20M phase.
- 2 Numbers represent an aroma peak intensity value.
- 3 Tentatively identified compounds in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italic* with a ? were tentatively identified by GC-MS.

Appendix 2 Concentrations in ppm<sup>1</sup> of compounds by retention times (RT), and Kovats indices (I20M)<sup>2</sup> found in Freon extracts of Pinot noir wines.

R. T.		1987			1988		Chemical
(min) [I20M]	early	middle	late	early	middle	late	name <sup>3</sup>
6.45 [1209]	19086	31533	20261	52218	44447	20474	3-methyl-1- butanol
7.33 [1244]	72	122	86	323	331	275	ethyl hexanoate
7.60 [1256]	15	10	25	67	87	46	n-pentanol
8.85 [1299]				41			
9.45 [1320]	11	16	14	40	26	23	3-methyl-1- pentanol
9.58 [1326]	6	9	8	60	32	57	
9.67 [1329]	4	12	8				
9.84 [1335]	17	25	24	74	52	39	
10.40 [1354]	2590	4981	3037	15997	11813	8172	ethyl lactate
10.56 [1360]	704	1391	872	3841	4912	3727	n-hexanol
10.96 [1373]	15	26	22	79	93	70	
11.35 [1385]	2		6	23	17		
11.45 [1391]	20	21	11	127	58	21	3-hexen-1-ol

R. T.		1987			1988		Chemical name
(min) [120 <b>M</b> ]	early	middle	late	early	middle	late	
12.14 [1412]	6						
12.21 [1415]	2	9	6	32	21	31	
12.48 [1425]	4	5	4	20	16	21	
12.90 [1439]	5	8	6	42			unknown ethyl octanoate impurity
13.29 [1454]	104	167	103	333	268	240	ethyl octanoate
13.42 [1458]						8	
13.60 [1463]	24	38	2	105	107	65	
13.86 [1474]	12	17	11	65	28	16	
14.18 [1483]	2		2				
14.61 [1500]	4	4	4	11			
15.02 [1515]	2	5	6	14	19	13	
15.18 [1520]	3	5	5				3-hydroxy butyric acid
15.52 [1534]	16	31	30	81	42	34	benzaldehyde
15.78 [1543]	1						

R.T.		1987			1988		Chemical name
(min) [120M]	early	middle	late	early	middle	late	
15.85 [1545]	5	5	17	35	147	215	unknown linalool impurity
16.00 [1550]	8	32	97	108	165	98	iinalool
16.22 [1557]	41	88	67	138	115	98	unknown linalool impurity
16.51 [1567]	18	43	36	61	56	54	4-methyl-2- pentanol ?
16.95 [1583]	121	190	135	513	256	181	2,3 butanediol
17.20 [1591]			11				
17.70 [1609]	3	6	8				
18.12 [1626]			2				
18.66 [1647]				503	482	373	gamma- butyrolactone
18.70 [1649]	171	338	233				gamma-butyro lactone and ethyl decanoate
18.74 [1651]				109	72	89	ethyl decanoate
19.13 [1665]	3		3				isovaleric acid
19.25 [1669]	12	13	12	25	24	24	
19.76 [1689]	736	1522	1427	2414	2119	1592	dlethyl succinate

R. T.		1987	**		1988		Chemical name
(min) [120 <b>M</b> ]	early	middle	late	early	middle	late	
20.10 [1701]	4			. 13			
20.30 [1708]	3	6	3	18	15	14	alpha- terpineoi
20.55 [1719]						15	
20.80 [1730]	40	58	24	42		16	methionol
20.98 [1735]	27		4	17			
21.36 [1751]	59	17	44	13	26	24	
21.87 [1770]	1	7					
21.97 [1773]	18	16	23	40	65	31	unknown methionol impurity
22.43 [1791]	6		3				unknown methionol impurity
22.55 [1795]	7		4				
22.97 [1811]	79	162	272	241	497	603	amyl-4-hydroxy butanoate?
23.46 [1832]	7	12	9	31	17	17	beta-phenyl ethyl acetate
23.62 [1838]	2	4	3	12	18	16	
23.91 [1850]	11	21	18	29	28	37	hexanoic acid

R. T.		1987	•		1988		Chemical name
(min) [120 <b>M</b> ]	early	middle	late	early	middle	late	
24.22 [1861]	4	10	8	26	21	11	ethyl dodecanoate
24.35 [1868]			24				n-butyl acetamide
24.67 [1880]	1	3	3	12			
24.88 [1887]	113	222	214	402	541	595	benzyl alcohol
25.21 [1899]	4		4				
25.53 [1914]	12	17	16	29	18	14	ethyl isobutyl succinate/ unknown beta- phenyl ethanol impurity
25.80 [1925]	8716	13357	10578	19405	16236	13953	beta-phenyl ethanol
26.25 [1943]		50.8	78				gamma-octa lactone
26.58 [1956]	1	113	54	19	19		
26.75 [1964]				8	78		
27.04 [1974]	3	44	20	18	136		
27.30 [1984]				56			
27.61 [1996]	4					177	

R. T.		1987			1988		Chemical name
(min)		1307			1300		Onemical name
[120M]	early	middle	late	early	middle	late	
28.92 [2051]	46	83	65	139	146	229	gamma-nona lactone
29.25 [2064]	3						
29.80 [2085]	1	6	4				unknown gamma- nonalactone impurity
30.12 [2099]	3		3				unknown gamma- nonalactone impurity
30.27 [2105]	1						
31.05 [2139]			2				
31.30 [2147]	448	154	284				
31.91 [2173]	81	221	100	202	417	165	
32.12 [2179]				853			
32.52 [2197]	2		4	504		295	
33.06 [2221]	6	7	7				
33.51 [2241]			3				
33.68 [2248]				53		64	ethyl palmitate
34.01 [2262]	23	32	26	61	39	52	

R.T.		1987			1988		Chemical name
(min) [120M]	early	middle	late	early	middle	late	
34.44 [2281]	7	12	9				
34.74 [2298]	113	194	131	259	178	198	
34.90 [2300]				21			
35.73 [2337]	13	20	11	41	18	15	octanoic acid
35.91 [2345]	9	25	21	44	92	141	
36.31 [2363]	54	15	6	12	22	27	decanoic acid
36.52 [2371]		265	138	23	24	28	
36.77 [2382]				13	15	21	
37.14 [2398]	13	62	26	25	208		
37.56 [2417]	3			142		97	
37.89 [2433]			2				
38.26 [2450]	6	8	13				
38.67 [2468]	9	8	6				
39.15 [2490]	6	4	4				

Appendix 2 (cont.)

R. T.	- · · · ·	1987			1988		Chemical name
(min) [120M]	early	middle	late	early	middle	late	
39.36 [2499]	5	11	16	47	17		
39.57 [2509]	6	4					
39.74 [2517]	3						
40.11 [2534]	5	9	3				
40.23 [2540]	32	47	27	96	23	33	
40.40 [2548]	7	13	9	31	18	49	
40.50 [2552]						36	
40.89 [2570]	9	10	9	40			
41.31 [2589]	22	19	26	54			
41.60 [2602]	11	72	30	214	54	113	methyl vanillate
41.70 [2606]	14	29	18				
42.00 [2617]	31	61	35	113	68	97	ethyl vanillate
42.60 [2641]	63	143	147	391	716	1227	aceto vanilione
43.60 [2660]	15	32	20	62	32	118	tridecanoic acid

- Only peak concentrations higher than 1 ppm, which is the minimum concentration reported by the integrator, were included in the table.
- 2 I20M means Kovats indices on Carbowax 20M phase.
- 3 Tentatively identified compounds in **bold** are those which chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italics* with a ? were tentatively identified by GC-MS.

Appendix 3.1 Early 1987 maturity Pinot noir, peaks detected by each panelists at least twice over the 4 replications.

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name <sup>2</sup>
09.96				fig, hay, pungent	
10.50	sweet, floral <sup>1</sup>	light floral, floral		,	1-hexanol
10.93		floral blossoms, perfumy			
12.82	floral		floral, fruity		unknown ethyl octanoate impurity
13.00			smokey, woody, toasty		
13.43		floral, perfumy, toasty			
13.99	herbal, grainny	sweet floral, herbal			
14.20	grainny, toasty	sweet floral, pungent			
14.44	wheat, toasty	floral, roses, fruity			
14.78	floral, spicy, pungent	floral, fruity, spicy	fruity, winy, vegetative		
14.93		floral			
15.20		lilac, sweet dried fruit			3-hydroxy butyric acid

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
16.16	linalool	linalool, roses, blackberry blossoms		floral, cinnamon	linalool
16.49		floral			4-methyl-2- pentanol?
17.70		floral, herbal, toasty			
18.03			smokey, tobacco, floral		
18.85		smokey, toasty, earthy, floral			
20.71	dirty socks, petroleum	floral, ripe fruit	toasty grainy, meaty, baked potato	apple, fruity	methionol
20.95	soapy, dirty socks		grainy, caramel, dirty socks	dirty socks	methionol
21.09		musty floral, wild floral			
21.29	dirty socks	floral, sweet ripe fruit	dirty socks	dirty socks	
21.48	cheesy, dirty socks floral	leather, wet dog			
21.68	dirty socks floral	musty, floral	dirty socks, fruity	dirty socks	
21.82		herbal honey, ripe cherry		dirty socks	
21.98	floral, grape		fruity, apple		

Appendix 3.1 (cont.)

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
22.04		ripe fruit	fruity, apple		
22.18		wild floral, roses			
22.63	citrus floral, fruity	ripe fruit		dirty socks, fruity	
23.42	sweet	sweet floral	apple, pear	fruity	beta- phenyl- ethyl acetate
23.55	sweet, dried fruit	toasty, sweet	cooked fruity, apple, pear	fruity, floral	
23.76		toasty bread		tobacco	
23.95	toasty bread, grainy	toasty bread, floral	tropical fruit, cherry		hexanoic acid
24.12		sweet terpine, sweet floral			ethyl dode- canoate
24.21		ripe fruit, wild blackberry			ethyi dode- canoate
24.48	herbal, vanilla	sweet toasty, ripe prune	smokey, metallic, floral		
24.92		floral, perfumy, roses			benzyl alcohol
25.06	floral	floral, perfumy			
25.20	floral, fruity	floral			

Appendix 3.1

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
25.60	roses	floral, herbal, roses	floral, lilac	floral, roses	ethyl isobutyl succinate/unk nown beta- phenyl ethanol
25.78	roses	floral, perfumy, roses	floral, lilac	floral, roses	beta-pheny! ethanol
25.92		nutty, floral	musty, floral		
26.27		nutty, sweet fruity			gamma- octalactone
26.50	floral, laurel		floral, roses, smokey	floral, roses	
27.30		sweet, ripe fruit, perfumy			
27.72		floral mixture, sweet ripe fruit			
28.59	dried fruit, cotton candy	ripe fruit, apricot melon, cotton candy	cotton candy, imitation cherry	fruity, cherry, cotton candy	
28.74	dried fruit artificial cherry	cotton candy	cotton candy, cherry	sweet fruit	
28.87	artificial cherry	fruittiness candy, sweet floral, ripe raspberry	cherry	fruity, cherry, blackberry	gamma- nonalactone
29.32		floral, fruity			
29.53	dried fruit	roasty, sweet, herbal floral			

Appendix 3.1 (cont.)

Time	Donalist	Donalist	Donalist	Donalist	Chaminal
Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
29.82	sweet, prune, grape	cotton candy, cherry	cotton candy, cherry berry	fruity, grape, blackberry, cherry	unknown gamma- nonalactone impurity
30.00	fruity, prune, dates	floral blossoms, ripe apricot prune	cherry berry	blackberry	
30.20	sweet citrus	floral, roses, ripe prune	fruity, raspberry grape	jammy, cotton candy	
30.97	sweet	floral	sweet		
31.15	floral	floral	sweet		
31.48		ripe fruit, floral, herbal		perfumy	
31.63	citrus floral, spicy	ripe fruit, floral, herbal	fruity, grape	chlorine	
31.84	soapy cardbord				
32.07	floral, gardenia	spicy, perfumy, sweet, herbal	heavy fruit, vegetative, herbal	floral, gardenia	
32.30	spicy, nutmeg, solvent	smokey, toasty, nutty			
32.76		floral, strange fruity musty, waxy			
32.91	floral, spicy, toasty	ripe fruit, smoked fruity			
33.18		fruity, smokey, musty	herbal, vegetative, grainy		

Appendix 3.1 (cont.)

Time	Panelist	Panelist	Panelist	Panelist	Chemical
(min)	1	2	3	4	name
33.26		floral, sweet, terpene, herbal		sweet fruit, grape bubble gum	
33.40	floral, soapy, grainy	sweet terpene	herbal, vegetative, smokey, grainy	fruity	
33.57	grape, herbal, toasty				ethyl palmitate
33.77		smokey, musty, sweet waxy		woody	
33.93		floral, sweet floral		woody	
34.21	herbal, solvent, pungent	sweet floral, smokey, sweet waxy	smokey, sulfury, stinky		
34.34	pungent			sausage, salami, smokey	
34.52	eucaliptus	pungent, black pepper, fruity backgroud	sweet, cooked fruit	meaty, smokey, herbal, black pepper	
34.81	spicy, nutmeg	herbal, floral, lavender lilac			
36.14	floral, herbal, spicy,toasty				
38.32	soapy, spicy				
39.70	herbal, floral, spicy, medicinal	herbal, faint floral, pungent			

#### Appendix 3.1 (Cont.)

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
40.23			fruity, floral, apple like		
41.12	floral, herbal, sweet	floral, sweet, musty	herbal, smokey	fruity	
41.29	sweet floral	sweet fruit, floral, perfumy	smokey, sweet floral		
41.48	ripe fruit, floral		heavy fruit, caramel, sweet floral		
41.88		ripe grape, sweet floral, herbal	sweet floral, herbal		

<sup>1</sup> Aroma descriptions used more often across replications per panelist.

<sup>2</sup> Compound names in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italic* with a ? were tentatively identified by GC-MS.

Appendix 3.2 Middle 1987 maturity Pinot noir, peaks detected by each panelist at least twice over the 4 replications.

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name <sup>2</sup>
07.35	floral, fruity, banana <sup>1</sup>	sweet fruity, floral	fruity, imitation fruity, cherry	sweet fruity, cherry, banana, grassy	ethyl hexanoate
09.70			floral		
10.45	sweet floral, fruity	musty, herbal, reduce compound	sweet floral, fruity	green sweet, sweet vegetative	ethyl lactate
10.66	ripe fruit, floral, spicy		floral, sweet	grainy	1-hexanol
10.96		pungent, reduce compound, musty			
11.61	spicy, herbal, ripe tomato				3-hexen-1- ol
12.38	floral, spicy, herbal				
12.69	egg, sulfuric	toasty, nutty, sweet pungent	•	grainy, cooked milk, oats meal	
12.78	reduce compound	reduce compound, kind of floral			
12.91			fruity, floral, smokey, carmelize, sulfuric		unknown ethyl octanoate impurity
13.18		floral, ripe fruit	floral, fruity, canned peach, pear		ethyl octanoate

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
13.46		sweet perfumy	smokey		
13.90	toasty, spicy, floral	floral	toasty, spoiled, brown sugar, floral		
14.03			spoiled, toasty, brown sugar, floral		
14.18	citrus floral, tutti fruity candy	sweet, floral, spicy, fruity	brown sugar, floral, heavy fruity, butter		
14.55	green, vegetative, grassy, herbal	herbaceous, herbal, spicy, fruity, smokey	green, vegetative, stemmy, smokey	vegetative, fresh vegetable	
14.75	toasty, herbal	smokey, to asty, reduce compound	cheese, stinky	rubbery	
15.06		musty, mousy, lousy, reduce compound			
15.52	floral, ripe fruit	floral, ripe fruit	heavy fruity, plum, sour		benz- aldehyde
16.05	caramel	waxy fruit, sweet floral	heavy fruity, prune plum		linalooi
16.20	toasty, linalool			sweet fruity, cherry berry peach	linalooi
16.29	floral	sweet, floral, ripe fruit, cherry peach	heavy sweet, maltol, sweet grainy	sweet fruity, cherry berry peach	unknown linalool impurity

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
16.94	floral, herbal, spicy	floral, fruity, waxy fruit	kind of burnt, smokey		2,3 bu- tanediol
17.27	spicy, toasty, vitamin B, pungent	floral, herbal, pungent			
17.80		toasty floral, pungent fruity, herbal, spicy, smokey			
18.73	to asty, vitamin B, cooked milk, butterscotch				gamma- butyrolacto ne/ethyl decanoate
20.20		floral, fruity, smokey, toasted, pun- gent			aipha- terpineoi
20.88	to asty, vitamin B, spicy, pun- gent, grassy	smokey, toasted nuts, pungent, vegetative	baked potato, earthy, toasty, grainy	meaty	methlonol
21.11	toasty, vitamin B	toasty	earthy, toasty, grainy	meaty	
21.30	floral, grassy, spicy, toasty			meaty, apple, sweet chemi- cal	
21.51				fruity, apple	
21.68		overripe fruit, floral, musty, pun- gent		dirty socks, cheese, fruity plastic	

Appendix 3.2 (cont.)

			·		· · · · · ·
Time	Panelist	Panelist	Panelist	Panelist	Chemical
(min)	1	2	3	4	name
21.81	dirty socks, toasty	meaty, floral, fruity musty	dirty socks, floral, stinky	fruity cheese, manure, vomit smell	
21.98	dirty socks floral		stinky	dirty socks, fruity	unknown methionol impurity
22.14	sweet fruity, prune apricot	floral, ripe apricot	dirty socks	dirty socks, fruity, sour apple	
22.46	baby vomit, floral	floral, herbal, musty	toasty, baked potato, smokey	malty, roasty	unknown methionol impurity
22.67	floral	waxy fruit, overripe fruit	fruity, still stinky	something	
22.94	floral	fruity	fruity stinky	manure	amyl-4- hydroxy butanoate?
23.36			citrus floral, fruity, apple, pear		beta-phenyl ethyl acetate
23.56	ripe fruit, apricot, prune	sweet fruity, floral	floral, fruity, apple	fruity, pineapple, apple, peach, tobacco	beta-phenyl ethyl acetate
23.74		floral, lilac, fruity, herbal			
24.09	herbal, toasty, grainy	herbal, black pepper, pungent			hexanolc acid
24.57	sweet, caramel, allspice, buttery		smokey, cheese, smokey meaty		
25.24	sweet floral				

Appendix 3.2 (cont.)

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
25.64				floral, roses, nutty, roasty	beta-phenyl ethanol
25.86	floral, roses, lilac	floral, ripe fruit, herbal	floral, lilac, fruity, vegetative	florai, roses	beta-phenyl ethanol
26.02		floral			
26.42	floral, lilac, grassy, spicy	floral, sweet ripe fruit, smokey	fruity, berry, raspberry		
26.64	spicy, pungent		floral, fruity, citrus floral, berry, raspberry, pungent		
28.38			sour aroma, kind of vegetative		
28.97	sweet spicy, herbal, caramel	floral, fruity, apricot, cherry, cotton candy	sweet, cotton candy, fruity		gamma- nonalactone
29.87	caramel, ripe fruit, prune	cotton candy, apricot, toasty, honey, nutmeg	cotton candy, fruity, banana		unknown gamma- nonalactone impurity
30.21	ripe fruit, molasses	herbal	sweet fruit		unknown gamma- nonalactone impurity
31.31	floral, soapy, ripe prune, caramel				

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
31.98	spicy, allspice, cinnamon, herbal, ripe prune	floral, ripe fruit, spicy, cinnamon, herbal, smokey	fruity	fruity, pineapple, citrus	
32.47	spicy, nutmeg, allspice				
33.44		floral, herbal, sweet, honey	fruity, winy, tropical heavy		
34.37			heavy, caramel, sulfuric, cheese		
34.66	spicy, black pepper, floral, herbal	black pepper, herbal, ripe fruit	fruity, apple, plum, heavy sweet		
35.80			fruity, floral		
36.14			cheese, sauerkraut		
38.20			burnt, sulfuric, pungent		
39.58			cheese, rancid, solvent like, fruity		
40.49	spicy, herbal, toasty		·		
41.51	vanilla, butterscotch	floral, fruity, candy	caramel, sweet		methyl vanillate

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
41.70	butterscotch				
41.85	spicy, herbal				ethyl vaniliate

- 1 Aroma descriptions used more often across replications per panelist.
- 2 Compound names in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italic* with a ? were tentatively identified by GC-MS.

Appendix 3.3 Late 1987 maturity Pinot noir, peaks detected by each panelist at least twice over the 4 replications.

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name <sup>2</sup>
07.29	floral, fruity <sup>1</sup>		floral, fruity, tropical fruity, pear, pineapple	fruity, banana	ethyl hexanoate
07.61			floral		1-pentanol
08.03			floral		·
08.43			fruity, floral		
08.82	toasty, grainy				
08.95	spicy, spicy fruity		floral, spicy floral		
09.56			floral		
10.24			floral		
10.54	floral, perfumy, lilac, gardenia	wild herbal, floral	floral, spicy floral		1-hexanol
10.89		sweet, waxy fruit	floral, spicy floral		
11.66		wheaty, smokey			
12.28		waxy skin fruit, toasty, smokey			
12.59	toasty, grainy, vitamin B, spicy	toasty, burnt, fruity floral pungent	floral, spicy floral	artificial orange, grainy, oats	

Appendix 3.3 (cont.)

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
12.80	toasty, grainy, vitamin B, spicy	toasty, burnt, fruity, floral, pungent	floral, fruity	artificial orange, grainy, oats	
12.95		sweet floral, ripe fruit			unknown ethyl octanoate impurity
13.05		perfumy, musty	floral, fruity	microlab, undesirable	
13.18		floral, perfumy	floral, citrus floral, floral fruity	peach	ethyl octanoate
13.31	ripe fruit, vitamin B	pungent, floral, musty	fruity, apple	peach	ethyl octanoate
13.57	hay, spicy, floral		floral, fruity, apple		
13.82	toasty, bread, grainy	toasty, smokey, reduce compound			
13.90		floral			
14.11	floral, fruity, cherry candy	floral, fruity, pungent	floral, fruity		
14.23		perfumy	floral, fruity, prune plum, toasted, grainy		
14.55	hay, grassy, cabbage, herbal			chemical, rubbery	

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
14.71		reduce compound, toasty, pungent, floral	toasty, grainy, rubbery, floral		
15.05		floral, stinky, rancid			
15.30	floral, ripe apricot prune				
15.72			floral, fruity		
15.95	spicy, floral, wheat, toasty	floral, ripe fruit, apple, prune			
16.11	linalool	floral	floral, nutty		iinalooi
16.21	wheat, vitamin B				
16.58			pungent, floral		4-methyl-2- pentanol?
16.98	kind of linalool, spicy, vitamin B, toasty	floral, rose lilac, herbal, fruity	floral, cooked fruit		2,3 butanediol
17.23	spicy, meaty, toasty		floral		
17.40		toasty, smokey			
17.90		floral, fruity, toasty, smokey			

Appendix 3.3 (cont.)

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
18.25	sweet floral, sweet fruity, to asty, nutmeg	sweet, buttery, floral	floral, fruity	pineapple	
18.39			floral, fruity	pineapple	
18.74	sulfuric, eggs, reduce compound	floral, perfumy, buttery, smokey			gamma- butyrolacto ne/ ethyl decanoate
19.49	spicy, toasty				
20.08		floral, ripe fruit, peach			
20.30		floral, ripe fig, toasty			alpha- terpineol
20.83		floral, ripe fruit, hay, toasty, smokey	meaty, buttery	grainy, malty	methlonol
21.07	citrus floral, hay, grassy, cabbage			apple, artificial orange, piney	
21.21		floral, hay, toasty	buttery, baked potato, cooked fruit	piney, resin, chemical	
21.38		sweet floral		apple, sour apple	
21.67	floral, dirty socks floral, sweet fruit	herbal	floral, cooked fruit, buttery	dirty socks	
21.84		ripe fruit, prune, hay			

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
22.15	ripe fruit, soapy	floral, roses, ripe fruit, herbal	floral, apple, buttery	dirty socks	
22.35	bouillon, floral, spicy	herbal, musty		dirty socks, musty	
22.57	floral, citrus	herbal, floral, ripe fruit, musty	earthy, woody, floral, sweet		
22.87	floral, ripe fruit		sweet, floral		
23.50	ripe fruit	floral, roses, ripe fruit	apple	berry, cherry, apple, jammy	beta-phenyl ethyl acetate
23.91	toasty, grainy	floral, herbal, toasty, smokey	floral, apple, buttery		hexanolc acid
24.50		spicy, herbal, floral, wild wheaty, ripe fruit			
24.80	allspice, nutmeg, herbal			caramel, brown sugar	benzyl alcohol
24.97		floral, spicy			
25.16	floral, ripe fruit				
25.82	roses, lilac	floral, herbal, fruity, spicy	roses, lilac	roses, carnation	beta-phenyl ethanol
26.43	floral, roses, lilac	floral, estery, herbal, spicy	floral, apple	floral, roses	

Appendix 3.3

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
26.58	roses, ripe fruit	floral	floral	floral, roses	
27.40	ripe fruit, spicy, hay, toasty	sweet pungent, ripe fruit			
28.13	floral, ripe fruit	floral	berry, floral		
28.30				spicy, fresh mushrooms	
28.68	ripe fruit, artificial orange	ripe peach apricot, cherry, raspberry, blackberry		sweet, blackberry	
29.26		cherry, fig, sweet			
29.83	ripe fruit. anise	peach, cherry, cotton candy		caramel, jammy, berry	unknown gamma- nonalactone impurity
30.08	ripe fruit, nutmeg, allspice	floral, cherry peach	sweet, floral	sweet fruit	unknown gamma- nonalactone impurity
30.30	sweet, heavy fruit, dates, grapes, anise	floral, roses	sweet, heavy fruity		
31.08	floral, fruity	sweet floral, fruity, herbal			
31.25	tropical fruit, prune, spicy	spicy, prune peach			
31.60	sweet fruit, prune	apple, prune, cherry, peach	peach berry	spicy	

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
31.86	minty, anise floral, spicy, green	fruity floral, herbal, black pepper		spicy, prune	
31.99				spicy, prune	
32.29	spicy, allspice, nutmeg				
33.08	blackberry, raspberry, spicy		fruity floral, apricot		
33.21	soapy, floral, roses, lilac	floral	soapy, floral		
33.40	soapy	floral, sweet	soapy, floral		
33.53	tea	sweet fruit, perfumy			ethyl palmitate
33.84	spicy, minty, hay	pungent, sweet	fruity, floral, apricot	spicy, herbal, petroleum	
34.31	floral, fruity, spicy, hay				
34.54	floral, spicy, hay	black pepper, wheaty, floral	heavy floral, winy, scotch, fruity	spicy, black pepper, green, herbal	
34.88	floral spicy, dry fruit	spicy, black pepper, pungent floral	floral, grandma perfume, plum	spicy, pepper	
35.40	floral, fruity	sweet	floral	woody, woodburn	
35.50			floral perfumy	woody, woodburn	

Appendix 3.3

Time (min)	Panelist 1	Panelist 2	Panelist 3	Panelist 4	Chemical name
35.69	floral spicy, heavy aroma	pungent, sulfury			octanoic acid
35.86	petroleum, solvent, chemical, spicy, waxy		something		
36.10			floral, cheese, meaty, vegetative, sulfury		
36.85			something		
37.30			floral, soapy		
39.34		floral, lilac, perfumy	floral		
39.62	spicy, floral, toasty				
39.98	spicy	floral, spicy, smokey, toasty	something, floral		
40.23	sweet, ripe fruit		fruity, plum, grape, cheese, smokey		
40.46	sweet, ripe fruit	floral	smokey	fermented apple	
40.55	apricot, prune, spicy				
41.38	vanilla, sweet fruit, sweet floral,	ripe fruit, floral, melon, lavender	sweet, caramel, cooked eggs, smokey		

Time	Panelist	Panelist	Panelist	Panelist	Chemical
(min)	1	2	3	4	name
41.84	floral, vanilla, buttery, sweet	floral, raspberry, herbal, sweet	fruity		ethyl vaniliate

- 1 Aroma descriptions used more often across replications per panelist.
- 2 Compound names in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italic* with a ? were tentatively identified by GC-MS.

Appendix 4.1 Early 1988 maturity Pinot noir, peaks detected by each panelist at least twice over the 4 replications.

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name <sup>2</sup>
6.45	spicy, toasty, spicy <sup>1</sup>	musty, vegetative	fusel oil	3-methyl- 1-butanol
7.28	floral	floral, sweet herbal	floral, fruity	ethyl hexanoate
10.36		pungent, kind of fruity		ethyl lactate
10.96	spicy, herbal, tobacco			
11.54	dried fruit, blackberry jam		floral, sweet	3hexen-1- ol
13.04		herbal, fruity		
13.42		smokey, vegetative		
14.00	floral, citrus, piney	floral, spicy, musty	rubbery, chemical	
14.07			chemical, butterscotch	
14.28		floral		
14.66		pungent, herbal, musty, smokey	rubbery, tobacco	ben- zaldehyde
14.90	dried fruit, sweet, toasty	ripe fruit, herbal	chemical, burnt match	unknown lilalool impurity
15.23	herbal spicy, toasty	herbal, perfume		
15.97	spicy, floral	ripe peach		linatoot
16.17	floral, linalool	floral, sweet	linalool, roses	linalool
16.63			smokey, floral	

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
19.47	herbal, floral			
20.68	cabbage, floral	floral, herbal vegetative	vegetative, herbal	methionol
20.91	dried fruit, floral	floral, fruity, sweet	floral, caramel	
21.03			tobacco, burnt match	
21.20	citrus, floral, dried fruit	spicy, fruity, herbal	floral, grassy	
21.44			floral, chemical	
21.87	cabbage, grassy hay			
22.00	cabbage, floral, herbal	vegetative, herbal	,	unknown methionol impurity
22.16	cabbage, herbal, medicine		chemical, tobacco	
22.32	cabbage, herbal, medicine	floral spicy, pungent herbal	caramel, chemical	
22.67	cabbage herbal	herbal, perfume		
22.78		toasty spicy, herbal, vegetative		
22.86	cabbage, anise dried fruit			
23.14	currant, anise, floral			
23.32	dried fruit, anise, floral	ripe fruit	floral, caramel	beta-phenyi ethyi acetate

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
23.44	currant		tobacco	beta-phenyl ethyl acetate
23.51	dried fruit		roses, tobacco, caramel	
23.70		floral, earthy, grassy		
23.90	floral, dried fruit, earthy	herbal, fruity, grassy	smokey, rubbery	hexanolc acid
24.15	toasty, anise earthy	fruity, vegetative		ethyl dode- canoate
25.13	dried fruit, perfume		caramel, smokey rubbery	
25.55	roses		roses	ethyl isobutyl succinate/ unknown beta-phenyl ethanol
25.64	roses	pungent, herbal vegetative, sweet, grassy	roses	beta-phenyl ethanol
25.91	roses			
26.04		floral	fiorai	
26.45	jasmine, roses		floral, vegetative	
27.16	roses, dried fruit, caramel			
27.27	nutmeg, fruity, caramel			
28.66	caramel, dried fruit	cotton candy cherry, dusty earthy		

Appendix 4.1 (cont.)

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
28.78	caramel, dried fruit			gamma- nonalactone
29.00		floral, fruity	caramel, fruity	
29.45	caramel, cotton candy			
29.74	caramel, cotton candy, dried fruit	ripe fruit, sweet candy		unknown gamma- nonalactone impurity
29.90	dried fruit	ripe fruit, sweet candy		
30.13	caramel, cotton candy, prunes	fruity, herbal spicy		unknown gamma- nonalactone impurity
30.55	prunes			
31.15	dried fruit		caramel, fruity	
31.68	dried fruit, cotton candy			
31.75	dried fruit	fruity		
31.95	floral, lilac	spicy, fruity		
32.85		earthy, musty, dusty		
33.20	floral	ripe fruit, candy, floral	methyl anthralilate	
33.35	soapy, spicy herbal musty	ripe fruit		
33.70		floral, fruity	coconut, smokey	ethyl palmitate
34.28	herbal, spicy			

- 1 aroma descriptions used more often across replications per panelist.
- 2 Compound names in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italic* with a ? were tentatively identified by GC-MS.

Appendix 4.2. Middle 1988 maturity Pinot noir, peaks detected by each panelist at least twice over the 4 replications.

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name <sup>2</sup>
6.45	pungent, herbal, spicy <sup>1</sup>	floral, smokey, nutty	chemical, fusel oil	3-methyl- 1-butanol
7.32	tropical fruit	sweet musty, perfume	floral	ethyl hexanoate
8.86	herbal, spicy, sweet, floral			
10.00	sweet, dried fruit		floral	
10.53	cinnamon, floral			1-hexanol
10.56	floral		floral	1-hexanol
13.05		floral		
14.04	piney	herbal, pungent, floral, nutty	rubbery, smokey, acrid	
14.06			butterscotch, caramel	
14.47	spicy	floral, pungent, smokey		
14.63			rubbery, caramel	
14.73	winey, toasty	herbal-smokey	rubbery,	
14.81			caramel, nutmeg	
14.95		herbal-smokey		
15.15			rubbery	
15.22		sweet, berry		3-hydroxy- butyric acid
15.60	citrus			ben- zaldehyde

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
16.17	linalool		linalool	linalool
16.67			floral	
18.15		perfume, smokey, vegetative		
18.89	floral			
19.11		musty, floral		isovaleric acid
20.68	cabbage		butterscotch, soapy, burnt match	methionol
20.75			burnt match, methyl anthranilate	methionol
20.85			butterscotch, floral	
20.96	grassy, dried fruit	fruity, floral.	roses, green, butterscotch	
21.29	cabbage, prunes,	floral		
21.68	cabbage	fragrance, herbal, pungent	floral, rubbery	
22.11	cabbage, dried fruit, prunes		caramel, linalool like	
22.24	cabbage, dried fruit, prunes		floral	
22.45			floral	
22.64	dried fruit	•		
22.76	cabbage, dried fruit, herbal, hay			
23.10	dried fruit, herbal, hay			

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
23.43	dried fruit	roses, ripe apple, herbal	rubbery, butterscotch, floral,tobacco	beta- phenyl- ethyl acetate
23.49	dried fruit	floral, ripe apple, herbal	burnt match, rubbery, tobacco	beta-phenyl ethyl acetate
23.66		herbal, minty, pungent		
23.90	herbal, nutty	herbal, minty, pungent		hexanolc acld
24.20		sweet		ethyl dode- canoate
24.41	floral, grape	sweet, fruity,	caramel, butterscotch	n-butyl acetamide
24.70		fruity, minty, sweet, spicy		
24.98			floral	
25.08			chemical, floral	
25.15			butterscotch, soapy	
25.25			floral	
25.59	roses	roses	roses	beta-phenyl ethanol
25.70	roses	roses	roses	beta-phenyl ethanol
26.27	sweet, dried fruit			
26.47			caramel, floral, tobacco	
27.26	chemical, cardboard			

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
27.47			floral	
28.36			tobacco, rubbery	
28.65	sweet, caramel, solvent, dates, fig	fig, cotton candy, floral	rubbery, caramel	
28.80	sweet, caramel, dates, fig	fig, cotton candy	tobacco, chemical	gamma- nonalactone
29.90	prunes, caramel	floral, fruity, caramel, herbal		unknown gamma- nonalactone impurity
30.11	prunes	floral, fruity, caramel, herbal		unknown gamma- nonalactone impurity
30.35	prunes, dried fruit	herbal, raspberry, cherry		
30.78		sweet, earthy		
31.10	meaty, soapy		sweet, linalool like, floral, vegetative	
31.27	prunes		floral	
31.54	grassy, cinnamon	floral, spicy, perfume	roses, caramel	
31.83	prune, floral		floral	
32.08	spicy, soapy	nutmeg, cinnamon, waxy		
32.87		floral, spicy, earthy		
33.30	cotton candy, floral, soapy, spicy, coconut	floral, fruity, vegetative, earthy	floral	
33.53		floral, minty, herbal		

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
33.75	coconut, spicy	herbal, spicy, sweet, musty	coconut, acrid	
34.13		herbal, floral, pepper	coconut, rubbery	
34.24	floral, nutmeg			
34.61	soapy, dill			
34.96	dried fruit			
35.23	stinky, caramel			
36.88	spicy			
38.33			floral, burnt	
39.41			chemical, floral	
39.83	tropical fruit, spicy			

- 1 aroma descriptions used more often across replications per panelist.
- 2 Compound names in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italic* with a ? were tentatively identified by GC-MS.

Appendix 4.3 Late 1988 maturity Pinot noir, peaks detected by each panelist at least twice over the 4 replications.

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name <sup>2</sup>
6.45	dried fruit, nutty toasty <sup>1</sup>		fusel oil, chemical	3 m et h y - 1 - butanol
7.10		floral pungent, slightly perfume	roses, chemical slighgtly floral	
7.28	tropical fruit, banana	floral pungent, nutty pungent	floral	ethyl hexanoate
8.07			rubbery, floral	
8.51	floral, citrus, lactic			
8.99	floral, herbal, spicy			
10.03			butterscotch, floral	
12.68		floral		
13.01		floral, pungent perfume		
13.44	nutmeg, piney, floral	floral, smokey	rubbery, chemical	
13.58			roses, chemical slightly floral	
14.00	linalool citrus, floral, spicy, piney, herbal, grassy	floral pungent	rubbery, caramel	
14.10	citrus, linalool, piney, spicy	floral, herbal	butterscotch, rubbery	
14.61		pungent, vegetative, herbal, smokey	burnt rubber	
14.68	toasty, spicy, caramel, nutty	herbal, smokey, pungent, toasty	rubbery	

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
14.85		toasty smokey, toasty pungent, herbal pungent	rubbery	
14.96	floral, nutty	herbal smokey, toasty, smokey pungent	rubbery, burnt rubber	
15.06	citrus, piney, spicy, floral		rubbery	
15.20		toasty, pungent, herbal		3-hydroxy butyric acid
15.54	spicy, floral, sweet		floral, smokey	benz- aldehyde
16.20	linalool	floral, sweet, herbal		unknown linalool impurity
16.79			floral	
19.72			rubbery, roses	dlethyl succinate
20.29	spicy, herbal, floral, cabbage caramel			alpha- terpineol
20.70	cabbage, dill		floral, vegetative, cabbage, butterscotch	methlonol
20.93		nutty, floral fruity, sweet floral	floral, sweet prunes	
21.02	floral, dried fruit, spicy	floral, sweet herbal, floral, smokey	vegetative, butterscotch	
21.13		ripe fruit, herbal smokey		

Appendix 4.3

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
21.26		ripe fruit	smokey, slightly floral, butterscotch	
21.92	caramel, dried fruit, floral, spicy, butterscotch		floral, like linalool	unknown methionol impurity
21.99			floral, butterscotch	
22.16	sweet floral, dried fruit		tobacco, floral	
22.34	spicy, herbal tobacco, cabbage	herbal	tobacco, chemical, floral	
22.96	cabbage, herbal, floral, spicy, toasty	floral, pungent, musty		amyl-4- hydroxy butanoate?
23.23	dried fruit, prunes, herbal			
23.37	dried fruit, minty	floral, fruity, spicy	butterscotch, cabbage	
23.44	dried fruit, minty	floral, fruity, spicy	tobacco, butterscotch	beta-phenyl ethyl acetate
23.50	dried fruit, minty	floral, fruity, spicy	tobacco, caramel	beta-phenyi ethyl acetate
23.64	dried fruit, minty	fruity, sweet, herbal, hay, smokey		
23.93	dried fruit, burnt match	ripe fruit, herbal, spicy, grassy, vegetative,	tobacco, smokey, burnt rubber	hexanoic acid
24.11	dried fruit, burnt match	smokey, spicy, herbs, ripe fruit	vegetative, chemical	hexanolc acid

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
24.35	prunes, nutty, nutmeg, medicinal	floral, ripe fruit herbal		n-butyl acetamide
24.53		sweet floral candy spicy, ripe fruit, herbal		
24.81	dried fruit, nutmeg	ripe fruit, herbal, spicy		benzyl alcohol
24.85		ripe fruit		benzyl alcohol
25.16	roses		roses, vegetative roses, burnt match	
25.26	roses	floral, fruity, herbal, spicy		
25.52	roses	roses, pungent, herbal	roses	ethyl isobutyl succinate/ unknown beta-phenyl ethanol impurity
25.66	roses	roses pungent, roses herbal		
25.75	roses	roses pungent, roses herbal	floral	beta-phenyl ethanol
25.95	sweet, roses	herbal floral, herbal fruity		
26.25	dried fruit	roses, fruity, herbal fruity		gamma-oc- talactone
26.52	dried fruit, spicy			
26.73		herbal, hay , earthy		
26.84	minty floral, sweet, nutmeg allspice			

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
27.13	sweet, eugenol			
27.25	tobacco, floral, dried fruit	floral herbal, floral fruity, stinky musty		
27.52	minty, dried fruit, herbal	floral, herbal		
27.96		spicy, ripe fruit, perfume		
28.15	sweet, dried fruit			
28.30		spicy ripe fruit, smokey musty		
28.47	dried fruit, candy, butterscotch			
28.70	dried fruit, cotton candy, butterscotch	cotton candy cherry	floral, butterscotch	gamma- nonalactone
28.84		cotton candy, apricot		gamma- nonalactone
28.96		cotton candy cherry apricot, floral herbal cinnamon, spicy cookies		gamma- nonalactone
29.06		apricot prunes cherry, cinnamon roses		
29.17		floral herbal, cherry prunes, floral cinnamon		
29.31	dried fruit	smokey spicy, cinnamon roses		•
29.38		spicy, smokey, floral, fruity		

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
29.70	dried fruit, cotton candy, nutmeg, herbal	figs, floral, herbal, cinnamon		unknown gamma- nonalactone impurity
29.80	dried fruit, cotton candy, nutmeg, herbal	figs , floral, herbal, cinnamon		unknown gamma- nonalactone impurity
29.88	dried fruit butterscotch, coconut, caramel	spicy, ripe fruit, smokey		
30.00	dried fruit, butterscotch, coconut, caramel	spicy, ripe fruit		
30.07		spicy, ripe fruit		
30.18	prunes, caramel nutmeg	spicy, floral, ripe fruit		
30.36	spicy, prunes currant			
30.51		spicy, herbal, floral		
30.63	spicy, dried fruit			
30.79	dried fruit, spicy, caramel, coconut			
30.99		floral, earthy smokey, fruity, spicy		
31.25	dried fruit, floral, minty, grassy, herbal	smokey, earthy, floral, pungent, musty, sl fruity	butterscotch, caramel, fusel oil	
31.54	dried fruit, nutmeg caramel	spicy, herbal, figs peach, floral, ripe fruit		

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
31.78	dried fruit, caramel	cinnamon, herbal		
32.04	floral, nutmeg, currant	spicy , herbal, smokey, floral, perfume		
32.27	burnt match, soapy, floral herbal	smokey, spicy, floral		
32.40		smokey, spicy		
32.53	nutmeg, nutty gardenia	smokey, spicy, musty		
32.77		spicy , herbal,floral	rubbery, coconut	
32.90	currant, sweet floral, grassy, vegetative nutty	spicy, herbal, floral		
33.26	spicy, herbal, dried fruit, floral	cinnamon, floral, fruity		
33.42		spicy, fruity, ripe fruit, floral		
33.53	candy caramel, toasty, herbal nutmeg	cinnamon, hay, floral		
33.72	coconut, gardenia, herbal nutmeg	spicy , herbal, pungent, ripe fruit	cabbage, sweet, coconut	ethyl palmitate
33.86	coconut, gardenia, herbal, nutmeg	spicy, herbal, pungent, ripe fruit	cabbage, coconut	
34.00		cinnamon, nutmeg, perfume, ripe fruit		
34.25	nutmeg, caramel, floral, herbal	cinnamon nutmeg, perfume, ripe fruit		
34.42		ripe fruit, perfume		

Time	Panelist	Panelist	Panelist	Chemical
(min)	1	2	5	name
34.57	vegetative, perfume	spicy, ripe fruit, herbal, vegetative		
34.85	dried fruit, anise, minty, spicy cabbage, perfume			
35.27	burnt match, cabbage, floral	ripe fruit, floral		
35.70	cabbage		floral	octanolc acid
36.33	dried fruit, minty			
36.48	soapy, sauerkraut, dried fruit			
36.89	coconut, butterscotch, sweet, dried fruit			
37.20	perfume, dried fruit			
37.36		floral, herbal, fruity		
37.58	dried fruit			
38.38	caramel, cotton candy			
38.58	sweet, cotton candy			
38.76			rubbery, floral	
39.09	chemical, cabbage, cotton candy			
39.23	sweet, cotton candy, caramei			

#### Appendix 4.3

Time (min)	Panelist 1	Panelist 2	Panelist 5	Chemical name
39.36	currant, cotton candy caramel			
39.60	dried fruit, spicy, cotton candy			
39.95	vanilla			

- 1 aroma descriptions used more often across replications per panelist.
- 2 Compound names in **bold** are those whose chemical name and aroma were confirmed. Compound names in plain letters were impurities found while running the pure compound. Compound names in *italics* were identified by GC-MS but could not be confirmed. Compound names in *italic* with a ? were tentatively identified by GC-MS.