RUM AROMA DESCRIPTIVE ANALYSIS

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ABSTRACT

Rum contains a large number of compounds that contribute to the complex aroma, some of which present in a very small amount. This fact represents a problem for the use of chemical analysis as a way to ascertain the quality of rum and to determine differences in rums products. Descriptive sensory analysis techniques can be useful in such circumstance, not only because they are easier to achieve, but because they give valuable information on the actual perceived aroma of the products. The first part of this work consisted of the development of lexicon for describing rums. A group of 5 semi-expert judges evaluated a variety of 15 commercial rums from different origins, raw materials and processing conditions, and created consensually a list of 33 terms with description useful for describing the aroma, flavor, and taste of rums. The second part of the work was focused on the creation of a method used to describe the aroma of different rum products, to discriminate among different rums, and to relate the perceived aroma of the samples prepared from different processing protocols. Using a group of 12 trained panelists, a modified descriptive analysis technique for the evaluation of rum aroma, and the adequate descriptors and references that were generated, it was possible to describe and discriminate rum samples. The results obtained from the evaluation of the 9 different commercial rums and one experimental sample can be used to relate the perceived aroma of the different rum samples to their processing protocol. Among the many possible applications of this study is the use as a tool for quality control, new product development, and brand identification. The information obtained from the description of the products can also be a useful tool for marketing purposes.

CHAPTER 1. INTRODUCTION

Rum is defined as "any alcoholic distillate from the fermented juice of cane syrup, sugarcane molasses or other sugarcane derivative, distilled at less than 190º proof (whether or not such proof is further reduced prior to bottling to no less than 80º proof) in such a manner that the distillate possesses the taste, aroma and characteristics generally attributed to rum and includes mixtures solely of such distillates" (27 CFR 5:21).

Rum production is centered in the West Indies. Traditional rum making countries are Jamaica, Martinique, Puerto Rico, Cuba, Barbados, Trinidad, Haiti, Guadeloupe, the Virgin Islands, the Dominican Republic, and Guyana. Other rum producing countries include Brazil, Peru, Mexico and parts of Asia and Africa (Lehtonen and Suomalainen, 1975).

Rum can be used for direct consumption in a pure form or as mixed drinks (Ruter, 1975). It can also be used as a flavoring agent in chocolates, liquors and in tobacco (cigars) and bakery products. Rum contains numerous compounds that contribute to the aroma. Some aroma compounds present in rum are higher alcohols, fatty acids, fatty acid esters, carbonyl compounds, phenolic compounds and lactone. Esters are the most important group of compounds in the aroma of rums. Many types of rums can be produced from using different processing protocols. For instance, heavy aromatic rums are generally produced in long-duration fermentations, and are distilled in pot-stills. The presence of certain bacteria also increases the amount of congeners in rum yielding a distillate, which upon aging yields a heavy aroma. Light rums are generally produced in short-duration fermentations and distilled in a series of continuous columns. Rums can

also be classified by their ester number, which is the concentration of esters (mg) in 100 ml of ethanol (100%) (Lehtonen and Suomailanen, 1977).

The types of raw material used, the method of clarification, fermentation conditions (rum yeast), distillation conditions, and aging conditions will affect the aroma of the final rum product. The objectives of the present study were:

1. To develop a lexicon to describe the aroma, flavor, and taste of rum;
2. To develop a methodology suitable for the aroma descriptive analysis of rum;
3. To describe the aroma of different rum products;
4. To relate the perceived rum aroma to the processing conditions of the products.

CHAPTER 2. LITERATURE REVIEW

2.1 Sugarcane

Sugarcane is a hybrid of several species of the genus *Saccharum*. The word *Saccharum* seems to originate form the Sanskrit word Sarkara. *Saccharum spontaneum* describes the wild cane varieties and S. officinarum L the developed varieties used for commercial sugar production. *Saccharum officinarum L.* has long been considered the "noble" cane, as for centuries it has been the major source of commercial sugarcane. It originates in New Guinea, from where it spread due to migration (Blackburn, 1984). Different breeding and selection techniques have been applied to sugarcane to improve sugar yield. Selections have been made based upon cane yield, sugar content, fiber content, habit, ratooning, disease resistance, insect resistance, and other characteristics such as flowering, spines, brittleness, and herbicide tolerance (Walker and Simmonds, 1984).

Sugarcane is a perennial tropical grass that produces unbranched stalks. It consists of root, stalk (with nodes), flower, and leaves (Jones and Scard, 1921). Its stalks can be 3-4 m tall and 5 cm in diameter. Sugar is extracted from these stalks of cane, and it is contained in the fibro-vascular bundles. The composition of sugarcane (*Saccharum officinarum L.*) varies depending on such factors as variety, soil condition, climate, and use of fertilizers (Ruter, 1975). The general composition of sugarcane is listed in Table 2.1.

2.1.1 Raw Sugar Process

The initial steps in sugar manufacture are: (1) handling of cane including harvesting, weighing, dispatching, and washing (if required); (2) cane preparation and

milling; (3) clarification of the juice; (4) evaporation and concentration; (5) sugar crystallization; (6) drying, storage, and packaging, and (8) steam and power production from bagasse.

Table 2.1 General Composition of Sugarcane

Millable cane:	
Water	73-76%
solids	24-27%
• fiber	11-16%
• soluble solids	11-16%
Juice composition:	
Sugars	75-92%
• Sucrose	70-88%
• Glucose	2-4%
• Fructose	2-4%
Salts	3.0-4.5%
▪ Inorganic acids	1.5-4.5%
▪ Organic acids	1-3.0%
o Carboxylic acids	1.1-3.0%
o Amino acids	0.5-2.5%
Other organic nonsugars	
▪ Protein	0.5-0.6%
▪ Starch	0.001-0. 1%
▪ Gums	0.30-0.60%
▪ Waxes, fats, phosphatides	0.05-0.15%
Other	3.0-5.0%

Source: Chen and Chu (1993).

2.1.1.1 Harvesting

Sugarcane cannot be stored without deterioration, thus manufacture is done immediately after harvesting. Sugarcane is commonly harvested during the cooler months in each hemisphere and before harvesting cane is commonly burnt to remove unwanted leaves (trash). Cane can be cut by hand using knifes, cutlass or machete. The advantage of cutting cane by hand is a reduction in trash entering sugar mills. Mechanical cut of

cane reduces labor, therefore is a common practice in developed countries. There are different machines employed to cut cane. Whole stalk harvesters cut the stalks at the bottom and the top. The cane stalk is thickest at the bottom and thus contains the most sugar, hence one wants to cut it close to the soil. The topping of the cane is to remove top leaves (trash). The tops contain the most reducing sugars. Chop harvesters gather the stalks as the whole stalk method, but cut them into pieces of 6 to 9 inches. Extraction fans blow off most of the leaves. Different methods and machines have been developed for specific regions according to their needs (such as climate) (Blackburn, 1984).

Table 2.2 Harvesting and Transportation Methods for Sugarcane

Harvest	Loading into Transport Vehicles	Transport System
Manual		
1. Green cane	Manual	Bullock-drawn carts
2. Burnt cane	Mechanical	Tractor –drawn trailers
		Trucks
		Rail trolleys
Mechanical		
1. Whole sticks	Mechanical	Tractor-drawn trailers
2. Chopped billets		Trucks
		Rail trolleys

Source: Hunsigi (1993).

After harvesting, sugarcane is transported to the sugar factory by various means (Table 2.2). The delivery vehicles are commonly weighed by electronically operated scales. A common system is usually sampling all trucks or railcars. These samples are analyzed for sucrose, fiber and dirt. Payment formulas include the net weight of the delivered cane, sugar content, fiber and trash. The most common methods for unloading cane are (1) unload of cane using an overhead moving platform, (2) tilting of trucks and

trailers using hydraulic platforms, and (3) chain net unloaders. Chopper harvested cane is usually transported into bin-type containers and unloaded by tipper or dumper trucks.

2.1.1.2 Cane Preparation and Milling

Harvesting of burned cane allows for harvesting and yields of cane with much less trash (dead leaves, weeds, etc). Cane should always be processed as soon as possible after harvesting to avoid quality deterioration. Cane may be further washed with water to remove clay and foreign materials. The high amounts of water used make it necessary to reuse the water. Washing in Louisiana cut chop cane typically causes 10-16 lbs of sucrose losses per ton. Cane preparation is required for good sugar extraction. This is commonly accomplished using rotary knifes and/or shredders, and crushers. The goal is to achieve a very high open crushers number in order to obtain better sugar extraction.

2.1.1.3 Sugar Extraction

Sugar extraction is achieved after crushing the mat of sugarcane in a mill tandem of multiples stages. Addition of water during milling improves the efficiency of the sugar extraction. The extraction efficiency of modern mills is about 92-96%. Higher extractions will result in the extraction of more non-sugar components that will have to be removed later on.

2.1.1.4. Juice Treatment and Clarification

The purpose of clarification is to remove the soluble and insoluble impurities from the cane juice. Lime and heat are commonly used for this purpose. Lime is used to neutralize the acidity in the juice by forming insoluble lime salts, mainly calcium phosphate. The heating above boiling temperature is used to will be coagulate albumina and other fats, waxes, and gums. The precipitate that is formed will entrap other particles

suspended in the juice. Once the precipitate settles and forms a mud, the juice is then filtered, and the remaining mud can be compressed and used as fertilizers.

The expressed juice obtained after milling contains many impurities that have to be removed. The impurities include floating solids, colloidal matter, phenols, coloring compounds, starch, glucose, fructose, minerals, and amino acids. Then the pH of the juice is raised from about 5.6 to 7 to prevent sugar inversion. The steps involved in the juice treatment are (1) initial heating to 70-75°C, (2) addition of $Ca(OH)_2$, (3) addition of dissolved phosphates, (4) bubbling of sulfur dioxide or calcium dioxide through juice, (5) second-stage heating to >100°C, (6) addition of polyelectrolites to aid coagulation of precipitates, (7) sedimentation and decantation of clear juice, and (8) sediment filtration to separate solids and reprocessing of juice. At the end of this process clear juice will be produced, it is a transparent, light golden liquid.

2.1.1.5 Evaporation

The clarified juice obtained has a soluble solid content (brix) of 13-15%, and it needs to be concentrated to a syrup with about 65 % solids. This can be accomplished by using a multiple effect evaporator. The number of effects used are normally four or five. The larger the number of effects, the higher the steam economy. The lesser the number of effects, the higher the evaporation capacity of each. The first body typically maintains a steam pressure of 1-1.5 kg cm^{-2} and a temperature of 115-125°C, while the last body has a vacuum of 650 mm Hg and a temperature of 55°C (Hunsigi, 1993). A feed forward process is used so the sugar solution, upon concentration boils at a progressively lower temperature. This avoids caramelization and other degradation reactions. Evaporators can be of (1) short-tube construction (Roberts type), with tubes of 2000 mm long.

Evaporators used in the raw sugar industry are typically of the short-tube type. The Robert or standard evaporator has short tubes and a large central down take. The external steam heats the juice in tubes, as a result its density decreases and it starts rising. Out of the tube flows liquid plus vapor. The latter moves overhead to the steam chest of the following vessel, which operates at a lower pressure. The juice returns through the central down take to the bottom of the evaporator. A portion, at the correct brix, flows forward the next evaporator where the process repeats itself. This way, one pound of steam can evaporate theoretically four pounds of water (3.5 practically). This technology was invented by Robert Rillieux around 1846 in New Orleans.

Long-tube rising or falling film evaporators are also available, just like rising or falling film plate evaporators. These have higher overall heat transfer coefficients and evaporate more water per square foot per hour than Robert type evaporator. Additionally, the residence time is significantly reduced, and thus there are color formation and degradation reactions.

2.1.1.6 Pan Boiling and Sugar Crystallization

Pan boiling and sugar crystallization is an important operation in a sugar factory. The syrup is concentrated to a super saturated stage where sugar crystallizes. This is done in single-effect vacuum-pans (CC) and at reduced boiling temperatures. The first pure or A^- solution is usually seeded with small sugar nuclei, while less pure solutions or B^- and C^- strikes (massecuite) are usually seeded with magma (pure juice + C^- crystals). The A^- strikes are centrifuged and separated into A^-sugar and A^- molasses. The latter is used to make a B^- strike, which is allowed to undergo additional crystallization in open horizontal crystallizers at lower temperatures, or in continuous vertical crystallizers. Next it is a

centrifugation step yielding B sugar and B molasses. The latter is used to make a C strike, which upon crystallization and centrifugation yields C sugar and C or blackstrap molasses. A and B sugar form the raw sugar, which is refined into white sugar at refineries. The final molasses is the chief raw material for rum production. During crystallization, it is important to obtain crystals of uniform size, shape and color. The presence of colloidal or coloring impurities trapped in the crystals should be minimized. Crystallization is carried-out in several stages because it is impossible to crystallize all sugar in syrup in one stage.

Commercial sugar is a disaccharide (sucrose) of two monosaccharides fructose and glucose. The empirical formula for sugar is $C_{12}H_{22}O_{11}$. It is soluble in water and ethanol.

2.1.1.7 Bagasse Use

As mentioned before, the steam and electricity demands of a sugar factory are generally covered by the use of bagasse as fuel. Bagasse has a calorific value of 2,200 kcal/kg on wet basis. Factories use baggase to produce high-pressure steam.

2.1.2 Sugar Refining

Raw sugar is priced according to the pol value, moisture content, ash, grain size, dextran content and color. It is normally delivered to the refiners in bulk in truck loads, or by rail, or by barge. The refining process of raw sugar is divided into several steps.

The first step is called affination. This process consists on the removal of a molasses film that is occluded in the raw sugar crystals. The molasses film contains most of the impurities in the sugar. The separation is carried out by using undersaturated syrup

at 75 Brix. The mixture is centrifuged and then washed with cold water. Then the washed raw sugar is mixed with water and melted.

The next step is the defecation or clarification of washed raw liquor in which all insoluble and colloidal matters are removed. The two types of clarification systems used in refineries are phosphatation and carbonation. Phosphatation is accomplished with the use of phosphoric acid and lime. It produces calcium phosphate floc difficult to filtrate. The common way of separating calcium phosphate is by using air flotation. Carbonation process consists of the addition of lime and bubbling carbon dioxide to produce a calcium carbonate precipitate in the washed raw melt liquor. The calcium carbonate crystals entrap and adsorb other impurities. Clarification can be aided by the addition of quaternary ammonium compounds.

The removal of impurities that imparts color in the raw sugar is called decolorization. Such impurities include phenolic compounds, melanoidins, caramels, and invert degradation products. Decolorization is accomplished with the use of adsorbents in which the aromatic structures with extensive conjugated double bond systems are adsorbed through hydrophobic bonding. The most commonly used adsorbents are carbonaceous adsorbents and synthetic resins. Decolorization can also be done with the use of cationic color precipitants.

The following step is the concentration of dark sweet-waters, wash waters and other materials with low sugar concentration not suitable for melting wash raw. Evaporation of multiple effects is commonly used for the concentration of such materials. After evaporation, further concentration is done in boiling pans. The next step is to centrifuge and wash white sugar.

The final step for white sugar production is the drying and conditioning. The obtained white sugar requires drying to remove moisture. The most commonly used dryer is the granulator. It consists of two drums in series, one for drying and the other one for cooling.

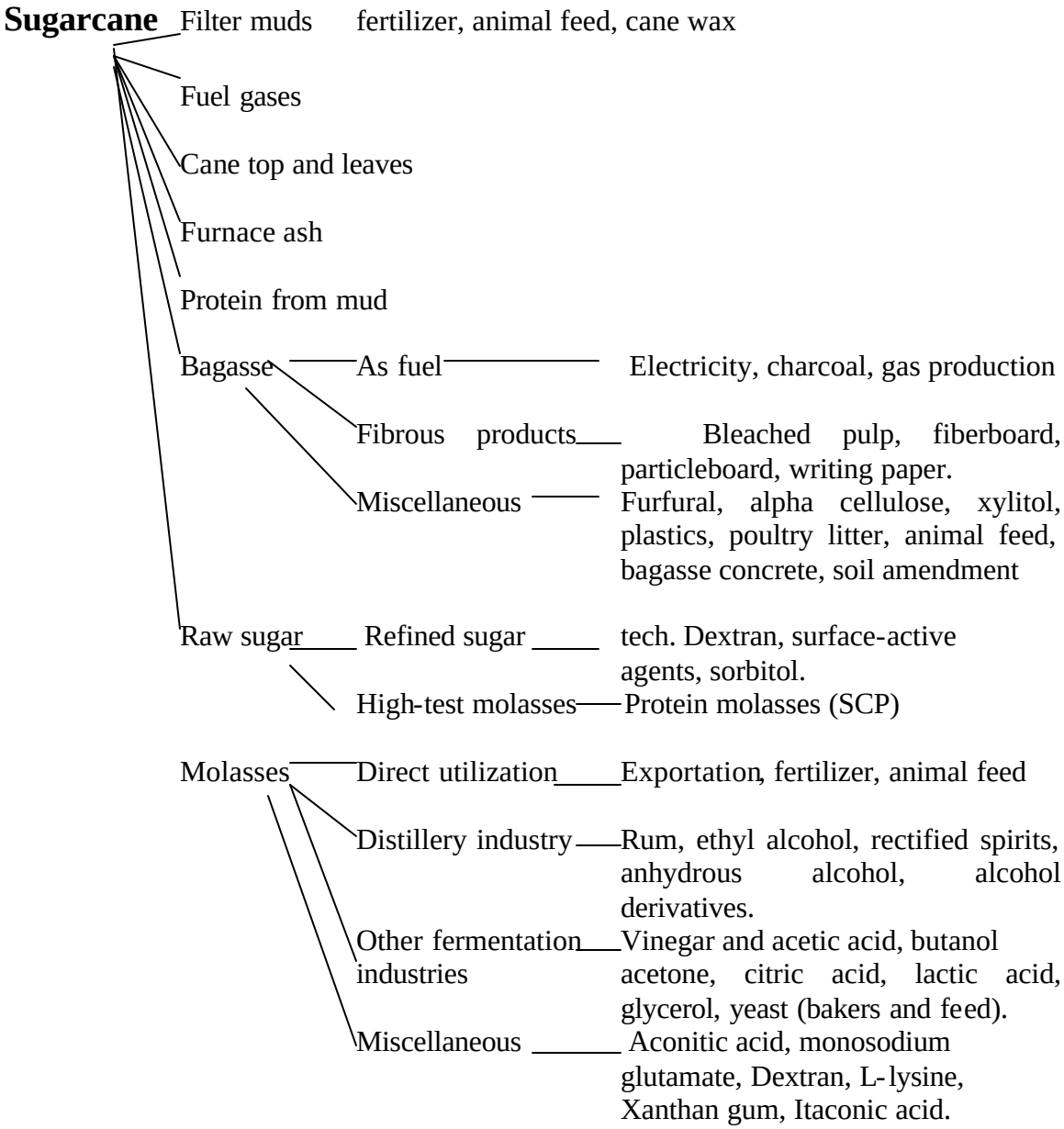


Figure 2.1 Sugarcane Parts and Its By-products. Sources: Manohar Rao (1997); and Hunsigi (1993).

2.1.3 By-products of Sugarcane Industry

A number of value-added products can be obtained from sugarcane in addition to sugar. Figure 2.1 shows some of the possible by-products from the sugarcane industry. The sugar industry can diversify to produce such product as glycerol, inositol, lactic acid, lysine, alcohol, baker's yeast, proteins, etc.

2.1.4 Blackstrap Molasses

Molasses is the main byproduct of the raw sugar process. It is a heavy viscous liquid from which no further sugar can be obtained by simple means. Per ton of cane, up to 8 gallons of molasses may be produced. Molasses contains about 50 to 60% total sugars, of which 16-17% is sucrose, the rest being glucose and fructose. The general composition of molasses is listed in Table 2.3

Table 2.3 Typical Composition of Louisiana's Cane Molasses

Dry matter	80%
Total sugar	52-58%
Sucrose	16-17%
Reducing sugars	34-38%
Total nitrogen	2-3%
Organic acids	3%
Gums (insoluble in alcohol)	≤2%
pH value	5-6
Ash	12-18%

Source: ASI molasses survey (2000).

The chemical composition basically determines the quality of molasses. In general good molasses should have a fresh and sweet aroma, and a pH value of 5-6. Molasses should be stored cooled as otherwise self-ignition becomes possible. Certain bacteria (exothermic) cause degradation reactions, which affect the quality of molasses.

Molasses can be used for animal feed, for the production of alcohol, rum, compressed yeast, and other organic compounds such as citric acid, acetone, butanol, lactic acid, itaconic acid, kojic acid, aconite acid and aconitates (Chen and Chu, 1993).

2.2 Rum

2.2.1 Rum History

The word rum showd up for the first time in 1612. The personal physician of the Duke of Meckenburry (Germany) had to travel to Hamburg. Arriving late he found the gates to the city closed. Some vendors were selling products, which he checked out. “A Dutch woman was selling rum, which tasted quite good”, he recorded in his diary. The rum was made from molasses imported from the West Indies. When the Dutch colonized a portion of Brazil (the Recife area) they produced rum. When the Portuguese conquered this area, the Dutch left and settled on different Caribbean Islands taking their “know-how” of making rum with them.

In 1650 Father du Terre, a French Priest visited the West Indies and gave the first description of a cane spirit. By 1722 Father Labat also described the spirit and said that the fermentation was spontaneous and scum cakes were added to molasses during fermentation which yielded so acid a wash that the house was called “vinagreirie”. He also stated that distillation was accomplished by using pot-stills and several distillations were done to produce a spirit before selling it. By this time the cane spirit was called ‘rum’, was produced in Barbados, Jamaica, Virgin Islands, and Santo Domingo, and was exported mainly to the United Kingdom (Clutton, 1974).

The origin of the word rum is uncertain, some possible sources were (1) derivation from the Latin word saccharum (sugar), (2) a corruption of the term ‘rombustion’

meaning ‘a strong liquid’, (3) from the Devonshire word ‘rumbullion’ brought to the West Indies by British sellers, and (4) from the Spanish ‘ron’, because it is likely that Spanish were distilling in the West Indies before the British arrived.

Even though rum was largely produced since the early 1600’s, its fist legal definition did not come until 1909 by Sir Algernon Aspinall; ‘Rum is a spirit distilled direct from sugarcane products, in sugar growing countries’. This definition was accepted by the Royal Commission on Whiskey and other Potable Spirits (Clutton, 1974). The definition of rum according to the labeling regulations of the U.S. Internal Revenue service (27 CFR 5:21) is “any alcoholic distillate from fermented juice of sugarcane syrup, sugarcane molasses or other sugarcane products, distilled at less than 190° proof (whether or not such proof is further reduced prior to boiling to no less than 80° proof) in such manner that the distillate possesses the taste, aroma and characteristics generally attributed to rum and includes mixtures solely of such distillates.”

2.2.2 Rum Manufacture

Rums can be produced from molasses, syrup and/or cane juice, with molasses being the chief raw materials. The type of rum produced depends largely on the type of raw materials, the treatment of the raw material, and the fermentation yeast used. Cane juice is obtained after pressing finely ground sugarcane. For the production of rum the juice can be used as it is obtained by pressing or after some concentration (Garnier-Larroche and Cottrell, 1975). Rum produced in continuous stills, whether from cane juice or molasses, typically posses a light aroma. Rum produced from long-duration fermentations, batch distilled and aged in oak wood for several years, possess a much heavier aroma. Molasses also contains a number of compounds that impact the aroma of

rum. Lehtonen and Suomalainen (1977) listed the compounds identified in sugarcane molasses, the list includes aliphatic and aromatic esters, aldehydes, alcohols, furan derivatives, nucleic acids, sugar alcohols, amino acids and other organic acids (Table 2.4).

Table 2.4 Aromatic Compounds Present in Molasses

Acids	**Esters**	**Nitrogenous compounds** (cont.)
Formic acid	Ethyl formate	Proline
Acetic acid	Ethyl acetate	Serine
Propionic acid	Isoamyl acetate	Threonine
n-Burtyric acid	Methyl benzoate	Tryptophan
n-Valeric acid	Ethyl benzoate	Tyrosine
Aconic acid	Benzyl formate	Valine
Benzoic acid	Phenethyl acetate	
Citric acid		
Glycolic acid	**Ethers**	**Phenolic compounds**
Lactic acid	Anisole	Phenol
Malic acid	Phenetole	m-Cresol
Mesaconic acid	Benzyl ethyl ether	Guaiacol
Succinic acid	Furfuryl ethyl ether	Salicylic acid
Tricarballyc acid		Resorcinol
	Nitrogenous compounds	Vainillic acid
Alcohols	Acetyl pyrrole	Syringic acid
Ethanol	Alanine	*p*-coumaric acid
Propanol	*b*-Alanine	Vainillin
2-Methyl-1-propanol	*g*- Aminobutyric acid	
2-Methyl-2-butanol	Asparagine	**Sugar alcohols**
3-Methyl-1-butanol	Aspartic acid	D-Arabitol
Furfuryl alcohol	Cystine	D-Erythriol
Melissyl alcoho1	Glucosamine	Myo-Inositol
Pheylethyl lcohol	Glutamic acid	D-Mannitol
	Glutamine	
Carbonyl compounds	Glycine	**Miscellaneous**
Acetaldehyde	Histidine	2-Acetylfuran
Furfural	Homoserine	4-Methyl-2-propyl furan
5-Methylfurfural	Isoleucine	
Acetyl benzaldehyde	Leucine	
o-Methoxybenzaldehyde	Lysine	
Furfuryl methyl Ketone	Methionine	
d- Valerolactone	Phenylalanine	
(-)-2-Decano-5-lactone	Pipecolic acid	

Source: Lehtonen and Suomalainen (1977)

Compounds found in molasses as hydroxymethylfurfural, and some fatty acids can adversely affect fermentation. They may retard or inhibit yeast fermentation depending upon its concentration and other factors such as pH (Lehtonen and Suomalainen, 1977). However, molasses is a rich and relatively inexpensive substrate for fermentation.

2.2.2.1 Pre-treatments

2.2.2.1.1 Clarification

The initial pre-treatment is clarification by precipitation to remove the colloidal matter especially calcium sulfate, which is the main component responsible for the blockage of the stripping columns during distillation. Clarification also ensures that fermentation and distillations processes are carried out at high efficiency. It can be accomplished by physical and chemical effects; by heating, acidification, and cooling of molasses. Molasses clarification consisted of diluting to 40 brix, heating to 92ºC, and reducing the pH with sulfuric acid to promote precipitation of insoluble materials. After clarification, insoluble impurities are removed by decantation and or centrifugation/desludging. Clarification can also be achieved by a special distillation method. Molasses is clarified to improve the fermentable sugar to non-sugar ratio to approximately 6.5.

2.2.2.1.2 Dilution with Water and Nutrient Addition

Yeasts require a number of nutrients to grow. They should be provided with a carbon source, a nitrogen source, minerals, and growth factors. Molasses possess a number of nutrients required by yeasts; however, further additions of nutrients are required by yeast to have an optimum fermentation. Sucrose, and further glucose and

fructose sugars are the main carbon source in molasses yeast fermentation. Molasses also provide a number of amino acids as organic nitrogen containing compounds. The presence of amino acids for rum production is important because they are used by yeasts to produce a number of flavor active and flavor precursor compounds. However, molasses has a low content of inorganic nitrogenous compounds, therefore ammonium phosphate, ammonium sulfate, or urea are commonly added to molasses (Bluhm, 1983).

The minerals required by yeast for growth and fermentation are phosphorus, potassium, sodium, calcium, sulphur, and magnesium. Cane molasses supply the requirement of those minerals for yeast growth. The phosphorus utilized by yeast will be mainly from phosphate. Sulphur taken by yeast is mainly from inorganic sulphate, but can be replaced partially or totally by other inorganic or organic sulphur sources (Rose and Harrison, 1971). Magnesium is the most common enzyme activator, particularly important is the activation of phosphate transferases and some decarboxylases (Bowen, 1966). Even though calcium is not required for yeast growth, it stimulates growth and fermentation (Rose and Harrison, 1971). Additionally cane molasses contain good amount of minerals.

Table 2.5 Composition of the Mash After Molasses Pre-treatments

Density (^{o}Brix)	15-17
pH value	5.5-5.8
Total sugar content (g/100ml)	>15
Nitrogen content (g/100ml)	2.0-2.5
Titratable acids (ml 0.1 N alkali/10 ml)	1.5-2.0
Phosphoric acid content (mg/100 ml)	600-750

Other additions made to the mash prior to fermentation may be ‘dunder’ and ‘skimmings’. ‘Dunder’ is the slop from a previous distillation, and ‘skimmings’ is the foam resulted after boiling of cane juice (Ruter, 1975) The typical composition of the mash prior to fermentation is shown in Table 2.5 Pasteurization is another pretreatment applied to molasses to destroy unwanted microorganisms.

2.2.2.2 Fermentation Conditions

Fermentation conditions will largely affect the quality of the rum and ethanol produced. Fermentation is normally carried out at 32-35ºC. Lower temperatures will result in low fermentation kinetics, and higher temperatures may destroy yeast. As an exception to this, the temperature in which rum production by wild fermentations is done can be as high as 37ºC (Lehtonen and Suomalainen, 1977). The pH value influences the fermentation and the metabolic products formed. A lower pH will yield relatively higher amounts of alcohol, while the production of a good aroma is achieved between pH 5.5-5.8. Low pH inhibits bacteria, and therefore when using a combination of yeast and bacteria for fermentation, the pH should not be much lower than 5 (Lehtonen and Suomalainen, 1977). Additionally, sulfuric acid will catalyze reactions within the fermentation such as esterification (Kampen, 1975).

Fermentation time is dependent on the quantities of yeast used in the inoculum, the quantities of bacteria present, the type of yeast used, the fermentation temperature, sugar concentration, and presence of nutrients. The normal fermentation time is 48-72 h but can be longer. It has been reported than fermentation can take as long as 14 days; however, only part of that time is necessary to complete fermentation, the rest of the time is used for the formation of aroma (Ruter, 1975). The reaction that takes place during

fermentation to convert sugars to ethanol and CO_2 is exothermic. Because of this effect, cooling is normally necessary to prevent the temperature from exceeding 35ºC.

During fermentation, compounds such as sugars can move into the yeast from the medium, and the metabolites move from the yeast to the medium, the movements to and from the yeast cell are regulated by the membrane. Some characteristics of the compounds such as size, branching, and lipophilic nature determine penetration through the membrane. Short chain compounds can pass easily through the membrane (Lehtonen, 1983).

2.2.2.2.1 Yeast

The selection of the type of yeast used will affect the quality of the rum obtained. For the production of heavy aromatic rums *Schizosaccharomyces* strains, fission-type top yeasts are best suited, while the quick-fermenting budding-type *Saccharomyces* strains are better for the production of light rums (Patrau, 1969). The best yeasts for rum production are isolated from molasses (Lehtonen and Suomalainen, 1977). Fermentation can be infected by the action of microflora present in the raw materials, equipment or air. That is why the fermenters used for light rums are of the closed type. Some 34 species of wild yeast have been isolated from molasses and cane juice (Table 2.6).

A successful natural-microflora fermentation occurs when aereophilic alcohol intolerant yeasts are active at the start of the fermentation, but gradually become dominated by fermentative yeast such as *Saccharomyces*.

Most modern day distilleries use proprietary cultured yeasts, some may use recycled yeast from previous fermentations. The yeast usually used is a strain of *S.*

cerevisiae (Lehtonen and Suomalainen, 1977; Watson, 1985), although some rum distilleries use *Schizosaccharomyces pombe* for the production of heavy bodied spirits.

Table 2.6 Yeasts Isolated from Molasses or Cane Juice

Candida guillermondii	*Saccharomyces cerevisiae*
Candida intermedia var. ethanophila	*Saccharomyces chevalieri*
Candida krusei	*Saccharomyces delbrucki*
Candida mycoderma	*Saccharomyces marxianus*
Candida parapsilosis var. intermedia	*Saccharomyces microellipsoides*
Candida pseudotropicalis	*Saccharomyces rosei*
Candida saccharum n. sp.	*Saccharomyces rouxii*
Candida tropicalis	*Saccharomycodes ludgwigii*
Endomyces magnusii	*Schizosaccharomyces pombe*
Hanseula anomala	*Torulopsis candida*
Kloeckera apiculata	*Torulopsis glabatira*
Pichia fermentan	*Torulopsis glabrata*
Pichia membranaefaciens	*Torulopsis globosa*
Saccharomyces aceti	*Torulopsis saccharum n. sp.*
Saccharomyces acidifaciens	*Torulopsis stellata*
Saccharomyces carlsbergensis	*Torulopsis stellata va. Cambresieri*
Saccharomyces carlsbergensis var. alcalophila n. Var.	

Source: Lehtonen and Suomalainen (1977).

Many distillers purchase commercial yeast, which is produced similarly to baker's yeast on molasses (Harrison and Graham, 1970; Rosen, 1989). Some indicators of the fermentative ability of yeast are the trehalose and glycogen content (Pollock and Holmstrom, 1951; Gadd *et al*, 1987; Pearce *et al*, 1989; Quain and Tubb, 1982). Yeast manufacturers must design cultivation programs that lead to longer fermentative activity in yeasts. This causes the fermentative action to occur not instantaneously (Watson, 1993).

Some distillers propagate their own yeasts using stirred-tank reactors with a substrate such as molasses. They generally operate in simple batch rather than a fed-batch mode. The production of distiller's yeast in closed aseptic systems to ensure inoculation will be free of a pure yeast culture. Inoculation can also be made with a previously finished yeast mash, therefore reducing laboratory propagation (Peppler, 1979). Table 2.7 shows the factors that have to be considered for the selection of distilling yeast.

Table 2.7 Desirable Properties of Distilling Yeast

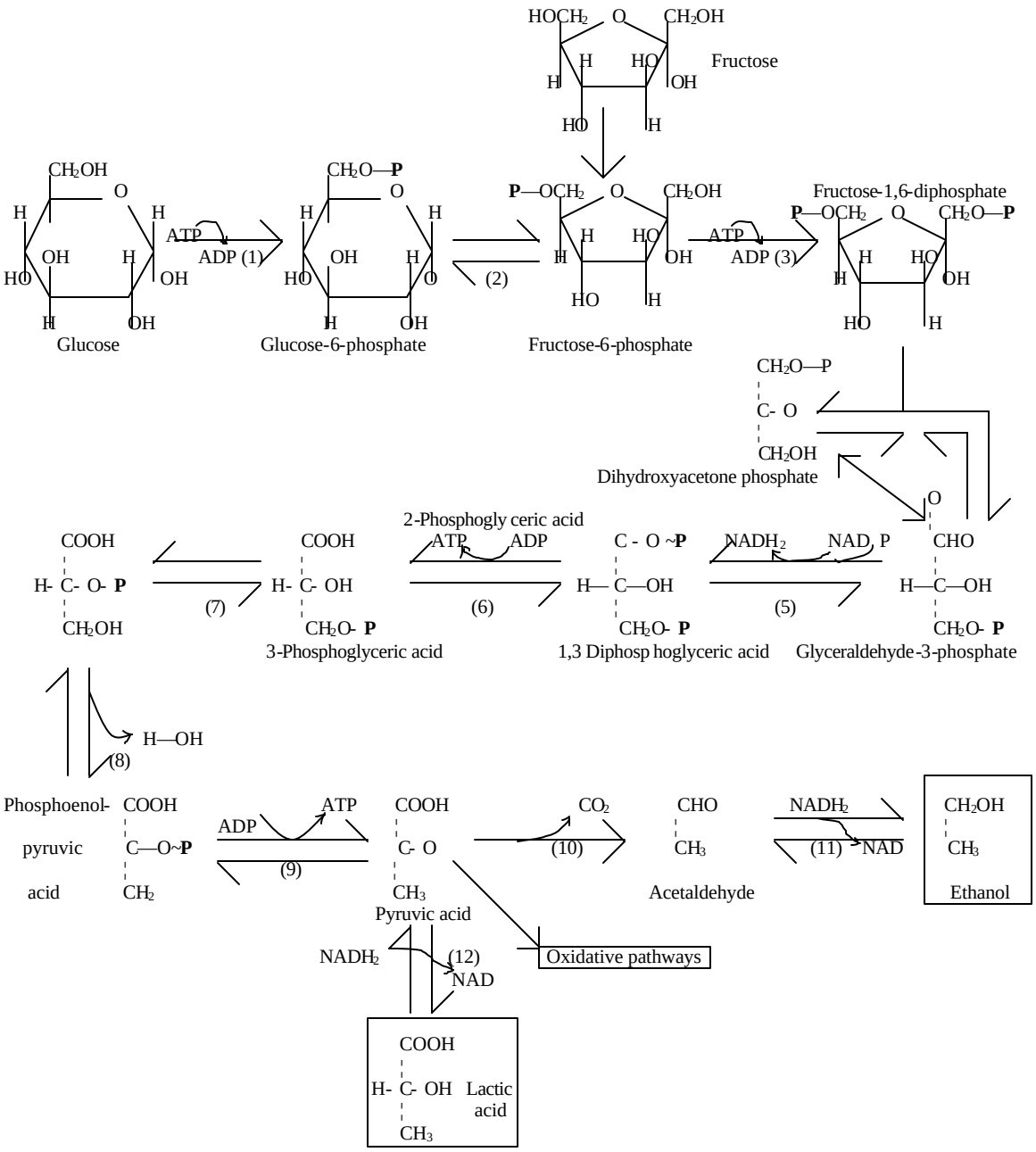
1. Optimal conversion of carbohydrates to ethanol, e.g. by reduction of conversion to biomass, glycerol, and other side products; increasing range of utilizable substrates.
2. Rapid fermentation rate, generally at higher temperatures, thereby minimizing fermentation, fermentation plant required for a particular distillation capacity.
3. High tolerance to osmotic pressure, ethanol, low pH, high temperature. All contribute to achieve points 1 and 2.
4. Production of correct aroma compounds or their precursors.
5. High biomass yield and retention of high activity from propagation prior to fermentation.
6. Flocculation, in cases of use of particular fermentation systems.

Source: Johnston (1990).

Another option for the production of distilled beverages is to use mixed cultures. By this means, a richer flavor can be obtained, with the down side of having more problems controlling fermentations.

2.2.2.2.2 Fermentation Rate and Efficiency

It is important to maximize the ethanol production while maintaining the desired product quality. The yeast used should be able to ferment the sugar at a fast rate. Most commercial strains of *S. cerevisiae* can utilize a number of sugars. Glucose, fructose galactose, maltose and maltotriose are transported across the cell membrane (Barnett, 1976), while sucrose is extracellularly converted to glucose plus fructose by β-D-fructofuranosidase (invertase). Since sugarcane molasses has a high sucrose content, good yeasts for rum production should have high invertase activity (Watson, 1985). In anaerobic conditions glucose and fructose are transported across the plasmalemma by constitutive facilitated-diffusion systems, galactose by inducible facilitated diffusion, and maltose and, in some strains, maltotetrose by inducible permease systems (Barnett, 1976) Hexose monosaccharides are then metabolized by the Emdben-Mayerhof-Parnas pathway (Figure 2.2) to pyruvate (Gancedo and Serrano, 1989). The pyruvate not required for yeast growth is then converted to acetaldehyde and ethanol, regenerating NAD+ from NADH, and thus maintaining the redox balance in the cell (Van Dijken and Scheffers, 1986). Other products of the EMP pathway are succinate and glycerol, and possibly lactic acid. These products remain mainly in the stillage after distillation. Glycerol is formed in order to regenerate NAD from the NADH formed in yeast growth reactions. It is believed that succinate is formed from oxaloacetate via malate and fumarate as a substrate for biosynthetic reactions in the tricarboxylic acid cycle (Gancedo and Serrano, 1989).



The enzymes involved in the reactions are: (1), glucokinase; (2), phosphoehexoisomerase; (3), phosphofructokinase; (4), aldolase; (5), glyceraldehydes-3-phosphate dehydrogenase; (6), phosphoglycerokinase; (7), phosphoglyceromutase; (8), enolase; (9), pyruvic kinase; (10), pyruvic carboxylase; (11) alcohol dehydrogenase; and (12), lactic dehydrogenase.

Figure 2.2 Embden-Mayerhof-Parnas Pathway of Glycolysis.

The overall fermentation effect is:

$$\text{Glucose} + 2\ \text{ADP} \rightarrow 2\ \text{Ethanol} + 2\ CO_2 + 2\text{ATP}$$

Theoretical fermentation yield from 1 g glucose is 0.51g ethanol and 0.49 g CO_2. In practice 10% of the glucose is utilized for biomass production, thus the ethanol and CO_2 yield is 90% of that theoretical value. ATP formed will be used to supply cell energy requirements.

Saccharomyces cerevisiae preferentially metabolizes glucose to fructose. Many factors affect yeast fermentation such as infection, available sugars, and genetic complement of the strain. Other environmental factors and the composition of the substrate also affect the fermentation rate. Lack of essential nutrients, especially available nitrogen, will limit cell growth, and thus slow fermentations. Low pH values, low temperatures, high contents of sulfur dioxide, minerals and certain fatty acids, and the presence of agricultural fungicides will inhibit fermentation rates (Jones *et al.*, 1986; Maiorella *et al*., 1984; Rose, 1987; Cantarelli, 1989; Laure and Lafon-Lafourcade, 1989). Yeast inoculation as well as physiological health of the yeast will also affect the initial fermentation rate (Watson, 1984 and 1985). Aerobically grown yeast tends to ferment more rapidly and survive unfavorable conditions better than anaerobically grown yeasts because of its higher reserves of cell constituents and ATP, and higher contents of sterols and unsaturated fatty-acyl residues (Rose, 1977; Korhola *et al.*, 1989; Rosen, 1989). Some of the techniques used to improve ethanol yield are vaccum, rapid and continuous

fermentation or immobilization of yeast, also low cost supplements can be used in conventional fermentation to improve the alcohol yield.

2.2.2.2.3 Environment

In the initial stage of the fermentation, with high sugar concentrations, the yeast is subjected to high osmotic pressure, while at the end of the fermentation, the yeast is subjected to high alcohol concentrations as well as elevated temperatures due to exothermic reactions. Molasses fermentations generally have high osmotic pressures, under these conditions, strains of *Schiz. pombe* are more tolerant than *S. cerevisiae* (Haraldson and Bjorling, 1981). The normal response of yeast to osmotic pressure is to produce glycerol, to work as osmoregulator (Meikle e*t al.*, 1988). The glycerol produced is leached out of the cell once the osmotic pressure drops (Panchal and Stuart, 1980).

In the presence of high ethanolic concentrations, especially under high temperatures, yeast cells may die, and can disrupt fermentation causing inefficient sugar conversion to ethanol (Casey and Ingledew, 1986; Pamment, 1989; van Uden, 1989). High alcohol concentrations also affect the fermentation by inhibiting and denaturing enzymes in yeast (Scopes, 1989). Yeast strains rich in sterols and double-unsaturated fatty-acyl residues in the plasma membrane have shown better resistance to the toxic effects of alcohol (Thomas and Rose, 1979; Casey and Ingledew, 1986).

2.2.2.2.4 Bacteria

In addition to the yeast used for fermentation, the mash may introduce significant levels of bacteria (Sharp and Watson, 1979). They may influence the fermentation characteristics and contribute to the sensory profiles of the final spirit.

The presence of bacteria during fermentation normally comes from raw materials (cane juice, molasses or process water). The bacteria will have metabolic activity parallel to yeast fermentation and ethanol production, affecting the kinetics and biochemistry as well as the sensory properties of rums (Fahrasmane and Ganou-Parfait, 1998). The presence of bacteria can be controlled by using thermal treatment, acidification, antibiotics, and by the fermentation yeast. The degree of infection during fermentation is normally kept well under 10% [(live yeast cells/live bacteria) X 100). Some of the compounds produced by bacteria parallel to alcohol fermentation by yeast is chiefly lactic acid, acrolein. They will not only affect the sensory characteristics, but they may add undesirable toxicity (Fahrasmane and Ganou-Parfait, 1998).

Some bacteria strains contribute favorably to the fermentation. The presence of *Clostridium saccharobutyricum* in the fermentation is found to accelerate fermentation due to an increased production of fatty acids and further corresponding esters (Arroyo, 1945). *Clostridium butyricucum* and C*lostridium kluyvery* can produce unsaturated fatty acids as metabolites (Scheuerbrant and Bloch, 1962). The ideal ratio of bacteria to yeast to produce the best rum yield is 1:5 (Arroyo, 1945).

2.2.2.3 Centrifugation

After fermentation the mash is allowed to settle for a few hours so that the yeast cells and other solids can precipitate. Centrifugation helps remove all the insoluble solids. Centrifugation is normally done with desludger (disk/nossle) centrifuges. Centrifugation also removes yeast cells that otherwise, at the prevailing high bottom temperatures in the stripping column (215 +°F), will decompose and may cause off-odors in the distillate.

2.2.2.4 Distillation

Separation of compounds can be achieved by creating various zones that differ in the conditions such as temperature, pressure, composition, and or phase state. Each compound reacts to different conditions, and therefore each compound establishes a different concentration at different zones. Distillation is a method of separation that uses the vapor and liquid phases at the same temperature and pressure for all the coexisting zones. These various zones can be created using packing or plates or trays that will put the two phases vapor and liquid in contact. These trays are stacked one over another in a column. After the flow material is introduced, the liquid, because of gravity will flow down, while the vapor flow up the column. The liquid is then reheated to vaporize after reaching the bottom of the column. The liquid that remained in the bottom is then removed. In the case of rum distillation, the remaining liquid after distillation is called "stillage" or "dunder". The vapor reaching the top of the column is cooled and condensed; it will then be removed as distillate, while some may go back as reflux.

During distillation, the lighter components (light boiling) tend to concentrate in the vapor phase, while the heavier (high-boiling) components will tend to remain in the liquid phase. Separation depends on the relative volatility of the components, the number of trays, and the ratio of the liquid-phase flow rate to vapor-phase flow rate (Perry *et al.*, 1984). In the cases where feed is introduced at a single point, the upper section of the column will be the rectifying sections, and the lower part the stripping section.

The distillation step can have a major effect on the aroma composition of the final rum product. Distillation can affect the composition by formation of new compounds such as products of pyrolysis. Another way distillation affects the aroma is by altering the

proportion of the compounds formed during fermentation. Also the presence of yeast during distillation can alter the ester content of the alcoholic beverage (Lehtonen and Jounela-Eriksson, 1983).

Rum can be distilled in batch (pot-still) and continuous distillation systems. The selection of the distillation procedure will depend on the type of product wanted (Ruter, 1975). Rum obtained by pot-still distillation is generally heavier and is mainly produced in English and French speaking areas of the West Indies. Continuous distillation produces lighter rum and is largely used for Cuban and Puerto Rican rums (Lehtonen and Suomalainen, 1977).

2.2.2.4.1 Pot-still Distillation

The simplest form of batch distillation consists of a single pot, a condenser, and one or more receiving tanks. In this case, no trays or packing are provided. The feed is charged into the pot and the mash is then brought to boiling. The vapors are condensed and collected in a receiver. Another variation of batch distillation contains pot stills in series. The fermented mash is pumped into the first vessel and distilled. The condensed distillate or "low wine" is collected in a second vessel. The "low wines" are subjected to distillation to obtain "high wines". "High wines" are rectified in a short rectifying column. "Dunder" or stillage remaining from distillation and fermented by the action of bacteria can be added back to the fermented mash prior to distillation.

2.2.2.4.2 Continuous Distillation

Continuous distillation normally consists of three columns (Figure 2.3). The purpose of the first column or 'stripping column' is to remove all the alcohol from the fermented mash. The overhead product is about 40-60 % alcohol by volume. At the head

of the second column or "purifying column" all the volatiles or low-boiling impurities are taken off, while the water/alcohol/congeners mixture purified will be taken from the bottom to the rectifying column. In the third or "rectifying column", the alcohol is concentrated and the remaining heavy compounds or fusel oils are removed. The upper trays of the column will contain the rum distillate while the above trays will contain fusel oils. The feed tray will contain (~70%) ethanol. This procedure can be modified by feeding water at the head of the purifying column and make it work as an extractive column (Unger and Cofey, 1975).

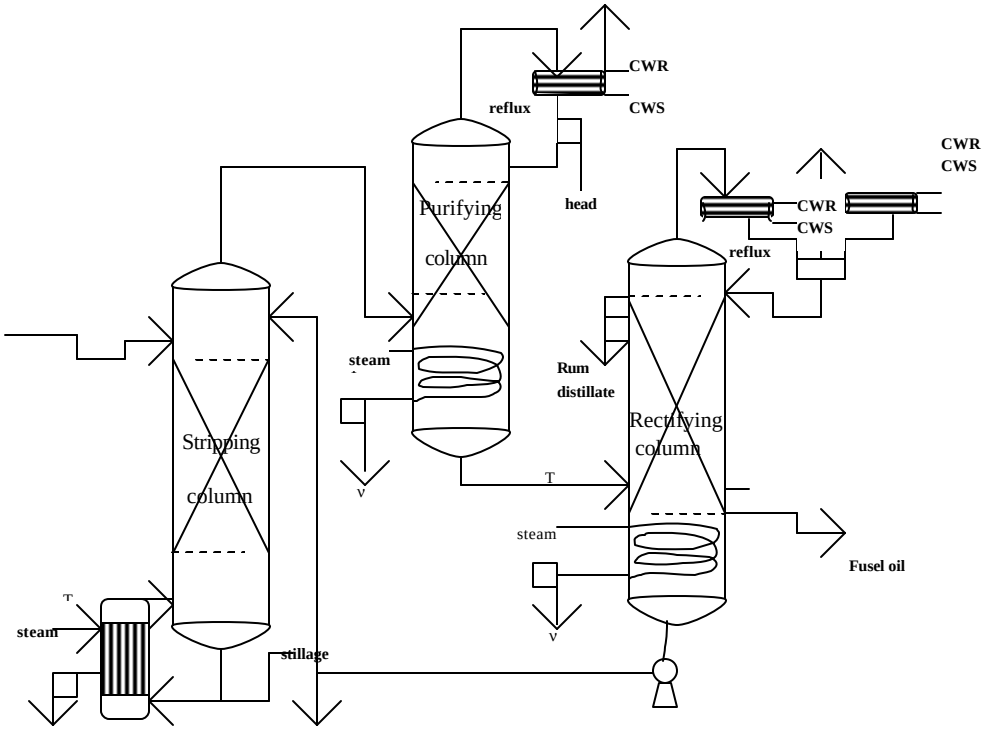


Figure 2.3 Example of a 3-Distillation Column System for Rum.

The distinctive taste from most flavored spirits comes from the acquired flavor rather than the base spirit, which is commonly a neutral spirit (Watson, 1985). Early

continuous still consisted of two columns. Modern stills contain six inter-connected columns. Continuous still can distill some 3,000 to 4,000 gallons per hour. The six columns of modern continuous still are: (1) The purifying column, which is a column filled with some 16 trays with hooded rectangular bubble-caps. The heated mash is fed at the top of the column, steam is introduced with a sparge from the bottom. As the mash flows down the trays, CO_2, other gases and aldehydes are removed for further condensation. The purified mash is removed from the bottom to the top plate of the second column; (2) The analyzing column is the second, and consists of 16-20 plates of grater diameter. The mash is fed from the top and flows down while the steam flows up, driving ethanol upwards. The mash is removed from the bottom with an ethanol content of 0.01%. The obtained liquid is known as stillage; (3) The aldehydes column can be a separate column or located at the top of the analyzing column. It concentrates aldehydes from the purifying column and returns ethanol to process; (4) The rectifying column is where the ethanol vapors are purified and concentrated. It removes congeners or impurities according to the distiller's requirements; (5) The preconcentrating column is used for neutral spirit production to remove impurities, mainly aldehydes before passing to analyzing or rectifying column. 6) The hydroselection column distills the mixture of the preconcentrating column with pure hot water to remove all the impurities from the alcohol. This produces an extra fine neutral spirit (L'Anson, 1976).

2.2.2.5 Aging

During maturation a series of many favorable changes occur steadily including removing harsh aromas in the rum distillates and developing a pleasant aroma and taste. It is necessary to have an initial distillate with good amounts of congener precursors in

order to have the opportunity of a series of chemical reactions to occur. This reactions will determine the progress of the aging process.

Rum is typically aged in oak barrels for at least 2 years, where many changes occur. According to Nishimura *et al* (1983), during maturation in oak casks, rum can overcome several changes. Those changes may be due to:

1. Wood components extraction;
2. Reaction of wood or wood components with the distillate;
3. Reactions within the aged distillate such as oxidation and condensation;

Some of the reactions that occur within the rum distillates are: 1) esterification, which requires the presence of acids and ethanol and other high alcohols; 2) polymerization and condensation, which requires aldehydes; and 3) formation of new acids and alcohols; which requires the presence of esters (Vargas Guzman, 1975). The amounts of acids increases rapidly during the first two years reaching concentrations up to ten times higher than the initial; however the increase in esters occurs at a slower rate. Higher alcohols will increase about 15-20% after 6 years, and aldehydes will be double the initial amounts during the same period of time (Vargas Guzman, 1975).

2.2.2.5.1 Oak Wood for Maturation

Although many woods have been used for spirit maturation, only oaks are important as maturation barrels (Singleton, 1974). Some of the qualities that make oaks suitable for liquid containers are the presence of multiseriate rays, tyloses, durable, tough, bendable wood with high extract to inhibit rotting organisms. The oaks used for cooperage are white oaks from the Leucobalanus or Lepidobalanus subgroups. European species are, *Quercus rubor*, and *Q. petraea*, in North America there are many species but

the most important one is *Quercus alba.* Red oaks are not suitable for cooperage due to the poor tylose formation. Tyloses are spheres of 28% lignin that expand from the cells to plug the sprinwood pores of the heartwood, this prevents the liquid from leaking out of the barrel. *Q. alba* is more fully plugged than the European species. Some other differences from American and European oaks are that European oaks have more extractable phenols, while American oaks have more aromatic compounds. In general the components of different oak species are similar, varying in concentration and relative amounts. Some conditions that may influence the composition of the oaks, and the maturation that can be achieved with the barrels are tree age, growing rate, and such.

Older trees have larger medullary rays which are barriers to the migration of liquids. This may be a reason why old, larger trees are better for staves. Wood chemical and physical composition varies between and within trees. The youngest heartwood, nearest to the sapwood has the highest and most diverse content of extractable components. As the tree becomes older, the heartwood near the pith diminishes in extractable and phenol compounds. Wood of slow grown trees is easier to work and bend, and has greater extracts, this is why coopers prefer it to faster growing wood. European staves have higher extractable solids (161%) than American oak, and have more phenol per unit (154%) (Singleton, 1974). The general composition of dry heartwood is 50% cellulose, 20% hemicellulose, and 30% lignin. Cellulose is the framework of the wood, hemicellulose the matrix, and lignin the encrustant. Cellulose is a polymer of glucose, hemicellulose is an heterogeneous polymer that includes xylose and other sugars, especially pentoses. Lignin in oak is a three-dimensional polymer of phenylpropane derivatives of guaical and syringyl units substituted in the fourth position with the

aliphatic side chain and cross-linked by oxidation. Some other compounds such as tannins and carbohydrates are attached to lignin (Rowell, 1984). In sufficient quantity, the extractives of the wood compounds affect the flavor of the final beverage. Water and ethanol extracts of oak wood contain color, carbohydrate derivatives, gallo- and ellagitannins, lignin fragments, together with their precursors and degradation products (Singleton, 1995). These compounds are flavorful and modify the aroma of the beverage during maturation. The soluble extractives of oak are depleted readily from the layer extracted, while the major structural polymers are broken down, if at all, during the process of maturation (Singleton, 1995).

The ideal ethanol concentration that yields the maximum solid extraction is approximately 55% (v/v) (Singleton and Draper, 1961). For the production of lighter products, lower proofs can be used yielding a product with less extractives and having a different composition. A barrel for 200 L has as much as 90 cm^2/L of wood surface. Evaporation, extraction, oxidation and component reactions are maturation effects related to the conditions of the barrels, and they will increase as more wood surface is in contact with a unit of beverage (Singleton, 1995). Each mm penetration of the beverage to the wood will contribute some 9 cm^3 of extracted oak or 5.4 grams of soluble oak solids, assuming a density of 0.6 g/cm^3, and some 10X flavor threshold (Singleton, 1995).

Once maturation results suffice, a common practice is to reuse or refill the barrels. After refilling, barrels never seem to lose their physical effects, and never seem to be depleted, even when they are not shaved off. This may be due to the faking and cracking that exposed some new wood (Singleton, 1995).

Some consideration has to be given to cooperage as the rays have to be tangential to the barrels circumference. The staves should not be dried too rapidly, in order to prevent checking. The ideal moisture content of the staves for cooperage is 8% to 10 % (Rassmussen and McMillen 1956). According to Francis *et al* (1992), woods that are seasoned in warmer, drier climates possess enhanced vanilla, buttery, caramel, and cedar notes. These notes plus nutty, and a decreased raisiny note are present in wood that is thermally treated at 105°C for 24 h. Other considerations are that due to tree-to-tree differences, the staves used for a single barrel should be randomly selected from several trees. This will decrease the barrel-to-barrel variation (Singleton, 1995).

Charring and toasting of the barrel wood produce various effects in the extractives which are transferred to the beverage during maturation. Charring is a more radical treatment and produces mainly carbon and some pyrolysis products as furfural and HMF. It may transfer some undesirable smoky flavor to the beverage. Toasting is a less drastic treatment and can be accomplished to different degrees and depths (Singleton, 1995). High temperature treatments cause a reduction in hemicellulose and increased furfural and HMF derivatives unless the heat treatment is so severe that the compounds polymerize (Hillis, 1984).

During maturation water and ethanol losses occur in the barreled spirits. About 2% to 7% loss in volume can be expected per year during maturation in standard barrels in the tropics. Many factors such as container size, temperature, relative humidity, and air circulation influence evaporation, other factors include water's liquid state, heat of vaporization, self association, adsorption to barrel carbohydrates and vapor pressure (Singleton, 1995). Given a certain temperature higher humidity lowers the water loss rate.

Ethanol is also lost during maturation. For a constant ethanol concentration in the beverage, the relative humidity should be between 65% and 70%. While water and ethanol are lost, the content of non-volatile compounds increases during maturation.

2.2.2.5.2 Contribution of Oak to the Aroma of Rum

Some of the components found in oak wood that contribute to the aroma of spirits are 4-methyl-γ-octalactone, γ-nonalactone, and eugenol. Table 2.8 lists compounds identified in oak wood. Acetic acid increases during maturation in oak casks, it is a main component of pyrolegneous acid, and it derives from the acetyl group of hemicellulose by degradation (Nishimura *et al*., 1983).

The products of lignin degradation obtained from charred oak are much larger than those obtained from uncharred oak. These components include extracts, with aromatic aldehydes such as vanillin, syringaldehyde, and sinapaldehyde. The higher the toasting temperature, the higher the amounts found. Aromatic aldehydes are directly extracted, while vanillin and syringaldehyde are products of the oxidation of coniferaldehyde and sinapaldehyde (Nishimura, 1983). Nishimura *et al* (1983), found that the ethanolysis of lignin yields more vanillic acid and syrinaldehyde, and less sinapaldehyde and coniferaldehyde than oak chips. Native lignin of low molecular weight yields about the same amounts than with oak chips. However, due to the relative low amounts of native lignin in wood, they suggested that this is not the main source of lignin-derived compounds. Later they studied oxidation and esterification of these aromatics, and suggested that the lignin-derived compounds are formed by the following pathways (Figure 2.4, and 2.5).

Table 2.8 Compounds Identified in Oak Wood.

Aliphatic hydrocarbons	**Aromatic hydrocarbons**	**Other aromatic compounds**
Tetradecane	Naphthalene	1-indanone
Pentadecane	α-methyl naphthalene	benzothiazole
Hexadecane	β-methyl naphthalene	methyl salicylate
Heptadecane	α-ethyl naphthalene	benzoic acid
Octadecane	β-ethyl naphthalene	phenyl acetic acid
Nonadecane	dimethyl naphthalenes	cinnamic acid
Eicosane	trimethyl naphthalenes	
	tetramethyl naphthalenes	**Furan compounds**
Aliphatic acids	biphenyl	Dibenzofuran
Acetic acid	acenaphthene	2-furoic acid
n-butyric acid	acenaphthylene	3-furoic acid
i-butyric acid	1,1-6-trimethyl-1-1,2-dihydronaphthlene	
n-valeric acid	fluorine	**Terpene compounds**
i-valeric acid	1,2-dmethyl-4-allyl benzene	α-Muurolene
caproic acid		γ-muurolene
heptanoic acid	**Phenols**	β-bisabolene
caprylic acid	Phenol	α-cadiene
nonenoic acid	guaiacol	γ-cadiene
nonanoic acid	o-cresol	δ-cadiene
caprilic acid	p-cresol	δ_2-cadiene
decanoic acid	p-ethyl phenol	α-curcumene
undecanoic acid	4-methyl guaiacol	calamene
lauric acid	eugenol	α-calacorene
myristic acid	i-eugenol	cadalene
tetradecanoic acid	chavicol	terpineol
pentadecanoic acid	syringol	borneol
pentadecenoic acid	4-methyl syringol	myrtenol
pentadecadienoic acid	4-ethyl syringol	elmol
palmitic acid	4-allyl syringol	epi-cubenol
	vainillin	β-eudesmol
Other aliphatic compounds	propiovainillone	α-eudesmol
Cis- 4-methyl-γ-octalactone		γ-eudesmol
Trans-4-methyl-γ-octalactone	**Other aromatic compounds**	α-cadinol
γ-nonalactone	Benzyl alcohol	T-cadinol
γ-decalactone	phenethyl alcohol	Verbonene
1,1-dimethoxynonane	phenethyl acetate	Geranyl acetate
1,1-dimethoxydecane	acetopehenone	
β-Ionone		

Source: Nishimura *et al.*, (1983).

Other effects of maturation on the composition of beverages are an increase in acetaldehyde, derived from ethanol + acetic acid. The already minor quantities of sulfur compounds, such as dimethyl sulfide, dimethyl disulfide, methionyl acetate, ethyl methionate, and dihydro-2-methyl-3(h)-thiophane decreased with aging. Toasting of oak wood produced other non-lignin volatile compounds such as furfural, 2-methylfurfural, guaiacol, and 4-methyl guaiacol (Nishimura *et al.*, 1983).

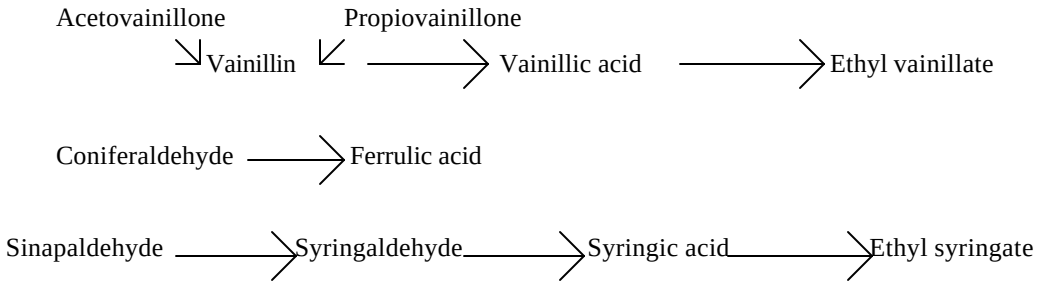


Figure 2.4 Reactions of Lignin Components During Storage in 60% Ethanol Solutions. (Nishimura et al, 1983).

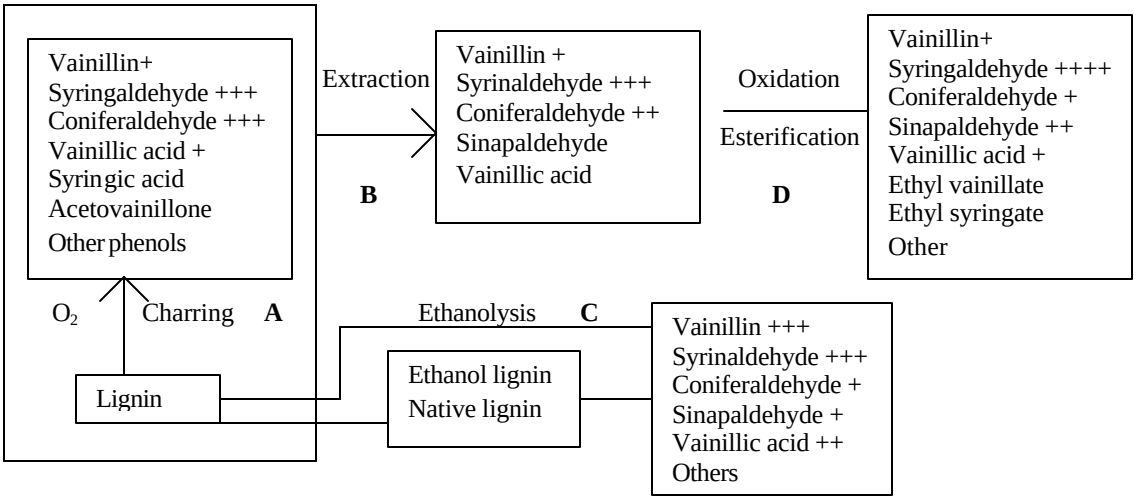


Figure 2.5 Pathways for Lignin Derived Compounds Formation (Nishimura *et al.*, 1983).

2.2.3 Flavor of Rum

The flavor of alcoholic beverages is composed of various volatile and non-volatile organic compounds. These compounds give the typical odor and taste to the beverage (Lehtonen and Jounela-Eriksson, 1983). Many of these compounds have been identified, and can be classified in several groups according to their chemical nature. Most compounds responsible for the aroma of distilled beverages are volatiles, and the typical flavor and chemical composition will be closely related to the manufacturing process used (Nykänen and Nykänen, 1991).

The fermentation stage is chiefly responsible for the basic aroma formation. The yeast metabolism is affected by the fermentation conditions. Distillation can largely affect the proportion of compounds recovered. Maturation influences the aroma of rum by adding new compounds formed in chemical reactions, while other compounds are condensed. The final rum product will be related to all the production stages.

2.2.3.1 Higher Alcohols

Higher alcohols are the most abundant aroma compounds in rum (Nykänen and Suomalainen, 1983). Higher alcohols such as n-propanol, and isobutanol are formed from their correspondent keto-acid, following the way ethanol is converted from pyruvate. Keto acids are formed from amino acids by transamination reactions, and then to alcohols (Watson, 1985). Strongly smelling aliphatic alcohols, such as 1-propanol, 2-methyl-1-propanol, 2-methyl-1-butanol, 3-methyl-1-butanol, and the aromatic alcohol phenethyl alcohol, may be formed from sugars by an anabolic process via the pathways the amino acids are synthesized. This formation seems to be suppressed in a medium with abundance of assymilable nitrogen (Nykänen, and Nykänen 1983). 3-Methyl-1-butanol

is the most abundant fusel oil in rum, followed by 2-methyl-1-propanol (Lehtonen and Jounela-Eriksson, 1983). Ter Heide *et al.* (1981) determined the presence of the homologous series of 1-alkanols from methanol to decanol in rum. The choice of yeast strain seems to be the main factor to control the formation of 1-alkanols (Parfait and Jouret, 1975), and the nature and quantity of the nitrogen source may also play a role (Parfait and Sabin, 1975). A fermentation mash low in inorganic nitrogen, and especially at high temperatures yields high fusel oil yields due to aminoacid deamination and decarboxylation (Figure 2.6).

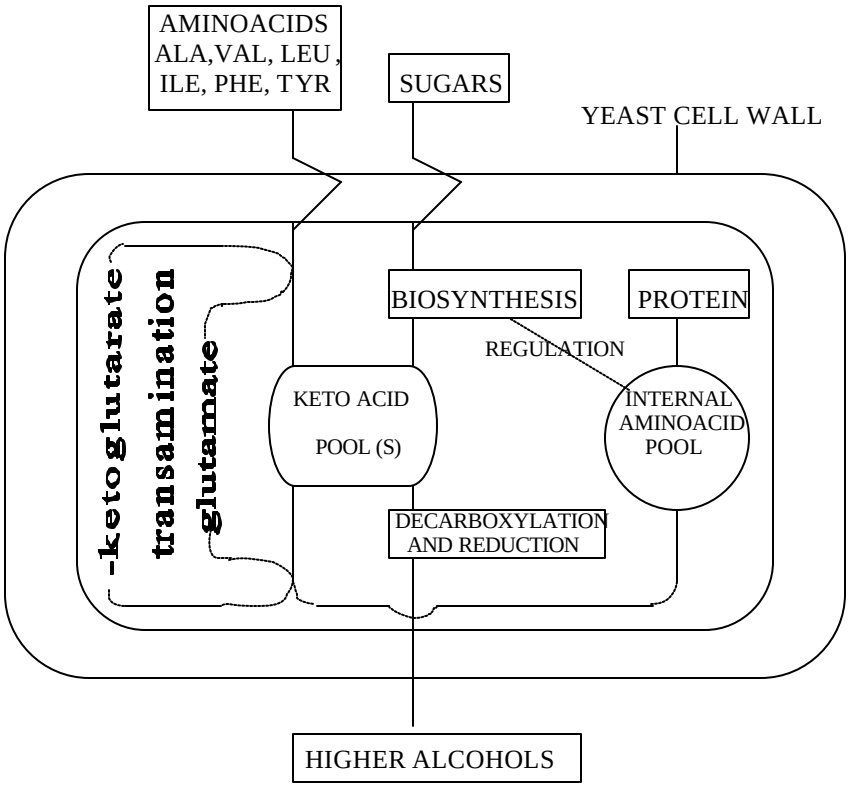


Figure 2.6 Diagram of the Formation of Higher Alcohols in Yeast Cells (Meilgard, 1975).

The distillation procedure is known to have an effect on the content of higher alcohols in rum. According to I'Ansosn (1971), larger amounts of higher alcohols were found in rums distilled in pot-stills, than in continuous stills. Some higher alcohols found in rum are included in Table 2.9.

Table 2.9 Higher Alcohols in Rum

3-methyl-1-butanol (isoamyl alcohol)	2-methyl-1-butanol
2-methyl-1-propanol (isobutanol)	Propanol
2-butanol	Isopentanol
1-propanol	

Source: (Nykänen and Suomailanen, 1983).

The presence of 2-butanol appears to be related to the action of bacteria during fermentation (Nykänen and Nykänen 1983). Aliphatic long-chain alcohols have hardly any effect on the aroma of rum (Nykänen and Nykänen 1983).

2.2.3.2 Organic Acids

Organic acids such as acetic and even-chained fatty acids are produced by strains of *Sacch. cerevisiae* through the fatty-acid synthesis pathway (Berry and Watson, 1987). Acids are important precursors of esters as well as being flavor-active by themselves (Berry and Watson, 1987). Fatty acids are produced during fermentation, and are easily transferred to the distillate during distillation. The total acid content in heavy bodied rums is about 100 to 600 mg per liter, with acetic acid being the predominant volatile acid. Heavy-bodied rums contain more volatile acids than light rums (Nykänen, and Nykänen 1983). The amount of acids was found to increase during maturation. The increase in acids was total, fixed and volatile acidity (Lehtonen and Jounela-Eriksson, 1983). Table

2.10 shows some of the fatty acids present in rums. Acids extracted with alcohol from oak casks include salicylic acid, 4-hydoxy-cinnamic acid, gallic acid and chlorogenic acid (Nykänen and Suomalainen, 1983). Table 2.11 includes other acids that may be identified in rum.

Table 2.10 Fatty Acids Present in Rum

Short-chain fatty acids	**Long-chain fatty acids**
Acetic acid	Octanoic acid
Propionic acid	Decanoic acid
Isobutyric acid	Lauric acid
Butyric acid	Myristic acid
Acrylic acid	Palmitoleic acid
Isovaleric acid	Palmitic acid
Valeric acid	Linoleic acid
2-ethyl-3-methyl	Oleic acid
butyric acid	Stearic acid
Hexanoic acid	
Heptanoic acid	

Source: Nykänen and Nykänen (1983).

Table 2.11 Aromatic and other Carboxylic Acids Identified in Rum

Benzoic acid	Gallic acid
Salicilic acid	Vainillic acid
p-Hydroxybenzoic acid	p-Hydroxycinnamic acid
Gentisic acid	4-Hydroxy-3,5-dimethoxy-benzoic acid
o-Pyrocatechuic acid	4-Hydroxy-3-methoxy-cinnamic acid

Source: Nykänen and Suomalainen (1983).

2.2.3.3 Esters

Esters are the key compounds in rum because of their contribution to the aroma. Esters are responsible for the presence of fruity-like aroma in rums. The main esters formed in rums (Table 2.12) are acetate-esters of alcohols (e.g. ethyl acetate), and ethyl

esters of fatty acids (e.g., ethyl decanoate). They are mainly from acid:alcohol esters, but there is a proposed mechanism of formation from enzymatic reaction between free alcohols and acetyl CoA derivatives of fatty acids (Watson, 1993)

The ester content of rum varies from 4 to 64% w/v, with ethyl acetate typically being the predominant ester. The ester content in rum is related to many factors. The esters formed during fermentation are related to the distribution of esters between the yeast cells and in the medium, and to factors affecting ester concentration. When yeast is present during distillation, the content of long-chain carboxylic acid esters derived from the yeast cells increases. The ester content of the beverage of rum also depends on the yeast.

Table 2.12 Esters of Aliphatic Monocarboxylic Acids in Rum

Ethyl formate	Ethyl valerate	Ethyl undecanoate
Methyl acetate	Hexyl acetate	Isobutyl dodecanoate
Ethyl acetate	Isopentyl propionate	Isobutyl duodecenoate
Isobutyl formate	Isobutyl butyrate	Ethyl laurate
Propyl acetate	Ethyl hexanoate	Isopentyl decanoate
Ethyl propionate	Ethyl heptenoate	Ethyl mysistate
Isopentyl formate	Isopentyl butyrate	Isopentyl laurate
Isobutyl acetate	Ethyl heptanoate	Ethyl pentadecanoate
Butyl acetate	Methyl octanoate	Methyl palmiatete
Sec-Butyl acetate	Phenethyl acetate	Phenetyl decanoate
Propyl propionate	Isopentyl valerate	Ethyl-9-hexadecenoate
Ethyl isobutyrate	Ethyl octanoate	Ethyl palmitate
Ethyl butyrate	2-Methylbutyl hexanoate	Propyl palmitate
Isopentyl acetate	Isopentyl hexanoate	Ethyl linoleate
Isobutyl propionate	Ethyl nonanoate	Ethyl oleate
Propyl butyrate	Methyl decanoate	Ethyl stearate
Ethyl 2-methylbutyrate	Ethyl decanoate	Isopentyl palmitate
Ethyl isovalerate	Isopentyl octanoate	

Source: Nykänen and Suomalainen, (1983).

Temperature affects the rate of ester formation in the medium (Nykänen, and Nykänen 1983). 2-ethyl-3-methylbutyric acid has been determined in rum and is believed to come from 3-methylbutyric acid, a compound found in molasses (Lehtonen *et al.*, 1977).

During distillation the middle aroma fraction consists of esters from ethyl hexanoate to ethyl laurate, esters of long-chained carboxylic acid with the acid part longer than 12 carbon atoms belong to the heavy aroma fraction of rum (Nykänen and Nykänen, 1983). Table 2.12 includes some of the most common esters of aliphatic monocarboxylic acids that have been identified in rum.

2.2.3.4 Carbonyl Compounds

It is believed that aliphatic carbonyl compounds are related to aroma nuances in distilled alcoholic beverages, especially when present in large amounts (Nykänen and Nykänen, 1983). Aldehydes can total up to 5 to 9% w/v in Jamaican, Puerto Rican, and Martinique rum, acetaldehyde being the predominant compound (Nykänen and Nykänen, 1983). Some of carbonyl compounds present in rum are shown in Table 2.13.

Table 2.13 Carbonyl Compounds in Rum

Higher aliphatic aldehydes	Ketones	Di-ketones
Propionaldehyde	Acetone	2,3-butanienone
Isobutyraldehyde	2-butananone	2,3-pentanedione
2-methylbutyradehyde	3-penten-2-0ne	
isovaleraldehyde	2-pentanone	
	4-ethoxy-2-butanone	
	4-ethoxy-2-pentanone	

Source: Nykänen and Nykänen (1983).

2.2.3.5 Acetals

As many as 43 acetals have been identified in rum (Nykänen, and Nykänen 1983). Most of the acetals are formed during distillation by reaction of a molecule of aldehyde and a molecule of alcohol, to form a hemiacetal. The hemiacetal formed is labile, and once it combines with another alcohol molecule it forms a stable acetal (Figure 2.7).

The reaction of the formation of acetals is reversible. Both, the acetal formation and hydrolysis reactions are catalyzed by acids (Nykänen, and Nykänen 1983). In rums, the amount of acetals present can be from 4.3 to 41.6 % w/v (Lehtonen and Suomalien, 1977). The most abundant acetal is the diethyl acetal of acetaldehyde.

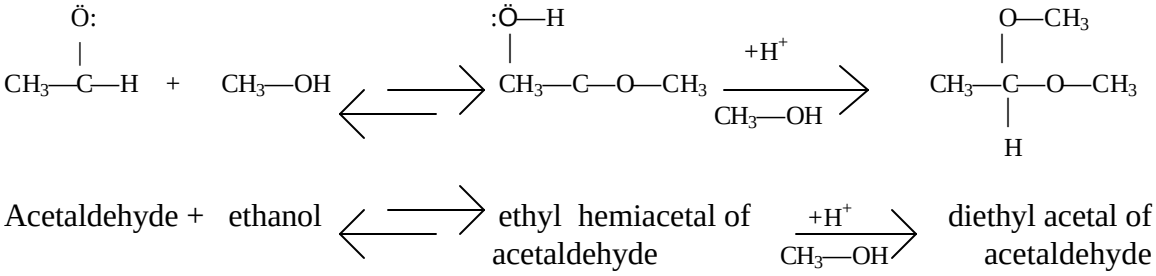


Figure 2.7 Formation of Acetal

2.2.3.6 Phenols

Phenols are compounds present in small amounts, but the contribution they make to the aroma of alcoholic beverages is significant (Lehtonen and Jounela-Eriksson, 1983). Phenols are mainly formed during maturation via alcoholic extraction from the oak casks, but they can also be formed during other production stages. During fermentation, p-ethylphenol and p-ethylphenol can be formed through decarboxylation of p-coumaric and

p-ferulic acid. Table 2.14 contains some of the phenols identified in rum. High temperatures, and some microorganisms as yeast and bacteria have been found responsible for the decarboxylation. Other phenols are formed from the lignin of oak barrels. Guaiacol, eugenol, vanillin, and m-creosol can be extracted from oak chips (Lehtonen and Jounela-Ericksson 1983).

Table 2.14 Volatile Phenols Identified in Rum

Phenol	2,4-Dimethylphenol
o-Cresol	p-Ethylphenol
m-Cresol	p-ethylguaiacol
p-Cresol	Eugenol
Guaiacol	p-(n-Propyl)guaiacol
o-Rthylpehnol	p-Methylguiacol

Source: Lehtonen and Jounela-Eriksson (1983).

2.3 Rum Aroma and Taste Perception

Alcoholic beverages contain numerous compounds that contribute to their aroma. "Olfaction is the ability to recognize and discriminate with great accuracy and sensitivity myriads of airborne molecules either by themselves or as complex mixtures" (Anholt, 1992). The olfactory system provides important information for survival by detecting and processing chemicals in the environment. The aroma is perceived when a volatile compound contacts the olfactory mucosa in the epithelium. The olfactory receptors axon through the cribform plate of the ethmoid bone terminating in the olfactory bulb. There is a large but limited number of olfactory receptors that transduce a large number of odorants. The olfactory receptors direct extensions of the olfactory nerve into the

environment. The stimulus signal of a molecule contacting the olfactory receptor cells has chemical specificity and it is translated to a neuronal electrical response. The transduction mechanism is still unknown (Thorngate, 1997). Odors can be perceived in two ways; direct, the molecules enter the nose from the front through the nosetrils by inhalation, and indirect via mouth and rear nasal passages (Pousias and Chabanon, 1974). The temperature rise of substances in the mouth releases more odorous compounds which reach the olfactory region by diffusion and through exhalation. These in-mouth odors are an important part of what is called flavor (Amerine and Roessler, 1983).

Man's olfactory system is far less sensitive than that of many mammals. The olfactory thresholds vary depending on the substance. Some factors like respiratory infections and migraine can increase olfactory thresholds. Individual threshold variations depend on various factors such as sex; women between adolescence and menopause have higher sensitivities, especially after ovulation. During stimulation by a particular odor, the threshold value rises above the stimulation concentration causing adaptation. Adaptation is caused by the higher centers of the brain, and it explains why professional smellers can perceive odors far longer than untrained persons (Carr, 1974).

Taste is a sensation transduced by a taste receptor cell. Taste receptors are located in the taste buds which are specialized aggregates of taste receptor cells. The distribution of the taste receptor is highly localized in the lingual margins. There are four tastes: sweet, bitter, salty, and acid. The upper edges of the tongue react to acids, the rear to bitter compounds, the tip of the tongue to sweet, and the sides to acid tastes.

The transduction of the taste sensation involves the interaction of sapid chemicals with receptor sites, which are believed to be localized at the apical microvilli membranes.

The stimuli must be dissolved in order to diffuse through the saliva and mucous which coats the apical membranes. Once proximate to the membrane, the stimuli may either bind to a membrane-bound receptor, block membrane on channels, or directly influx through ion channels (Thorngate, 1997).

The chemical sensations are astringency and irritations which are properly tactile sensations. They are perceived by the trigeminal nerve endings, located in the lingual mucous, pharingueal mucous, and the nasal mucous, including the yellow olfactory spot (Poisais and Chabanon, 1974).

In most vertebrates, the receptor cells are located in the olfactory epithelium, a pseudoestratified structure that lines a small portion of the nasal cavity. The olfactory epithelium is unique in that; 1) the receptor cell body is in direct contact with the external environment; 2) the olfactory neurons have a single, unbranched axon that projects to the telencephalon without intermediate neurons; and 3) the olfactory system replaces neural elements normally and when injured. The continued neurogenesys of olfactory cells is due probably to its constant exposure to the environment resulting in injure and damage of cells (Morrison and Constanzo, 1992). The neuroepithelium contains cells of three types: olfactory cells, supporting cells, and basal cells, arranged in a psudoestratified columnar organization.

The olfactory receptors have axons that terminate in the olfactory bulb and thus they have a more direct connection to the brain than do the receptors of any other senses. Their location in the upper part of the nostrils still allows for direct contact with the environment. As they are exposed to pollution, they have the ability to regenerate. There are about 6 million olfactory receptors in each nostril, enough to detect significant

environmental odors. They differ in the way they respond to different odorants, with each cell responding to more than one, but not all odorants. Therefore, each compound activates different sets of cells and generates unique patterns of neural activity that correspond to the odor quality and concentration (Anholt, 1992). The response to the odorant terminates when they are removed via mucociliary clearance and absorbed into the circulation (Getchell *et al.*, 1984; Hornug and Mozell, 1997; and 1980). In addition, the olfactory epithelium has a high activity of cytochrome, which inactivate odorants, and may also transform odorants into compounds of altered odorous quality and potency, therefore affecting the complexity of the stimulus and its perception (Dahl *et al*., 1982; Nef *et al.*, 1989; and 1990; and Dahl, 1988).

2.3.1 Odor Perception

Odor perception is multimodal, involving other sensations, including taste, and the relatively mild irritation from stimulation of the trigeminal nerve. “The sensory function of smell is overshadowed in man by his other abilities for interaction with the environment. Therefore, man has regressed developmentally in his olfaction ability as compared with most other higher life forms” (Estrem and Renner,1987). Human performance in detecting and identifying odors is less reliable than sight and hearing. However, the sense of smell is a sensitive impressionable faculty for assessing the chemical environment and for storing information. People can recognize familiar odors but are usually able to describe them only in general terms. One may recognize an odor as familiar without being able to pin down its identity, leaving one in the condition of uncertainty that is called tip-of the-nose (Lawless and Engen, 1977).

The pleasant and unpleasant qualities are not in the odors themselves but in the events or persons with which they are associated. Odor preferences are learned and they function because of the memory associations. Only familiar and identifiable odors are perceived as good. Odor memory is better than other types of memory because one can recapture the past, including the feeling of the remembered event. However, we cannot recall odors at will in the absence of such stimulation. A long-term odor memory can be established with only one exposure. Odor associations can be formed automatically and without conscious awareness. Having learned one association to an odor makes it difficult for us to replace the association with another one. Odors do not have their own names, but are described as smelling like something else. The special role of odors is to retrieve significant events regardless on when they happened. The sense of smell is very sensitive, learned quickly, and is not forgotten, but is not very discriminating and it has no judgment about what is important to remember and what is best forgotten. The human nose is in constant use because environmental odors must be in constant monitory. For each odor detected, a memory search is made to determine its identity. Familiar odors are hardly noticeable, only odors that are unusual or unexpected get conscious attention.

2.3.2 Persistence of Odors

Adaptation is caused by a decreased sensitivity to an odor because of a change in the function of the receptors, and this decrement may spread to the perception of odors through cross adaptation. However, depending on the pair of odors, exposure to one odor may also increase the response to a second odor, this is called facilitation. When the odor stimulus is removed or avoided, recovery from adaptation occurs. The concentration of the molecules can exceed the ability of the receptors to handle them, this is perceived as

adaptation, fatigue, or system failure. The two parameters that affect adaptation are strength of the stimulus and the duration of the exposure; the intensity and stability of odor stimulation are controlled by sniffing. Odor processing is variable even though one may be exposed to the same constant concentration of odor. It is believed that only 5 to 10% of the breathed air will reach the olfactory cleft, the rest is diverted below to the pharynx, past the epiglottis to finally reach the lungs. Sniffing serves a way to control the amount of air flowing trough the nostrils. By simulating gagging one can shut off the access of odorous molecules through the pharynx, between the mouth and the cleft. This prevents overexposure and keeps the sense of smell working. For any single odor, the longer the exposure, the less sensitive one is to it. This is a result of a change in the reaction of the olfactory receptors to odorous molecules, causing a rapid exponential decrement in sensitivity, but except for the weakest odors, sensitivity does not diminish to zero. (Berglund *et al*., 1978).

Naturally using the sense of smell will cause some fatigue, regarding some odors more than others, and it will change and not abolish perception. A properly working olfactory system prevents functional anosmia by avoiding extreme stimulation and by controlling the amount of stimulation through sniffing. Constant exposure to various odors affects sensitivity without interfering with the perception of significant odors. For certain pairs of odors an opposite effect can occur, this is a prior exposure to an odor increases the perceptual strength of a new one. This effect is called facilitation, synergism or potentiation. It has not been widely explained but it is believed to be involved the periferial olfactory mucosa, where an odor interacts with the receptors mucous. The possible mechanisms involved may be: 1) An odor may alter the mucus around the

olfactory receptors making them more accessible to new molecules, 2) the two odors involve the same receptor in a way in which their effects are summed, or 3) the first effect may create a priming effect that enhances general chemical communication. The sense of smell can recover from fatigue associated with the exposure to an odor after some period of time.

CHAPTER 3. DEVELOPMENT OF SENSORY DESCRIPTORS FOR RUM AROMA, FLAVOR, AND TASTE EVALUATION BY SEMI-EXPERT JUDGES

3.1 Introduction

Centuries ago, lexicon was a language to serve poetic styles and subjects of social conversations. Nowadays lexicon is used as expressing judgment and comparing products of mostly similar origin, and simultaneously creating a glossary allowing for the guidance and training of the less knowledgeable panelists (Boyazoglu, 1986.) These descriptors for products have not only been used by scientists, but also by wholesalers and retailers that employ the power of words to attract the attention of consumers.

The development of a meaningful lexicon is needed in order to have words that adequately interpret the description of the product. It is necessary that when those words are used, their meaning is clear to an audience, and finally, when a product is marketed, these terms will be used through ads and labels to educate the public about the product.

Lexicon development is widely used in Descriptive Sensory Analysis, techniques used for product development, quality control, and in laboratory practices (Amerine and Roessler, 1983). Among the Descriptive Analysis Techniques, Flavor Profile, Quantitative Descriptive Analysis (QDA), Texture Profile and Spectrum require a consensus among judges for the use of terms and definitions used to describe the product. Free choice profiling is a technique that does not require consensus among judges, but it allows for more descriptive terms that are not necessarily meaningful to all people.

For accurately describing a product, the words used should be objective and not subjective. They should not represent feelings (good, bad, etc.), because the interpretation made can be varied depending upon personal background and experiences.

Disagreement often takes place in applying terminology, therefore it is recommended that the panelists agree in applying the same terms to products (Lehrer, 1983). Having a set of references that can illustrate the meaning of the terms is necessary for stabilizing the panel's use of vocabulary. It is also useful for assisting in the training of new recruits on the use of terms. Therefore, suitable materials should be available to use as a standard (Lawless and Klein, 1991). The standard should: 1) contain a true representation of the favor note as it appears in the product, 2) contain the flavor note and no other note, and 3) be readily available in the required purity. It can be a pure compound or a complex material, the problem of using the later is that it might not be stable, and therefore not suitable for use (Lawless and Klein, 1991).

As stated by Bloomfield, (1939), "standardization of scientific language is obtained when a technical term is fixed by an agreement of definition which receives explicit formulation". When interpreting descriptors, it is important to consider that even experienced judges (particularly from different regions) are apt to have very different concepts about the terms (Amerine and Roessler, 1983). One must also consider that meaning of terms may change with time, for example a sweet wine in the past may not be so today (Ribéreau-Gayon, 1973). It is a mistake to think that every word has a fixed, specific meaning, where words have intra- and extra- linguistic relationships (Lehrer, 1983). Therefore, the importance of a term is based not only on its simplicity and clarity, but also mainly on its recognizable meaning (Amerine and Roessler, 1983).

One important thing to consider for developing a meaningful list of descriptors is to have a consensus among the judges on the terms and definitions. A disadvantage of

working on a consensus is that bias can be introduced by a dominant (assertive) member of the panel (Amerine and Roessler, 1983).

For the selection of descriptors it is important to consider that they should be meaningful; they accurately and precisely describe all required characteristics of the samples; they have to be understood by the assessors, and they should be agreed on their meaning; they can be easily identified with reliable standards, and they are understood by the recipients of the report (Lawless and Klein, 1991).

The aim of the study was to develop and evaluate a list of terms with their descriptions to describe rum products prepared from different substrates using different processing protocols.

3.2 Materials and Methods

For the development of a list of lexicon, 15 different commercial rums were selected to cover the spectrum and for comparative framework. For the actual product evaluation 6 commercial and one experimental sample were used. The experimental sample was made from molasses, distilled in continuous still and aged under an accelerated aging processing using oak chips (10g/L) for 2 ½ months at 30°C.

For the development of lexicon a panel of 5 semi-experts judges familiarized with evaluation of rum products and previously screened was used. A total of 9 sessions and 16 hours were required for training and development of the list of terms and definitions. Three sessions of 1 1/2 hours were used for evaluation of samples with the list of terms, definitions and references. All the judges were present in all sessions. The sessions were done in an air-conditioned room with around 67°F under fluorescent lighting with a round table. The time for the evaluations was between 3:00 to 5:00 PM.

The samples (1 oz) were presented coded and served in plastic sealed cups at room temperature. For the development of the list of terms, the judges were presented with 5 to 6 rum samples simultaneously. They were asked to evaluate them individually, and report the perceived character note with definition in a ballot provided. After this was done, the panelists had a group discussion to get consensus for the terms. From the individual sessions a pooled list of terms with definitions was prepared. The panelists were provided with the list of terms with definitions and the references, they were then asked to evaluate samples based on those descriptors, and to indicate the order in which they were perceived. In this case they evaluated a total of 7 samples including the experimental sample having two or three samples per session presented individually. The judges were ask to smell in short deep sniffs; first at a distance of 2.5 cm from the rim of the cup, then at the rim of the cup, next with the nose inside the cup, and finally they were asked to swirl the cup and sniff again with the nose inside it. Afterward the judges were asked to take a small sip, let it go to the back of the tongue, then slowly forward to the front of the mouth, take a deep breath, and swallow.

The perception of aftertaste was recorded after 1 minute. After evaluating each sample, the judges were asked to rinse their palates with spring water. In this case each judge checked the list of terms provided and determined the perceived notes, then a general consensus and conclusion for each sample was done, then the judges proceeded to evaluate the next sample.

3.3 Results and Discussion

3.3.1 List of Terms with Definitions for the Description of Rum Aroma, Flavor, and Taste

The panel of 5 semi-expert judges was able to determine the presence of the following attributes in the 15 rum samples. Definitions for some of the terms are based on Civille and Lyon (1996).

1. Allspice: spicy characteristic of ground allspice
2. Almond: aromatic associated with almond/almond extract
3. Apple: aromatic characteristic of various apple varieties
4. Apple/pear; fruity-like aromatic of pome fruits which cannot be recognized as any specific fruit
5. Artificial: aromatics or tastes considered artificial or not natural for rum
6. Astringent: chemical feeling factor
7. Banana: fruity aromatic characteristic of ripen banana
8. Bite: chemical burning sensation felt in tongue, mouth or throat
9. Burnt: chemical feeling factor associated with high concentration of irritants to the mucous membranes of the oral cavity
10. Butter: aromatic associated with fresh butter
11. Butterscotch: sweet aromatic typically having both buttery and caramelized notes
12. Caramel: an overall term reminiscent of chewy caramel
13. Chemical: associated with compounds such as solvents, cleansing compounds, and hydrocarbons
14. Cinnamon: sweet, woody aromatic of ground cinnamon bark

15. Pure ethanol: a pungent aroma associated with ethanol
16. Eucalyptus/mint: a sweet green aroma associated with fresh mint/eucalyptus leaves
17. Floral: fragrant associated with flowers
18. Fruity (general): aromatic associated with non-specific fruits
19. Fusel oil: aroma note associated with isoamyl alcohol, butanol
20. Gasoline: chemical solvent of hydrocarbons, reminiscent of gasoline, kerosene.
21. Leathery: aromatic associated with tanned animal hides
22. Medicinal: aromatic associated with band-aids, disinfectant-like (phenolic)
23. Musty: aromatic associated with closed air spaces such as attics (dry) and basements (wet)
24. Nutty: aromatic associated with nuts or nut meats
25. Pineapple: aromatic associated with fresh pineapple
26. Plastic: aromatic associated with plastic, polyethylene containers
27. Smoke: perception of any kind of smoke flavor
28. Smooth: clean, pleasant chemical feeling factor
29. Spicy: overall term associated with pungent spices
30. Sweet: taste on tongue stimulated by sugars and other high potency sweeteners
31. Syrupy: aroma associated with clean syrup, pancake syrup.
32. Vanilla: aromatic blend of sweet, vanillin, woody
33. Woody: aromatic associated with dry fresh cut wood, balsamic or bark-like.

For the development of the list of terms, it was very useful to provide the judges with a variety of references for each descriptor. This allowed the judges to have an open discussion on their perception, to homogenize their criteria, and also helped them to identify attributes they were not able to perceive before. In each session, the judges were presented with a list of the terms previously identified in the rum samples by them. This facilitated finding those descriptors in new samples, and allowed them to focus on the perception of new descriptors. All the attributes on the list were created from a consensus among all the judges.

3.3.2 Rum Evaluation Using Terms Developed by the Judges.

During evaluation sessions, the rums used were tested and checked for the presence of the different aroma, flavor and taste descriptors developed by the judges. The judges were also asked to determine the order of attributes perceived when sniffing the sample at 4 different instances: (1) at about 1-2 inch from the rim of the cup, (2) at the rim of the cup, (3) with the nose inside the cup, and (4) again with the nose inside the cup after having swirled the sample. Then the panelist were asked to take a small sip, bring it to the back of the tongue, slowly to the tip of the tongue, followed by a deep breath, and finally swallow the sample. Smelling at different levels allowed the judges to detect different aroma components naturally separated by the variation in their volatility. The fact that the judges were able to determine the instance they detected a specific descriptor allowed other judges to verify their perception, focus on that specific attribute, and be able to detect attributes easier. This also facilitated reaching a consensus among the judges for that sample.

According to consensus, their perception of the rum samples was as follows:

- Rum form Haiti from sugarcane juice double distilled in copper-pot stills and aged in Limousin oak casks for 15 years, with 86 Proof was perceived as "astringent", "bite", "caramel", "fruity", "pure ethanol", "smoke", "sweet", and "woody".
- Puerto Rican rum from molasses distilled in continuous columns, that consisted of blends of different aged distillates, with 80 Proof the following character notes: "apple/pear", "bite", "caramel", "cinnamon", "pure ethanol", "sweet", and "vanilla".
- Rum from Barbados made from molasses, double distilled in single distillations, blend of different distillates with 80 Proof. It was described as "bite", "pure ethanol", "fruity", "leathery", "smoked", "spicy", "vanilla", and "woody".
- Rum made in Jamaica from molasses distilled in copper stills, blend of different distillates. Aged for 12 years with 86 Proof, had the character notes: "bite", "fruity", "vanilla", and "woody".
- Rum made in Honduras from molasses distilled in continuous columns, aged for 7 years with 80 Proof was described as "bite" "butterscotch", "pure ethanol", "fruity", "sweet", "vanilla", and "woody".
- Cuban rum made from molasses distilled in continuous columns aged 7 years with 80 Proof was described as "butterscotch", "leathery", and woody".
- Experimental sample made from molasses distilled in continuous distillation, aged for 2 1/2 months with American oak chips at 30°C with

80 Proof was described as "artificial", bite", "chemical", "pure ethanol", "fusel oil", "leathery", "smoke", "vanilla", "woody".

3.4 Significance

There are a large variety of published terms for the description of wine and other alcoholic beverages. However, it is important to have a list of terms that are useful and specific for the sensory evaluation of rum. The aroma of rum is very complex and varies greatly according to the processing protocol, therefore this study aimed to create a lexicon for the aroma, flavor and taste of rum that includes attributes for the description of all the different types of rum produced. Other requirements for the list of lexicon were to be objective and easy to understand and perceive by different judges, this is the reason for the stress in the consensus of the created descriptors.

A set of lexicons and their description from this study is useful to describe a broad spectrum of rum products. It can be further used for quantification of sensory quality through descriptive analysis techniques.

CHAPTER 4. DESCRIPTIVE ANALYSIS OF THE AROMA OF RUM

4.1 Introduction

Rum is commonly examined and evaluated for quality control purposes and for ideal blending by highly trained and experienced expert blenders that work for each individual bottling company. Consumers usually based their criteria for the selection of rum on the raw material used, the origin, color, aging time, or brand characterization. In general, dark aged rums are believed to have more intense aromas than the light white rums. The light rums are most commonly used as mixers while dark rums are sipped straight. However, little is known on the differences in the aroma attributes perceived in these products.

Several techniques that focus on the description and quantification of the aroma of the products have been developed. Descriptive Analysis is the initial step of the characterization of food products, and is the basis for product testing. It categorizes the different senses, and provides a language useful for the communication of perceptions (Moskowitz, 1983). Quantitative Descriptive Analysis (QDA)® developed by Stone (Stone *et al*, 1974) allows for the statistical analysis of the obtained sensory evaluation. Quantitation of sensory responses requires the use of trained panels using the same vocabulary, as well as adequate definitions and standard references. In addition, it requires the use of the same scale to unify the evaluation criteria. Spectrum™ method of analysis consists of a complete description and accurate characterization of the sensory attributes of the product (Muñoz and Civille, 1999). It provides tools for designing a descriptive procedure for a given product category. It is a custom design approach to panel development, selection, training, and maintenance (Meigaard *et al.*, 1999).

Spectrum analysis uses a 15 cm scale anchored with several intensity reference points. It requires the participation of trained panelists with acute senses previously screened, the use of standard terminology and scales to rate the intensity of the perceived attributes (Muñoz and Civille, 1992).

Descriptive Analysis Techniques involve the characterization of the product by attributes and the intensity of those attributes. They have qualitative and quantitative components, both are necessary for the effective performance of the data analysis (Muñoz, and Civille, 1998). The references also have qualitative and quantitative characteristics, they include the background information and the reference points for intensity that evaluators will relate to when evaluating products. Without intensity references the panelists use their own criteria to rate intensities, resulting in higher variability (Muñoz and Civille, 1998).

Some specific descriptive analysis techniques have been developed or modified for the special needs of the evaluation of different alcoholic beverages. In most cases such methodologies are created for wine evaluation, such as the Traditional Quality Assessment (TQA) for wine, or the University of California Davis descriptive analysis (UCD DA) for the wine industry (Noble, 1998). Other methodologies have been applied to beer such as modified QDA for sensory profiling of beer (Mecredy *et al.*, 1974); to whisky (Piggot *et al* 1980 and 1985); and to gin (Piggot *et al* 1983). However, there has not been a methodology developed specifically for the descriptive analysis of rum products. The objectives of this study were to develop a methodology suitable for the descriptive analysis of rum, to describe the aroma of a variety of different rum products, and to relate the perceived rum aroma to the processing conditions of the products.

4.2 Materials and Methods

4.2.1 Panelist Screening

All participants were required to perform a screening test before training. The screening tests were completed in four days. The participants completed the screening test at the most convenient time for them, although they were asked not to come right before or after meals. The test was done in an air-conditioned room, in partitioned booths. The test was divided into 4 different sections. In the first section the participants were presented with 7 solutions and asked to identify the taste perceived in each of them (sweet, sour, salty or bitter). The solutions were prepared with sucrose (0.5%), citric acid (0.25%), salt (0.2%) and grapefruit juice (TexSun ruby red grapefruit juice, Citrus World Inc., Lake Wales, Florida). The concentration of the solutions was above identification threshold level. The samples were presented in capped plastic cups filled with ca. 20 ml, and coded with 3 digit random numbers. The participants were asked to write down the number of the solution on the line of the matching taste perceived. In the second part the participants were presented with four different sets of solutions and asked to rank intensities. Sucrose solution was used for sweet taste; white vinegar was used for sour taste; salt was used for salty taste, and grapefruit juice was used for bitter taste. 4 dilutions of each basic taste were presented. The following solutions were given to the participants; sugar solutions at 0.5%, 1%, 1.5%, and 2%; salt solutions at 0.2%, 0.4%, 0.6%, and 0.8%; white vinegar solutions at 0.25%, 0.45%, 0.65%, and 0.85%; and grapefruit juice (TexSun Ruby Red Grapefruit juice, Citrus World Inc, Lake Wales, Florida) diluted with water to 1/4, 1/3, ½, and pure grapefruit juice. The set of solutions were randomly coded with a 3-digit number, and presented in capped plastic cups with

ca. 20 ml of solution each. The participants were asked to rank the 4 solutions presented according to the intensity from the lowest to the highest. Each set was presented individually for evaluation. For the bitter taste ranking, the evaluation was performed under red light to minimize the stimulus error from the effect of color from the grapefruit juice. The purpose of the third part of the screening was to check the ability of the panelists to adequately describe perceived aroma of common food or ingredient samples. The samples, contained in 10 closed coded vials with a paper strip saturated with different extracts were given to the judges.

Figure 4.1. Screening

The panelists were instructed to sniff from the vial and describe the perceived aroma of the samples. The identity of the samples presented was: vanilla extract (Mixim Labs., Calle Sur No. 6 P.O. Box 3, Naucalpan, Mexico, Mexico); smoke flavor (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); artificial butter extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); cinnamon essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); oak essential oil extract (Robertet de Mexico, S.A. de C.V., Año de Juarez No. 65, Granjas San Antonio, 09070, Mexico City, Mexico); artificial almond extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); black pepper essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); artificial caramel extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); and artificial banana extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico). The final section of screening consisted of 2 different sets of samples coded with random 3 digit numbers. The participants were asked to sniff the first set of samples allowing time for rest after each sample, then sniffing the second set of samples, match the samples from the second set with the first one, writing down their corresponding number, and assigning to them a term from a provided list that best describes each sample. The identities of the samples were eucalyptus essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); clove essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); white vinegar; rose essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico

City, Mexico); artificial garlic extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); peppermint essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); artificial banana extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); artificial hazelnut extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); artificial strawberry extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico), and nutmeg essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico). After the screening test, the selected participants were informed in a detailed manner of the project, and briefly interviewed for their attitude towards rum and their availability for being part of the project. All participants were required to be over 21 years of age, and having good health. Since there was no other incentive for their participation in the project, the main criteria for the selection were their willingness and commitment to participate, and their availability to come to all the orientation, training and evaluation sessions.

4.2.2 Orientation

The orientation session took place in a conference room with a large central table. The session started at 4:30 PM and lasted about one hour. All the selected panelists were asked to come to an orientation session. There was a brief presentation on the objectives of the study, the definition and production of rum, the perception of aroma, the sensory technique and the methodology involved in this study. They were informed of the development of training and evaluation sessions, including dates, time, and location. During orientation all the participants were informed of the importance of their

commitment to the project and their presence during all the group-training sessions. Then there was a group discussion where they agreed on the best times for the group training sessions, and all their questions about the project were answered. Each of them was provided with copies containing all the information discussed during orientation including an overview of the project, the perception of aroma, and a work schedule.

Figure 4.2 Orientation Session.

4.2.3 Group Training Sessions

The objective of the first part of the training session was to develop an ability for the panelists to describe objectively and precisely the aroma of different samples. All the panelists were asked to be present during all training sessions, all the sessions started at

4:30 PM. The panelists were ask to work individually and in silence during evaluation, and to share openly their responses during discussions, paying attention to the responses of other panel members and checking the samples for new descriptors as the others present them.

During the first and second training sessions the panelist were presented with different samples contained in capped plastic cups. They were also given an additional cup filled with toasted coffee beans for neutralization. The panelists were instructed to smell each sample with short deep sniffs, write down the descriptors in the order they were perceived to describe the aroma/odor of the sample. Then they were asked to give a brief definition for each descriptor. In between samples they were required to sniff themselves (the collars of their shirts) the coffee beans cup, and themselves again, then rest for about 1-2 minutes, and proceed to the next sample. After all the panelists completed the evaluation, they were encouraged to share their responses with the group, and they had a brief discussion on what they perceived. Finally they were informed on the true identity of the samples and asked to smell all the samples again. The first session lasted about 1 hour, and the samples used were: (1) pureed pealed golden delicious apple; (2) fried apples with cinnamon and sugar; (3) liquid smoke (The Colgin Companies, P.O.Box 7779. Dallas TX. 75209); (4) ground pecans; (5) cane syrup (The C.C. Steen Syrup Mill Inc. P.O. BOX 339. 119 N. Main St. Abbeville LA. 70510); (6) molasses (The C.C. Steen Syrup Mill Inc. P.O. BOX 339. 119 N. Main St., Abbeville LA. 70510); (7) grapefruit juice; (8) mix of vanilla/butter extracts (McCormick & Co. Inc. Hunt Valley, MD. 21031-1100); (9) Dark Rum: (10) Light Rum. For the second training session the samples used were (1) butterscotch extract (Firmenich de Mexico, Luisiana No. 80,

Napoles, 03810, Mexico City, Mexico), (2) Caramel /almond extract (1:1) (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico), (3) Mixed pureed fresh banana and pealed Golden Delicious apple, (4) Mixed ground clove and black pepper, (5) Mixed oak wood extract (Robertet de Mexico, S.A. de C.V., Año de Juarez No. 65, Granjas San Antonio, 09070, Mexico City, Mexico) with smoke flavor (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) (6:1), 6) Vanilla/pecan extracts (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) (1:1), (7) Diet Pepsi (Pepsico, Inc.), (8) Minced fresh mushrooms, (9) Red Wine Vinegar. Spice Islands Gourmet Premium Red Wine Vinegar, containing red wine vinegar, water and sulfites (Burns Philip Food Inc. San Francisco Ca. 94108), (10) Cough Syrup: Tanafed Suspension with strawberry/banana flavors. It was contained in a plastic bottle (Horizon Pharaceutical Corporation Roswell, GA, 30076), (11) Floral Perfume extract (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico), and (12) Grapefruit Essential Oil. Expressed oil from fresh white grapefruit. The samples were presented in capped plastic cups. The second training session lasted 1hr 15 min.

In the third training session, the panelists were provided with 4 sets of samples with 3 samples each. They were asked to evaluate the samples as in sessions 1 and 2, and provide terms and their descriptions of terms to describe each set of samples. Then after a brief group discussion, the panelist were asked to agree on some terms that would be used to describe each set of samples, and rank the samples according to the intensity of each of the selected terms. The identity of the samples was: set (1) Molasses: Steen's Home Style Molasses: Undiluted, diluted 1:1 with water, and diluted 1:3 with water; set (2)

Mushroom/pecan/almond. 5 medium mushrooms were minced with ca. 20 ml water, 3 drops of artificial pecan extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) and 3 drops artificial almond extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) were added. The mixture was expressed and filtered with cheese-cloth. Dilutions at 100%, 50% and 33% were prepared; set 3) Vanilla/apple/smoke: a Golden Delicious Apple was cut in pieces and minced with 20 ml of water. 5 drops of vanilla extract: pure natural vanilla extract (3x) (Mixim Labs. Calle Sur No. 6 P.O. Box 3, Naucalpan, Mexico, Mexico), and 1 drop of smoke flavor (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico)) were added. The mixture was mixed thoroughly and filtered using cheese-cloth to obtain a clear liquid. Dilutions at 100%, 50% and 33% were used; (4) Rum: Ron Rico Extra Smooth Premium Gold Label. Dilutions at 100%, 50% and 33% were presented for evaluation. This session lasted for 1.5 hrs.

In the fourth training session a single rum sample was provided. The panelists were asked to evaluate the sample as in previous sessions, but they were encouraged to provide more terms and their corresponding definitions for each of them. They were then asked to sniff the sample several times and at different distances from the cup; (i.e., at 1 inch from the rim of the cup, at the rim of the cup, with the nose inside the cup, and sniff again with the nose inside the cup after swirling). The panelists were then asked to share their responses and reevaluate the samples again. Finally they had to agree on the terms that they were able to perceive. For the fifth training session, 35 samples contained in glass vials were given to the panelists for evaluation. They were asked to evaluate the samples by very quick sniffs, then they were asked to record terms for describing the

aroma and a brief definition for it. After they had evaluated all the samples a group discussion took place to agree on the term(s) to describe each sample. The identity of the samples used was: (1) ground allspice (Astor products Inc. Jacksonville, Florida, 32203) in a capped plastic cup; (2) anise seeds (Burns Philips Food Inc., San Francisco, Ca, 94108) in a capped plastic cup; (3) clove essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (4) black pepper extract (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (5) cinnamon essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16, Granjas Esmeralda, 09810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (6) artificial butter extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (7) artificial butterscotch extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial ; (8) artificial caramel extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (9) vanilla extract (Pure natural vanilla extract (3x) from Mixim Labs., Calle Sur No. 6 P.O. Box 3, Naucalpan, Mexico, Mexico) in paper strips contained in a closed glass vial; (10) Burnt sugar: 5 grams sugar with 5 ml water contained in glass vials, and cooked for different times in a microwave oven (1, 2, 2.25, 2.5, 2.75, 3, 3.5, and 4 minutes); (11) eucalyptus essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (12) peppermint essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810,

Mexico City, Mexico) in paper strips contained in a closed glass vial; (13) spearmint-flavored chewing gum (Wrigley's Company, Chicago, IL, 60611); (14) Artificial pecan extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (15) artificial hazelnut extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (16) artificial almond extract (McCormirck & Co., Inc., PO. Box 208 Hunt Valley, MD, 21030-0208) in paper strips contained in a closed glass vial; (17) finely crushed peanuts in a capped plastic cup; (18) artificial strawberry extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (19) artificial banana extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (20) artificial apple extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial; (21) artificial green apple extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in paper strips contained in a closed glass vial: (22) oak wood essential oil (Robertet de Mexico, S.A. de C.V., Año de Juarez No. 65, Granjas San Antonio, 09070, Mexico City, Mexico) in paper strips contained in a closed glass vial; (23) expressed juice of ground spinach; (24) expressed juice of ground cabbage; (25) ground fresh green beans; (26) mango type natural flavor (Flavors of North America Inc. 525 Randy Rd. IL, 60188) in paper strips contained in a closed glass vial; (27) expressed juice of minced mushrooms; (28) liquid smoke (The Colgin Companies, P.O.Box 7779. Dallas TX. 75209) in paper strips contained in a closed glass vial; (29) cough syrup (Tanafed Suspension with strawberry/banana flavors. Horizon

Phamaceutical Corporation Roswell, GA, 30076) in a capped glass vial; (30) paint thinner: few drops in a closed glass vial; (31) ethanol: few drops contained in a closed glass vial; (32) rubbing alcohol (isopropyl alcohol): a few drops contained in a capped glass vial; (33) methanol: a few drops contained in a capped glass vial; (34) propanol: a few drops contained in a capped glass vial; (35) Isoamyl alcohol: a few drops in a capped glass vial; (36) ethyl butyrate: a few drops in a capped glass vial; (37) new closed plastic bottle. This session lasted for 1hr 45 minutes.

For the sixth session the panelists were given 18 samples, they were asked to evaluate the samples as conducted in previous sessions, and to provide a larger number of terms with their definition for each sample. After they had finished, a group discussion took place. The identity of the samples used was: (1) artificial pecan extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (2) burnt sugar; (3) ground fresh green beans; (4) expressed juice of minced mushrooms; (5) cinnamon essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); (6) eucalyptus essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); (7) allspice (Astor products Inc. Jacksonville, Florida, 32203); (8) artificial hazelnut extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (9) artificial butterscotch extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (10) clove essential oil (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); (11) artificial banana extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (12) vanilla extract; (13) artificial apple extract (Firmenich de Mexico, Luisiana No.8,

Napoles, 03810, Mexico City, Mexico); (14) ethyl butyrate; (15) ground pecans; (16) Maple syrup (100% pure maple syrup, Maple Grove Farms Vermont); (17) clover honey; (18) soy milk. The samples were placed in glass vials. For those samples that contained pure extracts a paper strip with a few drops of the correspondent extract was placed in the vial. For session 7 the panelists were asked to evaluate samples as in session six. The identity of the samples provided was: (1) peppermint extract; (2) artificial strawberry extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (3) artificial almond extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (4) black pepper extract (AMCO Internacional, S.A. de C.V. Trigo No. 16 Granjas Esmeralda, 09810, Mexico City, Mexico); (5) vanilla extract (Pure natural vanilla extract (3x) from Mixim Labs., Calle Sur No. 6 P.O. Box 3, Naucalpan, Mexico, Mexico): (6) artificial caramel extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico); (7) Natural Oak Flavor (Wild Flavors Inc. 1261 Pacific Avenue, Erlanger, KY 41018); (8) paint thinner. A few drops of the extracts were placed on a paper strip in a capped glass vial. The second part of the session consisted on the evaluation of a single rum sample contained in a capped plastic cup. The panelists were encouraged to provide as many terms as they could come up with and provide a definition for each of them. At the end of the session they shared their responses, reevaluated the sample, and created a pooled list of terms and definitions. This session lasted for 1hr 15 min. For sessions 8, 9 and 10, different rum samples were given to the panelists in capped plastic cups, 2-3 samples per session. The panelist were asked to evaluate each sample as in previous sessions, they were reminded to sniff at different times and distances from the cup. They were encouraged to provide a list of terms as

extensive as they could, and to give a definition for each term. Final discussion of the terms assigned took place. Then the panelists were encouraged to sniff the samples again to verify the presence of each of the assigned terms. The terms that all the panelists perceived were kept on a final pooled list of terms with definitions. Training sessions 8, 9 and 10 lasted about 1 hr 15 min each. During the course of the group training and the development of the list of attributes for describing rum, records of the attributes detected by each panelist were taken as well as of those specific attributes some panelists had problems detecting.

Figure 4.3 Group Training. Development of a List of Terms for Describing Rum Aroma.

The final part of the group training consisted of presenting the panelist all the possible references for the evaluation of rum. They were asked to decide whether the aroma perceived in the references matches the aroma they perceived in rum. Different options for some references were presented, and references were presented in 40% ethanol solutions where possible. This is to mimic the whole complex rum sample. As a group the panelists concluded on which references would be most appropriate for the evaluation. They also agreed on the final definitions that would be used for describing each term for rum aroma.

4.2.4 Selection and Preparation of Standards and Scale Setting

After the panel had approved and agreed on the proposed references for all the rum attributes, the reference standards were prepared. For doing these, different solutions were prepared and presented to the panel. The panel agreed that ethanol based standards would best mimic the perceived complex aroma sensations in rum; therefore, the standards were prepared in 40% (v/v) ethanol (80 Proof) solutions, where possible. Different concentrations of each reference were prepared for every corresponding attribute. Once a wide array of concentrations were prepared, a group of 3 expert sensory analysts selected the concentrations that will be included as standards and assigned intensity values to them.

The creation of the scale was done considering that the possible maximum intensity for each attribute will have a value of 15 on a 15 cm line scale. All the rum samples were evaluated and compared against each set of standard solutions to determine the dilutions and intensity values that would be used as references.

All the standards were contained in glass vials unless otherwise specified. A volume of 5 ml was used in all glass vial standards. Ten samples of each of the standards were prepared. This allowed having fresh standard samples required for training and product evaluation. The standards were periodically checked and replaced with new vials as needed to make sure the volatiles are present sufficiently. Constant checking on the standards was a critical part as some of them were not stable under normal room storage conditions.

Figure 4.4. Reference Standards Preparation.

Caramel. It was defined as the aroma characteristic associated with caramelized sugar, and was prepared using artificial caramel extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico) in a 40% ethanol (v/v) solution.

Table 4.1 Preparation of References for Caramel Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
50 µl	4
75 µl	6
100 µl	8
120 µl	12

Medicinal. It was defined as a disinfectant-like aroma. A stock solution of 6.316g/l of Thymol was prepared in 40% ethanol.

Table 4.2 Preparation of References for Medicinal Aroma

Amount	Intensity on the scale
1/70 stock solution in 40% ethanol solution (90ppm)	3
1/50 stock solution in 40% ethanol solution (120ppm)	5
1/10 stock solution in 40% ethanol solution (631.6 ppm)	9

Butterscotch. It was defined as the aroma characteristic of butterscotch candy, and was prepared using artificial butterscotch extract containing vainillin, diacetyl, benzodihydro propane, valeric acid, butyric acid, ethyl formate, ethyl acetate, ethyl butyrate, pyrolognous acid, methyl ciclobentenotone. Dilutions were made in a 40% ethanol (v/v) solution.

Table 4.3 Preparation of References for Butterscotch Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
15 µl	8
30 µl	10
50 µl	12

Honey. It was defined as the sweet aroma of clover honey, and was prepared using pure clover honey (Deep South Products Inc. P.O. Box 1448 Fitzgerald, GA) in ethanol (40%).

Table 4.4 Preparation of References for Honey Aroma

Amount	Intensity on the scale
10g and 50 ml (40%ethaol)	3
25g and 41.6 ml (40% ethanol)	6
35g and 35ml (40% ethanol)	12
Pure honey (undiluted)	15

Vanilla. Defined as the aroma characteristic of vanilla beans. Pure natural vanilla extract (3x) from Mixim Labs (Calle Sur No. 6 P.O. Box 3, Naucalpan, Mexico, Mexico) in 40% ethanol (v/v) solution was prepasred.

Table 4.5 Preparation of References for Vanilla Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
75 µl	8
150 µl	10
300 µl	12

Woody. Defined as the aromatic characteristic of oak wood pieces. A stock solution of 10g of dried oak wood shavings in 100 ml of ethanol (40% v/v). The solution was steeped for 48 hours and filtered using cheese cloth.

Table 4.6 Preparation of References for Woody Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
5 ml	5
10 ml	10
20 ml	13
Stock (undiluted)	15

Fruity (artificial). Defined as an aroma associated with fruity characteristics that is perceived as artificial, such as the aroma of a bag of mixed-candy. Prepared using ethyl butyrate in 40% (v/v) ethanol solution.

Table 4.7 Preparation of References for Fruity-artificial Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
1 µl	2
2 µl	4
3 µl	6
5 µl	10

Almond. Defined as the aroma of freshly crushed almonds. Prepared using McCormick Imitation Almond extract, containing water, alcohol (36%), and benzaldehyde (McCormick &Co. Inc. Hunt Valley MD). Dilutions were made in a 40% (v/v) ethanol solution.

Table 4.8 Preparation of References for Almond Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
100 µl	1
200 µl	2
500 µl	5

Plastic. It was defined as the aroma characteristic of plastic containers, and was prepared using a new vinyl inflatable tire. The vinyl was cut to squares of ca. 1cm length. 16g of vinyl squares were soaked in 50 ml 40% ethanol solution.

Table 4.9 Preparation of References for Plastic Aroma

Amount of solution	Intensity on the scale
25 ml solution stepped for 5 h and 25 ml ethanol (40%)	5
Vinyl solution stepped for 5 h	8
Vinyl solution stepped for 24 h	10

Cinnamon. It was defined as the aroma of ground cinnamon bark, and was prepared using McCormick ground cinnamon (McCormick & Co. Inc. Hunt Valley MD). A stock solution of 1g ground cinnamon in 50 ml 40% ethanol (v/v) was prepared.

Table 4.10 Preparation of References for Cinnamon Aroma

Amount in (40% ethanol)	Intensity on the scale
10 ml stock solution	3
20 ml stock solution	5
30 ml stock solution	7
Undiluted stock solution	8

Butter. It was defined as the aroma of fresh unsalted full-fat butter, and was prepared using McCormick imitation butter flavor, containing water, propylene glycol, butyric acid, and other organic acids, diacetyl and other ketones, ethyl propionate and other esters, vainillin, and FD&C yellow #5 (McCormick &Co. Inc. Hunt Valley MD). Dilutions were made in 40% ethanol solution.

Table 4.11 Preparation of References for Butter Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
200 µl	3
400 µl	5
600 µl	7

Ethanol. It was defined as the pungent aroma characteristic of pure ethanol. Dilutions of ethanol in water were prepared.

Table 4.12 Preparation of References for Ethanol Aroma

Amount of ethanol (v/v)	Intensity on the scale
40%	4
60%	6
80%	12
100%	15

Green apple. It was defined as the aroma of green apples, and was prepared using green apple artificial extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico). Typically containing 6-hexanol and cis-3 hexenil acetate was diluted in 40% ethanol.

Table 4.13 Preparation of References for Green Apple Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
1 µl	5
2.5 µl	8
3.75 µl	10

Smoke. It was defined as the aroma characteristic of smoke or smoked products, and was prepared using Colgin Liquid Smoke. Containing water, natural hickory smoke flavor, vinegar, molasses, caramel color and natural flavorings (The Colgin Companies, P.O.Box 7779, Dallas, TX., 75209). Dilutions in 40% ethanol (v/v) were prepared.

Table 4.14 Preparation of References for Smoke Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
50 µl	2
150 µl	5
500 µl	8
Liquid smoke	15

Nutty. It was defined as the aroma of fresh ground pecans, and was prepared using artificial pecan extract (Firmenich de Mexico, Luisiana No. 80, Napoles, 03810, Mexico City, Mexico). Dilutions were made in 40% ethanol (v/v).

Table 4.15 Preparation of References for Nutty Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
5 µl	2
10 µl	3
25 µl	4.5

Cardboard. Defined as the characteristic aroma of paper or cardboard. Prepared using corrugated cardboard pieces of ca. 1 cm length. A stock solution was prepared with 40g of cardboard pieces soaked in 100 ml ethanol (40% by vol.) for 20 h.

Table 4.16 Preparation of References for Cardboard Aroma

Amount in 50ml (40% ethanol)	Intensity on the scale
25 ml	3
37.5 ml	5
Stock (undiluted)	10

Isopropanol. Defined as a chemical aroma foreign to rum. Prepared using 70% isopropyl alcohol solution (by vol.) (Top Care 70% isopropyl alcohol, Topco Associates Inc. Skokie, IL). Dilutions were prepared using water.

Table 4.17 Preparation of References for Isopropanol Aroma

Amount	Intensity on the scale
15%	4
30%	7
50%	10

Prune-like. Defined as the aroma characteristics of dried fruit such as prunes and raisins. Prepared using Sunsweet Bite Size Pitted Prunes. Dried Plums (Sunsweet Growers Inc. Yuba City, CA). 10g plums and 50ml (40% ethanol by vol.) were pureed and filtered with cheese cloth, the filtered liquid was recovered, and was assigned an intensity of 5 on the scale. The second reference was prepared using 20g of prunes pureed with 50 ml of ethanol (40% by vol.), the obtained pureed was assigned an intensity of 8 on the scale. The third reference was 3 whole bite size pitted prunes in a covered plastic cup, the assigned intensity on the scale was 12.

Pineapple. Aroma characteristic of canned pineapple. Prepared using Dole Pineapple Slices in its Own Juice.(Dole Co). Dilutions of the decanted liquid were made using 40% ethanol (by vol.) solution.

Table 4.18 Preparation of References for Pineapple Aroma

Amount in 50 ml (40% ethanol)	Intensity on the scale
0.5 ml	3
0.75 ml	4
1 ml	5

Pepper. Defined as the characteristic aroma of ground pepper. Prepared using ground white pepper (Deep South. Deep South Blenders Inc. Metaire, LA). Dilutions were prepared with 40% ethanol (by vol.) solution.

Table 4.19 Preparation of References for Pepper Aroma

Amount in 50 ml (40% ethanol)	Intensity on the scale
0.5 ml	3
0.75 ml	4
1 ml	5

Banana. It was defined as the aroma of a fresh-ground ripened banana, and was prepared using mashed ripened banana. A stock solution was prepared using 20 g of banana puree dissolved in 100 ml (40% ethanol) solution.

Table 4.20 Preparation of References for Banana Aroma

Amount of stock solution in 50 ml (40% ethanol)	Intensity on the scale
20 ml	3
30 ml	5
undiluted	8

The banana references were stored in a freezer. The references could be maintained in refrigeration and used the same day.

Ocean-like. It was defined as an aroma characteristic of sea breeze, and was prepared by blending 1.5 g roasted seaweed (Nagai Nori Co, Ldt. 2-10 Ohmori-Naka 3Chome Ohta-ku Tokyo 143 Japan) in 200 ml of 40% ethanol. That stock solution was diluted in 40% ethanol (v/v) to produce the different reference intensities.

Table 4.21 Preparation of References for Ocean-like Aroma

Amount of stock solution in 50 ml (40% ethanol)	Intensity on the scale
0.5	3.5
1	5
2	7

The reference for pungent was pure clover honey, for sweet aroma was brown sugar, and for sour (acid) was white vinegar. These standards were contained in plastic capped cups with ca. 5 ml each. No scaled reference for metallic aroma could be

produced given the instability of the samples. However, “metallic” aroma is defined as; the aroma characteristic of metals or metal pieces such as keys, coins, and cans, and the reference for the aroma were pulverized iron vitamin pills.

4.2.5 Individual Training Sessions. Use of References and Scales

The second part of the training consisted in individual training sessions. The panelists were required to sniff all the references (Figure 4.5) that were used for the evaluation of rum samples. The objective was that all panel members could become used to and memorize the aroma of the references and the scale to be used for product evaluation.

Figure 4.5 Standard References.

For the individual training sessions, the panelists were allowed to come at their most convenient time, but avoiding the times immediately before and after meals. They were provided with all the standards, and coffee beans cups for aroma neutralization. The panelists were asked to evaluate the set of standards for one attribute at a time, starting with that standard with the lowest intensity value, and continuing with the one with increasing intensity value. They were instructed to open the vials and take a with short deep sniffs, tightly close the vial, then smell themselves, and the coffee beans cup before moving to the next sample. Panelists were asked to evaluate the set of standards for all the attributes every session, and to pay more attention especially on those set of standards which corresponded to attributes they had problems detecting in the rum samples during the group training. This part of the training consisted of at least 9 sessions that lasted 30 minutes to 45 minutes each, and took place in individual evaluation booths.

4.2.6 Panel

Of a total of 19 initial participants, 12 panelists finished all the group and individual training required. The final composition of the panel consisted of 6 male and 6 female participants, ages 22 to 34, 75% of them were Hispanic, and 11 of them were students of different departments at Louisiana State University, the other one was a researcher.

4.2.7. Selection of Rum Samples for Evaluation

An extensive search over the internet, from rum literature, and in local markets was done in order to select an array of 9 commercial rum samples produced from different raw materials, using different processing protocols, produced in different regions, and that are of all price ranges. The selected rum samples (Figure 4.6 and Table

4.22) represent the different types and qualities of rum evaluated in this study. The selections include rums with no added spices or flavoring, as only true pure rum aroma was intended to be evaluated. An additional experimental sample was also used for evaluation. The experimental sample was made form molasses, and fermented over 72 hr. The distillation method used was a continuous system with two columns, were the initial "head" and final part "tail" of the distillation were removed. The raw distillate was aged for 3 months with toasted American oak chips under accelerated conditions. After aging it was stored in glass containers with plastic caps.

Figure 4.6 Rum Samples for Evaluation

Table 4.22 Rums Samples Selected for Evaluation

Code	Name	Origin	Raw Material	Distillation	Proof	Aging time	Aging conditions	Mixing	Refining	Type
174	Mount-Gay Eclipse	Barbados	Molasses	Two types: Continuous and Pot-stills up to 96% EtOH	80	Not specific (blend)	Charred American oak casks previously used for Bourbon	From different distillation methods/ aged for different lengths		
217	St. James Extra old	Martinique	Cane juice/dunder	Pot-stills	84	>10	Limousin oak casks			Dark
348	Barbancourt	Haiti	Cane juice	Pot-stills double distillation	86	8 years	Cognac method in Limousin oak casks			Amber
565	Experimental		Molasses	2 column continuous	80	~3 months	American oak chips 35ºC			
599	El dorado Superior	Demerara	Local molasses	Continuous and pot-stills	80	12	45 gallon casks	Continuous and batch distillations		Dark
722	Castillo Gold	Puerto Rico	Molasses	Continuous	80	≥ 3 years				Amber
796	Flor de Cana Dry	Nicaragua	Molasses	5 column continuous	80	4 years	Slow-aged (undisturbed) barrels used once			White
813	Ronrico Extra Smooth Premium Gold Label	Puerto Rico	Molasses	Continuous	80	>1	American oak casks			Amber
975	Myers	Jamaica	Molasses/ dunder	Pot-still	80	>5	White oak			Dark
983	Ron Bacardi Dry	Puerto Rico	Molasses	Continuous	80	>1	American oak casks		Double charcoal filter	White

4.2.8 Product Evaluation

A total of 9 commercial rums and an experimental samples were evaluated. For each session of evaluation of rum samples, the panelists were provided with all references used during training as well as 2 vials of two different rum samples (Figure 4.7). The rum samples were placed in 30 ml closed glass vials coded with 3 digit random numbers with 10 ml of rum sample.

Figure 4.7 Evaluation of Rum Samples.

For the evaluation of samples the judges were provided with a ballot containing all the attributes. The attributes that needed to be quantified had a 15 cm scale marked with decimal fractions; on the scale there were also marks for the intensities of the references provided. See appendixes for an example.

Before the evaluation, each panelist was reminded of the evaluation procedure as well as the use of the scale. Additional instructions were placed in each evaluation booth. Evaluation took place under red right to minimize the effect of color on the samples judging. The room was air-conditioned with positive air flow to prevent accumulation of aromas. Panelists evaluated the samples at the most convenient time for them, avoiding times before and after meals, and while in good health conditions. Each sample was evaluated 3 times in random order. A total of 15 sessions were required to finish all the evaluations. Each evaluation session lasted for approximately 40 minutes, resting for about 5 minutes in between samples. After each session was completed, the evaluation ballots were then checked for errors or missing data. Panelists were allowed to evaluate another session after 15 to 20 minutes. The first ten sessions were completed after 3 weeks. Some panelists were able to complete sessions 11-15 immediately after the previous ones, while others completed them 4 weeks after.

A total of 22 attributes were evaluated using the 15-cm line with the provided references. The evaluation of the presence of the chemical aroma sensations; pungent, sweet, and sour (acid), and the metallic aroma in the rum samples consisted on a yes/no section of the evaluation ballot.

4.2.9 Analysis of Data

The panelists evaluated all the samples and assigned intensities to each of the aroma attributes in every sample by marking a vertical line on 15 cm scale. The perception of chemical sensations (pungent, sweet, sour (acid)), and metallic aroma in the rum samples was analyzed by comparing the frequencies. This was done by assigning a number 1 to a positive detection and 0 when the panelist did not perceived the sensation. The obtained data was analyzed using analysis of variance (ANOVA) (SAS Institute Inc. 1999-2001) to check for significant differences in the intensities of each of the selected aroma attributes in the rum samples. The variation in the responses for the intensities of the aroma attributes was analyzed. The aim was to determine the reliability and uniformity of the judgment criteria used by the panelist. This is also a good parameter to determine the quality of the training sessions.

Multivariate Analysis of Variance (MANOVA) was performed to determine overall differences in the rum samples based on all the aroma attributes used to describe them. Further descriptive discriminant analysis was used to determine if the panelists were able to discriminate the rum samples using the selected attributes. The attributes that appear to have a major influence in the discrimination of the rum samples (ocean-like, cardboard, artificial-fruity, honey, caramel, woody, cinnamon, vanilla, and buttery) were selected.

The data for the selected attributes was used for further data analysis by Principal component analysis (PCA) (SAS Institute Inc., 1999-2001). The PCA plots that indicate the rum samples that are discriminated by the panelist from the group of samples were

made. The product-attribute plots were used to determine the aroma attribute that is responsible for the discrimination. All statistical analyses were conducted at alpha=0.05.

4.3 Results and Discussion

The attributes selected for the training and evaluation sessions were based on the consensus of the panel, and included the following terms woody, ethanol, buttery, butterscotch, caramel, honey, plastic, smoke, vanilla, almond, cinnamon. artificial-fruity, banana, prune, medicinal, pineapple, pepper, green apple, nutty, isopropanol, cardboard, ocean-like, metallic, and the chemical aroma sensations; pungent, sweet, sour. Some of the terms developed by the panel that did not reach consensus include berries, chemical, clove, coconut, grape, green, peppermint, clove, and coconut.

4.3.1 ANOVA Results

The data for the intensity of the aroma attributes in all the rum samples was analyzed using analysis of variance (SAS Institute Inc., 1999-2001) to determine if there were significant differences in the judgments for the intensity. The deviations in judgment from the mean intensity assigned to each attribute, and every sample were reported (Tables 4.23, 4.24, and 4.25).

The rum with the longest aging time (12 years) was perceived significantly more "woody" (9.53) than light rums aged for 2 years such as the two rum samples from Puerto Rico (6.28 and 5.98). The lowest woody intensity (5.10) was assigned to a white rum from Nicaragua aged for 4 years; it had a significantly lower woody aroma as compared with a rum from Martinique aged for 10 years, a rum from Demerara aged for 12 years, and a dark rum from Jamaica aged for at least 5 years. From these observations

we can conclude that the aging type as well as the time of rum (i.e., dark vs. amber or white) are related to the intensity of the "woody" aroma.

Table 4.23 Intensity of the Aroma Attributes (Part I) [a]

	Woody	Ethanol	Buttery	Butter-scotch	Caramel	Honey	Plastic	Smoke	Vanilla	Almond	Cinnam.
174	7.44^{ABC} (3.94)	8.42^{BA} (3.94)	3.71^{A} (2.39)	4.68^{A} (3.44)	4.49^{BA} (2.58)	2.91^{BCA} (2.40)	2.36^{A} (2.50)	1.25^{BA} (1.57)	5.91^{BA} (3.44)	1.44^{A} (1.65)	1.68^{A} (1.69)
217	8.4^{BA} (4.16)	8.85^{BA} (3.33)	2.69^{A} (2.42)	2.82^{A} (2.84)	4.32^{BA} (2.71)	3.68^{BAC} (3.10)	2.68^{A} (2.76)	1.54^{BA} (1.65)	4.48^{B} (3.16)	1.37^{A} (1.49)	2.32^{A} (1.95)
348	6.42^{BCA} (3.76)	7.91^{BA} (3.34)	2.66^{A} (2.01)	3.51^{A} (2.96)	4.24^{BA} (2.96)	2.59^{BAC} (2.46)	2.75^{A} (2.89)	1.67^{BA} (1.55)	4.49^{BA} (3.27)	1.28^{A} (1.55)	1.26^{A} (1.42)
565	7.87^{BCA} (4.17)	6.65^{B} (3.62)	3.64^{A} (2.57)	4.91^{A} (3.25)	5.63^{A} (3.09)	4.43^{BA} (3.08)	2.75^{A} (2.87)	1.24^{BA} (2.58)	7.05^{A} (3.83)	1.6^{A} (1.87)	1.83^{A} (1.75)
599	9.53^{A} (3.89)	8.18^{BA} (3.29)	2.31^{A} (2.01)	3.61^{A} (3.08)	4.77^{BA} (2.63)	3.59^{BAC} (3.11)	2.33^{A} (2.54)	1.91^{A} (2.03)	4.99^{BA} (2.98)	1.54^{A} (1.73)	2.22^{A} (1.81)
722	7.48^{BCA} (4.17)	7.93^{BA} (2.35)	2.46^{A} (2.15)	2.92^{A} (2.66)	4.03^{BA} (2.71)	3.31^{BAC} (2.25)	2.14^{A} (2.66)	0.7^{B} (2.71)	4.57^{BA} (2.25)	1.4^{A} (2.27)	1.49^{A} (0.66)
796	5.10^{C} (3.5)	8.8^{BA} (3.48)	2.19^{A} (2.25)	2.87^{A} (2.85)	3.14^{B} (2.77)	1.72^{C} (1.67)	2.3^{A} (2.17)	0.72^{B} (1.18)	4.05^{B} (2.88)	1.03^{A} (1.29)	1.15^{A} (1.37)
813	5.98^{BC} (3.64)	9.54^{A} (3.48)	2.11^{A} (1.79)	3.09^{A} (3.48)	3.92^{BA} (3.12)	2.39^{BC} (2.55)	1.86^{A} (2.13)	0.89^{BA} (1.11)	4.69^{BA} (3.06)	0.98^{A} (1.24)	1.30^{A} (1.55)
975	8.76^{BA} (4.49)	8.38^{BA} (3.33)	2.76^{A} (2.37)	3.41^{A} (3.46)	5.22^{BA} (3.07)	4.59^{A} (4.11)	3.71^{A} (4.12)	1.52^{BA} (1.78)	5.43^{BA} (3.37)	1.41^{A} (1.67)	2.18^{A} (1.81)
983	6.28^{BC} (4.11)	8.08^{BA} (3.12)	2.06^{A} (1.92)	2.53^{A} (3.07)	3.28^{B} (2.53)	2.32^{BC} (2.24)	1.99^{A} (2.09)	1.18^{BA} (1.44)	3.81^{B} (3.39)	1.18^{A} (1.53)	1.25^{A} (1.62)

[a] Numbers in parenthesis are the standard deviations. For each column, means with the same superscript letter are not significantly different ($p \geq 0.05$).

The highest ethanol aroma intensity (9.54) corresponded to that of an amber rum from Puerto Rico distilled in continuous distillation and aged for over 1 year. The second highest intensity was assigned to a rum from Martinique, that had the highest ethanol content. It was expected that the highest ethanol concentration would be related to the perception of ethanol aroma, but also a very light rum that does not have a complex

aroma mixture may allow for a more obvious detection of ethanol. The perception of lowest ethanol intensity (6.65) was in the experimental sample. A possible explanation for this was that it possessed a very complex aroma.

There was not a significant difference in the intensity of the "buttery" aroma of the samples, however the highest intensity (3.71) was perceived in a rum from Jamaica made from molasses, pot-stilled and aged for over 5 years. The lowest intensities (2.06, 2.11, and 2.19 respectively) were assigned to a white rum from Puerto Rico, an amber rum from Puerto Rico, and a white rum from Nicaragua, all of them made from molasses and distilled in continuous systems, those samples typically have short fermentation and aging times.

The experimental sample was significantly higher in the "caramel" aroma intensity (5.63), followed by a rum made in Jamaica, from molasses and pot stilled, and a rum from Demerara made from molasses with a blend of continuous and batch distillations and aged for 10 years (5.22, and 4.77, respectively). Lower intensities were perceived in a white rum, and amber rum both from Puerto Rico (3.28, and 2.89). The lowest "caramel" aroma intensity belonged to a white rum from Nicaragua. Similar tendencies were observed for the "honey" aroma attribute, where the dark Jamaican rum was perceived as having a statistically higher value than those of the white rums (1.72, and 2.32 for the Nicaraguan and Puerto Rican rums, respectively), as well as the rum from Haiti made from cane juice, pot stilled and aged for over 3 years (2.39). Higher "honey" aroma intensity was also observed in the experimental sample (4.43). A significantly higher "smoke" aroma was perceived in the rum from Demerara aged for 12 years, as compared to the amber Puerto Rican rum aged for over 1 year (0.7), and the

white rum from Nicaragua (0.72). The experimental sample (aged with toasted American oak chips) had significantly higher intensity of "vanilla" aroma (7.05) than those of the Puerto Rican white rum (3.81), the Nicaraguan white rum (4.05), and the rum from Haiti aged for over 3 years (4.48). The perception of "vanilla" attribute seems to be related to the aging time. However it is not aging time alone, but also aging conditions responsible for the "vanilla" aroma intensity. This is the case of the rum from Demerara aged for 12 years. A possible reason for these may be the use of uncharred barrels. As it was stated in the introduction, lignin degradation in the barrels that leads to the formation of aldehydes (such as vainillin) is higher in charred barrels as compared to uncharred ones (Nishimura, 1983). The toasting temperature also plays a role, as the temperature used increases so does the formation of those compounds. The type of oak wood used may also influence the formation of oak degradation compounds. This coincides with the idea that wood treated at higher temperatures will possess stronger vanilla notes as well as others (Francis *et al.*, 1992). It was observed that the intensity of the "vanilla" aroma in those rums aged in Limousin oak casks (the ones from Martinique and Haiti) was in general lower than that of the rums aged in American oak casks. In the perception of the almond aroma we observed the same pattern, where the amber Puerto Rican rum aged for 1 year, the white rum from Nicaragua, and the white Puerto Rican rums were given the lower intensities (0.98, 1.03, and 1.18 respectively), and while the differences were not statistically significant, the higher values corresponded to the experimental sample (1.6), and the rum from Demerara aged for 12 years. "Cinnamon" aroma also appears to be related to the aging conditions. The higher cinnamon aroma intensities were perceived in the rum from Martinique aged for over 10 years, and the rum from Demerara aged for 12

years, followed by the rum from Haiti aged for 8 years, all of them aged in Limousin oak casks using the cognac method. As expected, the lower intensities were observed in the white Nicaraguan and Puerto Rican rums (1.15 and 1.25, respectively).

There was no observed significant difference in the perception of plastic aroma, but the higher value was assigned to the Jamaican rum (3.71), followed by the experimental sample (2.75). Both samples were contained in bottles with a plastic cap, therefore that may be a possible source for the off-aroma transfer.

The rum sample having the statistically strongest "artificial fruity" aroma was the experimental sample. This sample was produced from molasses, with long fermentation times and distilled in a double column continuous system. The processing method had less controls and lacked the rectification of the composition one would expect in a commercial setting. This may have allowed for the formation of a distillate containing a larger variety of aroma compounds such as esters or their acid precursors, and in quantities larger than those in other distillates. Such composition will promote the formation of fruity-like aromas. There were not significant differences in the artificial fruity aroma of the other rums, however the weakest perceived aroma corresponded to that of the white Nicaraguan and Puerto Rican rums (1.15, and 1.25 respectively). Typically light rums are produced in short fermentation times, therefore less aroma compounds are formed, and they are distilled in continuous systems, where they are stripped of many congeners. The experimental sample was also assigned the highest banana aroma intensity (1.41), and it was significantly higher than those of the rum from Haiti made from cane juice and aged for over 3 years (0.31), as well as the ones for the

Nicaraguan and Puerto Rican white rums (0.39 and 0.31, respectively), both of them made from molasses and distilled in continuous systems.

Table 4.24 Intensity of the Aroma Attributes (Part II)[a]

	Artif. Fruity	Banana	Prune	Medici-nal	Pine-apple	Pepper	Green apple	Nutty	Isopro panol	Card-board	Ocean-like
174	2.03^{B}	0.84^{BA}	2.56^{BA}	0.91^{A}	1.57^{A}	1.12^{A}	2.64^{A}	1.58^{A}	1.72^{A}	0^{B}	0^{B}
	(2.68)	(1.62)	(3.21)	(1.01)	(1.54)	(1.08)	(2.73)	(1.29)	(2.54)	(0.0)	(0.0)
217	1.77^{B}	0.41^{BA}	3.69^{A}	1.42^{A}	1.63^{A}	1.62^{A}	1.96^{A}	1.85^{A}	2.31^{A}	0^{B}	0^{B}
	(1.84)	(1.01)	(4.04)	(1.59)	(1.73)	(1.49)	(2.29)	(1.37)	(2.67)	(0)	(0)
348	1.75^{B}	$.67^{BA}$	2.29^{BA}	1.34^{A}	1.18^{A}	1.43^{A}	1.62^{A}	1.41^{A}	2.23^{A}	2.44^{A}	0^{B}
	(1.92)	(1.24)	(3.09)	(1.64)	(1.28)	(1.1.9)	(2.21)	(1.44)	(2.31)	(2.49)	(0)
565	4.33^{A}	1.41^{A}	2.04^{BA}	1.68^{A}	1.85^{A}	1.24^{A}	2.67^{A}	1.75^{A}	1.24^{A}	0^{B}	0^{B}
	(3.12)	(2.12)	(4.17)	(3.62)	(1.66)	(1.37)	(2.15)	(1.31)	(1.98)	(0)	(0)
599	1.54^{B}	0.31^{B}	3.82^{A}	1.25^{A}	1.19^{A}	1.48^{A}	2.05^{A}	1.84^{A}	1.59^{A}	0^{B}	0^{B}
	(2.07)	(0.67)	(4.4)	(1.53)	(1.60)	(1.39)	(2.4)	(1.22)	(2.35)	(0)	(0)
722	2.13^{B}	0.52^{BA}	1.98^{BA}	1.05^{A}	1.66^{A}	1.09^{A}	2.72^{A}	1.5^{A}	1.81^{A}	0^{B}	0^{B}
	(3.17)	(1.42)	(2.5)	(1.31)	(1.65)	(0.97)	(2.37)	(1.36)	(1.97)	(0)	(0)
796	2.05^{B}	0.39^{B}	1.54^{BA}	1.58^{A}	1.74^{A}	1.09^{A}	2.44^{A}	1.26^{A}	1.92^{A}	0^{B}	2^{A}
	(2.18)	(0.7)	(1.77)	(1.59)	(1.60)	(1.23)	(2.06)	(1.27)	(1.93)	(0)	(1.83)
813	2.09^{B}	0.95^{BA}	1.19^{B}	1.44^{A}	1.20^{A}	1.15^{A}	1.98^{A}	1.18^{A}	1.55^{A}	0^{B}	0^{B}
	(2.24)	(1.53)	(1.88)	(1.70)	(1.69)	(1.16)	(2.85)	(1.51)	(1.53)	(0)	(0)
975	1.37^{B}	0.77^{BA}	3.69^{A}	1.82^{A}	1.59^{A}	1.62^{A}	2.35^{A}	1.44^{A}	1.81^{A}	0^{B}	0^{B}
	(1.79)	(1.74)	(4.01)	(2.58)	(1.9)	(1.46)	(2.82)	(1.29)	(2.39)	(0)	(0)
983	1.64^{B}	0.31^{B}	2.05	1.89^{A}	1.24^{A}	1.60^{A}	2.11^{A}	1.12^{A}	2.48^{A}	0^{B}	0^{B}
	(1.83)	(0.47)	(2.39)	(1.78)	(1.30)	(1.32)	(1.77)	(1.25)	(2.61)	(0)	(0)

[a] Numbers in parenthesis are the standard deviations. For each column, means with the same superscript letter are not significantly different ($p \geq 0.05$).

Those rums that had the higher "prune" aroma intensity were the rum form Demerara, blend of continuous and batch distillations and aged for 12 years (3.82), and a Jamaican rum distilled in pot-stills rum aged for over 5 years, both of them made from molasses. A possible reason for the high "prune" or "raisiny" aroma could be that the

wood used for maturation was not seasoned in a warm and dry climate, or it was not subjected to any additional high temperature treatment. Francis *et al*, (1992) stated that treatment of the wood using high temperatures reduces the "raisiny" notes of the wood. The prune aroma in the Demerara and Jamaican samples was statistically higher than that of the amber Puerto Rican rum aged for 1 year (1.19). The values in the Nicaraguan white rum (1.54) and the other Puerto Rican Gold rum aged for 3 years (1.98) also appeared to be low. The experimental sample was perceived as having the highest intensity for pineapple aroma among the rum samples, however the difference was not significant.

Table 4.25 Frequency of the Perception of some Aroma Sensations [a]

	Pungent	Sour	Sweet	Metallic
174	0.73^{BA}	0.36^{BA}	0.79^{BA}	0.45^{BA}
	(0.45)	(0.49)	(0.42)	(0.51)
217	0.88^{A}	0.45^{BA}	0.58^{B}	0.64^{BA}
	(0.33)	(0.51)	(0.50)	(0.49)
348	0.7^{BA}	0.45^{BA}	0.61^{B}	0.42^{BA}
	(0.47)	(0.51)	(0.5)	(0.5)
565	0.53^{B}	0.16^{B}	1^{A}	0.25^{B}
	(0.51)	(0.37)	(0)	(0.44)
599	0.79^{BA}	0.36^{BA}	0.76^{BA}	0.7^{A}
	(0.42)	(0.49)	(0.44)	(0.47)
722	0.64^{BA}	0.36^{BA}	0.82^{BA}	0.45^{BA}
	(0.49)	(0.49)	(0.39)	(0.51)
796	0.76^{BA}	0.58^{A}	0.64^{B}	0.48^{BA}
	(0.44)	(0.5)	(0.49)	(0.51)
813	0.82^{BA}	0.42^{BA}	0.67^{BA}	0.39^{BA}
	(0.39)	(0.5)	(0.48)	(0.5)
975	0.82^{BA}	0.33^{BA}	0.76^{BA}	0.45^{BA}
	(0.39)	(0.48)	(0.44)	(0.51)
983	0.64^{BA}	0.52^{BA}	0.64^{B}	0.39^{BA}
	(0.49)	(0.51)	(0.49)	(0.5)

[a] Numbers in parenthesis are the standard deviations. For each column, means with the same superscript letter are not significantly different ($p \geq 0.05$).

There were no observed significant differences in the intensities of the "medicinal", "peppery", "green apple", "nutty", and "isopropanol" aromas. The panelists perceived a slight (2.4) cardboard aroma in the rum from Barbados made from molasses, and distilled in pot-still, while the white rum from Nicaragua had an ocean-like aroma (2) (Table 4.25)

The sample more frequently perceived as having a pungent aroma was the rum from Martinique, which is the dark rum having a ethanol content of 84 %. This suggests that there may be a relationship between the alcohol content and the pungent aroma. However, the presence of other compounds in the rum may contribute to the pungent aroma either by masking the pungency of alcohol, or by increasing that perceived aroma. Dark rums have very complex aromas and are normally perceived as more pungent than white rums. White rums are in general characterized by a light taste. The rum most frequently perceived as having a "sour" aroma was the white rum from Nicaragua, followed by the white rum from Puerto Rico. The former one was statistically more frequently perceived as "sour" than the experimental sample, and this latter one having the least sour aroma perception. This may be directly related to their acid make-up. As with those short aging times, most of the acids that will normally be transformed into other chemical compounds after long maturation times (i.e., esters) may have remained intact. The aroma of the experimental sample was perceived as "sweet" by all panelists. Its sweet aroma was significantly different than that from the rums from Martinique and Haiti, both made from cane juice and distilled in pot-stills, and it was also different from that of the white rums from Nicaragua and Puerto Rico. It can be concluded that the raw material used has an effect on the perceived "sweet" aroma of the samples. Rums made

from molasses and aged for a long period are more frequently perceived as having a sweet aroma as compared to those made from cane juice. White rums typically have short aging times and lower concentration of congeners as compared to aged amber or dark rums. The aroma of rum from Demerara made from molasses and aged for 12 years was perceived as more "metallic" than the experimental sample. The experimental rum was aged by an accelerated method using American oak chips for three months. This method allowed for the extraction of large amounts of wood components into the distillate; however, it undoubtedly had the shortest aging time, and it was not aged in casks, as the other distillates were. The aroma of the experimental sample was less frequently perceived as "metallic", and it was significantly different in its' "metallic" aroma from those rums aged for longer periods. There appears to be a direct relationship between the time the distillate was maturating in the oak casks and the metallic aroma of the final rum.

Several problems contributed to the large variations in the results. One possible cause is the high complexity of the sample; rum contains about 40% ethanol, the ethanol content, as well as the general chemical composition will affect the aroma perception of specific aroma attributes. The complexity of the aroma of rum was also noticeable as seen by in the large amount of possible aroma descriptors the panelists assigned to rum. Handling such a large number of variables represented a problem for the analysis and interpretations. It also represented a problem during training and evaluation itself; the panelist had to smell the rum samples and the references for a very extended period of times to complete each session. In such conditions, it is very possible that the panelists were fatigued and may have been less sensitive due to affect the perception of aroma by

adaptation. Another possible cause of variation is the mere task of evaluating aroma. Rum contains a large number of volatile compounds that contribute to the aroma, in this case, the judges had to smell the sample and inhale the composition of the headspace of the bottle that contained the sample. It is not possible to provide a constant headspace composition even for the same sample, given that it will be affected by settling time and temperature, and eventually if opened and exposed to environment for long time, the initial aroma of the sample will no longer be maintained. According to Cain *et al.*, (1992) the concentration of the headspace varies proportionally with the odorant in the solution by a factor known as activity coefficient. This factor will differ among odorants diluted with the same solvent, among solvents, and among concentrations of the same odorant-solvent pair. Also, when subjects smell the headspace of a liquid solution contained in a recently opened bottle, some amount of air will be inhaled, causing the original stimulus to be diluted. According to Dravenkis (1975) a bottle left still will regain its headspace after 30 minutes or faster if the bottle is shaken. Cain *et al* (1992) stated that the problem of the equilibrium of the headspace is solved when 2 bottles are provided for alternative use. In the case of this study, 5 evaluation booths with the complete set of standards were used. Two sets of samples for evaluation were provided. Each set contained two containers for the same sample, so that the panelists could use it alternatively while evaluating for each and every attribute. The sets of samples were also used alternatively. Both the rum samples and the reference standards were periodically checked and replaced with new ones (refer to the methodology section). By providing many vials for the same sample the problem of having a representative headspace of the sample was minimized. The detailed instructions provided aimed to minimize the variation in the method of

evaluation used by each of the panel member. However, total control over those differences is difficult to achieve. The characteristics of this study, using a sample with such complex composition as in rum, and the complexity of the evaluation task itself demand a more intensive and extensive training in order to obtain results that are more significant, and that present less variation between panelists, and in the replications within the same panel member.

4.3.2 Overall Samples Differences

MANOVA was performed to determine if the overall differences existed in the rum samples differences based on all the aroma attributes used to describe them. The P value on the Wilk's Lambda (0.0001) revealed that the aromas of all 10 rum samples evaluated was different (Table 4.26).

Table 4.26 Multivariate Statistics and F Approximations

MANOVA Test Criteria and F Approximations for the Hypothesis of No Overall Form Effect

H= Type III SSCP Matrix for Form

E= Error SSCP Matrix

S=9 M=-0.5 N=151.5

Statistics	Value	F Value	Num DF	Den DF	Pr>F
Wilk's Lambda	0.15802275	8.08	81	1980	<.0001
Pillai's Trace	1.39254391	6.37	81	2817	<.0001
Hottelling-Lawley Trace	2.57477145	9.65	81	1304.5	<.0001
Roy's Greatest Root	1.19600156	41.59	9	313	<.0001

Discriminant analysis was used to determine the attributes responsible for the perceived differences. As it is shown in Table 4.27, in the first dimension of the pooled within canonical structure, "ocean-like" aroma (0.931) significantly contributed to the overall difference among rum samples. In the second canonical structure dimension, "cardboard" aroma appears to be responsible for the differences among samples. Further canonical structure dimensions reveal other aroma attributes that contribute significantly to the differences among samples. The most important attribute that contributes to differences among samples in the third canonical structure is "artificial fruity".

Table 4.27 Canonical Structure r's Describing Group Differences Among Samples[a]

Variable	Can1	Can2	Can3	Can4	Can5
Woody	-0.201	-0.146	-0.256	0.538	0.241
Buttery	-0.060	-0.012	0.297	0.342	0.755
Caramel	-0.140	-0.061	0.121	0.643	0.286
Honey	-0.169	-0.103	-0.057	0.687	0.169
Vanilla	0.081	-0.082	0.361	0.538	0.474
Cinnamon	-0.102	-0.116	-0.222	0.483	0.228
Art. Fruit	0.011	-0.043	0.680	0.537	-0.141
Cardboard	-0.093	0.994	-0.054	0.261	-0.082
Ocean-like	0.931	-0.007	-0.138	0.289	0.059

[a] Based on pool-within class variances. Highlighted numbers indicate aroma attributes which largely account for group differences in each dimension.

4.3.3. Principal Component Analysis

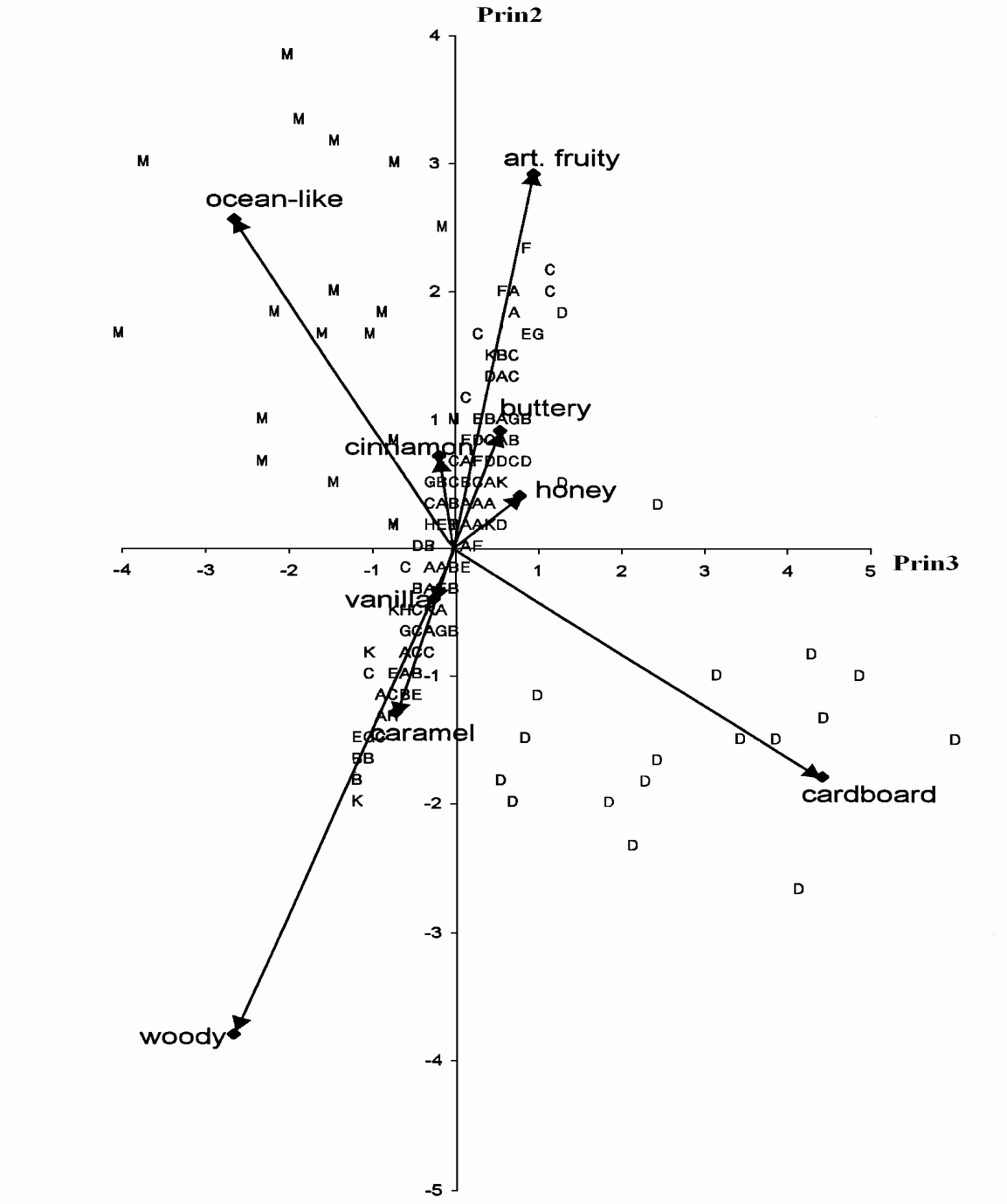


Figure 4.8. Plot of Prin2*Prin3.

* The symbols correspond to the value of the product. Sample 813 is product A, sample 975 is product B, sample 565 is product D, sample 722 is product E, sample 174 is product F, sample 983 is product G, sample 217 is product H, sample 599 is product K, and sample 796 is product M.

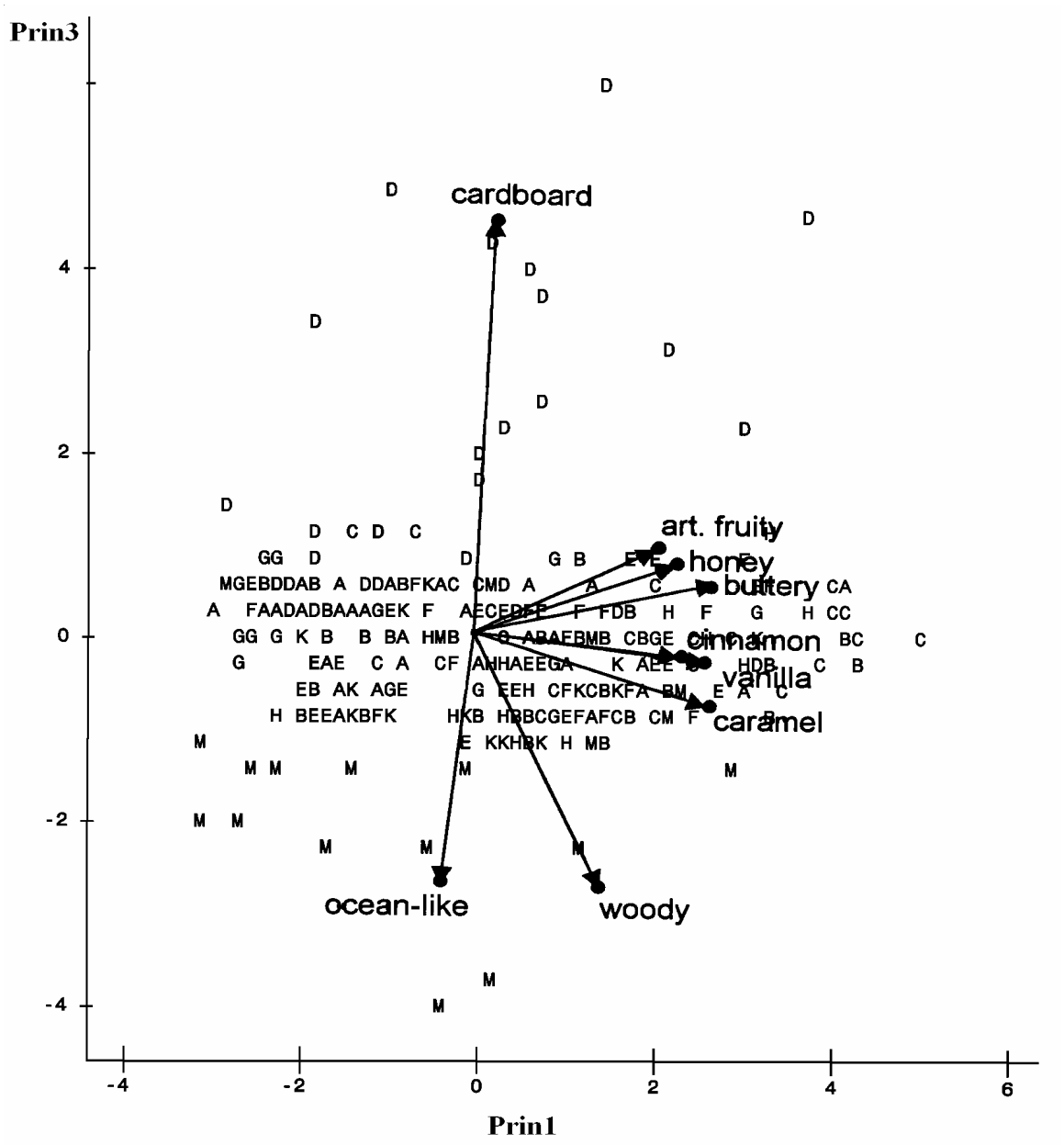


Figure 4.9. Plot of Prin3*Prin1.

* The symbols correspond to the value of the product. Sample 813 is product A, sample 975 is product B, sample 565 is product D, sample 722 is product E, sample 174 is product F, sample 983 is product G, sample 217 is product H, sample 599 is product K, and sample 796 is product M.

Principal component analysis was performed with the data for all samples. In the plots Prin2*Prin1 (Figure 4.8) and Prin3*Prin1 (Figure 4.9) we can observe that run 348 (D) and 796 (M) are different from the rest of the samples. Those rums appeared clearly separated while rest of the samples are scattered in between in the middle section of the plots. With the attributes plotted as vectors in the same principal component for sample plots, it is possible to determine that there is one attribute responsible for the discrimination of each of the two clearly distinct rums. Those attributes are "ocean-like" aroma for rum 796 (M), and cardboard aroma for sample 348 (D). Sample 796 was a white rum from Nicaragua made from molasses. What was peculiar of this sample was the use of a continuous distillation using 5 columns, and an undisturbed "slow-aged" method of aging that lasted for 4 years. Sample 348 was original of Haiti, was made of cane juice, double distilled in pot-still distillations, and aged in Limousin oak casks for 8 years. It is important to note that not only that those rums where clearly discriminated from the rest of the samples, but they appear to have the most differences in the perceived aroma.

4.4. Conclusion

Evaluation of rum, as with any other highly alcoholic beverage faces many problems, one of them is the impairment of the judges' ability to perform due to such quantities of ethanol will give the judge after tasting a large number of samples. This study proves that the mere aroma perceived by controlled sniffing of the rum samples can be used to evaluate and describe rums, and that such information can be used to discriminate between rum samples. Other problem faced when evaluating of rum is the overpowering effect that the aroma of ethanol may have on the samples. The use of

ethanol-based standards, with ethanol content similar to that in rum samples was an effective way to train panelists on the aroma of rum, and was also useful for the creation of adequate references that best mimic the perceived aroma of rum.

The developed method for descriptive analysis of rum products was effective in analyzing a larger number of samples than an aroma and taste evaluation would have allowed for, also it presented an advantage in recruiting panelists. People, who for different personal, cultural, or religious reasons, would not be part of a rum evaluation that required tasting can be part of an aroma evaluation test such as the one conducted for this thesis research. The use of this method with adequate training can provide comprehensive information on the aroma of rum.

With this descriptive analysis method and the developed list of aroma attributes, it is possible to discriminate the aroma of different rum samples. Significant differences in the intensities of attributes "woody", "ethanol", "caramel", "honey", "smoke", "vanilla", "banana", "prune", "cardboard", and "ocean-like" were found in the evaluated rum samples. There were also significant differences in the frequency of the perception of "pungent", "sour" (acid), and "sweet" chemical sensations, and "metallic" aroma. The most important attributes that have a significant effect in discriminating among samples were: "ocean-like", "cardboard", "artificial-fruity", "honey", "caramel", "woody", "cinnamon", "vanilla", and "buttery".

It is possible to analyze the information on the differences between samples and relate back the processing protocol of the different rum samples. This information can be a very useful tool for the development of new rum products that outstand the rest of the samples in a specific desirable aroma attribute, or that poses a unique aroma. The use of

rum descriptive analysis with the method used in this study can also be a good tool for the quality control of rum products, can be useful for brand identification, and can also be a helpful marketing tool.

CHAPTER 5. GENERAL CONCLUSIONS

Rum production constitutes a value addition of molasses with low economic value. It is important to determine the optimal conditions that yield desirable rum production with desirable aroma quality. The number of compounds present in rum is so large that it is impossible to single-out one component responsible for the aroma of rum, but it is important to know how the composition of volatile compounds, of and thus the aroma of rum will be impacted by the many steps involved in rum production.

Even though the raw material naturally influences the aroma of rum, the core of the aroma is formed during fermentation by the action of yeast, and also affected by the presence of bacteria. Other processing conditions have a major influence on the final quality of rum. Distillation affects the aroma of rum by altering the proportion of the aroma compounds originally present in the fermentation mash. The type of distillation, as well as the distillation temperature largely influence the aroma composition of the raw distillate. Reactions within the rum components, with wood components, and extraction of wood components alter the composition of the distillate, and have a large impact on the aroma and other sensory qualities of rum. The conditions of the maturation process, such as aging temperature, type of wood, and condition of the wood, as well as the aging time influence the perceived aroma of the final product.

As rum is a product with so many different compounds responsible for its aroma, and some of those compounds are present in a very small amount; it presents a problem for using chemical analysis alone for determining, achieving, and controlling the quality of rum. The developed list of descriptors for the aroma, taste and flavor of rum can be a good tool to describe a large variety of rum products.

The descriptive analysis of rum as used in this study was useful in describing different rum products, and to discriminate among rum samples. However, it is strongly recommended, for further studies, to the use of a reduced number of attributes, and to maintain the most important ones in order to make the training, evaluation, data handling, and interpretation easier, and to provide more significant information. "Ocean-like", "cardboard", "artificial-fruity", "honey", "caramel", "woody", "cinnamon", "vanilla", and "buttery" are the most significant attributes responsible for the discrimination among samples. However, significant differences in the intensity of attributes such as "smoke", "banana", "prune", and in the frequency of perception of "pungent", "sour" and "sweet" chemical sensations, and "metallic" aroma were also found.

The use of a larger panel size, and more intensive training is also recommended for further studies, given the complexity of rum, which made the description of the aroma a difficult task.

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APPENDIX A. BALLOT FOR THE EVALUATION OF RUM USING THE LIST OF TERMS BY SEMI-EXPERT JUDGES

Please mark the descriptor (if present) and indicate the order in which it was perceived.

	867	432	258	093	449	389	758
Allspice							
Almond							
Apple							
Apple/pear							
Artificial							
Banana							
Bite							
Burn							
Butter							
Butterscotch							
Caramel							
Chemical							
Pure Ethanol							
Eucalyptus/mint							
Floral							
Fruity (general)							
Fusel oil							
Gasoline							
Leathery							
Medicinal							
Musty							
Nutty							
Pineapple							
Plastic							
Rubbing alcohol							
Smoke							
Spicy							
Sweet							
Syrupy							
Vanilla							
Woody							

APPENDIX B. BALLOT FOR PANELIST SCREENING. PAGE 1

Name:_____ Date:_____
Phone:_____ e-mail:_____

Screening Part I:

Match each solution to one of the perceived tastes (sweet, sour, salty, or bitter)

Taste	Write down the solution number
Sweet	_____
Sour	_____
Salty	_____
Bitter	_____

Screening Part II:

1. Rank the sweetness intensity of the solutions from the least sweet to the sweetest. Write down the solution numbers on the space below.

_____ _____ _____ _____

Least sweet Sweetest

2. Rank the saltiness intensity of the solutions from the least salty to the saltiest. Write down the solution numbers on the space below.

_____ _____ _____ _____

Least salty Saltiest

2. Rank the sourness intensity of the solutions from the least sour to the most sour. Write down the solution numbers on the space below.

_____ _____ _____ _____

Least sour Most sour

2. Rank the bitterness intensity of the solutions from the least bitter to the most bitter. Write down the solution numbers on the space below.

_____ _____ _____ _____

Least bitter Most bitter

BALLOT FOR PANELIST SCREENING. PAGE 2

Screening Part III:

Sniff each sample and describe in your own words the perceived aroma.

Sample	**Description**
A	_____
B	_____
C	_____
D	_____
E	_____
F	_____
G	_____
H	_____
I	_____
J	_____

BALLOT FOR PANELIST SCREENING. PAGE 3

Screening Part IV:

Sniff the first set of samples allowing time to rest after each sample. Then sniff the second set of samples. Match the samples from the second set with the ones from the first set. Write down the sample numbers of the second set next to the matching pair in the first set. Then determine which term(s) from the list below best describes the aroma of the samples

Fist set	**Second set**	**Descriptors**
473	_____	_____
318	_____	_____
941	_____	_____
502	_____	_____
079	_____	_____
992	_____	_____
467	_____	_____
134	_____	_____
756	_____	_____
345	_____	_____

List of terms or descriptors:

Floral	Clove	Strawberry	Pepper
Hazelnut	Peppermint	Banana	Garlic
Rose	Cinnamon	Eucalyptus	Nutmeg
Pineapple	Vinegar	Allspice	

APPENDIX C. RUM AROMA DESCRIPTIVE ANALYSIS. ORIENTATION HANDOUT. PAGE 1

Rum Flavor Descriptive Analysis

1

Objective

Determine differences in flavor of rum products produced from different processing protocols

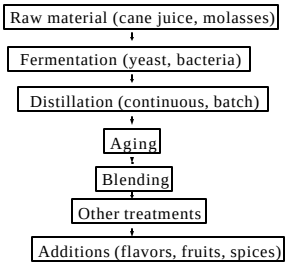
2

Rum

- Alcoholic Distillate
- Molasses, cane juice, cane syrup
- Distilled at < 190 Proof
- Posses tastes, aroma, and characteristics of rum

3

Rum Process



4

Spectrum Technique

- Discriminate
- Describe
- Quantify

} Flavor characteristics

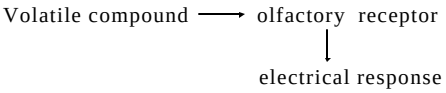
5

Requirements

- Expose panel to dimensions of flavor to ensure accurate evaluation
- Provide similar frame of reference in terminology and scaling

6

Flavor Perception



- Odors perceived:
  - Inhalation
  - Via mouth, rear nasal passages; flavor

7

Spectrum Method

1. Development of terminology
2. Review references and evaluation procedures
3. Product evaluation, discussion of results

8

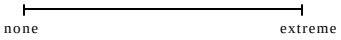
Development of Terminology

- Practice description of perceived flavor attributes
  - General samples
  - Samples related to rum (raw materials)
  - Evaluation products within rum category

9

Review of References, Evaluation Procedures

- Selection of adequate references
- Detailed definitions
- Group discussion → Selection terms
- Use of scale and references



10

Practice Sessions

- Review samples/terminology
- Review references/evaluation procedure
- Most of it individual training

11

Product Evaluation

Individual

9-10 sessions

Most convenient time

12

RUM AROMA DESCRIPTIVE ANALYSIS. ORIENTATION HANDOUT. PAGE 3

Location

- Orientation
- Training

} Room 201

- Evaluation → Individual booths

13

Results

14

Physiology of Aroma Perception

Rum products contain numerous products that contribute to their aroma. The aroma is perceived when a volatile compound contacts the olfactory mucosa, in the epithelium of the mucosa localized in the olfactory receptors, they axon through the cribform plate of the ethmoid bone terminating in the olfactory bulb. There is a large but limited number of olfactory receptors that transduce a large number of odorants. The olfactory receptors direct extensions of the olfactory nerve into the environment. The stimulus signal of the molecule contacting the olfactory receptor cells has chemical specificity, and it is translated to a neuronal electronic response. The transduction mechanism is still unknown (Thorngate, 1997). Odors can be perceived in two different ways; direct, the molecules enter the nose from the front through the nosetrils by inhalation, and indirect via mouth and rear the nasal passages (Pouisias and Chabanon, 1974). As the temperature of the substances in the mouth rises, more odorous compounds are released. These compounds will then reach the olfactory region by diffusion and through exhalation. The in-mouth odors are an important part of what is called flavor (Amerine and Roessler, 1983.)

Man's olfactory system is far less sensitive that that of many mammals. The olfactory thresholds vary depending on the substance. Some factors like respiratory infections and migraine can affect the olfactory thresholds. Individual threshold variations depend on various factors such as sex; women between adolescence and menopause have higher sensitivities, especially after ovulation. During stimulation by a particular odor, the threshold vale rises above the stimulation concentration causing adaptation. Adaptation is caused by the higher centers of the brain, and it explains why professional smellers can perceive odors far longer than untrained individuals (Carr, 1974).

References:

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RUM AROMA DESCRIPTIVE ANALYSIS. ORIENTATION HANDOUT. PAGE 5

Tentative Work Schedule

Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday
	1	2	3	4	5	6
7	8 Screening	9 Screening	10 Screening	11 Screening	12 Orientation	13
14	15 Group training	16 Group training	17 Group training	18 Group training	19 Group training	20
21	22 Group training	23 Group training	24 Group training	25 Group training	26 Group training	27
28	29 Indiv. Training	30 Indiv. Training				

Sunday	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday
			1 Indiv. Training	2 Indiv. Training	3 Indiv. Training	4 Indiv. Training
5	6 Indiv. Training	7 Indiv. Training	8 Indiv. Training	9 Indiv. Training	10 Indiv. Training	11
14	15 Evaluation	16 Evaluation	17 Evaluation	18 Evaluation	19 Evaluation	20 Evaluation
21	22 Evaluation	23 Evaluation	24 Evaluation	25 Evaluation	26 Evaluation	27
28	29 Evaluation	30 Evaluation				

APPENDIX D. BALLOT FOR GROUP TRAINING SESSION NO. 1 AND 2

Name:_____ Date:_____

Training Session No.__

Please smell each sample with short deep sniffs, and write down all the descriptors in the order perceived to describe the aroma/odor of each sample. Then give a brief definition for each term.

In between samples, sniff yourself, the coffee beans, and yourself again. Rest for about 1-2 minutes, then move to the next sample.

Sample No.____

Term	Definition
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Sample No.____

Term	Definition
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Sample No.____

Term	Definition
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

APPENDIX E. BALLOT FOR GROUP TRAINING SESSION NO. 3

Name:_____ Date:_____

Training Session No. 3

Please smell each sample with short deep sniffs, and write down all the descriptors in the order perceived to describe aroma/odor of the set of samples. Then give a brief definition for each descriptor.

In between samples, sniff yourself, coffee, and yourself again. Rest for about 1-2 minutes, then move to the next sample

Set No. ___

Term	**Definition**
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Selected Group Terms

_____
_____
_____
_____

Rate samples according to the intensity of each of the selected terms

Term	**Rank Order**		
_____	_____	_____	_____
	Less intense		More intense
_____	_____	_____	_____
	Less intense		More intense
_____	_____	_____	_____
	Less intense		More intense
_____	_____	_____	_____
	Less intense		More intense

APPENDIX F. BALLOT FOR GROUP TRAINING SESSION NO. 4

Name:_____ Date:_____

Training Session No. 4

Please smell each sample with short deep sniffs, and write down all the descriptors in the order perceived to describe aroma/odor of the set of samples. Then give a brief **definition** for each descriptor.

In between samples, sniff yourself, coffee, and yourself again. Rest for about 1-2 minutes, then move to the next sample

Term	**Definition**
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

APPENDIX G. BALLOT FOR GROUP TRAINING SESSION NO. 5

Name:_____ Date:_____

Training Session No. 5

Please smell each sample with a fast single short deep sniff, and write down one descriptor to describe aroma/odor of the samples. Then give a brief **definition** for that term, and rapidly proceed to the next sample.

After a few samples sniff yourself, the coffee beans, and yourself again. Rest for about 1-2 minutes.

Sample	Term	Definition	Assigned Term
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____
8.	_____	_____	_____
9.	_____	_____	_____
10.	_____	_____	_____
11.	_____	_____	_____
12.	_____	_____	_____
13.	_____	_____	_____
14.	_____	_____	_____
15.	_____	_____	_____
16.	_____	_____	_____
17.	_____	_____	_____
18.	_____	_____	_____
19.	_____	_____	_____
20.	_____	_____	_____
21.	_____	_____	_____
22.	_____	_____	_____
23.	_____	_____	_____
24.	_____	_____	_____
25.	_____	_____	_____
26.	_____	_____	_____
27.	_____	_____	_____
28.	_____	_____	_____
29.	_____	_____	_____
30.	_____	_____	_____
31.	_____	_____	_____

APPENDIX H. BALLOT FOR EVALUATION SESSION NO. 6

Name:_____ Date:_____

Training Session No. 6

Please smell each sample with a fast single short deep sniff, and write down one descriptor to describe aroma/odor of the samples. Then give a brief **definition** for that term, and rapidly proceed to the next sample.

After a few samples sniff yourself, the coffee beans, and yourself again. Rest for about 1-2 minutes.

Sample	Term	Definition	Assigned Term
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____
8.	_____	_____	_____
9.	_____	_____	_____
10.	_____	_____	_____
11.	_____	_____	_____
12.	_____	_____	_____
13.	_____	_____	_____
14.	_____	_____	_____
15.	_____	_____	_____
16.	_____	_____	_____

APPENDIX I. BALLOT FOR GROUP TRAINING SESSION NO. 7

Name:_____ Date:_____

Training Session No. 7

Please smell each sample with short deep sniffs, and write down a single term to describe aroma/odor of the sample.

Remember to sniff yourself, coffee, and yourself again. Rest for about 1-2 minutes, then move to the next sample

Sample	Term	Assigned Term	Group Definition
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____
8.	_____	_____	_____

Please smell the sample with short deep sniffs, and write down **all** the descriptors in the order perceived to describe aroma/odor of the sample. Then give a **definition** for each descriptor.

Term	**Definition**
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

APPENDIX J. BALLOT FOR GROUP TRAINING SESSION NO. 8, 9, AND 10

Name:_____ Date:_____

Training Session No. ___

Please smell each sample with short deep sniffs, and write down all the descriptors describe aroma/odor of the sample. Then give a brief definition for each term.

Remember to sniff yourself, coffee, and yourself again. Rest for about 1-2 minutes, then move to the next sample

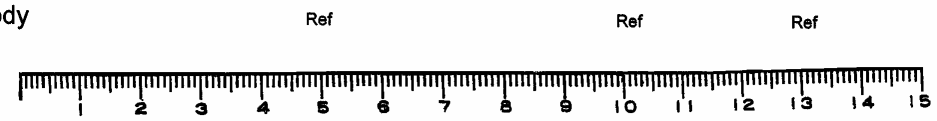
Sample: _____

Term	Definition

APPENDIX K. BALLOT FOR THE EVALUATION OF THE AROMA OF RUM. PAGE 1

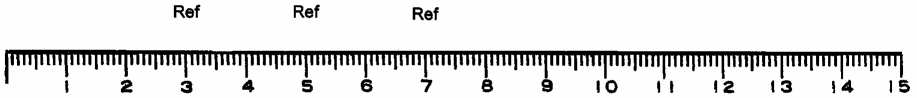
Sample No. Name:_____

Woody

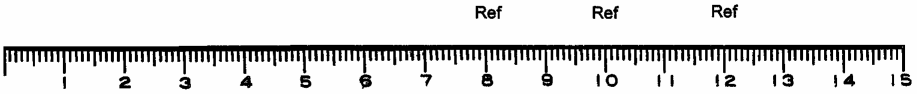


Ethanol

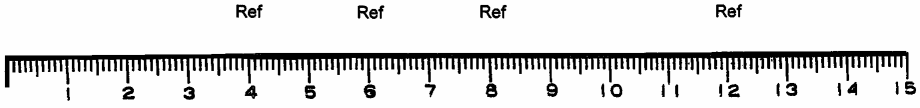
Buttery



Butterscotch



Caramel

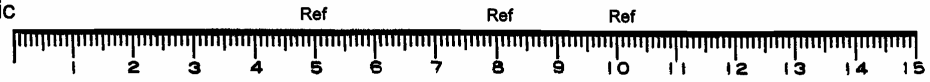


BALLOT FOR THE EVALUATION OF THE AROMA OF RUM. PAGE 2

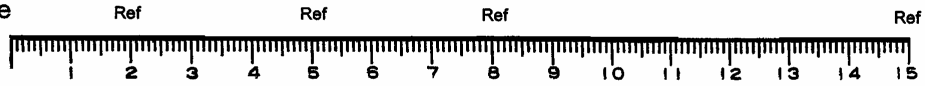
Sample No. Name:_____

Honey-like

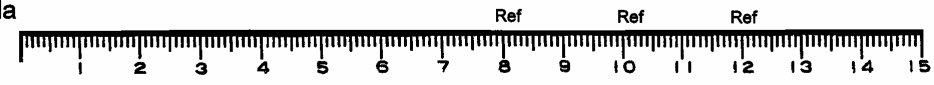
Plastic



Smoke



Vanilla

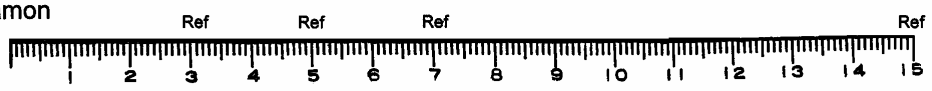


Almond

BALLOT FOR THE EVALUATION OF THE AROMA OF RUM. PAGE 3

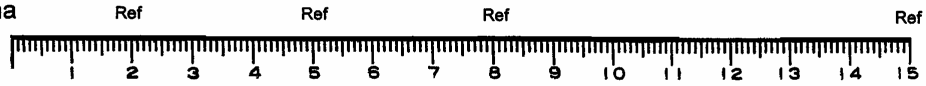
Sample No. Name:_____

Cinnamon

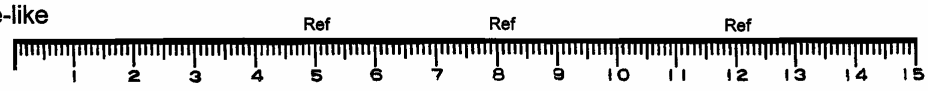


Fruity (artificial)

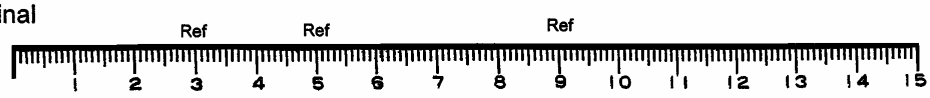
Banana



Prune-like



Medicinal



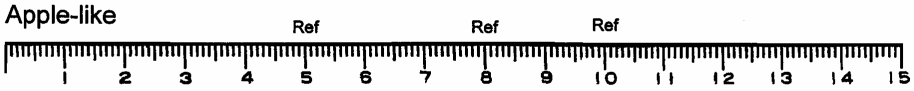
BALLOT FOR THE EVALUATION OF THE AROMA OF RUM. PAGE 4

Sample No. Name:_____

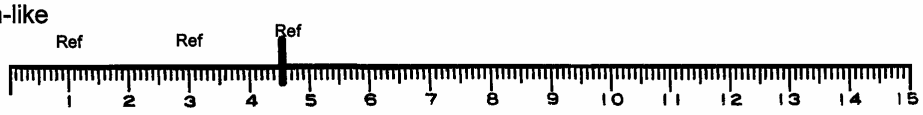
Pineapple-like

Peppery

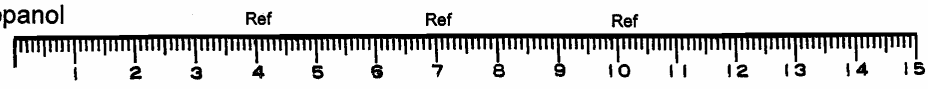
Green Apple-like



Pecan-like



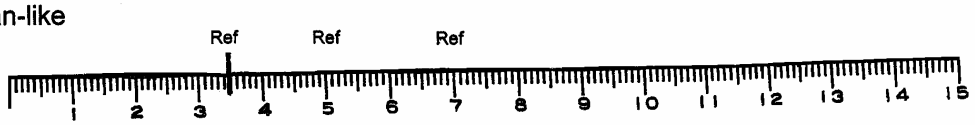
Isopropanol



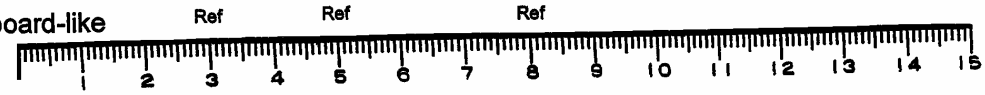
BALLOT FOR THE EVALUATION OF THE AROMA OF RUM. PAGE 5

Sample No. Name:_____

Ocean-like



Cardboard-like



Are you detecting the following sensations?

Pungent	Yes_____	No_____
Sour	Yes_____	No_____
Sweet	Yes_____	No_____
Metallic	Yes_____	No_____

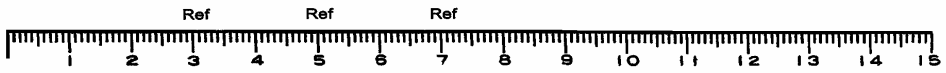
APPENDIX L. GUIDELINES FOR THE EVALUATION OF RUM AROMA

Guideline for Rum Evaluation

For every session you will be provided with 2 coded samples (2 vials each), the set of references, and the score sheets for the evaluation. Always check that the number of the sample corresponds to the number on the score sheet.

In the score sheet all the attributes (terms or descriptors) to be evaluated in rum are followed by a 15 cm scale. The left side of the line is the least intense, and the right side of the line has the highest intensity for that specific attribute. The intensity of the provided references are indicated on the scale:

Butter



Least intense Most intense

The reference intensity corresponds to the number below the "ref" notation except in one case where the intensity corresponds to 4.5. In that case the line was marked **only** to indicate the intensity value of the reference and should not be confused with the line you will have to trace for the intensity of that attribute (term or descriptor) in the rum sample.

For evaluating rum you should evaluate one sample at a time. You can start evaluating the samples for the attributes (terms or descriptors) in the order that they appear in the ballot, or in the order that you perceive them, however do not leave any line without score.

While evaluating the samples

1. open the vial
2. sniff with **one quick deep sniff** focusing on the specific attribute
3. close the vial tightly again
4. if you need so, you can check using the provided references, however try to avoid doing that in order for you to really focus on the rum sample and avoid excessive fatigue.
5. mark a vertical line exactly where you think the intensity of that specific attribute for that sample will be (0-15). Please make a clear straight line.

Remember to rest if you feel you cannot perceive any more:

- smell yourself
- smell the coffee sample
- smell yourself again,
- then continue with the evaluation.

Try alternating the vials for evaluating attributes. This will allow time for the release of aroma compounds in the headspace (empty space) of the vial.

The final attributes correspond to chemical sensations: sweet, sour, and pungent. In these cases you only need to check if you perceive it or not. References for these sensations are also provided.

Remember:

Try not to come right after eating or when very hungry
Avoid using perfume, lotions or other very aromatic personal-care products
When evaluating try to make yourself comfortable and take your time, if you feel you need to rest please do so.
Avoid other distractions or talking while evaluating.

Thank you

VITA

The author was born in Mexico City, Mexico, on August 22, 1975. She graduated from High School majoring in the chemical-biological area in 1993. She enrolled at the La Salle University, Mexico City, and completed an exchange semester at Louisiana State University in 1997. She graduated with a bachelor's degree in Chemistry with an emphasis in Food Science in 1998. She returned to Louisiana State University to pursue a master's degree. She will be awarded the degree of master of science in December 2002. She plans to continue her doctoral program in the Department of Food Science at Louisiana State University.