

AN ABSTRACT OF THE THESIS OF

Sonia M. Rubico for the degree of Doctor of Philosophy in

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Title: Perceptual Characteristics of Selected Acidulants by Different
Sensory and Multivariate Methods

Abstract approved: _____

Professor Mina R. McDaniel

The taste qualities of acidulants have not been studied in detail despite the fact that they are widely used by the food industry. Studies on characterizing the sensory properties of organic and inorganic acids are very limited. Reported studies are commonly on threshold, equi-sour and the time intensity values of sourness. A series of experiments were conducted to determine the sensory properties of selected acidulants by different sensory and multivariate methods.

First, the technique of Free-Choice Profiling was applied in order to characterize the sensory profile of some selected acids (adipic, citric, fumaric, glucono-delta-lactone, hydrochloric, lactic, malic, phosphoric, quinic, succinic, tartaric, citric:fumaric, citric:malic and fumaric:malic) on a weight (0.08% w/v or v/v) basis. Results analyzed through Generalized Procrustes Analysis indicate that on a weight basis (w/v or v/v), acids differed in their flavor and taste dynamics. Likewise, acids were described differently by individual panelists.

Second, the sourness power functions of the selected acidulants were generated from five molar concentrations by magnitude estimation

involving 16 trained panelists. Equi-sour concentrations were determined by regressing the log of the rescaled response (sensory) on the log of the stimuli (physical). The calculated equi-sour levels ranged from 0.48 ml/L for HCl to 2.34 g/L for glucono-delta-lactone when citric acid was set at 1.0 g/L. These theoretical equi-sourness were then tested by using an alternative sensory method, the directional difference from control test.

Third, the sensory profile of the acidulants at their equi-sour levels was characterized using two sensory methods, free-choice profiling and the conventional descriptive analysis. The former was analyzed by Generalized Procrustes Analysis while the latter was analyzed by Principal Component Analysis. The two sensory methods gave similar patterns of information regarding the acid samples. The similarities of several organic acids and their mixtures were very evident. Hydrochloric and phosphoric acids were astringent while succinic acid was bitter and had a monosodium glutamate taste. It was concluded that acids had other sensory properties aside from sourness that must be considered in a given food application.

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Perceptual Characteristics of Selected Acidulants by
Different Sensory and Multivariate Methods

by

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PERCEPTUAL CHARACTERISTICS OF SELECTED ACIDULANTS BY DIFFERENT SENSORY AND MULTIVARIATE METHODS

INTRODUCTION

Acids (sometimes called acidulants) are one of the most functional groups of ingredients used in the food processing industry (Andres, 1985). They are used in practically every group of formulated food products including beverages, gelatin desserts, meat and seafood products, dairy products, confectioneries, fruits and vegetables, jams and jellies, and even in processing fats and oils. They contribute a variety of functional properties that lead to the enhancement of food quality (Doores, 1990). Their many functions include: as an aid in sterilization, in food preservation, in chelation, providing leavening, as synergists to antioxidants, viscosity and melting modifiers and, one of its best known functions - as a flavoring adjunct. With the right amount of acid, flavor and palatability of many foods are enhanced and modified.

Acids may be added to foods, they may be a natural component of foods, or they may be produced during processing/preserving of foods by fermentation. Being a natural component of foods, many of the acidulants are considered GRAS (generally recognized as safe). Succinic, acetic, lactic, and fumaric acids arise from alcoholic fermentations in the production of wine. Citric acid occurs in almost all natural foods and is found in the cells of almost all plants and animals. Likewise, acidulants may be the key flavor-by-mouth components that could identify some of the tastes of various fruits like citric acid for lemon and lime, quinic acid for cranberries, tartaric acid for grapes and tamarind, malic acid for apples and adipic acid for beets.

The proper selection of acidulants in food is always dependent upon the properties or combination of properties of the acid as well as its cost. The last criterion is the main reason why food companies are trying to use inorganic acids such as hydrochloric and phosphoric acids which are much cheaper than organic or fruit acids (Toy and Walsh, 1987). Phosphoric acid has been associated with cola drinks, rootbeer and sarsaparilla although it has been the tendency of some food manufacturers to add citric or other organic acids in cola drinks. Nowadays, the trend is in combining different acidulants to improve the taste and more closely simulate the real taste, especially in formulated foods. This is understandable because food usually contains a mixture of different organic acids such as citric, malic, tartaric, lactic, fumaric and succinic acids. It is seldom that one finds a fruit or vegetable containing only one acidulant.

The use of acidulants as antimicrobial agents is receiving much attention today. By use of acidulants, processing parameters can be significantly altered and one can make a low acid food into high acid food. This is especially useful when controlling the growth of *Clostridium botulinum*, by lowering the pH below 4.6. However, one has to make sure that the addition of acid does not overwhelm the sensory properties of the food, i.e. does not make the food taste too sour. Glucono-delta-lactone is considered by food processors because it is less sour compared to other acids. The judicious use of acidulants will always be dictated by their sensory characteristics.

Studies on characterizing the sensory properties of organic and inorganic acids are very limited. Reported studies emphasize threshold values (Maeda and Nakao, 1963; Tada et al., 1972), power functions (Moskowitz, 1971; Straub, 1992; Straub and McDaniel, 1989), equi-sour concentrations (Furukawa

et al., 1969; Wani and Soma, 1961; Straub, 1992) and time-intensity (Straub, 1992). Organic acids have not been studied in detail with respect to their tastes despite the fact that they are very important ingredients in foods (Furukawa et al., 1969). Likewise, most of the studies have concentrated only on sourness, which is what acids are known for; however, there is research indicating that acids have other non-sour characteristics such as bitterness and astringency (Straub, 1992; Straub and McDaniel, 1989; Settle et al., 1986).

This study is important because the sensory properties of the acids have not been profiled before. A thorough understanding of the various acidulants is critical for both acidulant producers and their customers because even though the extent of sourness or pH may be the same, other sensory characteristics may often tip the balance as to which acid is most suitable for a given application. Thus, the different acidulants were characterized on an equi-sour basis to determine what other sensory characteristics may be present aside from sourness. The equi-sour levels were confirmed by an alternative sensory method, the directional difference from control test. Likewise, acids were characterized on a weight and on a molar basis but since the same pattern of information was gathered from the molar basis compared to the equi-sour basis, only the latter study is reported here.

The research goals of this three part study were to determine the sensory characteristics of some common acidulants using different methods of sensory profiling and multivariate analysis. The objectives chosen to meet these goals included:

Study 1

To investigate the sensory characteristics of selected acidulants using free-choice profiling and determine differences and similarities among the acids on a weight basis.

Study 2

To determine the sourness power function of each acid using a trained panel.

To determine equi-sourness levels at two levels of sourness for each acid.

To confirm the equi-sourness levels through an alternative sensory method, directional difference from control.

To compare the pH of the acidulants at various molar concentrations and at their equi-sour levels.

Study 3

To determine the sensory characteristics of some common food acidulants on an equi-sour basis using free-choice profiling and conventional descriptive analysis.

To investigate the differences, if any, between free-choice and the conventional descriptive analysis.

To find the underlying dimensions (structure) of the sensory characteristics used in flavor profiling.

To determine the relationships between sensory and chemical measures.

LITERATURE REVIEW

ACIDULANTS

Table 1.1 summarizes several key physical and chemical properties and Figure 1.1 shows the structures of selected acidulants.

•ADIPIC ACID

Adipic acid is a dibasic carboxylic acid that occurs naturally in beet juice (Ito et al., 1979). Low hygroscopicity and a lingering, high tartness that complements grape-flavored products and those with delicate flavors characterize this dry, white crystalline acidulant (Dziezak, 1990). It is used in gelatin desserts, powdered concentrates for fruit-flavored beverages, bottled beverages, jams and jellies, canned vegetables, confectionery products and in flavoring extracts (Andres, 1985). Other applications include evaporated milk, instant puddings, cheese analogues and fats and oils (Dziezak, 1990). It is also used in bakery products, in cheese products where it provides desired melt and texture, and as an acidulant in throat lozenges (Gardner, 1972)

Adipic acid imparts a slowly developing smooth, mild taste essential in supplementing foods with delicate flavors (Doores, 1990). The solubility of adipic acid is four to five times greater at room temperature than that of fumaric acid, another commonly used wetting agent. It is practically nonhygroscopic (Ito et al., 1979), a property that has the advantage of prolonging the shelf-life of a powdered product in which it is incorporated. It is used in beverages and in gelatin and pudding products to improve set and to maintain acidities within the pH 2.5-3.0 range (Gardner, 1972). It is an

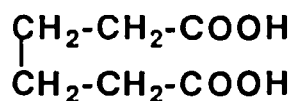
Table 1.1. Key Properties of Acidulants

ACID	EMPERICAL FORMULA	MOLECULAR WEIGHT	EQUIVALENT WEIGHT	NO. OF COOH	IONIZATION CONSTANT*
ADIPIC	C ₆ H ₁₀ O ₄	146.14	73.07	2	3.7 × 10 ⁻⁵ ^a
CITRIC	C ₆ H ₈ O ₇	192.12	64.04	3	8.2 × 10 ⁻⁴ ^a
FUMARIC	C ₄ H ₄ O ₄	116.07	58.04	2	1.0 × 10 ⁻⁴ ^a
GLUCONO-DELTA- LACTONE	C ₆ H ₁₀ O ₆	178.14	178.14	1	2.5 × 10 ⁻⁴ ^c
HYDROCHLORIC	HCL	36.46	36.46	0	
LACTIC	C ₃ H ₆ O ₃	90.08	90.08	1	1.4 × 10 ⁻⁴ ^b
MALIC	C ₄ H ₆ O ₅	134.09	67.05	2	4.0 × 10 ⁻⁴ ^a
PHOSPHORIC	H ₃ PO ₄	98.0	49.0	0	7.1 × 10 ⁻³ ^b
QUINIC	C ₇ H ₁₂ O ₆	192.0	192.0	1	2.0 × 10 ⁻⁴ ^a
SUCCINIC	C ₄ H ₆ O ₄	118.09	59.05	2	6.5 × 10 ⁻⁵ ^a
TARTARIC	C ₄ H ₆ O ₆	150.19	75.10	2	1.04 × 10 ⁻³ ^a

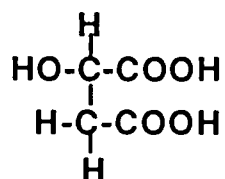
*K_a - the first dissociation constant is a measure of the extent to which the acid produces hydrogen ions when dissolved in water; ^aGardner, 1966; ^bArnold, 1975; ^cMerck Index, 1968

ACID STRUCTURES

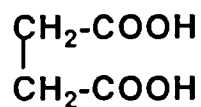
ADIPIC



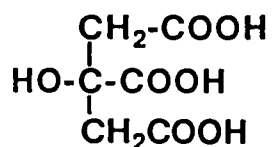
MALIC



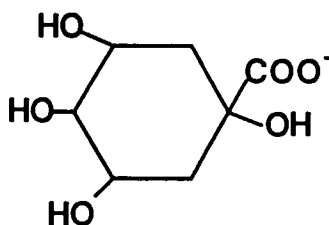
SUCCINIC



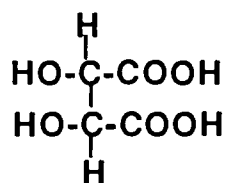
CITRIC



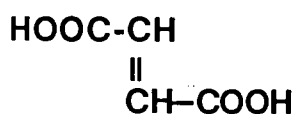
QUINIC



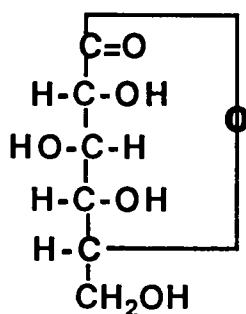
TARTARIC



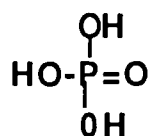
FUMARIC



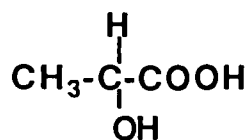
GLUCONO-LACTONE



PHOSPHORIC



LACTIC



HYDROCHLORIC



Figure 1.1. Structure of selected acidulants.

excellent, slow acting leavening agent that supports an even release of carbon dioxide in baked goods and can be used as an alternative to tartaric acid in commercial baking powders (Gardner, 1966). This acid is widely used in the food industry for buffering and neutralizing purposes (Ito et al., 1979).

No data have appeared in the literature to suggest that adipic acid has any unusual antimicrobial properties other than a pH effect (Doores, 1990). Yamamoto et al. (1987) reported the difficulty of preventing growth of lactic acid bacteria completely by the use of adipic acid alone. Growth of yeast was scarcely affected by any addition of adipic acid.

•CITRIC ACID

Citric acid is one of the most widely accepted and used organic acids in the industry (Andres, 1985; Dzeziak, 1990). It occurs in almost all natural foods and metabolites of both animals and plants. In humans, it is produced during the Krebs cycle which takes place during the metabolism of fats and carbohydrates (Dzeziak, 1990). Human blood normally contains about 25 ppm of citric acid (Arnold, 1975). Among plant sources, citric acid is most abundantly present in citrus fruits such as lemon (4.0-8.0%), grapefruit (1.2-2.1%), tangerines (0.9-1.2%) and oranges (0.6-1.0%) (Bouchard and Merritt, 1979).

Citric acid can be produced in numerous ways, from chemical syntheses to microbial fermentation techniques utilizing *Aspergillus niger* and specific *Candida species* (Dzeziak, 1990). Acids that once were obtained from lemons and canned pineapple waste are now mainly produced by fermentation of pretreated molasses or other starches with various strains of *Aspergillus niger* (Gardner, 1972; Kosobe, 1967).

Citric acid is the most versatile acidulant that can be used to advantage in practically every application because it is not optically active (Arnold, 1975), and the pleasant tart taste of citric acid is more compatible with citrus-flavored beverages than is malic acid. The other sensory characteristics of fumaric and adipic acids aside from fruit tartness are also reasons for citric acid preference. Fumaric has been described as metallic and adipic as chalky (Johnson and Peterson, 1974).

Citric acid is a valuable acidulant for dairy products where it is added to improve and protect the flavor and aroma of a variety of dairy products including cheese. It is also extensively employed in the preparation of carbonated beverages to bring out the flavor and impart a "tang" which is characteristic of many beverages. Citric acid continues to be one of the most popular acidulants for beverage powders and artificially flavored noncarbonated or "still" drinks. In wine, it maybe added to adjust acidity, prevent cloudy precipitates and inhibit oxidation (Gardner, 1972; Dzeziak, 1990).

In jams, jellies, fruit butters, and preserves, citric acid is used to control pH for optimum gel formation and at the same time serve as a flavoring agent. It is also employed to adjust the acidity of relishes, sauces, and other food products requiring particular flavor enhancement. It is incorporated in various types of candies and fondants to enhance the flavor of fruit berries and other ingredients, to prevent crystallization of sugar, to invert sucrose and to prevent oxidation of ingredients like nuts (Gardner, 1972; Kosobe, 1967).

Citric acid is used commercially as a synergist for antioxidants and as a retardant for browning reactions. It is also used as a plasticizer and emulsifier to provide texture to processed cheese and to enhance melting, to reduce heat

processing requirements by lowering pH, and to control acidity in pectin or alginate gel formation. However, citric acid is more hygroscopic than adipic or fumaric acids and can create storage problems in powdered products (Doores, 1990).

•FUMARIC ACID

Fumaric acid is widely distributed in nature in small amounts. The name fumaric was derived from fumitory (*Fumitoria officinalis*), a common English wild plant, whose leaves contain the acid (Arnold, 1975). Like citric and malic acids, it takes part in the Krebs metabolic cycle. It also occurs naturally in rice, sugar cane, wine, plant leaves, mushrooms and gelatin (Dzeziak, 1990).

Fumaric acid can be made by the aerobic fermentation of a carbohydrate with molds of the genus *Rhizopus* (Blenford, 1986; Arnold, 1975). The acid has also been produced as a by-product in the manufacture of phthalic and maleic anhydrides and by isomerization of the readily available maleic acid (Gardner, 1972). Alam Khan et al. (1987) obtained a yield of 95% fumaric acid by optimizing reaction conditions for HCL-catalyzed isomerization of maleic anhydride

Fumaric acid imparts a sour taste to food products and is one of the most acidic of the solid acids (Doores, 1990) on an equal weight basis. Its low solubility in cold water, however, is a limiting factor to its widespread use. Conversely, its low rate of moisture absorption aids in extending the shelf-life of powdered products (Gardner, 1966; Kosobe, 1967). A cold soluble form of fumaric acid can be attained in admixture with 0.3% dioctyl sodium sulfosuccinate and 0.5% calcium carbonate (Gardner, 1972). Fumaric acid is used in fruit drinks, in gelatin desserts, where it increases the gel strength of

gelatin, in pie fillings and biscuit doughs, and in wines. In addition to supplying acidity to the product, fumaric acid displays some antioxidant properties in fat-containing foods (Gardner, 1972). It can eliminate excessive hardening and rubbery texture in alginate-based desserts. It serves as an antioxidant, in preventing rancidity in lard, butter, cheese, powdered milk, sausages, roasted nuts and potato chips (Alam Khan et al., 1987). Fumaric acid was found more effective than citric, malic or succinic in lowering the pH of canned pimientos to pH 4.5 (Supran et al., 1966).

Among the solid (powdered) acidulants, fumaric acid is the most economical from the standpoint of cost and the quantities (g/L) required for giving the same sourness (Gardner, 1966). If solubility permits, it requires 60% less by weight formulation and 50% less in acid cost to achieve equal sourness compared to other organic acids.

Fumaric acid has been used as an antimicrobial agent (Pilone, 1975) in wines to prevent malolactic fermentation as well as a means of adding acidity (Ough and Kunkee, 1974). In wine, it can be used as an acidulant and clarifying agent at levels not exceeding 3.0 g/L of the finished product and in maraschino cherries, it imparts flavor in maraschino cherries (Dzeziak, 1990).

•GLUCONO-DELTA-LACTONE

Glucono-delta-lactone (GDL) is an intramolecular ester of gluconic acid marketed as a white crystalline powder. It has an initial sweet taste and a slightly acidic after taste (Dailey, 1978). Although it is not an acid, when it is dissolved in water it hydrolyzes slowly to an equilibrium mixture of gluconic acid and its delta- and gamma-lactones. When hydrolysis is complete, the solution contains 55 - 60% gluconic acid and 40-45% lactones, depending on the amount of glucono-delta-lactone added. Hydrolysis occurs slowly, taking

three hours to reach equilibrium at room temperature (Dzeziak, 1990). GDL is produced commercially by a fermentation process that utilizes enzymes or nonpathogenic, nontoxicogenic microorganisms such as *Aspergillus niger* or *Acetobacter suboxydans* to convert glucose to gluconic acid (Dzeziak, 1990).

GDL functions as an acidulant, an antimicrobial and a binding agent for water and metal ions and as starting material for various synthesis (La Belle, 1981). Its main applications are in cheese making, in meat, and in bakery products. It is widely used in Japan and Europe as a food additive (Tsuda and Nakanishi, 1983). In Japan, GDL is registered as a food additive and is widely used for a variety of purposes; in soybean curd productions as a coagulant, in biscuits and bread as a leavening agent, in jelly as an acidulant and in sausages as a pH-lowering agent (Anonymous, 1979). It is used as an acidulant in foods in West Germany while in Sweden it is used as an acid and maturing agent in foods (Hisao, 1981).

The slow hydrolysis rate of GDL in water and its acceleration with increasing temperature makes GDL the chemical of choice for use in leavening systems. These properties of glucono-delta-lactone made it the successful choice as an acidulant in baked goods utilizing a chemical raising action (Dailey, 1978). GDL can be useful in any product that needs acidification but requires a low flavor profile (Dzeziak, 1990). In fats and oils, GDL controls pH and complexes with trace metals that would otherwise promote oxidation (Dzeziak, 1990).

•HYDROCHLORIC ACID

Most hydrochloric (HCL) acid is obtained as a by-product of organic chlorination. It can also be made from its elements or by treating common salt with sulphuric acid. HCL is also the acid of the human stomach (0.25-0.5

per cent concentration) and is classified as a permitted acidulant for use in food (Arnold, 1975). It is used for souring off lye-peeled vegetables and for pH adjustment in foods. Basel (1982) found peas and snap beans that were stored for 4 months in an HCL solution of pH 1.2 - 1.6 maintained good quality. Acidified storage does not employ a heat treatment to kill microorganisms or inactivate enzymes and is one possible intermediate storage technique to delay final processing of snap beans and peas. HCL is also used in the direct acidification process during manufacture of mozzarella cheese (Micketts and Olson, 1974) and pigskin gelatin (Lee and Kim, 1986).

•LACTIC ACID

Lactic acid is one of the most widely distributed acids in nature and one of the earliest important organic acid used in foods (Gardner, 1972). It can be found in bread, milk, cheese, meat, beer and wine where it is formed in nature as an end-product of anaerobic carbohydrate metabolism (Andres, 1985). Lactic acid is produced naturally by bacterial action in wine, pickled food and dairy products. It also arises from alcoholic fermentation in the production of wine.

The methods used to commercially produce lactic acid are equally divided between natural fermentation and synthetic processes (Dzeziak, 1990). The first production method involves the controlled fermentation of highly refined sucrose or other less expensive carbohydrates such as potato starch, molasses, corn sugar or milk whey (Gardner, 1972). The mother liquor is purified by converting it to crystalline calcium lactate, which is then decomposed with sulfuric acid to give solutions of the pure acid.

Lactobacillus delbruckii cultures are employed in the production of lactic acid from starch or molasses. *L. bulgaricus* cultures grown on pasteurized

skimmed milk are used for fermentation in whey. Most producers employing fermentation techniques are based in Europe (Anonymous, 1985). Edible lactic acid is also manufactured synthetically by the hydrolysis of lactonitrile (acetaldehyde cyanohydrin or 2-hydroxypropionitrile). In the US, this compound is obtained as a by-product of acrylonitrile manufacturing while in Japan it is produced from acetaldehyde and hydrocyanic acid (Anonymous, 1964).

Lactic acid is highly soluble in water and is normally available as a concentrated aqueous solution (Andres, 1985). Food grade lactic acid is available in either 80% or 50% solutions (Dzeziak, 1990) or as lactic acid powders for dry mix applications (Andres, 1985). Lactic acid can be used as an antimicrobial agent, pH control agent, curing and pickling agent, flavor enhancer, flavoring agent and adjuvant, solvent, and vehicle (Doores, 1990). Its applications in the food industry are in confectionery products, as a component of pickles and olives, in soft drinks, in the baking industry, in dairy products and as a natural product of fermented foods such as sauerkraut (Andres, 1985). It is used in the baking industry in breads, buns, rolls, raisin bread, variety breads, etc. In the dairy industry, it is used in mozzarella and ricotta cheeses, processed cheese products, and cheese/fruit/vegetable combinations. Other applications include salad dressings, sauces, and marinated herrings. Since lactic is a normal constituent of food and an intermediate metabolite in humans, there is no limit set on its daily acceptable intake for humans (FAO/WHO, 1973). However, neither D(-) nor DL lactic acid is allowed to be used in infant food (FAO/WHO, 1973). Only the L (+) form is recommended for use in feeding premature infants (Ballabriga et al., 1970).

Lactic acid has a mild acid taste in contrast to the sharp, biting taste of some acids (Holten et al., 1971). This is advantageous because it does not mask or overpower the other weak aromatic flavors. It has a mild, creamy odor with a pleasant, sour taste (Gardner, 1972). It also has a mild fruity flavor, predominantly ascribed to yeast, lemons and sour apples.

Although lactic acid has been used more for its sensory properties, it has notable antimicrobial properties as well (Doores, 1990). For example, Dubos (1950) found that lactic acid has a significant bacteriostatic effect against *Mycobacterium tuberculosis* which increase as the pH is decreased. Rice and Pederson (1954) work on *Bacillus coagulans* (the causative agent in flat sour spoilage) in tomato juice showed lactic acid to be four times as effective as malic, citric, propionic and acetic acids at inhibiting growth.

•MALIC ACID

Malic acid is an acid that is also widely distributed in nature. It is as important as citric acid in fruit and, like citric acid, takes part in the Krebs metabolic cycle (Arnold, 1975). Malic acid is a white, triclinic, crystalline powder. The commercially synthesized product is a racemic mixture of the D- and L-isomers while the acid found in natural products is of the L-malic acid (Kosobe, 1967; Gardner, 1972). Malic acid is made from maleic anhydride by the very simple process of heating it with water under pressure at around 180°C, the temperature where an equilibrium mixture of malic and fumaric acids is produced (Arnold, 1975). The sparingly soluble fumaric acid is then filtered off and recycled while the malic acid in the filtrate is concentrated and crystallised.

Malic acid represents 100% of the acid found in watermelon and quince, 99% in plums, 97% in apples, 96% in cherries and peaches, 92% in

bananas, 87% in pears, 80% in orange peel and is found in most other fruits (Andres, 1985). It is also the predominant acid in apricots, grapes, broccoli, carrots, peas, potatoes, and rhubarb (Gardner, 1972). It is the second most prevalent acid in citrus fruits, in most berries, figs, beans and tomatoes.

Malic acid imparts a smooth, tart taste, with no burst of flavor and is commonly used in beverages, ices, candies, baked goods, canned foods, jams, jellies, and preserves (Gardner, 1966; Doores, 1990). Of all the flavoring materials, malic acid is among the few that gives a taste effect that closely resembles citric acid (Arnold, 1975). It has an unusual taste-blending characteristic that in some instances appears to have flavor-fixing qualities as well as masking undesirable aftertastes (Gardner, 1966). Due to its longer retention time, it has an effect in decreasing the strong aftertaste of artificial sweetener (Kosobe, 1967).

Smaller amounts of malic acid than citric acid are required to impart the same degree of acidity in food (Gardner, 1966). Although its degree of ionization in water is the same as citric acid for most practical purposes, malic acid has a much stronger apparent acidic taste than citric acid but less than that of fumaric acid (Gardner, 1972). Being naturally anhydrous, it is more preferred than citric acid for powder mixtures (Arnold, 1975). However, malic acid reacts with fructose to produce brown pigments (Kosobe, 1967). No unusual antimicrobial action is attributed to malic acid other than those associated with pH effects (Banwart, 1979).

•PHOSPHORIC ACID

Phosphoric acid is the only inorganic acid extensively employed as a food acidulant (Gardner, 1972). It is one of the least expensive acidulants and offers the following functions: it acidifies, it is used as a buffer to control

acidity, it imparts an acidic taste, and it complexes with metal cations that may promote product degradation (Dzeziak, 1990).

There are two methods of manufacturing phosphoric acid using phosphate rock, which is basically an agglomeration of fossil bones and other animal parts (Gardner, 1972). The older and more economical method involves treatment of phosphate rock with sulfuric acid. The other type consists of reducing the phosphate rock to elemental phosphorous in an electric or blast furnace, burning phosphorous to phosphorous pentoxide with air, hydrating the oxide by taking it up in 75 to 85% phosphoric acid and purifying the product with hydrogen sulfide to remove arsenic. Food grade phosphoric acid is available as 75%, 80% and 85% aqueous solutions (Dzeziak, 1990; Monsanto, 1991).

Phosphoric acid possesses unique chemical properties as quoted from Arnold (1975). " Its status as an acid is most unusual; the dissociation constants are respectively: 0.71×10^{-2} , 0.63×10^{-4} and 4.7×10^{-13} . The size of the first dissociation constant makes phosphoric acid a strong acid in comparison with most organic acidulants, but an extremely weak one in comparison with the common mineral acids, which are more or less completely dissociated in dilute solution (that is, the first dissociation constant approaches infinity); phosphoric acid can, in fact, form part of a buffer system. The second dissociation constant is only slightly larger than the second dissociation constant of citric acid, while the third dissociation constant has a value not too far removed from that of water. Thus the third hydrogen atom possesses no acidic properties to speak of, and orthophosphoric acid is best regarded as a dibasic acid".

The tartness that makes carbonated drinks such as cola drinks, root beer, sarsaparilla and similar soft drinks so refreshing is attributed to phosphoric acid (Toy and Wash, 1987). Cola contains 0.057% to 0.084% of 75% phosphoric acid with a pH of about 2.3-2.5. Root beer contains 0.013% phosphoric acid with a pH of 5.0. In comparison, the human stomach has a pH of 2.5 during digestion. In a sample test, one pound of phosphoric acid

was found to have the equivalent sourness of 4.25 lb citric acid or 5.5 lb of tartaric acid. Phosphoric acid is also much cheaper than fruit acid. In 1984, one lb of citric acid cost 81 cents as compared to 7 cents for one lb of 75% phosphoric acid. While sulfuric and HCL acids are the cheapest among the acid group, neither is used because their addition in food is regarded as questionable by nutritionists. Hence, phosphoric acid ranks as the most economical choice as a food additive by default.

Besides its major use in soft drinks, phosphoric acid is used in cheese and in brewing to adjust pH, to enrich and preserve fodder, as an ingredient of bread dough, as a yeast stimulant, to neutralize the caustic peeling of fruit, to clarify and acidify collagen in the production of gelatin, in the purification of vegetable oils, and to a small extent in the manufacture of jams and jellies (Gardner, 1972).

•QUINIC ACID

Quinic acid (1, 3, 4, 5 - Tetrahydroxycyclohexanecarboxylic acid) is a naturally occurring organic acid that is found in many plant species including cinchona bark, tobacco leaves, carrot leaves, apples, peaches, pears, plums, etc. (Merck Index, 1968). Ryan and Dupont (1973) identified and analyzed the major acids from fruit juices and wines and found quinic acid to be the main acid in prunes, the second most important acid in apples and of very high concentration in cranberries. Likewise, it was also reported by Bazzarini et al. (1986) that quinic acid was the characteristic acid of blueberries.

Quinic acid is one of the main fruit acids in the berries of the wild Northern species of *Empetrum* (Kallio and Markela, 1982) and *Vaccinium* (Fernandez-Flores et al. 1970; Coppola et al., 1978). It even accounted for 60% of the organic acid content of crowberry (*Empetrum nigrum* coll.) which

could be an indirect explanation for the well known good keeping quality of berry juice. Quinic acid is a polyhydroxylated derivative of benzoic acid, an approved antimicrobial agent that is widely used in the food and beverage industry. However, its role as an antimicrobial agent needs further investigation since conflicting results were reported by Schanderl (1962) and Kallio et al. (1985). Schanderl reported that 100 ppm of quinic acid was inhibitory to yeast fermentation in wine while Kallio et al. (1985) reported that quinic acid concentrations of up to 10,000 ppm had no effect on the growth of 3 yeast species and 3 mold species.

In Japan, the addition of quinic acid at a 1:100-1000 (quinic acid:organic acid) ratio was found to improve the flavor of products and is thus highly recommended (Takasago, 1981). In the United States, it is not GRAS yet, although its role in the food and beverage industry is known.

Coppola and Starr (1988) found that quinic acid is the most important and characteristic cranberry acid although malic and citric acids were also present in substantial amounts. As quinic acid is not currently available at prices to make it practical for use as an adulterant, its concentration in commercial juices can be used as an estimate of cranberry juice content (Wrolstad et al., 1988; Hong and Wrolstad, 1986; Coppola and Starr, 1988). The total acid content of kiwi fruit is about 17-18 mg/g fresh weight with quinic and citric acids as its main acids. MacRae et al. (1989) reported that for the outer cortex, inner cortex and core of kiwi fruit, respectively, the quinic acid concentrations (mg/g fresh weight) were 8, 5 - 6 and 3 compared to 6 - 8, 10 - 14 and 6 - 8 for citric acid.

Quinic acid is found in large quantities in the peel of citrus fruits (Ting and Attaway, 1971). Apple juice is not particularly acidic with malic acid as the dominant acid (about 71 to 94% of the total acids) followed by quinic acid

which is also present in substantial concentrations (Lee and Wrolstad, 1988). Hulme (1971) reported that in certain immature fruits, the levels of quinic acid were greater than that of malic acid. Quinic acid was found to be the main component of peach accounting for 25-35% of total organic acids with malic and citric acids as the other components (Tsugi et al., 1985).

Quinic acid can occur as a free acid or in esterified form. Free quinic acid is commonly found at levels in the range of 0.4 - 0.7%, but may be as high as 1.5% in discolored green coffee beans (Clifford, 1985). The esters, known as chlorogenic acid (CGA), form a quantitatively important fraction of green and roasted coffee beans, soluble coffee powders and coffee brews. CGA can be hydrolyzed to one molecule of caffeic acid and one molecule of quinic acid (Mabrouk and Deatherage, 1956; Sivetz, 1963) thus increasing the acid content of the coffee brew. Such acid changes are also accompanied by coffee flavor changes (Memory, 1968).

Sakata et al. (1986) first isolated (-)quinic acid salt as one of the major constituents of tea. Its contribution to the taste of tea remains to be clarified. Quinate in tea is not an artifact produced during tea processing. The reason that so much quinate accumulates in young tea leaves remains to be elucidated.

•SUCCINIC ACID

Succinic acid is one of the natural acids found in broccoli, rhubarb, beets, asparagus, fresh meat extracts, sauerkraut and cheese (Gardner, 1972). Its content in foodstuffs of plant origin is low in comparison with other carboxylic acids, e.g. citric, malic, tartaric acids, etc. (Velisek et al., 1978). Succinic acid is affirmed as generally recognized as safe (GRAS) under the Federal Food, Drug and Cosmetic Act and is used primarily as a flavor

enhancer and pH control agent in condiments, relishes, and meat products (Federal Register, 1979).

Fruits, vegetables, and non-alcoholic beverages have been shown to contain as much as 70mg/100 g of succinic acid (Fernandez-Flores et al. 1970; Heatherbell, 1974). In ciders and wines, 0.1% succinic acid is common while 0.03% in beers is considered a rather high concentration (Whiting, 1975). Its importance as a flavoring compound in dairy products is also well known (Randler, 1975). Succinic acid is an important flavoring substance for sake, a fermented beverage in Japan, (Kodoma, 1970). Succinic acid or sodium succinate have also been employed for preparing artificial flavorings, seasonings or condiment mixtures, and for giving foodstuffs a meaty flavor (Velisek et al., 1978). Yeast autolysates also contain relatively large proportions of succinic acid.

Succinic acid is manufactured by the catalytic hydrogenation of maleic or fumaric acid (Gardner, 1972). It has also been produced commercially by aqueous alkali or acid hydrolysis of succinonitrile. Succinonitrile is derived from ethylene bromide and potassium cyanide.

Succinic acid is odorless and has a sour, acid taste (Doores, 1990). Its apparent taste characteristics in foods appear to be similar to the other acidulants of this type, although pure aqueous solutions have been described as slightly bitter (Gardner, 1972). It is a nonhygroscopic acidulant with a slow taste buildup. It can extend shelf life of dessert powders without damaging flavors. It readily combines with proteins in modifying the plasticity of doughs and aids in the production of edible fats with desired thermal properties (Gardner, 1972).

Like malic acid, succinic acid has not been used extensively in food products for its antimicrobial activity (Doores, 1990). Cox et al (1974) have

found that succinic acid at levels of 3% or 5% at 60°C are as effective as acetic acid in decreasing the microbial load on chicken carcasses. Despite the antimicrobial nature of the compound, it is not commercially used because even at this concentration, it can adversely affect the appearance of the product.

Succinic acid can also be used as an index of microbial spoilage (Stijve and Diserens, 1987). For example, Littman et al. (1982) found that microbial spoilage of eggs is closely associated with an increase in succinic acid concentration.

•TARTARIC ACID

Tartaric acid is very seldom found in nature (Arnold, 1975) although it is found in many plants and is the predominant acid in grapes (Andres, 1985) and tamarinds (Hasan and Ijaz, 1972). The DL, Meso, D, and L are the four types of tartaric acids. The D type tartaric acid is the one present in nature (Kosobe, 1967) while the L-configuration is the tartaric acid of commerce (Gardner, 1972). Tartaric acid is poorly if at all absorbed in the mammalian gut and is excreted sometimes unchanged (Arnold, 1975). Natural tartaric acid, which is the ordinary type used in the industry is a (+) -enantiomorph. It is quite a strong acid, and dissolves freely in water. The racemic acid, often called Racemic acid, probably does not occur in nature and has much lower solubility. However, tartaric acid racemises with great ease, and a certain amount is always formed in the working-up of (+)-tartaric acid. The internally-compensated isomer, meso-tartaric acid, has been reported in apples.

It is necessary to select and control conditions precisely so that pure DL-tartaric acid (racemic acid) is obtained free from meso-tartaric acid.

Consumption of tartaric acid as the L(+) form or its salt form is limited unconditionally for humans up to a level of 30 mg/kg body weight (FAO/WHO, 1973). Subcutaneous administration of 0.25-1.0g of D-tartaric acid as the sodium salt to rabbits produced chemistry changes (Doores, 1990) .

The tartaric acid of commerce can be derived from insoluble tartrates precipitated during the processes of wine manufacture (Arnold, 1975). Very recently, the world's first plant for the production of synthetic tartaric acid was opened up in South Africa, using a process developed in England whereby maleic anhydride (acid) is oxidized in aqueous solution by hydrogen peroxide in the presence of a catalyst (Bewsey, 1977). The U.S. imports tartaric acid and tartrates from wine-producing countries such as Spain, Italy, France, and West Germany (Dzeziak, 1990). However, because of high prices and limited supplies, the use of tartaric acid has declined in the food industry (Anonymous, 1985).

Tartaric acid is the most water-soluble of the solid acidulants (Gardner 1972). Tartaric acid has a strong, tart taste and an ability to augment the flavors of fruits in which it is a natural constituent. It is widely used in grape- and lime-flavored beverages as an acidulant and as a flavor enhancer. Tartaric acid acts synergistically with antioxidants to prevent rancidity and discoloration of cheese (Gardner, 1972). It also has good sequestering power and stabilizes colors and vitamins. It is widely used in the baking industry and is also used in confectionery products, fruits, vegetables, gelled products, ice cream products and fats (Andres, 1985). Antimicrobial uses for this acid are limited (Doores, 1990)

TASTE PERCEPTION OF ACIDULANTS

Although sourness is the most common characteristic of all acids, many terms were generated by different panelists for evaluating malic, acetic, tartaric, fumaric and hydrochloric acids (Straub, 1992). Due to the panelists' disagreement in the use of other terms, astringency and sourness were chosen from among the lists. This shows that there were more characteristics to describe an acidulant and perception varies among the panelists which could have affected the sourness and astringency perception of the panelists. Likewise, a number of studies suggested that at least some "sour" stimuli produce sensations other than sour, including bitter and salty (McBurney and Schick, 1971; McBurney et al., 1972; Meiselman and Dzendolet, 1967), providing some explanation for the apparent "misidentification" of sour stimuli as bitter in forced test situations (McAuliffe and Meiselman, 1974; Robinson, 1970). Furthermore, this confusion is most readily observed near threshold (Gregson and Baker, 1973). Jellinek (1985) confirmed differences among panelists in evaluating citric acids with some describing citric acid as bitter, sweet or astringent.

Settle et al. (1986) reported the sour and non-sour taste sensations elicited by six concentrations of each of seven acids: citric, hydrochloric, sulfuric, lactic, malic, phosphoric and tartaric. In general, the acids differed significantly in their ability to elicit sour, salty and bitter sensations, with sulfuric and hydrochloric acids producing the smallest proportions of perceived sourness. Bitterness was found to be the largest non-sour sensation produced, followed by saltiness.

Sourness Perception

The perception of the sourness of acidic compounds has been studied extensively but the mechanism involved is still not clear (Ganzelves and Kroeze, 1988). Acids, when dissolved in water dissociate into ions according to the relationship:



in which HA is an undissociated acid molecule, H^+ is a hydrogen ion and A^- is the acid anion. Strong acids like HCl are completely dissociated while in organic acids the dissociation is not complete and the dissociation constant $K = k_1/k_2$ is related to the chemical structure. With the above equations, there are three possible candidates that may contribute to sourness: (1) H ion, (2) anion of the acid and (3) undissociated form of the acid. However, studies have been reported that acid sourness can also be related to the physiological reactions which occur at the receptor sites.

Taylor et al. (1930) studied the relative permeability of different acids in order to show the influence of various substituents in the acid molecule. He assumed that only the undissociated molecules of the acid can pass through the membrane and the physiological stimulus is due to the hydrogen ion concentration in the interior of the cell. Crozier (1916), on the other hand, suggested that the potentially ionizable hydrogen is a factor influencing sourness and cell penetration power. However, the probability that the mere presence of hydrogen completely defines the sour taste perception is small (Price and De Simone, 1977) since anion effects on acid taste are recognized (Beidler, 1967; 1971).

Beidler (1967) hypothesized that the importance of the anion to sourness perception was because its presence enhanced further binding of the hydrogen ion by preventing membrane charging. He explained that the

hydrogen ion interacts with many sites on the taste cell membrane (Beidler, 1971), some of which are involved in receptor stimulation. As hydrogen ions are adsorbed, the membrane becomes less negatively charged and decreases the rate of hydrogen ions approaching other unfilled receptor sites. If the negatively charged portion (anion) of the acid also binds, then the membrane becomes more negative and the number of hydrogen ions that can interact at a given pH is increased. Thus, not all acids are equally effective at equal pHs.

There have been several proposals attempting to explain the factors affecting the acid taste. Early studies conducted by Kahlenberg (1898) and Richards (1898) showed that the perception of sourness is due primarily to the hydrogen ion concentration but this was disputed by Furukawa et al. (1969). In a study by the latter group on the organic acids (butyric, fumaric, acetic, malic, lactic, ascorbic and acetic), the intensity of acid taste was found to be highly correlated with the dissociation constant (K) in an equimolar concentration.

Ganzelves and Kroeze (1987) stated that aqueous solutions of strong acids (i.e., K is large) taste more sour than weak acids (i.e. K is small) when the molar concentrations are equal. If hydrogen ion concentrations are equal, the sourness of weak acids is higher than that of the strong acids.

Kenrick (1931) and Beatty and Cragg (1935) proposed that the sourness of various acids is proportional to the respective amounts of phosphate buffer required to adjust equimolar solutions of each acid to a pH of about 5.0. Buffered solutions are equally or more sour than unbuffered solutions at the same pH. Fabium and Blum (1943) found that equal volumes of buffer were needed to titrate acid solutions of hydrochloric, malic and lactic acids which were at threshold concentrations. Beidler (1952) supported that perhaps the salt itself contributes to the sourness of buffered acid solutions. He postulated

that with higher anion concentration (the result of adding a buffer), the hydrogen ion could bind more readily to the receptor cells because of less membrane charging (Beidler, 1958; 1967). Pfaffman (1959) reported that buffered acid solutions retained their sour taste longer than unbuffered solutions. However, Ganzelves and Kroeze (1987) found that suppression of hydrogen ions by buffering acid solutions had no effect on sourness.

CoSeteng et al. (1989) reported that monocarboxylic acids were more sour than the dicarboxylic acids which in turn were more sour than tricarboxylic acid. Chauncey et al. (1967) reported that equi-molar concentrations of tartaric, lactic, acetic and citric acids produced significantly different salivary flow and with salivary flows correlated with sourness perception. Pangborn (1963) found that concentrations of lactic, tartaric and acetic acids of equal sourness were not equal in molarity.

The composition and flow rate of human saliva has been associated with sourness perception. Cragg (1937) found that tasters with more alkaline saliva required a higher concentration of HCl to match an acetic acid standard. It was found that buffering capacity of saliva differs among subjects. Bicarbonate as well as other salivary buffers may play a significant role in the perception of sour substances (Christensen, 1986). Preliminary results clearly showed that saliva significantly increased the pH of acidic tastants.

Moskowitz (1971) measured the psychological functions of acid sourness of 24 carboxylic acids. It appeared that no simple relationship exists between sourness and physico-chemical properties of the acids. This was also reported by Straub (1992). Harvey (1920) asked the question, "what do we taste in acid solutions, hydrogen ion concentration or total acidity?". He said that in the ingestion of such acidic foods as tomatoes or citrus fruits, the tendency

to produce hyperacidity is dependent also upon the total acidity or quantity of acid ingested and not necessarily upon the hydrogen ion concentration alone. Joslyn (1974) also reported that the sour taste of acids is influenced more by titratable acidity than by pH.

Shamil et al. (1987) hypothesized that taste is related to the compatibility between the stimulus and water structure. They reranked stimuli according to their apparent specific volume and found that as the measurement increases, taste perception ranged from salty to sourness, then sweetness and finally bitterness. However, they found that lactic and acetic acids fell into the sweet/bitter category depending on their specific molar volume. They attributed this to the fact that these molecules exist as dimers.

Many years of research have produced a complex pattern of information on the perception of sourness but so far no clear answer has emerged to the basic question of what stimulates sourness perception.

Astringency Perception

Astringency has been classified since the time of Aristotle as a basic taste. The word astringency comes from the Latin phrase, *ad stringere*, meaning "to bind". Bate-Smith (1954) defined astringency as "concerned with the sense of touch, and is due to the coagulation of proteins of the saliva and mucous epithelium in combination with the astringent body". It was defined by Joslyn and Goldestein (1964) as the mouth drying and puckering feeling produced by the interaction of polyphenols with the proteins of the mouth. However, others define astringency more as a taste rather than a tactile sensation, e.g. by Moncrieff 's (1971) reference to "a contracting or drying taste". It is still debatable whether, astringency is a tactile or a taste sensation because, like sourness, the precise mechanism of astringency is still not very

well defined. Historically, astringency has been considered of equal importance to the classic four taste qualities of sweet, sour, salty and bitter (Bartoshuk, 1978). Schiffman et al. (1991) reported that the chorda tympani nerve (taste nerve) rather than the trigeminal nerve transduces the signals for astringent compounds.

Astringency is an important sensory attribute of foods and beverages that contain astringent tannins, e. g., coffee, tea, beer, wine, apples, ciders, many berry crops, and nuts (Haslam and Lilley, 1988). Other astringent materials include salts and multivalent cations (Al, Cr, Zn, Pb, Ca, B), mineral acids, and dehydrating agents such as alcohol and dimethyl ketone (Haslam and Lilley, 1988). It is also one of the main sensory attributes of wine (Guinard et al., 1986).

The American Society for Testing and Materials (ASTM) Committee on Sensory Evaluation of Materials and Products defined astringency as "the complex sensations due to shrinking, drawing or puckering of the epithelium as a result of exposure to substances such as alums and tannins "(American Society for Testing and Materials, 1989). Previous research on the topic has not attempted to break down astringency into its multiple sub-qualities during psychological evaluation, allowing subjects to simply rate astringency by itself or some of its aspects such as dryness of the mouth (Lee and Lawless, 1991).

The chemical reactions underlying astringent sensations are poorly understood, although several mechanisms have been proposed (Lee and Lawless, 1991) involving polyphenols (McManus et al., 1981; Hagerman and Butler, 1981). One of the most popular theories is that polyphenolic compounds such as tannins form complexes with salivary proteins and/or mucopolysaccharides, which either precipitates them or causes sufficient

conformational changes so that they lose their lubricating power, and thus make the mouth feel rough and dry (Bate-Smith, 1973).

Crozier (1920) reported that acids exhibit a characteristic astringency in solutions so dilute that they are no longer sour. Likewise, with our reported astringency of acids (particularly hydrochloric and phosphoric acids) an expanded mechanism or set of mechanisms for astringency that takes into account not only tannin-protein interaction but also acid-protein interactions, also needs to be studied (Rubico and McDaniel, 1992). One difficulty pointed out in the literature is the common practice of studying astringency in complex media or beverages such as wine (Arnold et al., 1980; Noble et al., 1984; Guinard et al., 1986). While the approach is understandable from a practical point of view, caution must be exercised since other chemical components in the food may interact with the astringent substance which can confuse the results (Lee and Lawless, 1991). The interference of other components in the food may alter not only the salivary flow but also the degree of oral lubrication (Lyman and Green, 1990). Another difficulty in studying astringency is that observers tend to confuse astringency and bitterness.

Bitterness Perception

Bitterness is one of the four so-called "basic tastes" which is usually associated with alkaloids, certain amino acids and peptides, some phenolic compounds (including glycosides), urea and derivatives, certain diterpenes, and the hop iso-alpha and alpha-acids (Leach, 1984). Alkaloids comprise many bitter compounds including caffeine and quinine. Most large phenolic compounds are bitter and astringent while some of the naturally occurring glycosides are solely bitter like naringin, the compound responsible for the

bitterness of grapefruit. Other bitter amino acids and peptides include arginine, valine, leucine, and tryptosine (Kirimura et al., 1969). Likewise, picric acid which derives its name from the Greek word "pikros", meaning "bitter" is often described as bitter.

With the diversity in chemical structure of bitter substances, it is difficult to find a chemical structure common to all of them (Kurihara, 1987). As a result, Teeter and Brand (1987) even hypothesized that unique receptors may not actually exist for bitterness. Many bitter compounds are lipophilic. This means that the perception of bitterness may be mediated by the solubilization of the stimulus within lipid bilayer of the taste receptor-cell membrane (Koyama and Kurihara, 1972) resulting in a change in surface potential (Kumazawa et al., 1985) and/or activation of a second messenger system.

Correlations between intensity and for example, oil/water partition ratios or other hydrophobicity-solubility indices have been developed for many bitter compounds (e.g. Gardner, 1979; Greenberg, 1980). Biedler and Gross (1971) reported that there is no specific theory that accounts for all bitter molecule receptor site interactions. Kubota and Kubo (1969) found certain bitter molecules that react in a manner similar to that shown by Shallenberger for sugars except that the AH-B distance in the receptor site is assumed to be 1.5Å rather than 3Å. This finding is particularly interesting since many very sweet molecules can be changed to very bitter by a slight change in structure (Beidler and Gross, 1971).

The outstanding difference between bitter and other sapid substances is that the bitter ones are detectable in much higher dilutions (Moncrieff, 1970b). Moncrieff (1970a) reported the detection threshold concentrations for typical "basic" taste substances as follows:

Sour	0.007% HCL
Salt	0.25% NaCL
Sweet	0.5% Sucrose or 0.001% Saccharine
Bitter	0.00005% Quinine

This detection sensitivity for bitterness even at very low concentrations may be a human evolutionary adaptation to help us avoid hazardous foods (Akabas et al., 1988). Another interesting difference is the way the surface of the tongue responds to sweet, salt or sour. The changes occurring in the gustatory (taste) nerves in response to these three taste categories results in changes in measured electrical currents. However, bitter tastes provoke no such current flows (Akabas et al., 1989). So, even after decades of research, chemists barely understand the mechanism by which the bitter sensation is perceived on the surface of the tongue. Bitter still remains the least understood among the basic tastes (Mor et al., 1989).

Umami Perception

A variety of taste qualities have been postulated since ancient times. For example, Aristotle stated that there were only two primary tastes; sweet and bitter (Kurihara, 1987), leaving out salty and sour. Nowadays, debate still continues as to whether there are only four primary tastes i.e., sweet, sour, salty, bitter or more. According to Henning's (1916) model, taste can be formally viewed as a tetrahedron, with the four basic tastes placed at each corner. Henning believed the tetrahedron to be hollow because no compounds have been identified to produce all four tastes. However, until now, no conclusive evidence in favor of pure primaries has been found.

Schiffman and Erickson (1980) believed that the idea of taste is more "continuously" organized along many tastes other than the traditional four.

This alternative model is more consistent with existing research data whether studied at the stimulus, neural or psychological levels. The existence of this controversy may be in part due to the fact that the biochemical events associated with taste have not been fully resolved (Mela and Mattes, 1988a).

A more complete overhaul of the tetrahedral model may be brought about by the existence of a fifth taste category. The most likely candidate for this is the umami taste, which is generally accepted as a basic taste in Japan. The conclusions made from multivariate measures indicated that the spatial locus of MSG lay outside the tetrahedron generated by the four primary tastes (Yoshida, 1963; Schiffman and Gill, 1987). This was also true for sodium succinate (Schiffman and Gill, 1987) and succinic acid which was confirmed by our studies as exhibiting a distinct umami taste (Rubico and McDaniel, 1992).

Umami, derived from the Japanese word meaning "deliciousness", refers to the characteristic sensation elicited by monosodium glutamate (MSG). This taste is common to meat, fish, certain vegetables, mushrooms, and cheese, but in its more generally recognized definition, it is a taste quality represented typically by glutamates and 5'-nucleotides (Kare, 1987). MSG interacts sensorially with other food components and this may be the reason for the uncertainty that exists about its role as a basic taste or flavor enhancer (Birch, 1987).

In a study by Halpern (1987), the quality terms used to describe MSG are "bouillon" and "fish" but the terms "meat" or "meatlike" were absent. He explained that the absence of meat-related descriptions for the MSG solution was perhaps related to Boudreau et al.'s (1979) suggestion that several different taste receptor populations may respond to umami stimuli. In our study, the Oriental panelists described succinic acid to have an MSG taste while the Caucasians used the term 'savory' and 'brothy' (Rubico and

McDaniel, 1992). In English, umami taste has been described more commonly as 'Chinese food taste', meaty or 'bouillion like' (O'Mahony and Ishii, 1985).

Umami substances do not usually have high pleasantness ratings in simple solutions but earn high pleasantness scores in foods and solutions compatible with the added umami substance (Kawamura and Halpern, 1987). The uniqueness of MSG taste (O'Mahony and Ishii, 1987) is said to be independent of the four primary tastes (Kawamura and Halpern, 1987).

SENSORY PROFILING

Sensory evaluation resolves into: (1) evoking, (2) measuring, (3) analyzing and (4) interpreting sensory responses (Powers, 1984). The most advanced and sophisticated sensory method applying all of the above functions is called sensory profiling. Sensory profiling has moved a long way since it was first conceived by A.D. Little Corporation in the 1950's (Caul, 1957) and was named "The Flavor Profile" method.

Since Flavor Profile was developed, many attempts have been made to modify or improve upon the method. Nowadays, descriptive analysis and free-choice profiling are the two most commonly used techniques in sensory profiling although the latter was introduced only in the early 1980's. Both of these methods, their differences, similarities and corresponding statistical analyses, will be reviewed in this chapter.

Descriptive Analysis (DA)

Descriptive analysis, also referred to as quantitative sensory (Powers, 1988), consensus (Tunaley et al., 1988), or fixed-choice (MacFie, 1987) profiling,

is the method by which information about products has been obtained by sensory scientists for many years (Oreskovich, et al., 1991). It is the sensory method by which the attributes of a food material or product are identified, described and quantified using human subjects who have been specifically trained for this purpose (Einstein, 1991). The modifications for the Flavor Profile are as follows: texture profiling (Brandt et al., 1963; Szczesniak, 1963; Szczesniak et al., 1963; Munoz, 1986), Quantitative Descriptive Analysis (Stone et al., 1974; Stone and Sidel, 1985) and Spectrum (Meilgaard et al., 1987). Likewise, several extensive reviews have been written on these different methods (Einstein, 1991; Gatchalian, 1989; Powers, 1988; Piggott and Canaway, 1981).

All the descriptive methods follow these procedures: a) selection and training of subjects b) development of a language c) evaluation sessions and d) data analysis and interpretation. In descriptive analysis, the panelists develop a common vocabulary to characterize the product. However, even with panelists training, these sources of variation might not be eliminated completely (Arnold and Williams, 1986): i) assessors vary in their overall level of scoring; ii) assessors use descriptors in different ways; iii) assessors vary in their range of scoring; iv) assessors vary in their use of terms and scales between sessions and v) assessors might perceive different stimuli in the same products. Likewise, several drawbacks of this method are the tremendous amount of time needed to develop a common product-oriented language and not all assessors use a given descriptor (Quarmby and Ratkowsky, 1988).

Free-Choice Profiling (FCP)

The most important difference from conventional profiling is that with free-choice profiling (FCP) each assessor is free to come up with and use his/her own descriptive terms in a more natural way. It is not necessary that the assessors first learn a common language to be able to assess the product in a consistent way. Thus, this approach is faster than conventional profiling because it lacks the need for elaborate training (William and Langron, 1984).

Free-choice profiling was first developed by Williams and coworkers (1981). This method was used to assess commercial ports (William and Langron, 1984), coffee (Williams and Arnold, 1985), fish (Quarmby and Ratkowsky, 1988), sweeteners (Tunaley, 1988), chocolate (McEwan et al., 1989), cheese (Marshall and Kirby, 1988), whiskey (Guy et al., 1989), beer (Gains and Thomson, 1990), milks (Raats and Shephard, 1992) and cider (Piggott and Watson, 1992).

The procedures being followed in free-choice profiling has a lot of similarity with descriptive analysis. Panelists are recruited, selected and trained in scale usage. They must be capable of using scales for intensity rating, must be objective in their assessment and once having defined an attribute must be consistent in its use (Williams and Langron, 1984). Usually, the number of attributes are different among panelists and these large number of terms usually makes interpretation of results difficult and time consuming (Tunaley, 1988). However, Williams and Arnold (1985) showed that FCP with scores analyzed by General Procrustes Analysis (GPA) (Gower, 1975) demonstrated similar results to conventional profiling and similarity scaling. Similar conclusions were also reported by Scriven and Yak's (1991)

study among the usage behavior of meat products by Australians and Hong Kong Chinese when FCP and Consensus Profiling were compared.

FCP permits the use of untrained assessors, because there is no need for training in the use of descriptors. Thus, it can be applied to consumer research (Guy et al. 1989). They recruited 100 subjects to study the profile of 8 Scotch whiskies and found that FCP was useful for consumer research, though difficulties were experienced in the interpretation of the results. In addition, some assessors have difficulty in generating sufficient descriptors (Piggot et al., 1991).

STATISTICAL ANALYSIS

General Procrustes Analysis

For analyzing free-choice profile data, Procrustes analysis is the only statistical method used. It is a technique that allows for individual differences and can give information about these differences (Gower, 1975). The method is named after *Procrustes*, the innkeeper in Greek mythology who stretched his guests or lopped off their limbs to make them match the inn beds (Digby and Kempton, 1987).

The goal of Procrustes analysis is to blend each individual's configurations into a common space. It allows transformations to be performed while maintaining the shapes of configurations, thus, enabling one to compare discriminating ability independent of adjective scores and scales used. The analysis consists of three logical stages (Arnold and Williams, 1986; Gains et al., 1988; Dijksterhuis and van Buuren, 1989): firstly, the centroids of each assessor's data are matched so as to eliminate the effect of

use of different parts of the scales; secondly, isotropic scale changes remove the differences in the scoring range used by different assessors; thirdly, the configurations are matched as closely as possible by rotation and reflection of axes (Arnold and Williams, 1986). This produces a perceptual space for each assessor, which is matched as closely as possible with other assessors. Then the average configuration from all the assessors is calculated to produce a consensus configuration usually simplified as a reduced dimensional plot by principal component analysis (PCA). GPA can show the intersample distances, and whether the panelists had agreements/ disagreements on their ratings based on the calculated residuals. Likewise, GPA can indicate whether the panelists used a narrow or wide range of descriptors or scores, i.e, whether he/she is an extrovert or an introvert panelist.

Principal Component Analysis

When performing descriptive analysis, several multivariate statistics are applicable, e.g., principal component analysis (PCA) being the most common one. PCA has been used in the evaluation of the following products: whiskies (Piggott and Jardine, 1979), beer (Clapperton, 1979), meat (Horsfield and Taylor, 1976), poultry products (Frijters, 1976). Syarief et al. (1985) recommended that 1/3 of the original attributes can be used in profiling foods after PCA analysis because the attributes used to describe the flavor profile of beefsteaks, fish gels, frankfurters, peanut, peanut butters and baked sweet potatoes were not completely independent. They explained that a lot the terms used by the panelists were redundant and the deletions of the terms can avoid panel fatigue and can reduce both the time and cost of evaluation.

Principal component analysis can be applied to only one data set at a time which is usually the averaged data across panelists, or across panelists and replications, for each attribute (Powers, 1984). Use of a large single data set is not recommended (MacFie, 1987) because the resulting configuration becomes very crowded and maybe difficult to interpret (Oreskovich et al., 1991).

The specific goals of PCA are to summarize patterns of correlations among observed variables, to reduce a large number of observed variables to a smaller number of factors, to provide operational definition (a regression equation) for an underlying process by using observed variables, or to test a theory about the underlying process (Tabachnik and Fidel, 1989; Johnson and Wichern, 1988). Its function is to reduce a set of individual items into components such that the first component has a maximum correlation with all the variables and accounts for the greatest variance, the second-largest amount of variance, etc., until as much of the variance has been accounted for as is reasonable (Powers, 1984).

Tabachnik and Fidel (1989) listed two criteria for choosing the number of components: 1) size of eigenvalues and 2) scree test. Eigenvalues represents variance so a component with an eigenvalue less than one is not as important. The scree test is a plot of eigenvalue plotted against the factors or components (Cattell, 1966). As a rule of thumb, only variables with loadings of 0.30 and above are interpreted (Cliff, 1987) and the greater the loading, the more the variable is a pure measure of the factor (Tabachnik and Fidel, 1989). However, interpretation of lower loadings is warranted if the sample produces similar scores on the observed variables (Tabachnik and Fidel, 1989). The .30 cutoff corresponds to about 10% of the variance but sometimes .20 is used as a cutoff or sometimes raised to .40 (Cliff, 1987).

Another "rule of thumb" is to include just components explaining 90% of the total variation (Mardia et al., 1982). However, this should depend on the interpretability of results (Gauch, 1982). Gauch reported that in ecological data, there are cases where 90% of the variance may be accounted for but the result is meaningless or distorted while a 5% variance for the first couple of PCA can be quite informative ecologically. So, Tabachnik and Fidel (1989) believed that a good PCA is the one that makes sense and a bad one does not.

PCA is very useful for studying interdependency and the underlying dimensions in flavor profile analysis (Syarif et al., 1985). This could help reduce the number of terms in designing a ballot for further experiments (Ennis et al., 1982). Reduction of the number of attributes being evaluated can improve reliability and validity, avoid panel fatigue, and reduce both the time and cost of evaluation.

According to Johnson and Wichern (1988), "Analyses of PCA are more of a means to an end rather than an end in themselves because they frequently serve as intermediate steps in much larger investigations. For example, principal components may be inputs to a multiple regression or cluster analysis." Alternatively, it has been recommended that, since PCA and GPA provide different information, the former can be used in conjunction with Procrustes Analysis (Oreskovich et al., 1991).

Sensory Evaluation of Acids By Free-Choice Profiling

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ABSTRACT

The technique of Free-Choice Profiling was applied in order to characterize the sensory properties of some common organic and inorganic acids. Analysis of panelists' scores by generalized Procrustes Analysis (GPA) provided information on the relationships among samples and assessors for both the consensus and individual configurations. Results indicated that on a weight basis (w/v or v/v), acids differed in their flavor and taste dynamics. Acids were described differently by individual panelists.

The GPA resulted in three important principal axes (PA). The first PA had astringency/mouthfeel as the most important factor, while bitterness and sourness were the most important for the second and the third PAs, respectively. At 0.08% (w/v or v/v), the inorganic acids, hydrochloric and phosphoric, were more astringent than sour. The bitterness of succinic (S) was intense as was the sourness of fumaric, malic and the combinations of fumaric:malic (FM), citric:malic (CM) and citric:fumaric (CF). The sensory characteristics of adipic and quinic were perceived to be very weak at this concentration. The relationship between astringency and pH was more evident than was the relationship between pH and sourness.

INTRODUCTION

Acidulants have more functional uses than any other class of ingredients (Andres, 1985). Their preserving, buffering, chelating, and acidulating role in leavening systems (Gardner, 1966) are just some of the many functions they possess, but one of the best known functions is as a flavoring adjunct. With the right amount of acid, flavor and palatability of many foods are enhanced and modified. Thus, knowledge of the sensory attributes of acids is critical for both acidulant producers and their customers.

Organic acids have not been studied in detail with respect to their tastes despite the fact that they are very important ingredients in foods (Furukawa et al., 1969). Recently, Straub (1992) and Straub and McDaniel (1989), reported the equi-sourness levels and time-intensity ratings on sourness and astringency of seven organic and one inorganic acids. Different acids were described differently by individual panelists and disagreements arose in the use of descriptors except for the terms astringency and sourness. The acids differed significantly in their ability to elicit sour, salty and bitter sensation, with sulfuric and hydrochloric acids producing the smallest proportions of perceived sourness (Settle et al., 1986). Differences are due to the chemical and physical properties of the acids as well as to physiological differences among individuals.

The latest and only technique for describing a product that takes into consideration differences among panelists' perception is the method of free-choice profiling (Langron, 1981; Arnold and Williams, 1986). The assessors are allowed to use their own words and phrases and not those proposed by others. The panelists require less training and hence the total process takes less time. The method also takes into account minor individual variation

while highlighting those panelists who respond differently from the rest (Marshall and Kirby, 1988). Free-choice profiling (FCP) has been used to assess the appearance, flavor and aroma of commercial ports (Williams and Langron, 1984), coffee aroma (Williams and Arnold, 1985), flavor and odor profile of fish spoilage (Quarmby and Ratkowsky, 1988), perceptual characteristics of sweeteners (Tunaley, 1988), appearance, flavor and texture of chocolate (McEwan et al., 1989), cheese texture (Marshall and Kirby, 1988), consumer profiling of whisky (Guy et al., 1989) and canned lager beers (Gains and Thomson, 1990).

Williams and Arnold (1985) showed that FCP with scores analyzed by Generalized Procrustes Analysis (GPA) (Gower, 1975) demonstrated similar results to conventional profiling and similarity scaling. The goal of GPA is to blend the individual configurations into a common space. The analysis consists of three logical stages (Arnold and Williams, 1986; Gains et al., 1988; Dijksterhuis and van Buuren, 1989): first, translation to correct for variations in using different parts of the scale; secondly, rotation/reflection to remove variations for the differences in terms used; and thirdly, an isotopic scaling to correct for the range effect. GPA provides information on the relationships among samples and assessors including the consensus and individual configurations.

The objectives of this study were to investigate the sensory characteristics of some common food acidulants using free-choice profiling, and determine differences and similarities among the acids.

MATERIALS AND METHODS

Samples

Fifteen samples of acids (eight organic, three organic mixtures and two concentrations of glucono-delta-lactone plus two inorganic) were evaluated. Anhydrous citric, DL-malic and fumaric acids (FCC) were obtained from Haarmann and Reimer (H&R) Company (formerly Miles Inc., Elkhart, IN), adipic and phosphoric acid (75%) from Monsanto (St. Louis, MO), and glucono-delta-lactone from Finnsugar Bioproducts (Schaumburg, IL). Hydrochloric (NF-FCC, 36.5-38.0%) and lactic acids (USP-FCC, 85-90%) were purchased from J. T. Baker Inc. (Jackson, TN), D-quinic acid from ICN Biomedicals, Inc. (Cleveland, OH) and succinic and tartaric from Penta Manufacturing Co. (East Hanover, NJ). The acid levels were 0.08% w/v except for lactic, phosphoric and hydrochloric (0.08% v/v) and two higher concentrations of glucono-delta-lactone (0.16% and 0.24% w/v). The latter was due to its weak sensory characteristics at 0.08%. The chosen concentrations were based on preliminary sensory evaluations. The panelists reported that a 0.10% concentration was too strong for most of the acids while 0.05% was too weak. Aside from the pure acid, solutions of 50/50 citric:malic, 50/50 citric:fumaric and 50/50 malic:fumaric were prepared. The acids were diluted with spring water (Opal Spring, Culver, OR).

Panelist

The panel was comprised of four male and eight female graduate students and staff from the Department of Food Science and Technology at Oregon State University. All had panel experience as trained panel members or as a panel leader.

Presentation of Samples

All samples were clear, colorless solutions which were presented at room temperature in coded 85 ml plastic cups. During initial vocabulary development, all 15 samples were coded and evaluated in random order. For the actual assessment, 15 samples were divided into 3 sets (A, B, C) of five and presented in counterbalanced order among the 12 panelists. For every session, five samples were evaluated. One replication (15 samples) was completed within one week with three sessions a week. Four replications were done. The groupings of the acid and their codes were as follows: Group A - adipic (A), citric (C), hydrochloric (H), lactic (L), fumaric:malic (FM); Group B - 0.16% glucono-Delta-Lactone (G1), phosphoric (P), fumaric (F), quinic (Q), citric:malic (CM) and Group C - malic (M), succinic (S), tartaric (T), 0.24% glucono-delta-lactone (G2), citric:Fumaric (CF). The combinations (FM, CM, CF), the two inorganic acids (H and P) and the two glucono-delta-lactones (G1 and G2) were placed in separate groups.

Assessment Procedure

The concept of free-choice profiling was introduced to the panelists during the first of four practice sessions. Different concentrations of citric acid were given to the panelists in order to practice scoring the magnitude of different characteristics on a 16 point intensity scale (0=none, 7=moderate, 15=extreme). This scale was used for the entire experiment. Then all 15 samples (coded) were presented twice at one sitting and panelists were asked to list all sensory characteristics which described the perceived attributes of the samples. Panelists were seated in separate, well ventilated daylight-illuminated booths. Panelists had to define their own terms for use in subsequent testing. A score sheet was then developed by each panelist for use during actual assessment.

When tasting all the acid solutions, the 'sip-and-spit' method was applied. The panelists were instructed to sip the sample and manipulate in the mouth for 7 s, then expectorate and rinse the mouth three times with spring water. To minimize adaptation effect a resting period of 60 s was imposed between samples.

Statistical Analysis

The data were analyzed by Procrustes Analysis using Procrustes-PC Version 2.0 (Dijksterhuis and van Buuren, 1989) and by Statistical Analysis System for Personal Computer (SAS, 1987, Cary, NC). For the free-choice profiling experiment the data from each of the 12 panelists were assembled into matrices of 60 rows (15 samples assessed over 4 replications) by n columns where n represented the number of attributes for each panelist. Using these matrices a generalized Procrustes analysis was carried out. Analysis of variance on the principal axis scores was used to ascertain differences among samples and, where appropriate, least significant difference.

pH and Total Titratable Acidity Determinations

The pH of the 15 acid samples was measured by a pH electrode with a microprocessor pH/mV meter (Orion Model 811) equipped with a combination pH electrode (Ross Model 81550). Total titratable acidity was determined using a glass electrode and titrating with 0.1N NaOH to an endpoint of pH 8.2

RESULTS AND DISCUSSION

Sensory Results

Free-choice profiling of the 13 acid samples on an equal weight basis (0.08%) and two higher concentrations of glucono-delta-lactone (0.16 and

0.24%) generated between seven (Panelist 2) and fifteen (Panelist 12) terms with an average of ten terms per panelist (Table 2.1). As expected, not all individuals behaved similarly and the number of dimensions required to describe differences among the acids varied. Sour, astringent and bitter were the common terms used by the panelists. Even with different numbers of descriptors, the General Procrustes Analyses (GPA) can maximize the agreement among the panelists with respect to the way in which they scored each individual sample.

The final result of a GPA is a consensus configuration of the samples for the different principal axis combinations as graphically presented in Figures 2.1 and 2.2, for the first principal axis (PA) vs. second PA and first PA vs. third PA, respectively. The figures illustrate the intersample distances. To supplement the graphical information from GPA, an analysis of variations (ANOVA) of the scores on each of the first four principal components of the consensus was carried out; results are given in Tables 2.2, 2.3 and 2.4 for the first, second and third components, respectively. The scores for the fourth axis were not significant and, therefore not reported.

Examination of the combination of descriptors used by each of the panelists in defining the axes enables one to get some indication as to their meaning in terms of each individual's own terminology. Likewise, the combination explains what descriptors are important in differentiating the different acid samples for every principal axis by each panelist as summarized in Table 2.5. The more important attributes have high loadings and are highlighted.

The first three axes explained only 36% of the variation. This percentage was low in comparison with the reported values of 58% for consumer and 74% for trained panel data in evaluating eight whiskies (Guy et

Table 2.1. Terms Generated by Each Panelist in the Free Choice Profiling of Acids.

PANELISTS											
Pan 1	Pan 2	Pan 3	Pan 4	Pan 5	Pan 6	Pan 7	Pan 8	Pan 9	Pan 10	Pan 11	Pan 12
1. acid	acid	sour	sour	sour	sour	sour	sour	sour	sour	sour	sour
2. astringent	astringent	astringent	astringent	strong	astringent	astringent	astringent	astringent	sharp	astringent	astringent
3. bitter	medicine	bitter	bitter	mild	bitter	green tart	bitter	bitter	bitter	bitter	bitter
4. sour	bitter	salty	cold taste	slow feeling	bright sour	sweet	salty	salty	lemon	fruity	mild
5. baking soda	citrus	metallic	vinaigre	strange sour	fast astringent	citrus	acid	impact	smooth	mouth puckering	citric
6. sweet	apple	lemony	piquant	stick	sweet	chalky	aspirin	lemon	tartaric	biting initially	aspirin
7. salty comfortable	fruity	cherry	beer	sour but	sharp puckering	vinegar aftertaste	length	linger	mouth	clean	numb
8. maple		clean	fermented		fruity	sickening bad	viscosity	smooth	linger/astringent	metallic	abrasive
9. lemon		aspirin			dull sour			tooth astringent	aspirin		metallic
10. musty		savory			brothy			msg			msg
11.		length			tooth astringent						quick
12.					metallic						aftertaste
13.											lingering
14.											acetic
15.											spoil

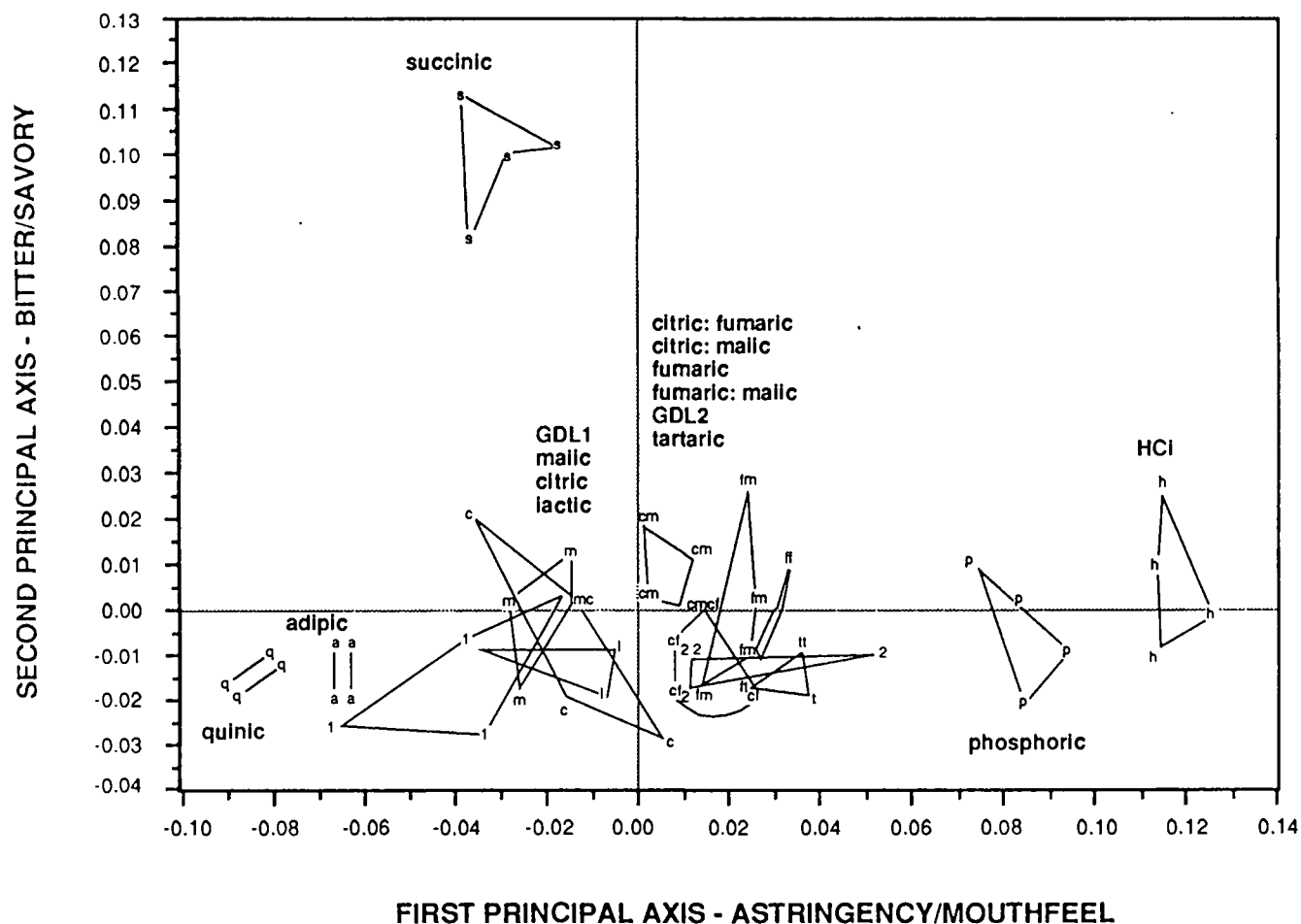


Fig. 2.1. Sample consensus plot for free-choice profiling of different acids (0.08%) following Generalized Procrustes analysis (GPA) : Principal Axes 1 vs 2. a-adipic, c-citric, f-fumaric, h-hydrochloric, l-lactic, m-malic, p-phosphoric, q-quinic, s-succinic, t-tartaric, x-citric:fumaric, y-citric:malic, z-fumaric:malic, 1-0.16% glucano-delta-lactone (GDL), 2-0.24% GDL. Four points represent four replications across 12 panelists.

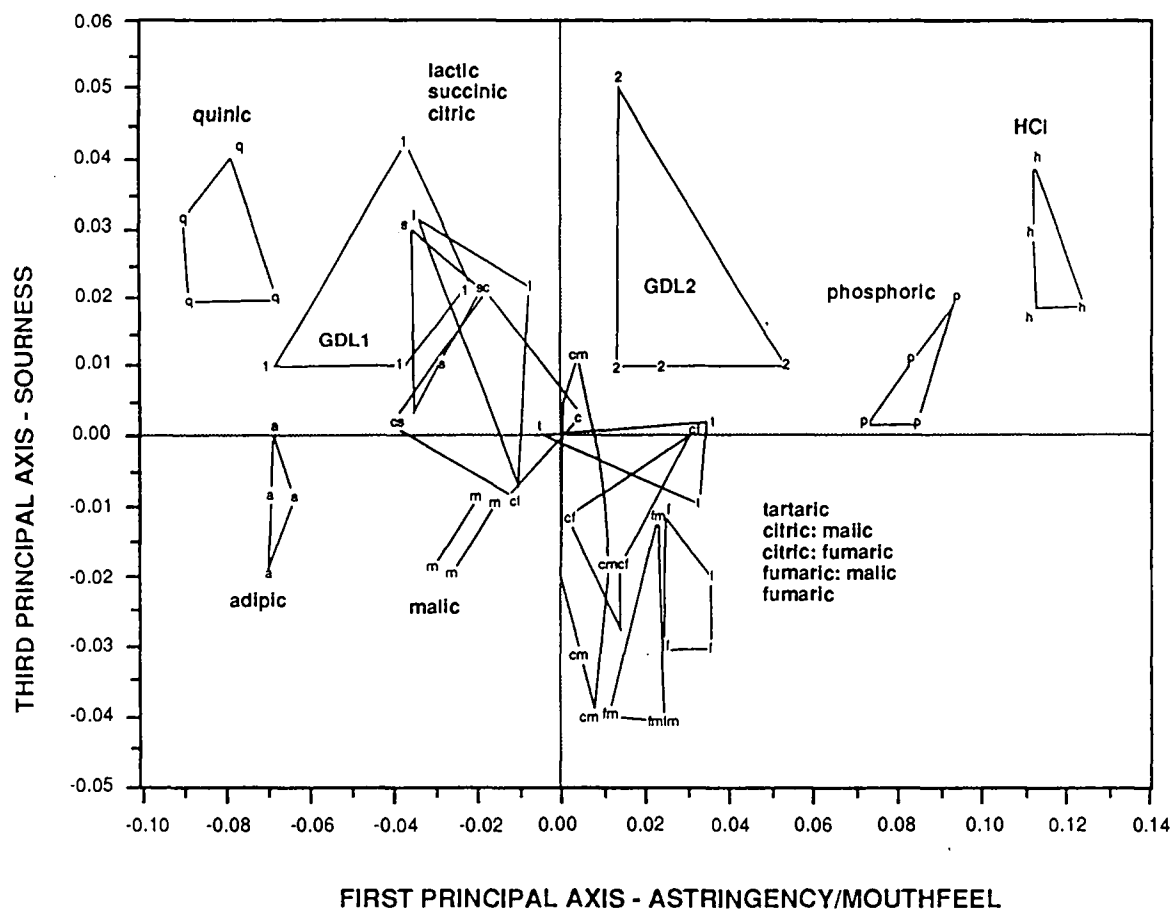


Fig. 2.2. Sample consensus plot for free-choice profiling of different acids (0.08%) following Generalized Procrustes analysis (GPA) : Principal Axes 1 vs 3. a-adipic, c-citric, f-fumaric, h-hydrochloric, l-lactic, m-malic, p-phosphoric, q-quinic, s-succinic, t-tartaric, x-citric:fumaric, y-citric:malic, z-fumaric:malic, 1-0.16% glucano-delta-lactone (GDL), 2-0.24% GDL. Four points represent four replications across 12 panelists.

Table 2.2. Principal axis (PA) 1: mean scores for different acids.

ACID ^a	MEAN SCORE ^b
Hydrochloric (H)	0.1125 ^a
Phosphoric (P)	0.0800 ^b
Fumaric (F)	0.0250 ^c
Glucano-Delta-Lactone (0.24%) (G2)	0.0225 ^c
Fumaric:Malic (FM)	0.0175 ^{cd}
Tartaric (T)	0.0175 ^{cd}
Citric:Fumaric (CF)	0.0125 ^{cd}
Citric:Malic (CM)	0.0050 ^d
Lactic (L)	-0.0175 ^e
Citric (C)	-0.0200 ^e
Malic (M)	-0.0250 ^e
Succinic (S)	-0.0325 ^{ef}
Glucano-Delta-Lactone (0.16%) (G1)	-0.0425 ^f
Adipic (A)	-0.0700 ^g
Quinic (Q)	-0.0825 ^g

^a Different letter superscripts indicate significant differences at $p < 0.05$

^b All acids at 0.08% unless noted

Table 2.3. Principal axis (PA) 2: mean scores for different acids.

ACID ^a	MEAN SCORE ^b
Succinic (S)	0.0755 ^a
Hydrochloric (H)	0.0075 ^b
Citric:Malic (CM)	0.0075 ^b
Citric:Fumaric (CF)	0.0000 ^{bc}
Fumaric (F)	-0.0025 ^{bc}
Malic (M)	-0.0025 ^{bc}
Phosphoric (P)	-0.0050 ^{bc}
Tartaric (T)	-0.0050 ^{bc}
Citric (C)	-0.0075 ^{bc}
Citric:Fumaric (CF)	-0.0100 ^{bc}
Lactic (L)	-0.0125 ^c
Glucano-Delta-Lactone (0.24%) (G2)	-0.0125 ^c
Adipic (A)	-0.0150 ^c
Quinic (Q)	-0.0175 ^c
Glucano-Delta-Lactone (0.16%) (G1)	-0.0175 ^c

^a Different letter superscripts indicate significant differences at $p < 0.05$

^b All acids at 0.08% unless noted

Table 2.4. Principal axis (PA) 3: mean scores for different acids.

ACID ^a	MEAN SCORE ^b
Quinic (Q)	0.0275 ^a
Hydrochloric (H)	0.0275 ^a
Glucano-Delta-Lactone (0.16%) (G1)	0.0200 ^{ab}
Glucano-Delta-Lactone (0.24%) (G2)	0.0200 ^{ab}
Succinic (S)	0.0150 ^{abc}
Lactic (L)	0.0150 ^{abc}
Phosphoric (P)	0.0075 ^{abc}
Citric (C)	0.0250 ^{bcd}
Tartaric (T)	-0.0025 ^{cdef}
Adipic (A)	-0.0100 ^{defg}
Malic (M)	-0.0150 ^{efgh}
Citric:Fumaric (CF)	-0.0150 ^{efgh}
Citric:Malic (CM)	-0.0200 ^{fgh}
Fumaric (F)	-0.0225 ^{gh}
Fumaric:Malic (FM)	-0.0325 ^h

^a Different letter superscripts indicate significant differences at $p < 0.05$

^b All acids at 0.08% unless noted

al., 1989), and 59% for five chocolates (McEwan et al., 1989) for the first three axes. Also, with 96% for eight ports (Williams and Langron, 1984), 60% for eight yoghurts (Dijksterhuis and Punter, 1990), 86% for six coffee varieties (Williams and Arnold, 1985), and 80% for five cheeses (Marshall and Kirby, 1988) for the first two axes. The lower percentage of variation explained could be due to the differences in the GPA program used. Other researchers (except Dijksterhuis and Punter) used the GENSTAT macro which is based entirely on the Gower (1975) method while the PROCRUSTES-PC is based on the Berge and Knol (1984) and Peay (1988) methods. Dijksterhuis and Punter (1990) discussed the differences between these two methods. The lower percentage could also be partially due to both the number and nature of acid samples evaluated in this study. The panelists evaluated 15 acids, almost twice the number of samples in other FCP reported studies. Likewise, difficulty arose in differentiating samples which are similar sensorily, particularly the acid combinations. In addition, some panelists may have used an excessive 'check-list' of highly correlated descriptors which is a common problem in the use of FCP (Gains et al., 1988). However, the large number of attributes used was probably because there were very large differences in the sensory characteristics of the 15 acid samples. In some cases, a particular attribute was only applicable to one sample, thus, creating a number of zero scores for the other samples. These data were analyzed by removing those attributes that were not significant in the ANOVA ($P < 0.05$) and had less than 0.30 loading. However, interpretation was very similar, improving the tightness of the groupings only marginally while the variation explained increased just slightly from 36% to 41%. Thus, the analysis of the original data is reported here.

The first PA had astringent as the descriptor with the highest positive loading for panelists 2, 3, 6, 7, 8 and 9 (Table 2.5). It also appears to be important for panelists 1, 4 and 10. This axis was also described by other panelists as biting, stick, abrasive, sharp and mouth puckering. These terms probably also described the mouthfeel when tasting an astringent sample. Four groupings were evident in the consensus configuration (Figure 2.1). Hydrochloric (H) and phosphoric (P) acids which formed the first group were very astringent compared to the other acids. The combinations citric:fumaric (CF), citric:malic (CM), fumaric:malic (FM) together with fumaric (F), 0.24% glucono-delta-lactone (G2) and tartaric (T) formed the second group. They were also high in astringency/mouthfeel characteristic (Table 2). The CM combination was significantly lower than F and G2 but was not significantly different from FM, CF and T. The third group based on first PA comprised lactic (L), citric (C), malic (M), succinic (S) and 0.16% glucono-delta-lactone (G1) which were significantly lower than the members of the second group. The CF, CM and FM combinations were more astringent than C and M alone. Quinic (Q) and adipic (A) were the last group and weakest in this characteristic.

The above findings were in agreement with Straub's (1992) report that HCl was the most astringent compared with other organic acids and some of the acids were perceived to be much more astringent than sour. The duration of astringency response (23 to 50 s) was sometimes almost twice as long as the duration of sourness response (15 to 26 s) (Straub, 1992). Likewise, Settle et al. (1986) reported that sulfuric and hydrochloric acids produced the smallest proportions of perceived sourness compared with six organic acids. Solutions of strongly ionized mineral acids not only taste extremely sour but, in

Table 2. 5. Loadings* of the attributes for the first three principal axes following free choice profiling for 15 acids.

Panel list	Principal axis 1	Principal axis 2	Principal axis 3
1	Bitter (0.56)+acid (0.46) +sour (0.35)-musty (0.33) +astringent (0.48)	Lemon (0.52)-sweet (0.57) +baking soda (0.30) -musty (0.37)	-Salty (0.72)-sour (0.37) -musty (0.49)
2	Astringent (0.84)+acid (0.42) +medicine (0.34)	Medicine (0.90)-acid (0.33)	-Citrus (0.64)-acid (0.52) +astringent (0.50)
3	Astringent (0.55)+sour (0.50) +length (0.55)+aspirin (0.33)	Bitter (0.65)+savory(0.63) +salty (0.33)	-Sour (0.66)-salty (0.46) bitter (0.37)
4	Sour (0.66)+astringent (0.55) -bitter (0.35)	Vinaigre (0.63)+bitter(0.53) +fermented (0.31)	Cold taste (0.63)-piquant (0.31) +astringent (0.53)-vinaigre (0.38)
5	Stick (0.71)+strange sour (0.47) +strong (0.46)	Strange sour (0.73) +mild (0.38)-strong (0.56)	-Sour (0.89)+strange sour (0.30) -slow feeling (0.33)
6	Fast astringent (0.61) +bright sour (0.46)+sour (0.48) +tooth astringent (0.38)	Bitter (0.61)+metallic (0.43) +brothy (0.43)+dull sour (0.36) +sour (0.31)	-Astringent (0.57) -dull sour (0.56) -fast astringent (0.32)
7	Astringent (0.69)+sour (0.55) citrus (0.34)	Sickening bad (0.78) +vinegar (0.52)	Chalky (0.57)-sour (0.53) -green tart (0.30) +astringent (0.41)
8	Astringent (0.73)+sour (0.58) +aspirin (0.31)	Bitter (0.81)+length (0.35)	-Aspirin (0.53)-sour (0.38) +astringent (0.49) -length (0.39)
9	Teeth astringent (0.53) +impact (0.46)+sour (0.49) +astringent (0.44)	Linger (0.53)+bitter(0.50) +msg (0.48)	-Lemon (0.80)-sour (0.30)
10	Sour (0.51)+sharp (0.42) +linger/astringent (0.38) mouth puckering (0.48)	Bitter (0.68)-tartaric (0.43) -linger/astringent (0.31)	-Sour (0.60)-linger/ astringent (0.64) -aspirin (0.30)
11	Biting (0.63)+astringent (0.32) +mouth puckering (0.47)	Metallic (0.52)-sour (0.39) -clean aftertaste (0.54) -fruity (0.35)	Metallic (0.52)-sour (0.50) +mouth puckering (0.39) -astringent (0.48)
12	Abrasive (0.63)+numb (0.32) +sour (0.33)+lingering (0.42) +astringent (0.30)+	Aftertaste (0.56) +msg (0.54) +spoil (0.54)	-Aspirin (0.61)-sour (0.37) -acetic (0.55)

*Attributes with loadings < 0.30 were not included in the table

addition, stimulate the feeling of astringency and 'set the teeth on edge' (Moncrieff, 1971).

The ASTM Committee on Sensory Evaluation of Materials and Products defined astringency as 'the complex of sensations due to shrinking, drawing or puckering of the epithelium as a result of exposure to substances such as alums or tannins' (American Society for Testing and Materials, 1989). Historically, it has been considered of equal importance to the classical four taste qualities of sweet, sour, salty and bitter (Bartoshuk, 1978). Schiffman et al. (1991) reported that the chorda tympani nerve (taste nerve) rather than the trigeminal nerve transduces the signals for astringent compounds. Others have suggested that astringency is a tactile sensation that results from precipitation of salivary proteins or activation of oral or lingual mechanoreceptors (Lyman and Green, 1990). The chemical reactions underlying astringent sensations are poorly understood, although several mechanisms have been proposed (Lee and Lawless, 1991) involving polyphenols (McManus et al., 1981; Hagerman and Butler, 1981). One of the most popular theories is that polyphenolic compounds such as tannins form complexes with salivary proteins and/or mucopolysaccharides, either precipitating them or causing sufficient conformational changes so that they lose their lubricating power, thus making the mouth feel rough and dry (Bate-Smith, 1973).

With our findings on the astringency characteristics of acids, particularly hydrochloric and phosphoric acids, an expanded mechanism or set of mechanisms for astringency that takes into account not only tannin-protein interaction, but also acid-protein interactions, must be studied. A difficulty in the literature is the common practice of studying astringency in complex media or beverages such as wine (Arnold et al., 1980; Noble et al.,

1984; Guinard et al., 1986). It is understandable from a practical point of view, but it may confuse the picture insofar as other chemical components in the food may interact with the astringent substance (Lee and Lawless, 1991).

Human subjects are known to confuse sour and bitter tastes (Moncrieff, 1971). This misidentification of sour stimuli as bitter in forced test situations (McAuliffe and Meiselman, 1974; Robinson, 1970) may be explained by a number of studies suggesting that at least some 'sour' stimuli produce sensations other than sour, including bitter and salty (McBurney et al., 1972; Meiselman and Dzendolet, 1967).

The second PA is mainly differentiated by a bitter characteristic as described by most panelists with bitter samples appearing at the top of the graph (Figure 2.1). Based on their individual configurations S had a very unique taste that was described as medicine (Pan 2), savory (Pan 3), vinaigre (Pan 4), strange sour (Pan 5), brothy/metallic (Pan 6), sickening bad/vinegar (Pan 7), metallic (Pan 11), linger/MSG (Pan 9), aftertaste/MSG/spoil (Pan 12). Two Caucasian panelists (3 and 6) considered S as savory and brothy, respectively, while two Oriental panelists (9 and 12) used the term monosodium glutamate (MSG). Although different terms were used, the panelists had a consensus in referring to S as the sample primarily describing principal component two which was significantly ($P < 0.05$) different from the rest of the samples (Table 2.3).

PA 2 scores of H and CM were significantly ($P < 0.05$) higher than L, G2, G1, A and Q, but the former two samples were not different from the other organic acids and their combinations. The greater disparity (spread) over the replicates was more evident in PA 2 than in PA 1 (Figure 2.1). This may be because panelists were more uncertain about terms to describe their perceptions, confirming the confusion between sour and bitter (Moncrieff,

1971; Jellinek, 1985) and between astringent and bitter (Lyman and Green, 1988). Peyron (1971) reported that most of the pure chemical substances do not have a single taste, but provide a complex taste. It was also argued by Moncrieff (1970b), and Schiffman and Gill (1987) that taste could not be analyzed in terms of just the four basic tastes (sweet, sour, bitter and salty).

The good replication for Q and A may be due to their overall very low flavor impact at the concentration used while the same concentration of S imparted a strong bitter aftertaste which made it separate out from the group. Bitterness lingers and is generally detectable at lower concentrations than other basic taste stimulants (Moncrieff, 1970b). S is recognized as an important flavoring substance in sake (Kodoma, 1970), is found at a very high concentration in shellfish (Jo and Park, 1985) and the seasoning characteristic of its salt resembles the mollusc extract (Anonymous, 1967). Likewise, the latter group reported that S with salts of basic amino acids have a pronounced salty flavor. The strong, salty, bitter flavor in addition to the acidic taste of S is detrimental to the flavor of a number of fermented beverages depending upon the concentration. S is commonly found in ciders and wines at 0.1%, but 0.03% in beer is considered too high (Kaneuchi et al., 1988). Thus, the 0.08% concentration used in this study was found objectionable by the panelists.

The two Oriental judges, nos. 9 and 12, described a MSG note in S which Japanese refer to as the umami taste, commonly described in English as 'Chinese food taste, meaty or bouillion like' (O'Mahony and Ishii, 1985). It was similarly described by Pan 3 as savory and by Pan 6 as brothy. In a study by Velisek et al (1978), there was no significant difference between the effects of MSG and sodium succinate on the flavor of beef broth, and the flavor of beef was enhanced by its addition. Umami substances do not usually have

high pleasantness ratings in simple solutions but earn high pleasantness scores in foods and solutions compatible with the added umami substance (Kawamura and Halpern, 1987), thus, explaining the 'sickening bad' and 'spoil' ratings by Pans 7 and 12, respectively. The uniqueness of MSG taste (O'Mahony and Ishii, 1987) is said to be independent of the four primary tastes (Kawamura and Halpern, 1987).

The meaning of the third dimension is less easy to summarize. The exact interpretation varied from panelist to panelist (Table 2.5) and there was a wide disparity over the four replicates among the samples (Figure 2.2). This axis appears to be predominantly sour (Pan 3, 5, 6, 10, 11) being expressed also as citrus (Pan 2), lemon (Pan 9) and acetic (Pan 12). The higher positive loadings of astringent in this axis were a contrast with the negative loadings for sour for Pans 2, 4, 7 and 8, whereas these two terms were related to Pan 6, 10 and 11. Aspirin (Pan 8, 12), salty (Pan 1) and cold taste (Pan 4) also had high loadings. Since these attributes had high negative loadings, the samples appearing at the bottom of the graph (FM, CM, CF, F and M) possessed these characteristics while Q and H were high on the positive (opposite) side (Figure 2.2). The sour characteristics probably referred to by the panelists in this third principal axis were the fruit sourness (e.g. lemon, citrus) and not the strong acid taste of the inorganic acids such as those imparted by P and H. The panelists described the inorganic acids as more astringent than sour. Similar findings were also reported by Straub (1992) on her equi-sourness and time-intensity studies on astringency and sourness of H, M, C, T, L, F and acetic acids. The fruit acids, namely M, C and T, did not differ from each other in any sourness time-intensity parameters which agree with the findings in this study.

The three combinations (CF, CM, FM) were very close in sensory attributes as they clustered together in the first three axes. These combinations and F were more astringent and sour than M and C alone, but when C and M were combined, those sensory properties were enhanced. Likewise, from a sensory standpoint, this CM combination could be a good substitute for tartaric acid based on our experiment in comparing these acids in synthetic grape juice.

Straub (1992) determined that C, fumaric QD, H, L, M and T were of equal sourness at 0.091, 0.076, 0.023, 0.056, 0.075 and 0.08 %, respectively, while Furukawa et al. (1969) determined equi-sourness for 0.05, 0.03, 0.16, 0.06, 0.04 and 0.05 % for C, F, GDL, L, M and S, respectively. C was the least sour among the acids tested on an equal weight basis while the differences among M, T and L were not clear (CoSeteng et al., 1989).

pH and Titratable Acidity Results

The pH and titratable acidity of the 15 acid samples are given in Table 2.6. Among the 14 acids, it was evident that at the concentration tested, A had the highest pH while H had the lowest pH. The pH may not be the best chemical property with which to correlate sourness. A was the least acidic (highest pH) among the acids but not the least sour at 0.08% concentration. However, there may be a strong relationship between astringency and pH for these acids. Those with very low pHs were the more astringent samples. Straub (1992) found similar relationships wherein the pH of an acid was more related to astringency than sourness. She found correlations between pH and time-intensity parameters for astringency ratings but none between pH and sourness time-intensity ratings.

The perception of sourness of acidic compounds has been extensively studied but the mechanism involved is still not fully understood

Table 2.6. Molarity, total titratable acidity (TTA) and pH of different acid samples at 0.08% concentration.*

ACID	Molarity	TTA	pH
Adipic	0.0055	0.075 (0.001)	3.842 (0.015)
Citric	0.0042	0.074 (0.002)	3.15 (0.038)
Fumaric	0.0069	0.08 (0.012)	2.90 (0.035)
Glucono1 ^a	0.0090	0.137 (0.011)	3.273 (0.089)
Glucono2 ^a	0.0135	0.214 (0.014)	3.175 (0.083)
Hydrochloric	0.0097	0.033 (0.002)	2.183 (0.051)
Lactic	0.0094	0.056 (0.003)	3.282 (0.052)
Malic	0.0060	0.076 (0.001)	3.190 (0.047)
Phosphoric	0.0097	0.085 (0.007)	2.452 (0.055)
Quinic	0.0042	0.064 (0.003)	3.455 (0.068)
Succinic	0.0068	0.074 (0.001)	3.635 (0.05)
Tartaric	0.0053	0.074 (0.002)	3.075 (0.087)
Citric:Malic	0.0051	0.074 (0.001)	3.188 (0.028)
Citric:Fumaric	0.0055	0.074 (0.001)	3.068 (0.063)
Fumaric:Malic	0.0064	0.076 (0.001)	3.040 (0.069)

 *Numbers in parentheses are standard deviations; mean values based on four replications.

^aGlucano1 and Glucano2 were 0.16 and 0.24%, respectively.

(Ganzeloves and Kroeze, 1988) as compared with sweetness. (Shallenberger and Acree, 1967) reported an AH-B type reaction when the sweet tasting molecules complexes with the proteins in the taste receptors (Price and DeSimone, 1977). Kurihara (1973) suggested that lipids maybe the receptors for bitter, salt and sour stimuli. He found that lipids can be more easily extracted from papillae of bovine tongue epithelium when pretreated with sour, salty or bitter compounds than with sweet compounds. Beidler (1967) proposed the interaction of protons with proteins or phospholipids.

Acids dissociate into protons and acid anions when dissolved in water. ($[HA] \rightleftharpoons [H^+][A^-]$). In organic acids the dissociation is not complete and the dissociation constant $K = [H^+][A^-]/[HA]$ is related to the chemical structure. Ganzeloves and Kroeze (1988) reported that aqueous solutions of strong acids (i.e. K is large) taste more sour than aqueous solution of weak acids (K is small) when the molar concentrations are equal. If hydrogen ion concentrations are equal, the sourness of the weak acids is higher than that of the strong acids.

Several researchers suggested that there is no simple relationship between the concentration of free protons $[H^+]$ and perceived sourness (Ganzeloves and Kroeze, 1986; McBurney et al., 1972; Pangborn, 1963; Furukawa et al., 1969). Makhloaf and Blum (1972) reported that pH had little to do with sourness based on salivary flow rate measurements. In the study of Norris et al. (1984), they concluded that sourness intensity was more dependent on titratable acidity than on pH. The importance of anion in eliciting sourness (Beidler, 1967; Norris et al., 1984; Noble et al., 1986) was reported based on the different responses on acid solutions at equal pH. Beidler (1958, 1967) postulated that with higher anion concentration, the

hydrogen ion could bind more readily to the receptor cell because of less membrane charging.

Panelist and Sample Variation

The GPA can be used to determine whether or not the panelists were in agreement in evaluating the 15 acid samples. The percentage consensus and percentage within (residual) is illustrated in Figure 2.3. Panelists who performed differently from the rest could also be identified by examining their percentage residual variation. No panelists had residuals that were consistently greater than the rest, thus none of their residuals are reported. In Figure 2.3, the lower, darker part of the histogram represents the percentage consensus variation while the upper, lighter part is the residual within variation of the corresponding acids. A high percentage consensus means high agreement among the panelists, while a high percentage within means greater residual error or high disagreement among the panelists. The panelists had good consensus for H, P, S, Q and A and very low consensus for CM, CF, FM, C, M and F. The latter could be due to the high percentage residual (error) that resulted from panelists' difficulty in differentiating those samples. This is clearly illustrated in the overlapping of intersample distances, and the big disparity over the replicates (Figures 2.1 and 2.2). The high astringency ratings for inorganic acids H and P, the uniqueness and strong bitterness of succinic, and very weak characteristics of Q and A made them easy to discriminate from the other organic acids.

Q and A had lower residual error, meaning a higher agreement among the panelists. There was high disagreement on S probably due to the difficulty in describing the product. Although panelists had good consensus on S as a unique acid, its taste was hard to describe because of the combined

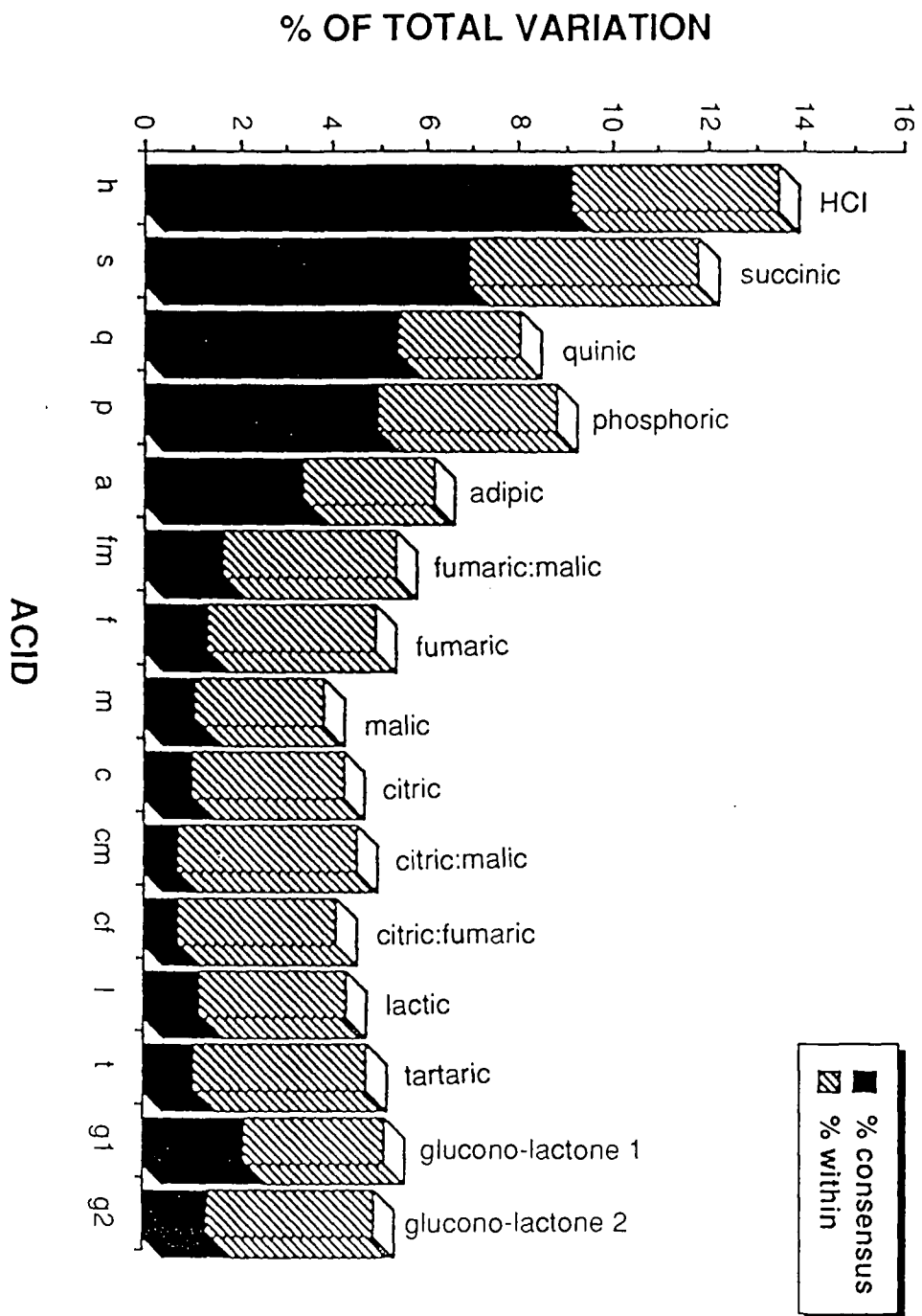


Fig. 2.3. Percentages consensus and within (residual) variation distributed over the fifteen samples.

bitter, savory and sour notes. Some of the panelists felt that it was both sour and bitter but others referred to it as just bitter without any sourness.

Likewise, the high residual for P and H may be explained by the difference of opinion between 'astringency' and 'sourness'. Some panelists were able to distinguish between astringent and sour samples, but for others the two terms were related as shown by the same signs of the loadings in Table 2.5.

The residual error increased with a concomitant decrease in consensus variation from the 0.16% (1) to the 0.24% (2) concentration of GDL (Figure 2. 3). This was an indication of the difficulty in evaluating GDL at a higher concentration. At 0.16%, the sensory properties were weaker like Q and A while at 0.24% the properties were more close in sensory properties to the acid combinations and F.

CONCLUSIONS

This research demonstrates conclusively that acids differ in sensory character. Likewise, the concentration of the acids will greatly influence the characteristic of the products. In some acids, some sensory properties may stand out at either very low or very high concentrations, such as for the weak properties of A and Q and the very strong properties of H and P at 0.08%. However, some acidulants tend to have similar properties such as M, F, T and their combinations at 0.08%. Then combining acids, particularly C and M, produced a synergistic effect which could be a good substitute for T. So in deciding which acidulants to add to formulated foods, one must consider several things: the effect that the acids will have on the food system, their effect on other flavors in the food and also what will be their function because even though the pH or the extent of sourness may be the same, other sensory

characteristics often will tip the balance as to which acid is most suitable for a given application.

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The Use of Power Functions and a Directional Difference from Control Test to
Determine Equi-sourness Levels of Selected Acidulants

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ABSTRACT

Acidulants have not been studied in detail with respect to their tastes despite the fact that they are used widely by the food industry. This study was designed to determine the equi-sour concentration of the selected acidulants (adipic, citric, fumaric, glucono-delta-lactone, hydrochloric, lactic, malic, phosphoric, quinic, succinic, tartaric, citric:malic, citric:fumaric and fumaric:malic). These were generated from the power functions of each acid based on their five molar concentrations evaluated by 16 trained panelists using magnitude estimation. Citric acid served as a reference throughout the experiment so that the acids could be related to each other. Equi-sour concentrations were determined by regressing the log of the rescaled response (sensory) on the log of the stimuli (physical). The calculated equi-sour levels ranged from 0.48 ml/L for HCl to 2.34 g/L for glucono-delta-lactone when citric acid was at 1.0 g/L. These theoretical equi-sourness levels were then tested by using an alternative sensory method, the directional difference from control test.

INTRODUCTION

Acidulants have not been studied in detail with respect to their tastes despite the fact that they are used widely by the food industry. The authors have focused their attention on several organic and inorganic acids and have tried to clarify their taste properties on several bases such as on a weight (Rubico and McDaniel, 1992) and on a molar basis (Rubico and McDaniel, 1991). Results indicated that on both weight (w/v or v/v) and molar bases, acids differed in their sensory characteristics primarily on sourness, astringency and bitterness attributes. Hydrochloric and phosphoric were more astringent than sour while succinic had bitterness with monosodium glutamate taste. Likewise, sourness intensity was significantly different among the acids.

Since acids are known to impart sourness to foods, the sourness of different acidulants is often compared. However, based on our previous studies, other sensory attributes may greatly influence the taste of the final product. Determining the sensory profile of these acidulants on an equi-sour basis can help separate this sourness effect from other taste and textural characteristics which define their uniqueness.

In this study, the equi-sourness levels were determined based on power functions generated for each acid using magnitude estimation with 0.005M citric acid as the standard. Beebe-Center and Waddel (1948) were the first to use magnitude estimation while Stevens (1957) developed and used magnitude estimation to support his psychophysical law - the power function. Numerous studies have shown that the perceived intensity of a

stimulus (S) increases as a power function of the physical intensity (I), (Stevens, 1956; Moskowitz and Sidel, 1971).

In presenting evidence for his power law, Stevens (1957) plotted in log-log coordinates, the relation between scale value and molar concentration of tartaric acid and found a straight line with slope (exponent) of approximately 1.0. The relationship between a stimulus and its perceived response has been established in a variety of sensory modalities (Meiselman, 1968; Moskowitz and Arabie, 1970; Moskowitz 1970; Moskowitz, 1971; Sauvageot, 1987; Yau and McDaniel, 1990). Power functions define or describe the relationship between the concentration of the acid and the perceived intensity. It is known to be relatively constant and can therefore be used to compare intensity estimates from separate taste sessions (Moskowitz et al., 1972).

Although ratio scaling of taste intensity has routinely yielded power functions for a number of taste stimuli, wide discrepancies have been reported in the exponents of the functions (Meiselman, 1971). For citric acid, the exponent ranged from 0.46 (Smith, 1969) to 1.29 (Straub and McDaniel, 1989), while the published exponents for sodium chloride have varied from 0.41 (McBurney, 1966) to 1.59 (Ekman, 1961) and for quinine sulfate from 0.30 (Smith, 1969) to 1.0 (Stevens, 1969)

Other studies, using direct magnitude estimation of sensory magnitude, produced different exponents for acids, from a low value of 0.5 to higher value of 2.02 (Straub, 1992). The discrepancies may be due to procedural differences for stimulus presentation (Meiselman, 1971), size of modulus (Straub, 1992) and range of concentrations used.

This study covers two experiments. The first experiment was the generation of power functions for each acid. The second part of the study was undertaken to validate the equi-sourness values resulting from the power

function determination. In the power function determination, panelists were asked to concentrate only on the sourness of the samples, ignoring other attributes. A second test was used to provide an additional direct sourness comparison in order to validate the calculated equi-sourness levels. These theoretical equi-sourness levels were then tested by using a directional difference from control method. Each concentration was rated for how more or less sour it was than a citric acid reference. This method is similar to difference from control test.

Objectives:

Experiment 1

1. To determine the sourness power function of each acid using a trained panel.
2. To determine equi-sourness levels at two levels of sourness for each acid.
3. To compare the pH of the acidulants at various molar concentrations and at their equi-sour levels.

Experiment 2

1. To confirm the equi-sourness levels through an alternative sensory method, directional difference from control.
2. To compare the pH of the acidulants at their equi-sour levels.

Materials and Methods

Samples

Power functions of fourteen samples of acids (9 organic and 3 organic mixtures plus 2 inorganic) were generated. The lists of the acids and the source (name of company) are as follows: anhydrous citric, DL-malic and fumaric acids (FCC) were obtained from Haarmann and Reimer (H&R)

Company (formerly Miles Inc. Elkhart, IN), adipic and phosphoric acid (75%) from Monsanto (St. Louis, MO), and glucono-delta-lactone from Finnsugar Bioproducts (Schaumburg, IL). Hydrochloric (NF-FCC, 36.5-38.0%) and lactic acids (USP-FCC, 85-90%) were purchased from J. T. Baker Inc. (Jackson, TN), D-quinic acid from ICN Biomedicals, Inc. (Cleveland, OH) and succinic and tartaric acids from Penta Manufacturing Co. (East Hanover, NJ). The acids were diluted with bottled water (Aqua-Cool, Portland, OR).

Panelists

Sixteen panelists (7 males and 9 females), all of whom were student and staff from the Department of Food Science and Technology at Oregon State University, were recruited to form the panel. All had previous trained panel experience.

Experiment 1 - Power Function Determination

a. Samples

The 14 acid samples evaluated in this study and the five molar concentrations for each sample are listed in Table 3.1. All but glucono-delta-lactone (GDL), hydrochloric (H) and lactic (L) acids had the same molar concentrations (0.00125, 0.0025, 0.005, 0.01 and 0.02M). The molar concentrations for both GDL and L were increased up to 0.04M because of their weak sensory properties while H at 0.02M was already too strong for the panelists to taste, so 0.000625M was prepared instead. The acid combinations were citric:fumaric (CF), citric:malic (CM) and fumaric:malic (FM); calculation for each acid in a combination was based on molar concentration divided by two.

A solution of 0.005M citric acid was presented as a reference and was assigned an intensity score of 100. All acid solutions were prepared 16 hours

Table 3.1. The concentrations (g/L or ml/L) of the different acids used to generate power functions.

ACID	M.W/ [M]	<u>MOLAR CONCENTRATION [M]</u>						
		0.000625	0.00125	0.0025	0.005	0.01	0.02	0.04
ADIPIC	146.14	-	0.18	0.36	0.73	1.46	2.92	-
CITRIC	192.12	-	0.24	0.48	0.96	1.92	3.84	-
FUMARIC	116.07	-	0.145	0.29	0.58	1.16	2.32	-
GLUCANO- LACTONE	178.14	-	-	0.445	0.89	1.78	3.56	7.12
MALIC	134.09	-	0.167	0.335	0.67	1.34	2.68	-
QUINIC	192.00	-	0.24	0.48	0.96	1.92	3.84	-
SUCCINIC	118.09	-	0.148	0.295	0.59	1.18	2.36	-
TARTARIC	150.19	-	0.188	0.375	0.75	1.50	3.00	-
LACTIC	11.70M	-	-	0.21	0.427	0.85	1.71	3.42
HCL	12.08M	0.05	0.10	0.20	0.41	0.83	-	-
PHOSPHORIC	12.05M	-	0.10	0.20	0.41	0.83	1.66	-
CF*		-	0.195	0.385	0.77	1.54	3.08	-
Citric (C)	192.12	-	0.12	0.24	0.48	0.96	1.92	-
Fumaric (F)	116.07	-	0.075	0.145	0.29	0.58	1.16	-
CM*		-	0.205	0.41	0.82	1.63	3.26	-
Citric (C)	192.12	-	0.12	0.24	0.48	0.96	1.92	-
Malic (M)	134.09	-	0.085	0.17	0.34	0.67	1.34	-
FM*		-	0.155	0.32	0.63	1.25	2.50	-
Fumaric (F)	116.07	-	0.07	0.145	0.29	0.58	1.16	-
Malic (M)		-	0.085	0.17	0.34	0.67	1.34	-

*Calculation for each acid was based on molar concentration divided by 2.

prior to tasting by dissolving the appropriate amount of acid in one liter of water in a one liter volumetric flask.

b. Procedure

All panelists were introduced to magnitude estimation in several practice sessions. Five molar concentrations for each of the 14 acid samples were then rated for sourness using magnitude estimation (Stevens, 1957). All of the acids were rated against a citric acid standard and therefore could be related to one another. Each session consisted of the presentation of the reference sample with five concentrations of a particular acid. The samples were served at 22°C in randomized order using 60 mL plastic cups coded with three-digit random numbers. Panelists evaluated two sets (replicates) of acids per day and tasted three days per week.

c. Data Analysis

Due to the large panel variance that often results from magnitude estimation data, a normalization procedure was applied prior to data analysis. The data were normalized by a method similar to modulus equalization (Lane et al., 1961; McDaniel and Sawyer, 1981). Since the distributions tend toward log-normal, the geometric mean of each panelist's ratings was calculated. Each panelist's individual geometric mean across acids was divided into each of their respective raw values. The original ratios between magnitude levels were maintained. The data were then transformed to log values. Powers et al. (1981) found that only when the number of observations approached 200 was the logarithmic transformation unnecessary.

Power functions were established by regressing the log of the response (sensory) on the log of the stimuli (physical). The stimuli were the molar

concentrations of a particular acid while the response was the intensity ratings (magnitude estimation data) of the 16 panelists across two replications.

d. Equi-sour Determination

Equi-sour concentrations were estimated by picking a subjective intensity on the y-axis of the plot of the power functions and substituting this value into the power function equation to get the appropriate concentration from the x-axis for each acid.

e. Chemical Measurements

The pH of each sample was measured by a pH electrode attached to a microprocessor pH/mV meter (Orion Model 811) equipped with a combination pH electrode (Ross Model 81550).

Experiment 2 - Confirmation of Equi-Sourness Level

Two equi-sourness levels were selected for the confirmation study. Table 3.2 contains those two levels with their corresponding pH. The same panelists from Experiment 1 participated in this study (experiment 2). The 14 acid samples were divided into three sets (A, B, C). Citric acid was in both sets A and C to make up 5 samples/set. Set A comprised adipic (A), citric (C), hydrochloric (H), lactic (L) and fumaric:malic (FM) acids. Glucono-delta-lactone (GDL), phosphoric (P), fumaric (F), quinic (Q) and citric:malic (CM) acids were in Set B, while Set C was composed of malic (M), succinic (S), tartaric (T), citric (C) and citric:fumaric (CF) acids. Two replications were done per session using only one set per session. The 16 panelists were divided into three groups (two groups with five panelists and one group with six panelists) and one group evaluated one set. The presentation of the five acid samples per set were randomized for all the panelists. The panelists evaluated the acids in comparison with citric acid (0.8 and 1 g/L for Levels 1 and 2,

Table 3.2. Concentration (g/L or ml/L) and pH of the different acids on an equi-sour basis.

ACID	LEVEL 1		LEVEL 2	
	CONC. g/L or ml/L	pH	CONC.	pH
ADIPIC	0.90	3.35	1.12	3.20
CITRIC	0.80	3.02	1.00	2.72
FUMARIC	0.50	2.80	0.62	2.66
GDL	1.87	2.92	2.34	2.81
MALIC	0.58	3.00	0.72	2.86
QUINIC	1.41	2.90	1.76	2.75
SUCCINIC	0.54	3.33	0.68	3.18
TARTARIC	0.54	2.85	0.68	2.71
LACTIC*	0.78	2.94	0.98	2.82
HCL*	0.38	2.37	0.48	2.19
PHOSPHORIC*	0.43	2.50	0.54	2.36
CF	0.56	2.88	0.70	2.74
Citric	0.35			
Fumaric	0.21			
CM	0.66	2.92	0.82	2.78
Citric	0.39			
Malic	0.27			
FM	0.58	2.86	0.72	2.74
Fumaric	0.27			
Malic	0.31			

* ml/L

respectively) which served as the reference (R) using the directional difference from R ratings. The ratings ranged from -4 (extremely less sour than R), with 0 as same as R to +4 (extremely more sour than R). The data were analyzed using analysis of variance (ANOVA) and Least Significant Difference (LSD) if appropriate using SAS. Mean comparisons test were done by testing the null hypothesis (H_0 : mean = 0) using PROC GLM (SAS, 1987). To test this hypothesis, the NOINT (no intercept) command in PROC GLM was included in the model statement.

RESULTS AND DISCUSSION

Experiment 1 - Power Function Determination

The power function parameters [slopes (b) and intercepts (a)] and their corresponding standard errors and correlation coefficients (r) for each acid are listed in Table 3.3. The power function is normally expressed as (Stevens, 1970):

$$\log y = n \log x + \log k$$

In this equation, y represents the magnitude of response, x represents the magnitude of stimuli; n represents the exponent which implies the rate of the change of response with the change of stimuli; and k represents a constant.

The exponent n has been a parameter of interest to sensory scientists and psychologists. If n is greater than one, the response is an accelerating function of concentration. Conversely, if the n is less than one, the response

Table 3.3. Power function parameters (intercepts and slopes), correlation coefficient (r), and equi-sour molar concentration.

ACID	r	INTERCEPT*	SLOPE*	MOLAR ^a CONC.
Citric	0.93	2.93 (0.09)	1.23 (0.04)	0.004148
Adipic	0.89	3.07 (0.13)	1.39 (0.06)	0.006185
Fumaric	0.89	2.98 (0.12)	1.26 (0.05)	0.004314
Malic	0.90	2.93 (0.09)	1.24 (0.04)	0.004336
Glucano	0.91	2.79 (0.11)	1.41 (0.05)	0.0105
Quinic	0.82	2.56 (0.16)	1.20 (0.07)	0.007356
Succinic	0.89	2.90 (0.12)	1.24 (0.05)	0.004584
Tartaric	0.91	2.79 (0.10)	1.14 (0.04)	0.00357
HCL	0.85	3.32 (0.19)	1.41 (0.07)	0.00442
Lactic	0.92	2.57 (0.09)	1.26 (0.04)	0.009127
Phospho- ric	0.80	3.32 (0.13)	1.45 (0.06)	0.000513
COMBINATIONS				
CF	0.85	2.70 (0.13)	1.11 (0.06)	0.003695 C - 0.00185 F - 0.00185
CM	0.89	2.96 (0.12)	1.24 (0.05)	0.004101 C - 0.00205 M- 0.00205
FM	0.89	2.90 (0.12)	1.24 (0.05)	0.004584 F - 0.002292 M- 0.002292

^a Molar Concentration when Y=0 based on the equation
 $\log Y = \log a + (n) \log x$

* Values in paranthesis are standard error

is a decelerating function of concentration. All of the 14 acid samples had exponents greater than one, with values ranging from 1.11 to 1.45 (Table 3.3).

The exponent for citric acid in this study was 1.23 which was close to the reported value of 1.29, 1.25 and 1.18 by Malcolmson and McDaniel (1980), Straub (1992) and McDaniel and Harasym (1979), respectively (Table 3.4). Although Straub used a narrower range of molar concentrations (0.0008M to 0.001M) for her power function studies compared to this study (0.000625 to 0.02M), our exponents were almost in close agreement in all acids except hydrochloric acid (Table 3.4).

The similarities of these results may support the Moskowitz et al. (1972) report that a power function is relatively constant and can therefore be used to compare intensity estimates from separate taste sessions. However, previous studies have reported sourness power functions to be lower than the present findings and less than one. Moskowitz (1971) reported values which ranged from 0.64 to 0.84 while Ganzeloves and Kroeze (1988) reported values from 0.42 to 1.07. For the present study the results indicate that the response has increased at a faster rate than the stimulus; for the Moskowitz (1971) study, the opposite is true, although he reported an exponent of 1.0 from his earlier study (Moskowitz, 1968). The most commonly reported power function among the acids was for citric acid with 0.5 as the lowest (Feallock, 1966) and 1.29 as the highest (Straub, 1992) (Table 3.4).

Several differences in methodology could account for the differences in the magnitude of the slope. For example, Moskowitz's panelists had to evaluated 40-48 acids in one session and Ganzeloves and Kroeze's panelists to evaluated 44. In the present study, only five samples were given per session; and eight samples were presented at one time for Straub's study. With so

Table 3.4. Exponents (n) of the power relation ($S=KC^n$) between sourness and molarity.

ACIDS	RUBICO	MOSKOWITZ	STRAUB	Fealock	Meiselman	Moskowitz	Malcoimson &	McDaniel &
	1992	1971	1991	1965	1968	1968	1980	1979
Adipic	1.39							
Citric	1.23	0.72	1.29	0.5			1.25	1.18
Fumaric	1.26	0.86	1.25					
Glucono-delta-lactone	1.41	0.64						
Malic	1.24	0.77	1.25					
Quinic	1.2							
Succinic	1.24	0.76						
Tartaric	1.14	0.82	1.19			1		
Lactic	1.26	0.84	1.25					
HCL	1.41		2.02	0.5	0.8			
Phosphoric	1.45							
Citric:Fumaric	1.11							
Citric:Malic	1.24							
Fumaric:Malic	1.24							

*Range of Concentrations (M) used:

Rubico, 1992 0.000625M, 0.00125M, 0.0025M, 0.005M, 0.01M and 0.020 M

Moskowitz, 1971 0.003M, 0.006M, 0.0125M, 0.025M, 0.05M, 0.10M

Straub, 1992 0.0008M---0.001M (Tartaric) 0.001M---0.01241M (Fumaric)

many samples evaluated per sessions in Moskowitz's study, adaptation could have taken place especially with the higher concentrations used (0.003M to 0.1M). In this study the panelists expressed discomfort about the strong sensory characteristics of acidulants higher than 0.01M. A wider range of stimuli could cause a flattening of the slopes (Moskowitz, 1983). Poulton (1968) found exponents to be higher in concentration ranges closer to threshold levels of the stimulus.

Ganzelves and Kroeze (1987) used a different method of stimulation in their study. The panelists evaluated the samples by placing a circular piece of filter paper on the frontal part of the tongue. In this study, the panelists 'sip-and spit' the acid samples thus exposing all of their taste buds to the stimulus. This could increase the sensitivity to the sourness solutions which could explain the steeper slopes. Meiselman (1971) studied the change in power functions for basic tastes with three procedures; sip, anterior dorsal tongue flow and whole mouth flow. Exponents produced by the sip method were consistently higher than the other two methods suggesting that presentation procedure has an influence on power function parameters.

The various sourness functions are diverging lines in log-log coordinates (Fig. 3.1A). For a better illustration, the different power functions were separated and presented in Figures 3.1C and 3.1D. The similarities of the power functions of C, F, M and their mixtures (CF,CM, FM) and the differences among inorganic acids compared with GDL and Q were clearly illustrated in Figures. 3.1D and 3.1C, respectively.

The concentrations used in this study are similar to the acid levels added to formulated products like carbonated beverages. The percent concentration of acids used in carbonated beverages ranges from 0.02 to 0.15

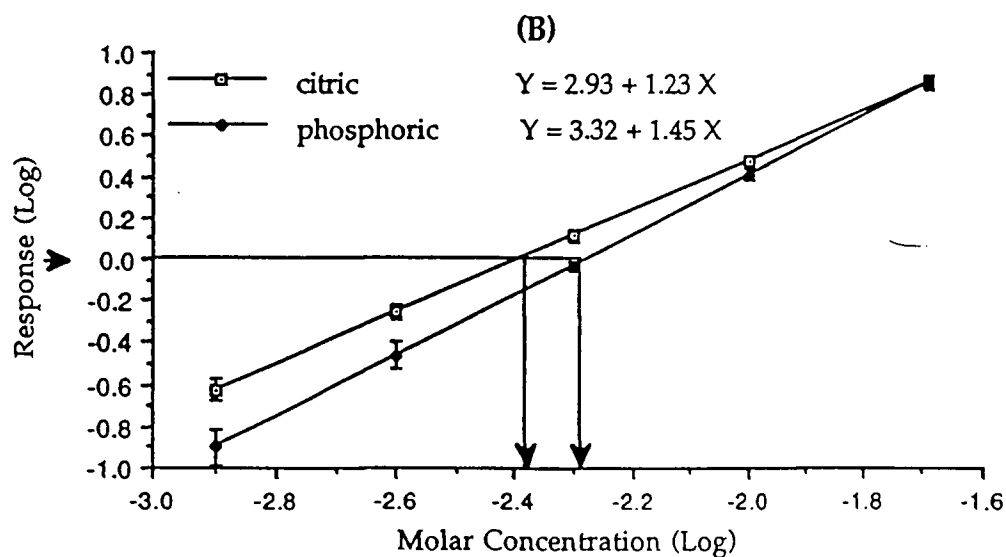
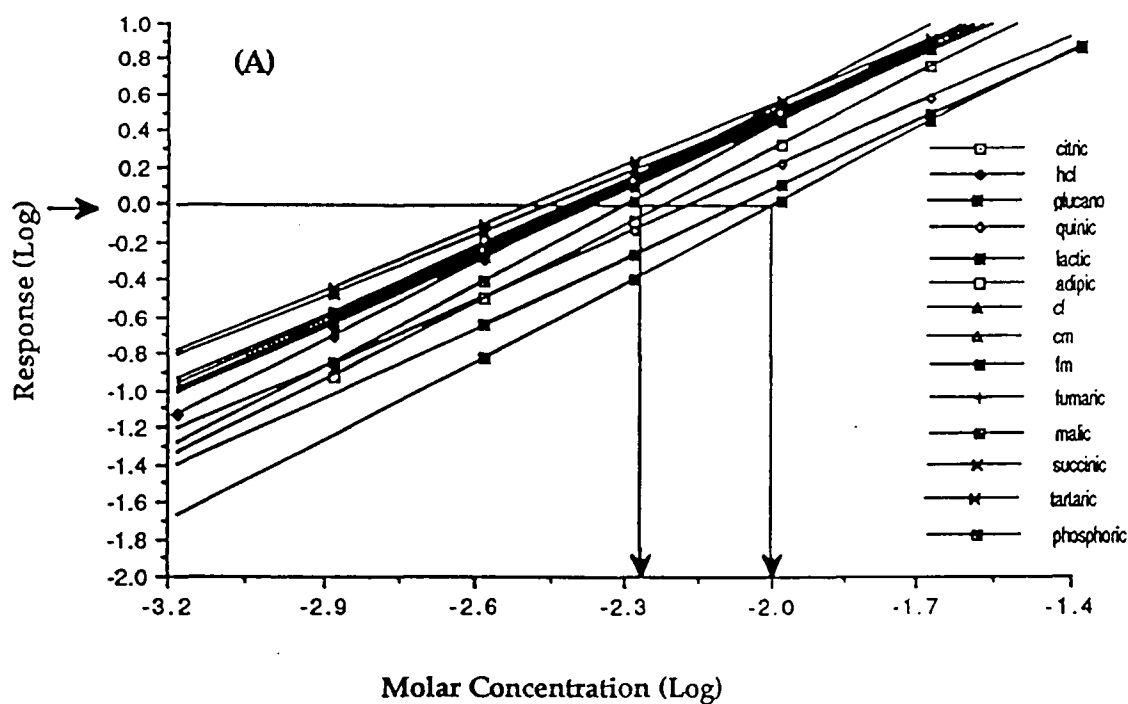


Figure 3.1 (A) Power functions for fourteen acids (B) Power functions for quinic and citric acids.

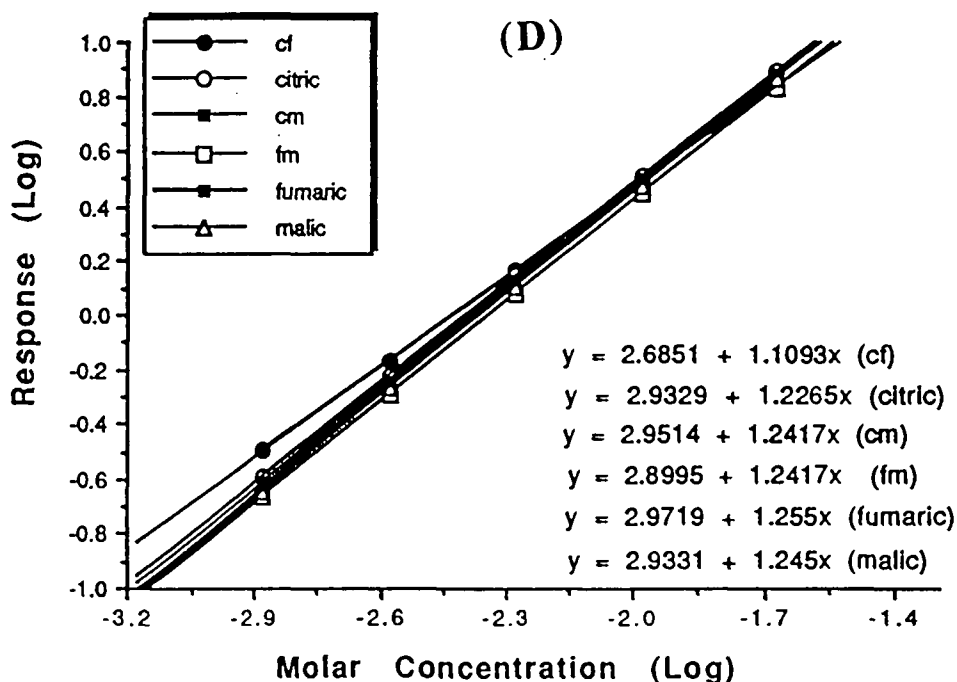
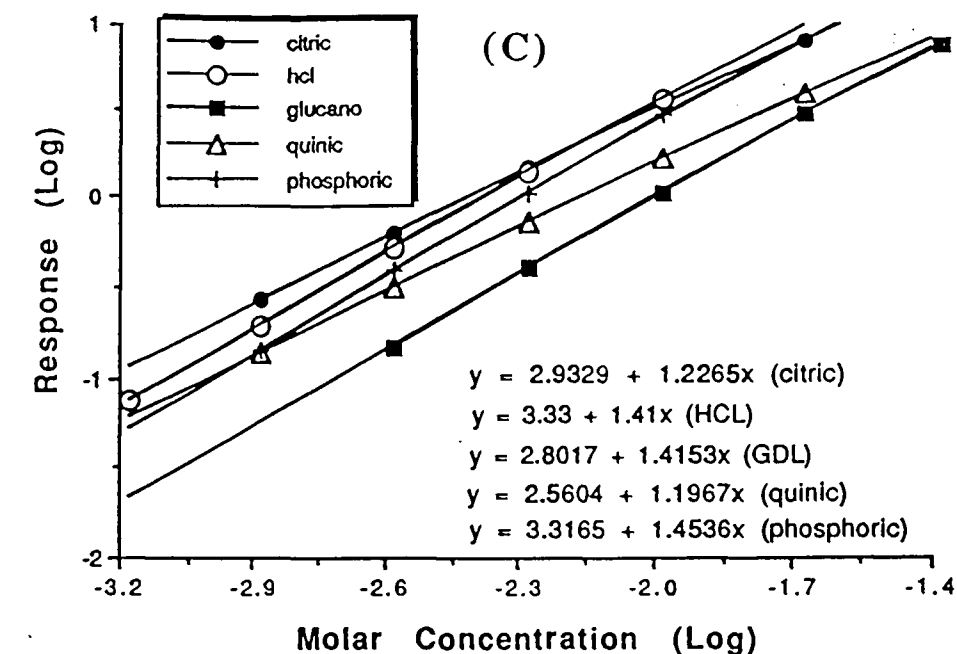


Figure 3.1 (C) Power functions for citric, hydrochloric, glucon-delta-lactone, quinic and phosphoric acids (D) Power functions for citric, citric:fumaric, citric:malic, fumaric:malic, fumaric and malic acids.

percent (Peterson and Johnson, 1978) which is encompassed by the ranged (0.005 to 0.38 percent) used in this study.

The intercept k will vary with the size of the modulus used and the numbers that panelists used to rate the stimuli. The intercept can change from experiment to experiment without affecting the exponent (Stevens, 1960) as was illustrated by the results of the citric acid exponent from this study and from other researchers (Straub, 1992; Malcomsom and McDaniel, 1980). Thus, determining the difference among the intercepts will not be helpful in this study because the intercepts were out of the range of the data set used to calculate the regression equation.

The power function can be used to compare sourness intensity estimates of different acids (Figures 3.1C and 3.1D). To give an example from Table 3.3, the power functions for C and P acids were expressed as follows:

$$\text{C: } \log(y) = 1.23 \log(x) + \log(2.93)$$

$$\text{P: } \log(y) = 1.45 \log(x) + \log(3.32)$$

The correlation coefficients (r) (Table 3.3) were 0.93 and 0.80 for C and P, respectively, suggesting a strong linear relationship as graphically presented in Fig. 3.1B. With the use of Fig. 3.1B, the equi-sour level can be determined graphically by drawing one straight line horizontally across the power function lines from the y -axis and drawing perpendicular lines from the power function lines down to the x -axis. To calculate the value, substitute the same value for y in the power function equation and solve for x . For example, substituting $y = 0$ in the relationship equation for C,

$$0 = 1.23 \log(x) + 2.93$$

$$x = \text{antilog}(-(2.93/1.23)) = 0.000415\text{M}$$

or to get the g/L, multiply 0.00415M by 192.12 (MW of C)

$$x = 0.80 \text{ g/L}$$

Then, using the same procedure for all the acids, (i.e, substituting $y=0$ in the equations) the equi-sour concentrations (in g/L or ml/L) were calculated. The values are given in Table 3.3. For the acid combinations: CF, CM and FM, the concentration was based on the MW of each acid after the molar concentration (M) was divided by 2. Since the MW of the three acids (C, M and F) were different, the calculated g/L varied for each acid.

Table 3.2 contains the concentrations of the different acids to be used for equi-sourness when the intensity of C is taken as 1.00. Only GDL, quinic (Q) and adipic (A) acids needed more acid by weight (g/L) with 2.3, 1.76 and 1.12, respectively. For contrast, the two inorganic acids (H and P) needed only half the amount (ml/L) of C (g/L) to achieve the same sourness intensity.

Chemical measures of equi-sourness must be sought if the goal is to substitute the more expensive organic acids with the inexpensive inorganic acids. P is one of the least expensive acidulants (Gardner, 1972) and much cheaper than fruit acids (Toy and Walsh, 1987). The low pH of P and H acids (Figure 3.2) may have a detrimental effect on some functional properties of the formulated foods. Results in Figure 3.2 (Table 3.5) can help the acidulant users and producers determine the pH change at different molar concentrations of the selected acidulants. For instance, A and S are the acids of choice for acidulants intended for low acid food formulations. Of course, the cost may play a great role here because almost twice the amount of A is needed to be equi-sour to S acid (Table 3.2). Likewise, this is assuming that these two acids were sensorially the same that the equi-sourness determination was accurate; these issues will be discussed in experiment 2.

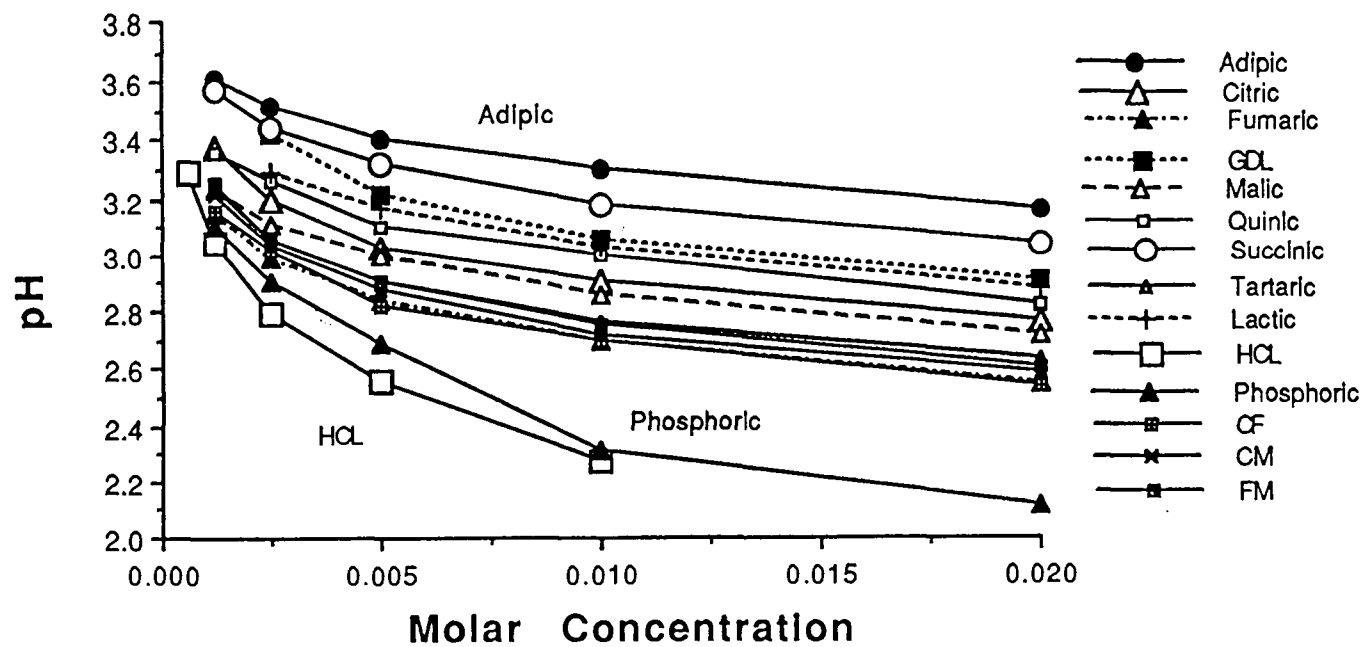


Fig. 3.2. pH of the acids at different molar concentrations.

Table 3.5. pH of the acids at different molar concentrations.

ACID	MOLAR CONCENTRATION [M]						
	0.000625	0.00125	0.0025	0.005	0.01	0.02	0.04
ADIPIC	-	3.59	3.50	3.38	3.28	3.14	-
CITRIC	-	3.26	3.08	2.91	2.78	2.64	-
FUMARIC	-	3.18	3.02	2.87	2.73	2.58	-
GLUCANO-DELTA-LACTONE	-	-	3.42	3.22	3.06	2.90	2.82
MALIC	-	3.22	3.10	2.99	2.85	2.71	-
QUINIC	-	3.29	3.19	3.03	2.93	2.75	-
SUCCINIC	-	3.60	3.47	3.35	3.21	3.07	-
TARTARIC	-	3.16	2.98	2.82	2.68	2.55	-
LACTIC	-	-	3.33	3.21	3.07	2.92	2.82
HCL	3.31	3.07	2.81	2.58	2.29	-	-
PHOSPHORIC	-	3.13	2.93	2.72	2.34	2.13	-
CF	-	3.22	3.08	2.88	2.76	2.60	-
CM	-	3.32	3.14	2.98	2.82	2.68	-
FM	-	3.31	3.12	2.96	2.81	2.66	-

Experiment 2 - Confirmation of Equi-Sour Levels

Two levels of sourness were selected; one at a low-moderate intensity and the second at a higher but not offensive level. The low level, level 1, 0.08% (0.8 g/L) was the concentration used in Study 1 (Chapter 2) for the free-choice profiling of acids on a weight basis. The value was the calculated concentration of C acid when $\log y=0$ in Experiment 1 (Chapter 3) (see equation C). The panelists found Level 1 to have low to moderate intensity ratings for the different sensory attributes. A second, a higher equi-sour level (Level 2), where C acid was equivalent to 1.0 g/L was also selected. The panelists reported Level 2 to have a moderate to large intensity rating.

The two sourness levels (Table 3.2) are also graphically presented in Figures 3.3 (Level 1) and 3.4 (Level 2) with their corresponding pH. Acid concentration is indicated by the bar graph while the pH is noted above. At equi-sour levels, only GDL, Q and A needed a higher concentration than C. The other acids needed a lower concentration than C, except for L which had an amount similar to C. However, it was very interesting to note the close pH values of the remaining acids, except for the high pH for A and S and the low pH for H and P.

The directional difference from control test was used to confirm the calculated equi-sour levels. Two null hypothesis testings were used to evaluate the results of the confirmation test. The first null hypothesis test determined through ANOVA and LSD, whether the means were equal, i.e. $H_0: \text{mean}_1 = \text{mean}_2 = \text{mean}_3 \dots$. The second method tested the null hypothesis that the means were all equal to 0, ($H_0: \text{mean} = 0$), because theoretically, a sample equal in sourness to the reference would be assigned a value of 0.

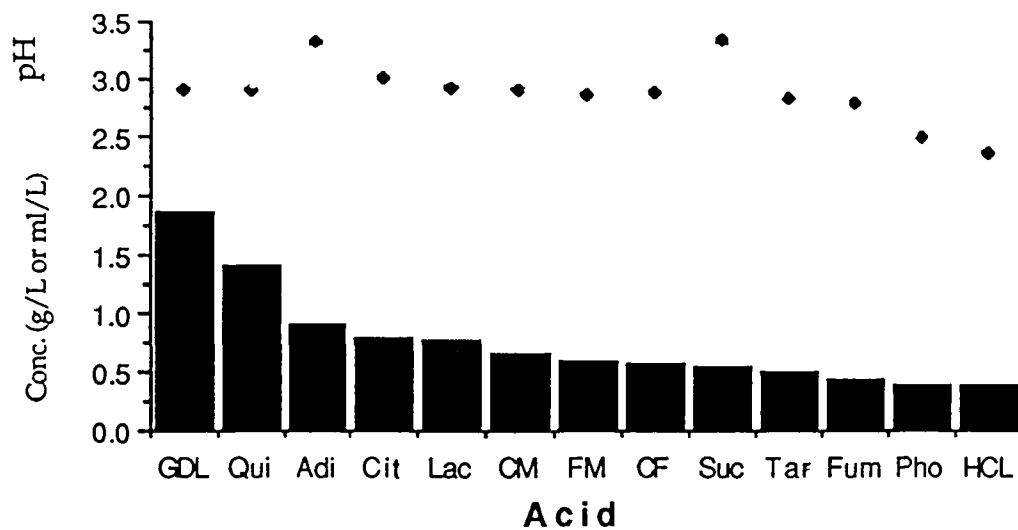


FIG. 3.2. Equi-sour level 1: concentration and pH.

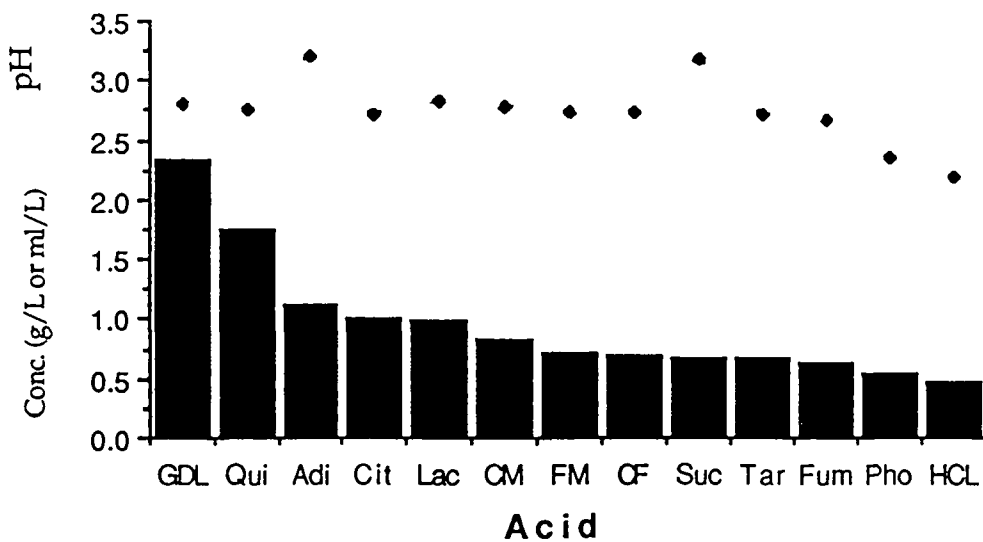


FIG. 3.3. Equi-sour level 2: concentration and pH

The results of the directional difference from control test for testing null hypothesis 1, for equivalence of means are given in Figures 3.5 A and 3.5B. The y-axis was the mean rating with 0 rating represents same as reference, positive (+) values represent more sourness and negative (-) values represent less sour as compared to the reference. Those acid samples with the same letters across the the bar graph are not significantly different. The standard error of the mean and mean values of the intensity ratings across 16 panelists and two replications are listed in Table 3.6.

There were two citric acid samples given to the panelists, Cita (as part of group A) and Citb (as part of group C). Since they were in two separate groups, the data gathered from the two citric acid samples were not pooled during the analysis. Although Cita (0.56 - 0.59) had a slightly higher mean scores for the two acid levels compared to Citb (0.37 - 0.44), the differences were not significantly different. The mean ratings of two replications for all the 16 panelists are illustrated in Figures 3.7A (Level 1) and 3.7B (Level 2). More panelists thought C was slightly more sour than the reference while only one to three panelists gave negative scores (less sour). However, among the acid samples tested, citric acid had the most number of zero scores particularly, Cita (level 2), wherein half of the panel (8 panelists) indicated that Cita was same as reference. The difference found may be due to some psychological errors during sensory testing such as errors of habituation/anticipation and error of expectation (Stone and Sidel, 1985; Meilgaard et al., 1987). Errors of habituation and of anticipation arise from the panelist's possible shift in judgement after a series of evaluations, even with similar products. In the expectation error, the panelists may report differences at a

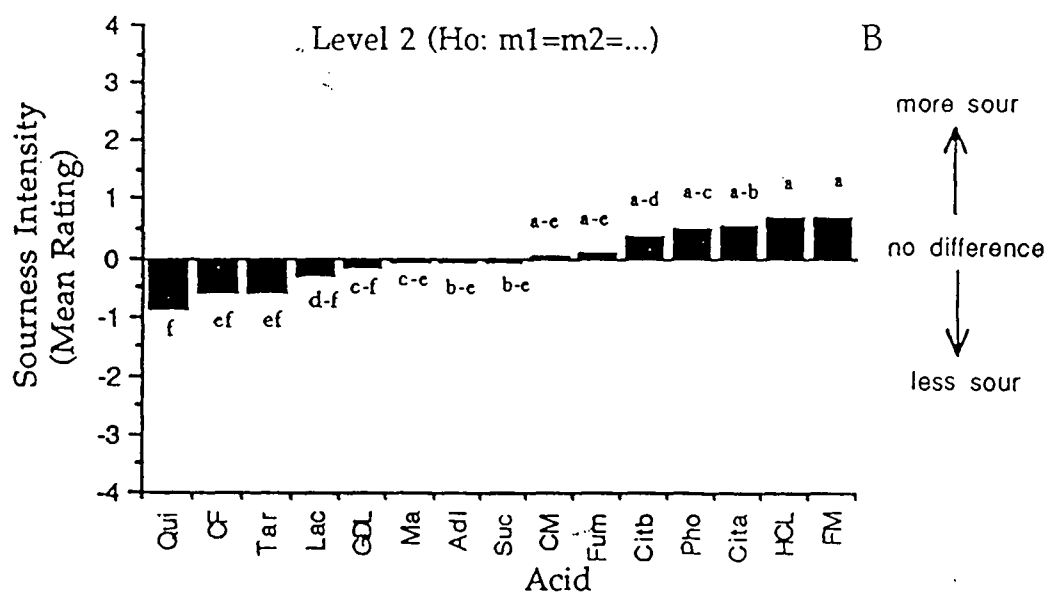
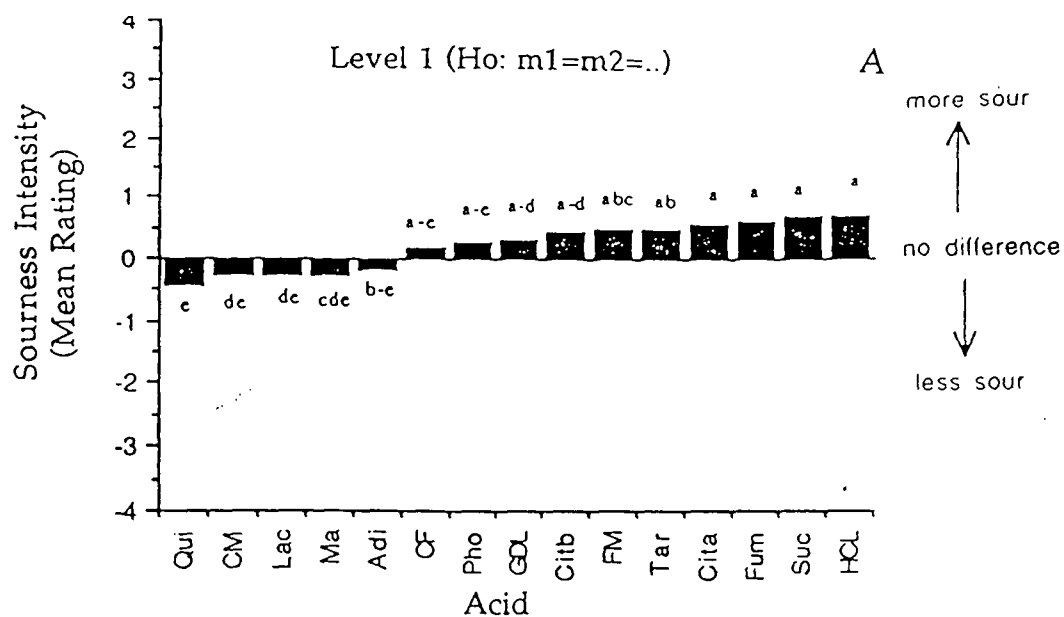


Fig. 3.5. Directional difference from control mean: (A) level 1; (B) level 2. Bars with different letters are significantly different at $p < 0.05$. ($H_0: \text{mean}_1 = \text{mean}_2 = \dots$)

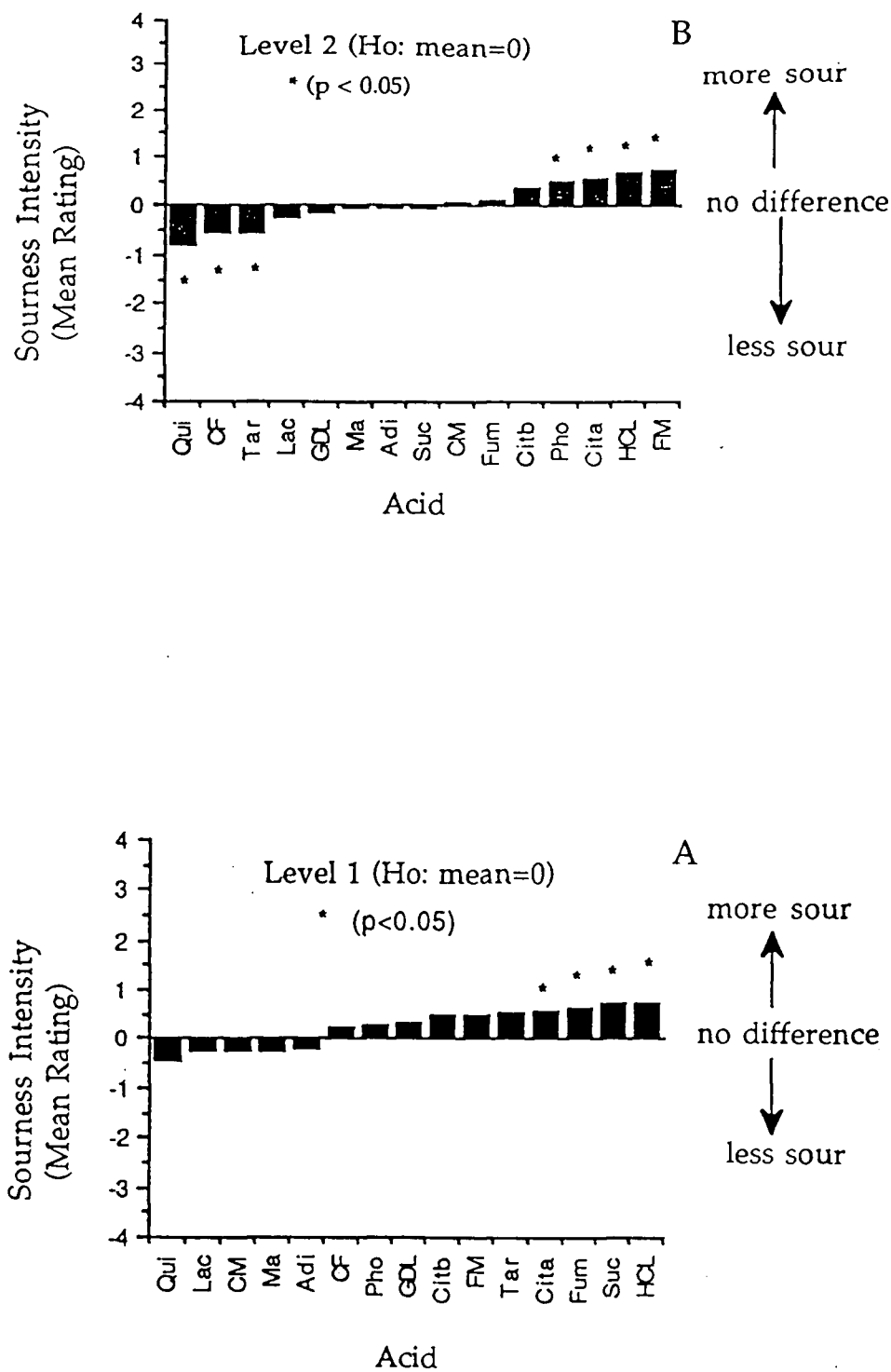


Fig. 3.6. Directional difference from control mean: (A) level 1; (B) level 2. Bars with asterisk (*) are significantly different at $p < 0.05$. (Ho: mean = 0).

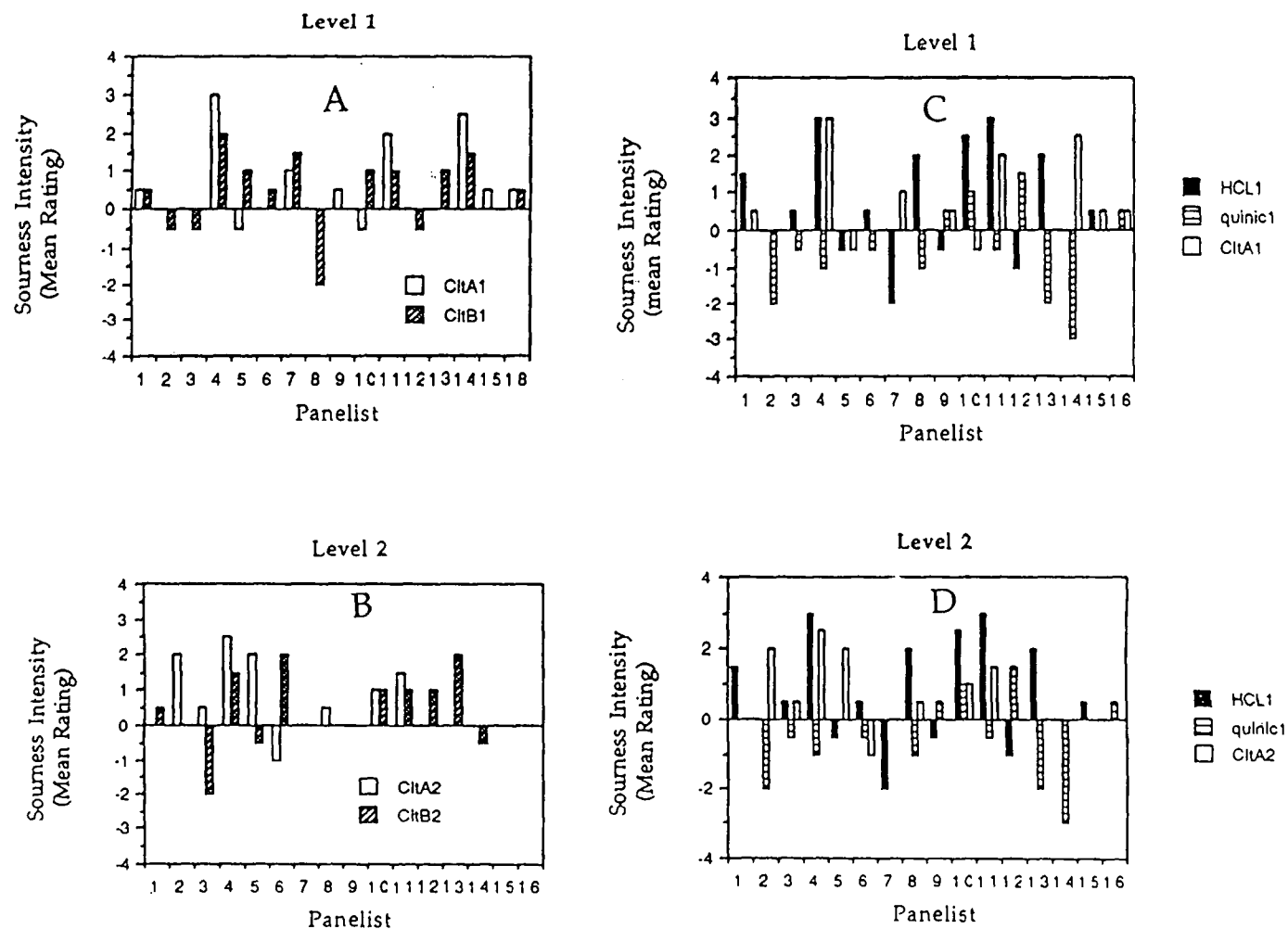


Fig. 3.7. Directional difference from control mean (A) level 1 citric acid (Cita and Citb); (B) level 2 citric acid (Cita and Citb); (C) level 1 hydrochloric, quinic and Cita; (D) level 2 hydrochloric, quinic and Cita.

Table 3.6. Directional difference from control mean and standard error (s.e) for levels 1 and 2 equi-sour concentrations*.

ACID	MEAN ^a	
	LEVEL 1	LEVEL 2
Adipic	-0.19 (0.38)	-0.06 (0.40)
Citric (a)	0.59 (0.34)	0.56 (0.33)
Citric (b)	0.44 (0.33)	0.37 (0.40)
Citric:Fumaric	0.19 (0.32)	-0.59 (0.37)
Citric:Malic	-0.28 (0.33)	0.03 (0.34)
Fumaric	0.62 (0.38)	0.09 (0.35)
Fumaric:Malic	0.47 (0.42)	0.72 (0.43)
Glucono-delta-lactone	0.31 (0.31)	-0.16 (0.34)
Hydrochloric	0.72 (0.46)	0.69 (0.46)
Lactic	-0.28 (0.40)	-0.28 (0.42)
Malic	-0.25 (0.42)	-0.06 (0.32)
Phosphoric	0.25 (0.50)	0.53 (0.43)
Quinic	-0.44 (0.42)	-0.84 (0.33)
Succinic	0.72 (0.51)	-0.06 (0.50)
Tartaric	0.50 (0.36)	-0.59 (0.35)

*Mean of 32 observations (16 panelists x 2 replications)

^aRating scale: extremely less sour than reference (R) (-0.4), same as R (0), extremely less sour than R)

much smaller concentration than expected since there is a tendency to expect differences in samples that are presented.

For acid Level 1 and testing null hypothesis 1, panelists found no significant differences between the sourness intensity of Citb and the rest of the acids except for Q (Fig. 3.4A). However, the other Cita was significantly different not only from Q but also from CM, L, M, and A. This indicated that the groupings of the different acids may have influenced the ratings of the panelists. Cita was grouped with these acids, A, H, L and FM while M, S, T, and CF for Citb. While in comparing the mean rating to 0 for testing null hypothesis 2, only four (H, S, F and Cita) out of fifteen acid samples were significantly different from 0.

For acid Level 2, only Q, CF, and T were significantly different from Citb (Fig. 3.5B) while the same three samples plus Citb, P, H and FM were significantly different from the mean equal zero ($H_0: \text{mean}=0$) (Fig. 3.6B). But Figures 3.5B and 3.6B indicate that the panelists found more similarity in Level 2 than Level 1 because more acid mean ratings were closer to 0 compared to Level 1 (Figures 3.5A and 3.6A). Likewise, the panelists indicated that the concentration of Level 1 was very weak in sensory properties compared to Level 2, thus, the latter was then selected for the next study on characterizing the acids on an equi-sour basis.

Although this study was designed to determine equi-sour levels, the findings of small but significant differences among acids was understandable. First, the mean ratings for all acids ranged only from -0.44 to 0.72 for Level 1 and from -0.84 to 0.72 for Level 2. These values indicate that the difference from control was very slight since the ratings could range from -4 to +4. However, it was interesting to note that Q in both levels was rated less sour by eight and eleven panelists with ratings of -0.44 and -0.84 for Levels 1 (Fig.

3.7C) and 2 (Fig. 3.7C), respectively . In contrast, H was rated more sour than the reference by more than half of the panelists. Its mean rating was the highest for Level 1 and second for Level 2 as well (Table 3.6).

The explanations for these differences may become more clear after doing the descriptive analyses (Chapter 4) because our previous study (Chapter 2) indicated that the sensory properties of these acids were different and the concentration of the acids also affected these properties too. For instance, GDL at 0.16% was closer in sensory properties to 0.08% C but at 0.24%, it was more similar to 0.08% F, FM, CM and CF.

This study illustrated the usefulness of the power function as well the directional difference from control methods in evaluating equi-sourness. Some differences may be explained through the panelists' usual comments that some of these acids had different taste qualities than the citric acid standard which may have interfered with the sourness ratings.

Aside from the power function method, paired comparison method has also been used to determine equi-sourness. However, it is still undetermined which method is better. Beatty and Cragg (1935) and Pangborn, (1963) calculated the equi-sourness using paired comparison by having their panelists rate the sourness of the acids against a reference. Beatty and Cragg (1935) used hydrochloric acid as a reference and Pangborn (1963) used citric acid. They plotted the percent of the responses considering the reference acid more sour than another acid presented at a range of concentrations. The point where 50 percent of the panelists felt that the reference acid was more sour than the test acid solutions was considered equi-sour. The findings of Beatty and Cragg (1953) were in agreement with the present study that hydrochloric must be present in greater amount (molarity) to be equal in sourness to tartaric acid. They reported 0.005M HCL to be of equi-sour to 0.003 of tartaric

acid while in the present study it took 0.00442 M of HCL to be equally sour to 0.00357 M of tartaric acid.

Since we are interested in knowing these other sensory properties aside from sourness, this experiment is very important to determine what level to use in future characterization of the acids on an equi-sour level. The equi-sour levels of the various acidulants would help the acid industry relate one acid to another in terms of sensory properties. However, the importance of chemical properties like pH must not be overlooked because of their obvious differences or similarities at various concentrations. With the same molar (Fig. 3.2) concentrations, the two inorganic acids, HCL and phosphoric, had the lowest pH while adipic acid had the highest pH. Likewise, the same trend was observed at their equi-sour (g/L or ml/L) concentration (Fig. 3.8).

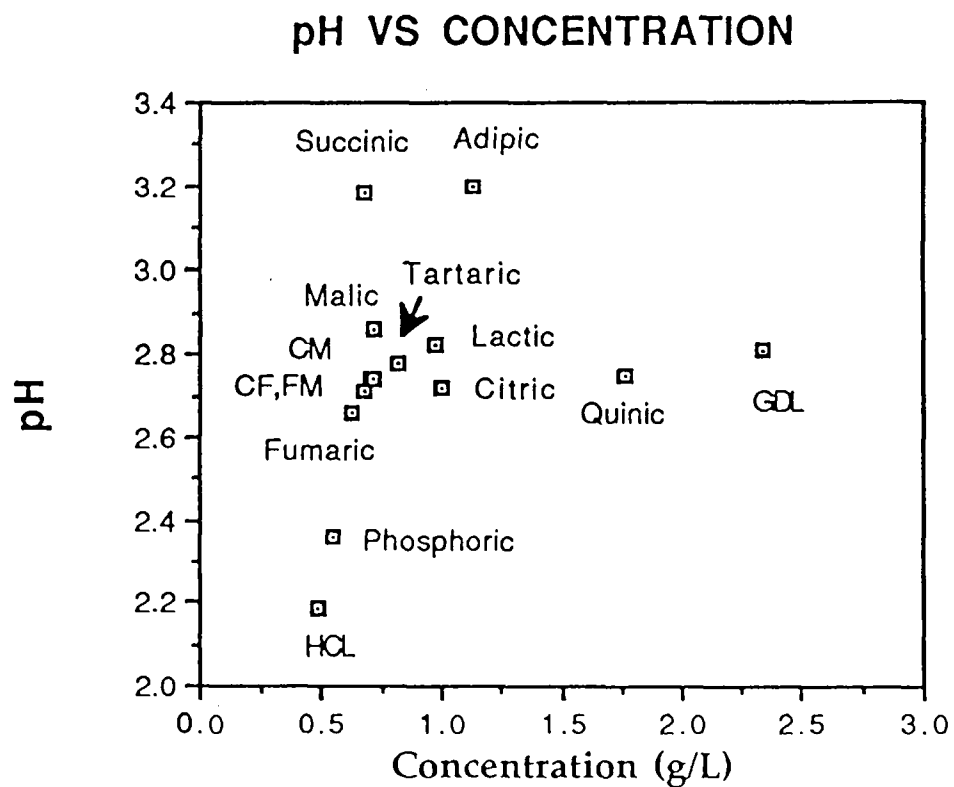


Fig. 3.8. pH of the different acids at their equi-sour concentrations

SUMMARY AND RECOMMENDATIONS

1. Power functions were generated for each acid to evaluate the relationship between the concentration of the acid and the perceived intensity of sourness. The correlation coefficients (r) ranged from 0.80 to 0.93 suggesting a strong linear relationship between the two parameters.
2. Two equi-sour levels (Levels 1 and 2) were chosen based on citric acid concentrations with Levels 1 and 2 when citric acid was equal to 0.08% and 0.10%, respectively. The equi-sour values reported were expressed in molar concentration and on a weight basis when citric acid is given a value of 1.0 g/L.
3. Only glucono-delta-lactone, quinic and adipic acids needed more acid by weight than citric when the latter was taken as 100 resulting in ratios of 2.3, 1.76 and 1.12, respectively. Hydrochloric and phosphoric acids needed only half the amount to get the same sourness intensity.
4. The acid combinations (citric:fumaric, citric:malic and fumaric:malic) apparently increased the sourness intensity in comparison with just citric acid alone.
5. Using the directional difference from control test, no significant differences were found on the sourness intensity of most of the acids compared to citric acid for the two levels. Only quinic acid was significantly different for both levels. The finding of still significant differences among the acids on the supposed equi-sour levels were due to the possible effects of other sensory properties and some psychological errors.
6. Level 2 was selected because panelists found this concentration more similar to the citric acid reference.

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Free-Choice Profiling vs Conventional Descriptive Analysis in Characterizing
the Sensory Properties of Acids on an Equi-Sour Basis

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FREE-CHOICE PROFILING VS CONVENTIONAL DESCRIPTIVE ANALYSIS
IN CHARACTERIZING THE SENSORY PROPERTIES OF ACIDS ON AN
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ABSTRACT

Previous studies on the tastes of acids have focused on threshold, equi-sour levels and time intensity values of sourness. Studies on characterizing the sensory properties of organic and inorganic acids have been very limited. This study was designed to determine the sensory profile of selected acids (adipic, citric, fumaric, glucono-delta-lactone, hydrochloric, lactic, malic, phosphoric, quinic, succinic, tartaric, and citric:fumaric, citric:malic and fumaric:malic combinations) on an equi-sour basis while comparing two sensory methods, free-choice profiling and conventional descriptive analysis. The former method was analyzed by Generalized Procrustes Analysis and the latter with Principal Component Analysis with the principal axes scores analyzed by analysis of variance and least significant difference. The relationship between chemical measures and sensory data was also reported.

The two sensory methods gave the same pattern of information regarding the acid samples. The acids differed in their flavor and taste dynamics even at their equi-sour concentrations with succinic acid having a very intense bitterness and monosodium glutamate taste. Hydrochloric and phosphoric acids were more astringent than sour. While the similarities of several organic acids and their mixtures were very evident, no chemical measure could explain the differences well. It was concluded that acids had

other sensory properties aside from sourness that must be considered in a given food application.

INTRODUCTION

Acidulants contribute a variety of functional properties that lead to the enhancement of food quality (Doores, 1990). Their many functions include aid in sterilization, in food preservation, in chelation (Johnson and Peterson, 1974) and as a flavoring adjunct (Andres, 1985). However, the optimal amount of acidulant that can be added for any food formulation is dictated by the acid's sensory characteristic. Thus, knowledge of these attributes is critical for both acidulant producers and their customers.

Studies on characterizing the sensory properties of organic and inorganic acids have been very limited. Reported studies determined the threshold values (Tada et al., 1972; Maeda and Nakao, 1963), power functions (Moskowitz, 1971; Straub, 1992; Straub and McDaniel, 1989), equi-sour concentrations (Wani and Soma, 1961; Furukawa, et al., 1969; Straub, 1992) and time-intensity properties (Straub, 1992). However, these studies covered only acid sourness except for Straub's (1992) inclusion of astringency in her research.

Recently, the characterization of the sensory properties of 15 acid samples using free-choice profiling was reported by Rubico and McDaniel (1992). Our results, analyzed by general Procrustes analysis (GPA), indicate that on a weight (0.08% w/w or v/v) basis, acids differ in their sensory profiles. The acids separated on three important principal axes (PA) which could be designated as astringency/ mouthfeel, bitterness and sourness axes for the first, second and third PAs, respectively. The astringency/mouthfeel characteristic was the attribute that separated the inorganic acids (hydrochloric and phosphoric acids) from organic acids, while bitterness separated succinic acid from the rest of the acids. The sourness characteristics on the third axis

were the fruit sourness (e.g. lemon, citrus) and not the strong acid taste of the inorganic acids. Thus, the fruit acids were found to be similar to each other and to be significantly different from the inorganic acids.

Since the panelists found the concentration of hydrochloric, phosphoric and succinic to be very strong in sensory characteristics other than sourness, the sensory profiling of acidulants on a molar basis (0.005M) was continued using the same sensory method as the study on a weight basis (Rubico and McDaniel, 1991). On a molar basis, the equivalent concentrations in % w/v or v/v range from 0.04% (hydrochloric and phosphoric) to 0.10% (citric and quinic). The panelists still found significant differences in astringency, bitterness and sourness, although similarities of the fruit acids were more evident. With the goal of acidulant producers and consumers to compare these acids more on their degree of sourness for which they are basically used, the third study characterizing the acids on an equi-sourness level was designed. This characterization determined what other predominant sensory characteristics may be present in the acidulants at their equi-sour level. This will give additional information to the acidulant users on whether one acid can be substituted with another without affecting the sensory properties of the formulated products.

For more in depth in characterization of the various acidulants, two methods of sensory profiling were used, free-choice profiling (FCP) and conventional descriptive analysis (CDA). In FCP, panelists independently develop individual vocabularies while in CDA, a consensus on descriptors is obtained from the panel (Tunaley, 1988). FCP, although a relatively new method, has been used to assess commercial ports (Williams and Langron, 1984), coffee (Williams and Arnold, 1985), fish (Quarmby and Ratkowsky, 1988), sweeteners (Tunaley, 1988), chocolate (McEwan et al., 1989), cheese

(Marshall and Kirby, 1988), whiskey (Guy et al., 1989), beer (Gains and Thomson, 1990), meat products (Scriven and Yak, 1991), milks (Raats and Shepherd, 1992) and cider (Piggott and Watson, 1992).

CDA, in existence for many years, started when Arthur D. Little developed the flavor profile (Cairncross and Sjostrom, 1950; Caul, 1957). Then later, several methods evolved such as texture profiling (Brandt et al., 1963; Szczesniak, 1963), Quantitative Descriptive Analysis (Stone et al., 1974; Stone and Sidel, 1985), and Spectrum (Meilgaard et al., 1987). Many laboratories develop their own style of CDA using portions of the earlier methods.

General Procrustes Analyses (GPA) is the only multivariate statistical method designed to analyze FCP data while several multivariate statistics are applicable for CDA data, with Principal Component Analysis (PCA) being the most common. The GPA method has been described extensively by other investigators (Oreskovich et al., 1991; Gower, 1975; Arnold and Williams, 1986; Gains et al., 1988; Dijksterhuis and van Buuren, 1989) while several illustrations of PCA use in the literature were made on the following products: whiskies (Piggott and Jardine, 1979); beer (Clapperton, 1979); meat (Horsfield and Taylor, 1976); poultry products (Frijters, 1976).

Objectives:

1. To determine the sensory characteristics of some common food acidulants on an equi-sour basis using free-choice profiling and conventional descriptive analysis.
2. To investigate the differences, if any, between free-choice and conventional descriptive analysis.
3. To find the underlying dimensions (structure) of the sensory characteristics used in flavor profiling.

4. To study the relationships between sensory and chemical measures.

MATERIALS AND METHODS

Samples

Fourteen samples of acids (eight organic, three organic mixtures plus two inorganic) were evaluated at their equi-sour level. An equi-sour concentration of each of the different acids was prepared based on the methods used by Rubico and McDaniel (1993). Five molar concentrations of each acid were evaluated by 16 panelists by comparing each concentration to 0.0005M citric acid. The reference was assigned an intensity score of 100 and by using magnitude estimation, the panelists were asked to rate the samples in comparison with the reference. Power functions were established by regressing the log of the response (sensory) on the log of the stimuli (physical). Equi-sour concentrations were estimated by picking a subjective intensity on the y-axis of the plot of the power functions and substituting this value into the power function equation to get the appropriate concentration from the x-axis of each acid. The source of the different acid samples, their codes and the equi-sour concentrations (g/L or ml/L) are listed in Table 4.1.

Panelists

The panelists was comprised of four male and eight female graduate students and staff from the Department of Food Science and Technology at Oregon State University. All had previous trained panel experience. The same group of panelists were used first for FCP and then for CDA. Only panelists who were able to identify the four basic tastes were selected.

Table 4.1. Details of the samples used

Code	Acid	Label Description	Supplier	Concentration (g/L or ml/L*)	pH	TTA
C	Citric	Anhydrous, FCC	Haarmann and Reimer (H&R), Elkhart,IN	1.00	2.72	0.97
M	Malic	DL	Haarmann and Reimer (H&R) Elkhart,IN	0.72	2.86	0.70
F	Fumaric	FCC	Haarmann and Reimer (H&R) Elkhart,IN	0.62	2.66	0.62
A	Adipic	75%	Monsanto, St. Louis, MO	1.12	3.20	1.11
P	Phosphoric		Monsanto, St. Louis, MO	0.54*	2.36	0.59
G	Glucon-delta Lactone		Finnsugar Bioproducts, Schaumburg, IL	2.34	2.81	2.03
H	Hydrochloric	NF-FCC,36.5-38%	J.T.Baker Inc., Jackson,TN	0.48*	2.19	0.42
L	Lactic	USP-FCC,85-90%	J.T.Baker Inc., Jackson,TN	0.98*	2.82	0.70
Q	Quinic	D	ICN Biomedicals Inc., Cleveland, OH	1.76	2.75	1.69
S	Succinic		Penta Manufacturing Co. East Hanover,NJ	0.68	3.18	0.66
T	Tartaric		Penta Manufacturing Co. East Hanover,NJ	0.68	2.71	0.66
CF	Citric:Fumaric	50/50		0.70	2.74	0.67
CM	Citric:Malic	50/50		0.82	2.78	0.82
FM	Fumaric:Malic	50/50		0.72	2.74	0.71

Presentation of samples

All samples were clear, colorless solutions which were presented at room temperature in coded 85-ml plastic cups (30-ml sample). During the initial vocabulary development for FCP, all the 14 samples were coded and evaluated in random order. For the actual assessment, 15 instead of 14 acid samples were evaluated to even up the three acid groupings. This was done by putting an additional citric acid in one group. The 15 samples were divided into three sets of five and presented in counterbalanced order among the 12 panelists. The groupings of the acid were as follows: Group A - A, C, H, L, FM; Group B - G, P, F, Q, CM; and Group C - M, S, T, C, CF. Five samples were evaluated at each session. Three replications were done for FCP.

Free-Choice Profiling

Panelists attended four 30-minute sessions where they were instructed in the FCP procedure and presented with 14 samples. They were asked to taste the samples and to describe, using their own terms, the flavor and mouthfeel of the acid samples. During the training sessions, the panelists were instructed to write of their terms and definitions which were included in their individual ballot. Different concentrations of citric acid were given to the panelists in order to practice scoring the magnitude of different characteristics on a 16-point intensity scale (0 = none, 7 = moderate, 15 = extreme). A rating sheet was then developed by each panelist for use during the actual assessment. When tasting all the acid solutions, the 'sip-and-spit' method was applied. The panelists were instructed to sip the sample and manipulate it in the mouth for 7 s, then expectorate and rinse the mouth three times with bottled water. To minimize any adaptation effect, a resting

period of 60 s was imposed between samples. The panelists were seated in separate, well ventilated daylight- illuminated booths.

Conventional Descriptive Analysis

Panelists attended 10 one hour training sessions. At the start of the training session, the panelists discussed and defined all the sensory characteristics perceived in the acids. Standards or references were created and tested and thus used to help develop a vocabulary and definitions (Table 4.2). After discussions with all the panelists, the lists of descriptors and definitions were consolidated into a consensus vocabulary of 8 terms perceived when the samples were in the mouth (overall intensity, sourness, astringency, bitterness, sweetness, saltiness, monosodium glutamate (MSG), metallic) and 6 terms for the samples after expectoration (astringency, sour, bitter, metallic, chalky, persistent). Then, using these attributes were rated using same 16 point scale as in FCP.

Statistical Analysis

The FCP data were analyzed by Procrustes Analysis using Procrustes-PC Version 2.2 (Dijksterhuis and van Buuren, 1991) and by Statistical Analysis System for Personal Computer (SAS, 1987). For the free-choice profiling experiment the data from each of the 12 panelists were assembled into matrices of 45 rows (15 samples assessed over three replications) by n columns, where n represented the number of attributes for each panelist. Using these matrices a generalized Procrustes Analysis was carried out.

Table 4.2. Consensus descriptors selected for aqueous solutions of fourteen acid samples, the composition of standards used to represent them and their consensus definitions. F = Flavor when in mouth; A = aftertaste.

DESCRIPTOR	COMPOSITION OF STANDARD	INTENSITY RATING	DEFINITION
F - OVERALL INTENSITY			The overall impact
F/A - SOUR	CITRIC ACID		Taste stimulated by acids such as citric
	0.05%	2	
	0.08%	5	
	0.15%	10	
	0.20%	15	
F/A - ASTRIN- GENT	ALUM		The shrinking or puckering of the tongue surface caused by alum
	0.15%		
	0.25%		
F/A - BITTER	CAFFEINE		The taste stimulated by caffeine which is perceived primarily at the back of the tongue
	0.05%	2	
	0.08%	5	
	0.15%	10	
	0.20%	15	
F - SALTY	NaCl		The taste stimulated by NaCl
	0.20%	2.5	
F - SWEET	SUCROSE		The taste stimulated by sucrose
	2.0%	2	
F - MSG	MONOSODIUM GLUTAMATE		The taste stimulated by MSG
F /A- METALLIC	FERROUS SULFATE		The taste of metal. Characterized by eating a boiled egg with certain type of metal spoon, or aluminum foil touching a tooth filling
A - CHALKY			
A - PERSISTENT			The length of time the sensation lasts

Analysis of variance (ANOVA) on the principal axis scores was used to ascertain differences among samples, and least significant differences.

The CDA data were analyzed by PCA using SAS (SAS, 1987) and by PC-ORD (McCune, 1990). The means for each acid on all attributes were calculated across all 12 panelists and data were assembled into matrices of 60 rows (15 samples assessed over 4 replications) by n columns, where n represented the number of attributes. Data were analyzed by PCA and principal axis scores was analyzed by ANOVA and by Least Significant Difference (LSD).

Likewise, PCA was also run for just the mean scores across all panelists and replications using PC-ORD. Powers (1984) pointed out that PCA is commonly run for a data set consisting of sample means averaged across panelists and over replications for each attribute. Use of a large single data set is not recommended (MacFie, 1987) because the resulting configuration is very crowded and may be difficult to interpret (Oreskovich et al., 1991).

Chemical Measures

The following chemical parameters were included in the PC-ORD analysis with their corresponding abbreviations as follows:

-pH	-Number of COOH (NoCOO)
-Titratable Acidity	-Number of Hydrogen Ion (NoH)
-Normality (Norma)	-Weight or Volume (Weight)
-Molarity (Molar)	

The pH of the 14 acid samples was measured by a pH electrode with a microprocessor pH/mV meter (Orion Model 811) equipped with a

combination pH electrode (Ross Model 81550). Total titratable acidity was determined using a glass electrode titrating with 0.1N NaOH to an endpoint of pH 8.2.

RESULTS AND DISCUSSIONS

Free-Choice Profiling

The panelists listed descriptors when the acid samples were in the mouth and after expectoration. The number of descriptors used varied from three (Panelist 2) to fifteen (Panelist 8), with an average of nine terms per panelist (Table 4.3). Some panelists appeared to have difficulty in generating sufficient descriptors, a situation also reported by Piggot (1991). McEwan et al. (1989) found that panelists, in the isolation of sensory booths, find it difficult to describe their perceptions.

Sour, astringent and bitter were the most common terms used. As expected, individuals behaved differently in selecting descriptors and the number of dimensions required to describe differences among the acids. All panelists except Pan 1 and Pan 2 had terms unique to themselves like *musty* for Pan 3, *soda water* and *plastic* for Pan 4, *cold taste* for Pan 5, *sulfur* for Pan 6, *vinegar* for Pan 7, *warming* for Pan 8, *takes off enamel* for Pan 9, *MSG* and *abrasive* for Pan 10, *acid* for Pan 11 and *spoiled* for Pan 12. This situation usually creates a problem for a panel leader using the CDA method during ballot development. Achieving consensus on what attributes to include generally requires extensive discussion, particularly if several members of the panel have strong personalities. Free-choice profiling avoids this communication problem. Difficulty in interpretation of results may be encountered, especially if some terms are unclear like the terms *spoiled*,

Table 4.3. Terms generated by each panelist in the free-choice profiling of acids.

	Pan 1	Pan 2	Pan 3	Pan 4	Pan 5	Pan 6	Pan 7	Pan 8	Pan 9	Pan 10	Pan 11	Pan 12
When In Mouth												
1	Sour	Sour	Sour	Sour	Sour	Sour	Sour	Sour	Sour	Sour	Sour	Sour
2	Astringent	Bitter	Astringent	Astringent	Salty	Astringent	Bitter	Astringent	Bitter	Astringent	Acid	Bitter
3	Bitter		Bitter	Bitter	Bitter	Bitter	Yucky	Sickening	Aspirin	Bitter	Bitter	Lemony
4			Fruity	Citrus	Cold Taste	Salty	Chalky	Drying	Sharp	Mild	Salty	Salty
5			Musty	Aspirin	Aspirin	Lemon	Citrus	Refreshing		Abrasive		Fruity
6			Sweet	Soda Water		Sulfur	Vinegar	Warming		Citric		Sharp
7				Sweet				Salty		Spill		
8										MSG		
9										Aspirin		
After Expectoration												
1	Astringent	Astringent	Astringent	Astringent	Dry	Astringent	Astringent	Sour	Astringent	Astringent	Aftertaste	Linger
2	Sour		Fruity	Bitter		Bitter		Astringent	Bitter	Abrasive	Metallic	Chalky
3			Smooth	Plastic		Lingering		Refreshing	Goes away fast		Salty	Yuk
4			Linger					Warming	Metallic			Smooth
5			Mouth puckering					Sickening	Takes enamel			Spilled
6								Drying	off teeth			Mouth
7								Sweet				puckering
8								Salty				

warming, and cold taste that are listed in Table 4.3. With this subjectivity of individual panelists, analyzing the FCP data by GPA can figure out the interpretation of the results more objectively. GPA groups the unique terms and assists the panel leader in understanding the meaning of those terms.

The final result of a GPA is a consensus configuration of the samples for the different principal axis (PA) combinations as graphically presented in Figure 4.1 for the first two dimensions which accounted for 31% and 25% of the original variance. The third dimension accounted for a further 16% but could not be readily interpreted and will not be discussed further. The entire analysis was conducted at the individual level, but for clarity, only the consensus across individuals and replicates is shown.

To supplement the graphical information from GPA, an analysis of variance (ANOVA) of the scores on each of the first four principal components of the consensus was carried out with the results given in Tables 4.4 and 4.5 for the first and second components, respectively. The scores for the third and fourth axes were not significant and, therefore, not reported.

To interpret the main perceptual dimension of the spaces, the vector loadings for each individual panelist were examined by inspection. These loadings simply provide a weighting for each attribute on a principal axis and they can be used to decide which attributes are important. Thomson and McEwan (1988) stated that it is reasonable to select those attributes that have loading coefficients (weightings) greater than 0.3 or less than -0.30. The combinations of descriptors for the first two principal axes are summarized in Table 4.6.

The first PA showed a contrast between bitter/aspirin, MSG/salty, and yucky/sick/spoil/musty on the positive side with astringent and sour on the negative side. The negative axis was also described as sharp, takes off enamel,

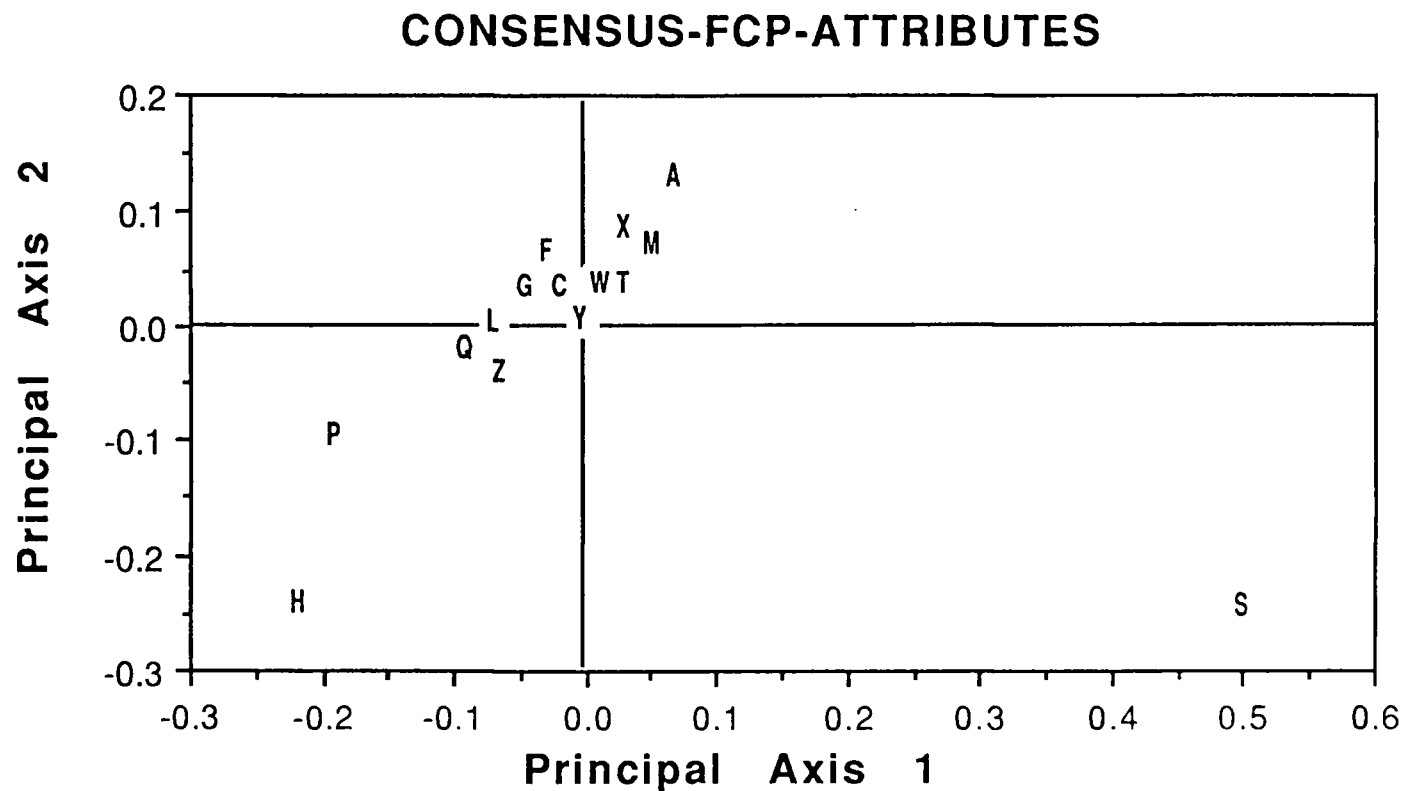


Figure 4.1. Sample Consensus Plot for Free-Choice Profiling of Different Acids on an Equi-Sour Basis-Following Generalized Procrustes Analysis (GPA) : Principal Axes 1 vs 2. A-adipic, C-citric, F-fumaric, H-hydrochloric, L-lactic, M-malic, P-phosphoric, Q-quinic, S-succinic, T-tartaric, X-citric:fumaric, Y-citric:malic, Z-fumaric:malic, G-glucono-delta-lactone W-citric.

abrasive, mouth puckering and chalky. These terms probably also described the mouthfeel when tasting an astringent sample. All the characteristics on the positive axis referred to succinic acid which was found to be significantly different (Table 4.4) compared to the rest of the acids oriented towards the negative axis (phosphoric and hydrochloric) and to the group of acids on the middle axis (Fig. 4.1). There were three major groupings of acids based on the letter superscripts (Table 4.4) on the first PA, with succinic as the sole member of the first group, then the bulk of the organic acid plus the mixtures from adipic to fumaric:malic as the second group. The inorganic acids comprised the last group with lactic and quinic acids leaning towards both the second and third groups. This was an indication that on an equi-sour basis, succinic, hydrochloric and phosphoric acids had other predominant attributes aside from sourness.

Table 4.4. Principal axis (PA) 1: mean scores for different acids

Acid	Mean Score
Succinic	0.497 ^a
Adipic	0.067 ^b
Malic	0.057 ^{bc}
Citric:Fumaric	0.030 ^{bcd}
Tartaric	0.027 ^{bcde}
Citric:Malic	-0.004 ^{bcde}
Citric	-0.013 ^{bcde}
Fumaric	-0.030 ^{bcde}
Glucono-delta-lactone	-0.033 ^{bcde}
Fumaric:Malic	-0.064 ^{cde}
Lactic	-0.070 ^{def}
Quinic	-0.093 ^{ef}
Phosphoric	-0.187 ^{fg}
Hydrochloric	-0.220 ^g

* Different letter superscripts indicate significant differences at $P < 0.05$.

Table 4.5. Principal axis (PA) 2: mean scores for different acids

Acid	Mean Score
Adipic	0.134 ^a
Citric:Fumaric	0.093 ^{ab}
Malic	0.078 ^{abc}
Fumaric	0.070 ^{abc}
Tartaric	0.053 ^{abc}
Glucono-delta-lactone	0.043 ^{abc}
Citric	0.040 ^{abc}
Citric:Malic	0.131 ^{abc}
Lactic	0.067 ^{abc}
Quinic	-0.013 ^{abc}
Fumaric:Malic	-0.037 ^{bc}
Phosphoric	-0.087 ^{cd}
Hydrochloric	-0.237 ^d
Succinic	-0.243 ^d

* Different letter superscripts indicate significant differences at $P < 0.05$.

Table 4.6. Interpretation of the first two principal axes from GPA of data from FCP panel.

Principal Axis	Loading +/-	Description	
1	positive (+)	Bitter (6)	Yucky (2)
		Af-Bitter (2)	Musty
		Aspirin	Af-sick
		Sour	Sick
		Salty	Spoil
		Af-salty	Sulfur
		MSG	Af-linger (2)
			Dry
	negative (-)	Astringent (3)	Sour (5)
		Af-astringent (4)	Citrus
		Sharp	Bitter
		Abrasive	Af-plastic
		Chalky	
		Mouth Puckering	
		Takes off enamel	
2	positive (+)	Sour (3)	Af-astringent (2)
		Af-sour	Goes away fast
		Vinegar	Mild
		Acid	
	negative (-)	Astringent (3)	Bitter (5)
		Af-astringent (4)	Sour (3)
		Chalky (2)	Af-sour
		Yucky (2)	Salty (2)
		Abrasive	Af-sick
		Musty	Sick
		Spoil	Af-linger (2)

*Where more than 1 panelist had a loading of greater or equal to 0.30, number is given in brackets

Af-descriptors after expectoration

The second PA was not as easy to interpret as the first PA based on the attribute groupings. However, the positive axis was described by sour characteristics (vinegar and acid). The acids that had positive mean scores were the organic acids and their mixtures starting from adipic to lactic acid; the acids on the negative axis were succinic, hydrochloric, phosphoric, FM and quinic, although the differences among the organic acids other than succinic were not significant (Table 4.5). The attributes on the second PA were astringency, mouthfeel and the bitter/salty. The presence of sour characteristics on the negative axis may be due to panelists' difficulty in distinguishing sourness from bitterness or bitterness from astringency (Powers, 1984). Likewise, Robinson (1970) pointed out the confusion that exists between designating a sensation as sour or bitter. This may be more true with the FCP method because panelists were not trained and no standards were given.

The groupings of the attributes are shown in Table 4.6. Based on their GPA loadings, we were able to successfully decipher the terms used by the panelists even though there were no definitions given for each attribute. The panelists agreed in their description of succinic as bitter with an aspirin, MSG and salty taste (to some panelists); the negative descriptors like musty, yucky, sick, spoil, sulfur were used to describe similar characteristics by different panelists. Likewise, the astringency of hydrochloric and phosphoric acids may also relate to such descriptors as abrasiveness, sharp, chalky, mouth puckering and 'takes off enamel'. Different panelists used different terms to describe the same sensation. Conducting the free-choice profiling section of the study first, prior to conventional descriptive analysis, allowed us to cull the number of terms significantly. It was also very helpful in minimizing the training time of the panelists since the same group of panelists were used.

Conventional Descriptive Analysis

Principal component analysis (PCA) has been the most popular method in analyzing CDA data. Basically it has two main functions: first, to indicate the relationship among groups of attributes in a data set, and second, to show the relationship between objects or samples (Piggott and Sharman, 1986). Aside from Canonical Correlation, only PC-ORD can relate two matrices (e.g. sensory data and chemical data) set up in one data set. This is not possible with some of the most popular statistical packages available (e.g., SAS and Statgraphics). So by using PC-ORD, the PCA could satisfy an additional objective, to determine the relationships between sensory and chemical data.

The first two principal components (PC) accounted for 72.31% of the explained variation, with 44.84% and 19.29% for the first and second components (PC), respectively (Table 4.7). The third and fourth dimensions accounted for a further 16% of the variance, but they could not be satisfactorily interpreted.

The first PC had MSG, bitterness, persistent and metallic aftertaste as the most influencing attributes (Fig. 4.2) which basically described solely succinic acid (Fig. 4.3). This axis was mainly a contrast between succinic acid and the rest of the acids, i.e., succinic acid was significantly different than the other acids (Table 4.8).

The second PC was a contrast between sourness aftertaste (negative axis) versus astringency, astringent aftertaste and overall intensity (positive axis) as shown in Table 4.7 and Figure 4.2. It was critical to note here that the sourness when the acid samples were in the mouth was not an important discriminating attribute in the second PC, but sourness after expectoration was. This means that the acid samples oriented towards the negative axis of

the second PC such as adipic, fumaric and citric:malic (Fig. 4.3) had sourness that was more lingering than other acidulants. Thus, the importance of separating the characteristics before and after expectoration was very important. This may be one of the reasons why even on an equi-sour basis, slight significant differences were noted when the confirmation study was done using the directional difference from control test (Chapter 3). This can be confirmed by doing determining time-intensity characteristics of these acidulants on their equi-sour level.

For the second PC, the inorganic acids, HCL and phosphoric were significantly more astringent than the organic acids, although HCL was significantly more astringent than phosphoric acid (Table 4.9). The fact that sourness aftertaste was the key attribute for the second PC rather than the sourness in the mouth, was an indication that most of the acid samples had almost the same sourness intensity. This means that we were successful in our study on equi-sour level determination (Chapter 3) and the differences on the sourness intensity was more on the lingering aftertaste of sourness after expectoration.

The clear separation of inorganic acids (HCL and phosphoric) and succinic acids on the different PCs was again a confirmation from the results of our previous studies on profiling the acids on a weight basis (Rubico and McDaniel, 1992) and on a molar basis (Rubico and McDaniel, 1991) that the inorganic acids are astringent and that succinic acid has msg/bitter taste. These characteristics are more predominant than sourness.

HCL was also reported by Crozier (1920) to exhibit a characteristic astringency in a dilute solution to a point that the panelists described it not to

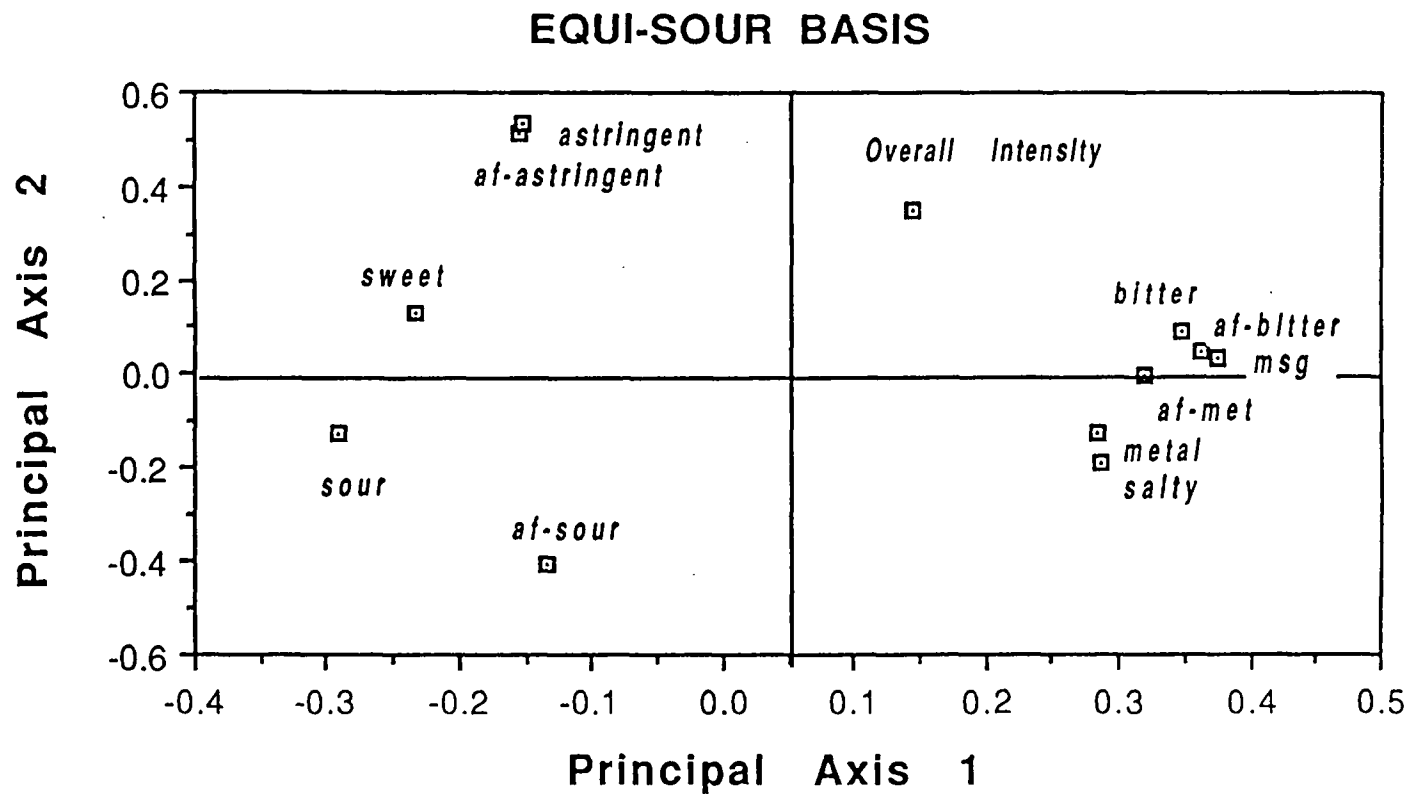


Figure 4.2. Loadings of the terms used for descriptive analysis of 15 acid samples on the first two principal components. Af-indicates attributes after expectoration.

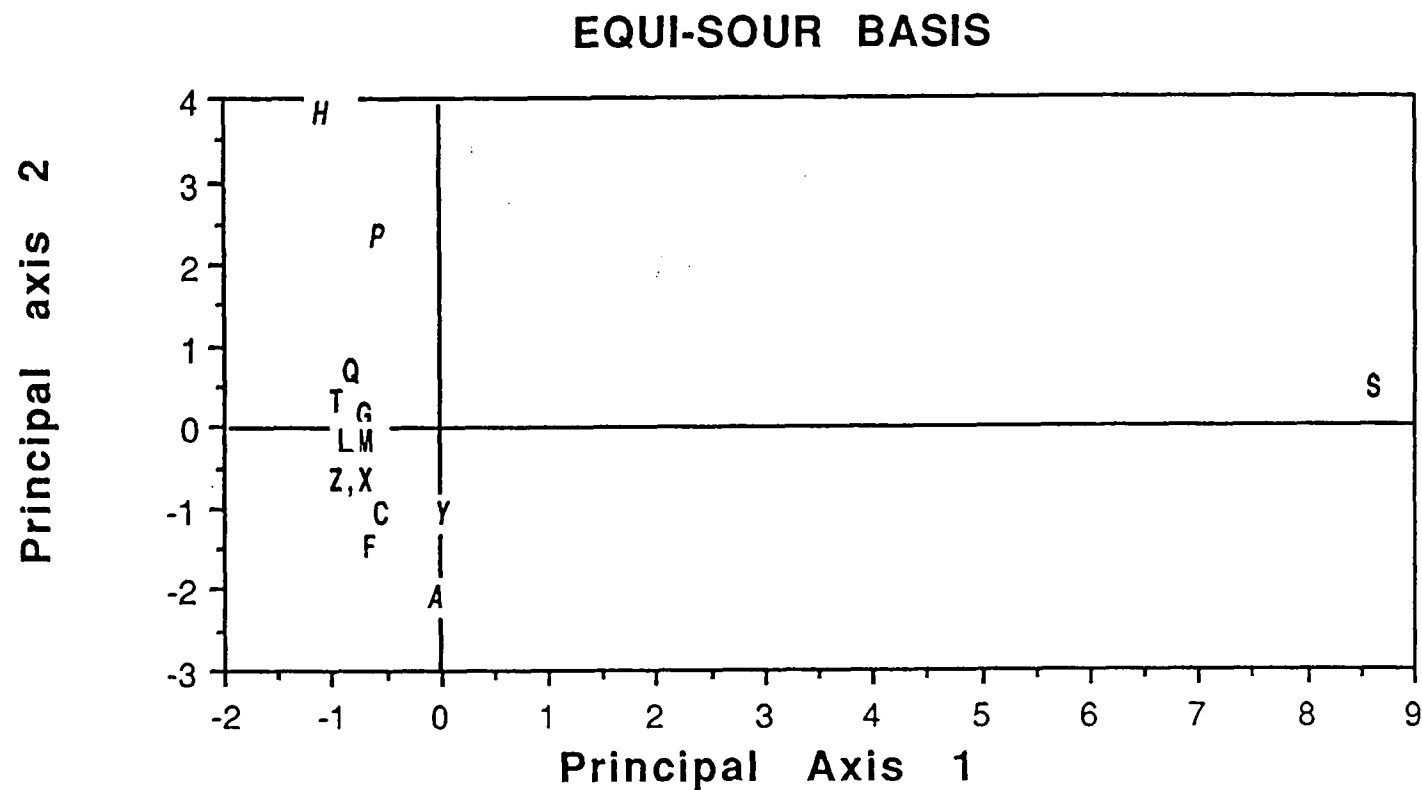


Figure 4.3. Consensus configuration on first (horizontal) and second (vertical) dimensions for the 14 acid samples following PCA of descriptive analysis data. A-adipic, C-citric, F-fumaric, H-hydrochloric, L-lactic, M-malic, P-phosphoric, Q-quinic, S-succinic, T-tartaric, X-citric:fumaric, Y-citric:malic, Z-fumaric:malic, G-glucono-delta-lactone

Table 4.7. Eigenvectors (loadings) and eigenvalues of the different principal component axes.

ATTRIBUTE	PRINCIPAL COMPONENT	
	1	2
When in the Mouth		
Overall Intensity	0.144	0.349
Sour	-0.290	-0.132
Astringency	-0.151	0.533
Bitter	0.349	0.089
Sweet	-0.232	0.128
Salty	0.287	-0.195
MSG	0.376	0.026
Metallic	0.285	-0.125
After Expectoration		
Astringency	-0.155	0.516
Sour	-0.134	-0.409
Bitter	0.363	0.043
Metallic	0.321	-0.010
Chalky	-0.010	-0.031
Persistent	0.339	0.251
Eigenvalue	6.28	2.70
Proportion (%)	44.84	19.29
Cumulative (%)	44.84	72.31

Table 4.8. Principal Component (PC) Axis 1: Mean Scores for Different Acids.

ACIDS ^a	MEAN SCORE
Succinic (S)	8.678 ^a
Citric:Malic (CM)	0.056 ^b
Adipic (A)	-0.007 ^b
Phosphoric (P)	-0.476 ^b
Citric (C)	-0.547 ^b
Fumaric:Malic (FM)	-0.630 ^b
Fumaric (F)	-0.649 ^b
Citric (C2)	-0.654 ^b
Lactic (L)	-0.677 ^b
Citric:Fumaric (CF)	-0.746 ^b
Quinic (Q)	-0.783 ^b
Malic (M)	-0.788 ^b
Tartaric (T)	-0.794 ^b
Glucono-delta-lactone (G)	-0.917 ^b
Hydrochloric (H)	-1.067 ^b

^a Different letter superscripts indicate significant differences at $p < 0.05$

Table 4.9. Principal Component (PC) Axis 2: Mean Scores for Different Acids.

ACIDS ^a	MEAN SCORE
Hydrochloric (H)	3.917 ^a
Phosphoric (P)	2.300 ^b
Quinic (Q)	0.656 ^c
Succinic (S)	0.486 ^{cd}
Glucono-delta-lactone (G)	0.256 ^{cde}
Tartaric (T)	0.034 ^{cdef}
Lactic (L)	-0.038 ^{cdef}
Malic (M)	-0.360 ^{cdefg}
Citric:Fumaric (CF)	-0.507 ^{defg}
Fumaric:Malic (FM)	-0.548 ^{defg}
Citric (C2)	-0.695 ^{fgh}
Citric (C)	-1.037 ^{fgh}
Citric:Malic (CM)	-1.080 ^{fgh}
Fumaric (F)	-1.371 ^{gh}
Adipic (A)	-2.013 ^h

^a Different letter superscripts indicate significant differences at $p < 0.05$

be sour after all. This was also true for succinic acid for having an umami (mono-sodium glutamate) taste. The predominance of these two additional tastes, astringency and umami, supports the conclusion by Schiffman and Erickson (1980) that taste is not limited to sweet, sour, salty and bitter domains.

This study shows that acids, even at their equi-sour level, may have a different influence on the quality of a product. Even though one acid may be less expensive than the other, substitution may not be possible because of differences in taste dynamics such as the difference in the time-intensity parameters. A good example was the lingering sour aftertaste of adipic acid compared to GDL. Likewise, their chemical and physical properties have to be taken into consideration. Some acidulants may have caused a dramatic change in the pH of the formulated food which consequently may affect the functional properties of the product.

Overlays as shown from Figures 4.4 to 4.11 offer another flexible way of showing patterns of variables on the ordination axes by using variously sized circles or ellipses to indicate differences in relative magnitudes (McCune, 1990). Overlay plotting ability is another advantage of using the PC-ORD statistical package which can help explain clearly and in more detail the differences among the acidulants. The bigger the bubble size, the higher the intensity characteristic of an acidulant. For example, on an equi-sour basis, succinic acid appears not to be sour at all (Fig. 4.4), but very persistent (Fig. 4.5), quite salty (Fig. 4.6) and has an MSG taste (Fig. 4.7) based on its bigger bubble size compared to other acids. Since the panelists were forced to rate the sourness intensity of succinic acid compared to citric acid when the equi-sour level determination study was conducted, the equi-sour concentration might not be the correct estimate after all. Succinic acid may not even have

sourness but more of the bitterness and MSG as the predominant characteristics. Succinic may be considered as another acid that is not predominantly sour, like picric acid and other amino acids that are bitter but not sour. The same problem was also encountered in comparing the inorganic acids with citric acid. This was again well illustrated in the overlay plots of astringency characteristic when the acid samples were in the mouth. The overlay plotting confirmed the more astringent characteristic of HCL compared to phosphoric and other acids (Fig. 4.8) Among the organic acids, quinic was more astringent based on its bigger bubble size compared to citric and especially to the least astringent samples, adipic and succinic. The astringency of quinic may contribute to the astringent aftertaste of cranberry, kiwi and several immature fruits where it is reported to be present in high concentration.

Another interesting finding here was the chalky aftertaste of some acids, particularly phosphoric acid, followed by tartaric and malic acids (Fig. 4.9). This attribute was not a predominant one in the first two axes. Thus, this additional information from the overlay plot may be useful in differentiating the acidulants.

Since the acids were rated on an equi-sour basis, as expected, almost all of the acids except for succinic had the same bubble size for sourness (Fig. 4.4) when the sample was in the mouth. However, sourness after expectoration (aftertaste) did not have the same trend, with HCL and succinic acids disappearing quickly while adipic acid stayed the longest followed by fumaric, citric and the acid combinations (Fig. 4.10). By comparing the overlay plots of sourness intensity when the samples were in the mouth (Figures 4.4) to the sour aftertaste (Fig. 4.10), different information is given on the characteristics of the different acidulants as was discussed previously. The overlay plots

helped explained the results of PCA results better. This underlies the importance of not relying on just one method or program when analyzing the data which has been observed in a lot of publications wherein conclusions from sensory data were just based on either ANOVA or other multivariate method such as PCA.

Since PCA is the most popular statistical test for CDA data, many researchers are using PCA as if this method can explain everything, or as what Johnson and Wichern (1988) explained, "that analyses of PCA are more of a means to an end rather than an end in themselves because they frequently serve as intermediate steps in much larger investigations". Thus, Tabachnik and Fidell (1989) recommended the use of both multivariate and univariate statistics in data analysis.

Sensory Parameters vs Chemical Measures

Finding a chemical measure that could predict the sensory properties of a product, e.g. sourness of a formulated food, has always been a challenge. It remains a challenge because there is no clear answer to the basic question of what stimulates sourness perception. Moskowitz (1971) found no simple relationship between sourness and physico-chemical properties of 24 carboxylic acids. Whether it is pH, titratable acidity or the combinations of these chemical parameters is still unresolved. Harvey (1920), Plane et al. (1980) and Joslyn (1974) believe that the sour taste of acids is influenced more by titratable acidity than by pH.

To evaluate these relationships, the correlation coefficient (r) and the coefficient of determination (r^2) were evaluated and tabulated in Table 4.10. The r value explains how good the relationship was [0 value as no correlation and a maximum value of +1 (perfect positive correlation) or -1

(perfect negative correlation)] while the r^2 multiplied by 100 indicates the amount of variation (from the total) in Y (sensory data) that was explained by the independent variable X (chemical measure data). Based on the Pearson and Kendall Correlation, no chemical measure could explain well the first PC (the MSG, bitter, metallic axis) while a negative correlation was evident between the second PC (astringency) and pH (-0.70), explaining 49 % of the variation, followed by normality (-0.63). This was also clearly illustrated in the overlay plot of pH (Fig. 4.11) and PC 1. These negative relationship may have been driven by the very low pH of HCL (2.19) followed by phosphoric acid (2.36). HCL in contrast to adipic acid had the highest pH (3.20) and the sample with the least astringent characteristic (Fig. 4.8). Similar to findings in our previous study, the relationship between astringency and pH was more pronounced than was the relationship between pH and sourness both on the sensory profiling of acids on a weight basis (Rubico and McDaniel, 1992) and on a molar basis (Rubico and McDaniel, 1991). Straub (1992) also found that the pH of an acid is more related to astringency than sourness. Since there is not a complete understanding of the mechanism involved in sourness perception, it is difficult to fully explain the results.

Comparison between FCP and CDA

Results from this study clearly show that the two sensory methods gave almost the same pattern of information regarding the acid samples. Although interpretation of results for free-choice profiling was more difficult because of the large number of terms representing each dimension, the use of free-choice for characterizing acids also had advantages. It not only provided adequate descriptive information, but it allowed the freedom to investigate individual differences in perception. Starting our study with the free-choice

profiling allowed us to determine how panelists from different cultures and backgrounds perceived the various acids. The Oriental panelists contributed the MSG term to the acid attributes which the non-Oriental panelists (Caucasian, Hispanic, etc.) became aware of this during the training.

From the long lists of terms developed during FCP, we were able to greatly reduce the number of attributes for CDA, especially terms that meant the same thing (particularly astringency and mouthfeel characteristics). Since we used the same panelists for the two studies, training time was vastly shortened. In our study, the FCP essentially became a training ground for the CDA. Those wanting to do conventional descriptive analysis would be well advised to start with FCP before going to CDA, especially if panelists come from diverse backgrounds and cultures. The FCP results aid the panel leader in getting a broad range of usable attributes and which may be culled after GPA.

The success of FCP may also be dependent upon the samples to be evaluated. If the samples are plentiful and have a broad range of characteristics, then FCP is recommended, as we found with the sensory profiling of acids on a weight basis (Rubico and McDaniel, 1992). In that study, we were able to successfully differentiate the 15 acid samples in three dimensions. However, with samples that have similar characteristics particularly most of the organic acids in this study, interpretation was more difficult.

For industry practice, the choice between FCP and CDA must be carefully studied. While consensus profiling provides clearer, more concise information that is easier to relay and understand, the length of time to conduct a study and, consequently the higher costs, makes this method difficult for some companies. R&D and marketing professionals want quick

results because of the fast turnaround in developing a new product. Basically, the method of choice depends primarily on the objectives of the project. For instance, if the goal is to relate sensory data and chemical measures, there is no doubt that CDA is the method of choice because there is no guarantee that the same terms used in FCP may have the same meaning for different panelists. So relating the groups of data set will be misleading. Since standards were provided in the CDA, the panelists knew what the term meant. Likewise, statistical tests available to evaluate FCP data and chemical measures at the same time are limited. For the CDA data, several statistical methods can be applied such as canonical correlation (SAS, 1987) and PCA in PC-ORD (McCune, 1990). However, if the project's goal is to compare the sensory properties of a particular brand of product to those of the competitors, running FCP may save time and money for the company.

In conclusion, neither method can be considered superior, because both have their advantages and disadvantages. Likewise, this is true for data analysis, where the use of several techniques (univariate such as ANOVA and several multivariate like PCA and GPA) is more appropriate than concentrating on just one method.

Table 4.10. Pearson and Kendall correlation coefficient (r) and coefficient of determination (r^2) of the different chemical measures with ordination axes (PCA 1 and PCA 2) for 14 acid samples.

Chemical Measures	Principal Components			
	1		2	
	r	r^2	r	r^2
pH	0.54	0.29	-0.70	-0.49
Weight	-0.15	0.02	-0.18	0.32
Molarity	-0.14	0.02	0.04	0.01
No. of COOH	0.20	0.04	0.16	0.03
Normality	0.15	0.02	-0.63	0.40
Titrateable Acidity	-0.11	0.12	-0.30	0.09

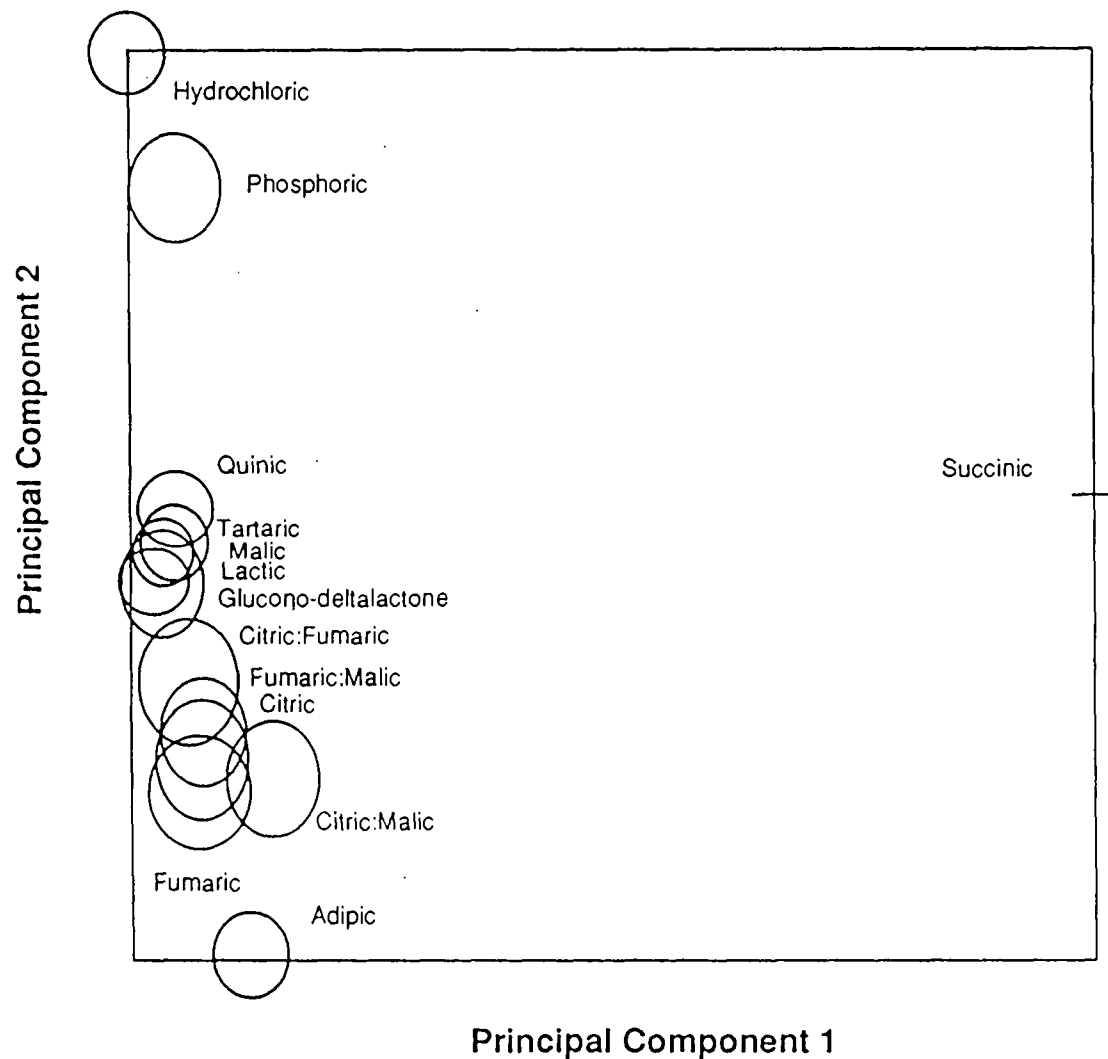


Figure 4.4. Overlay plot of sourness of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (_).

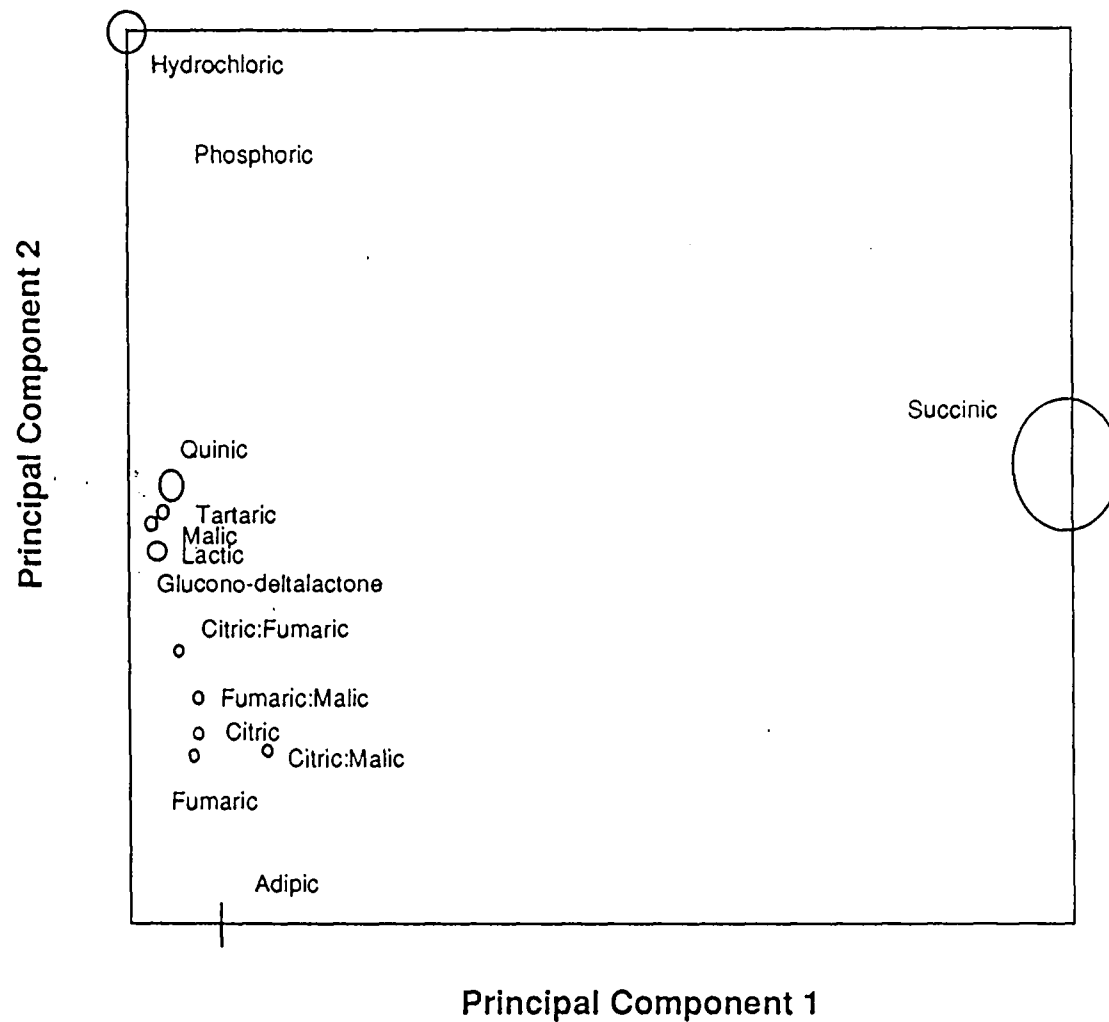


Figure 4.5 Overlay plot of persistence of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (—).

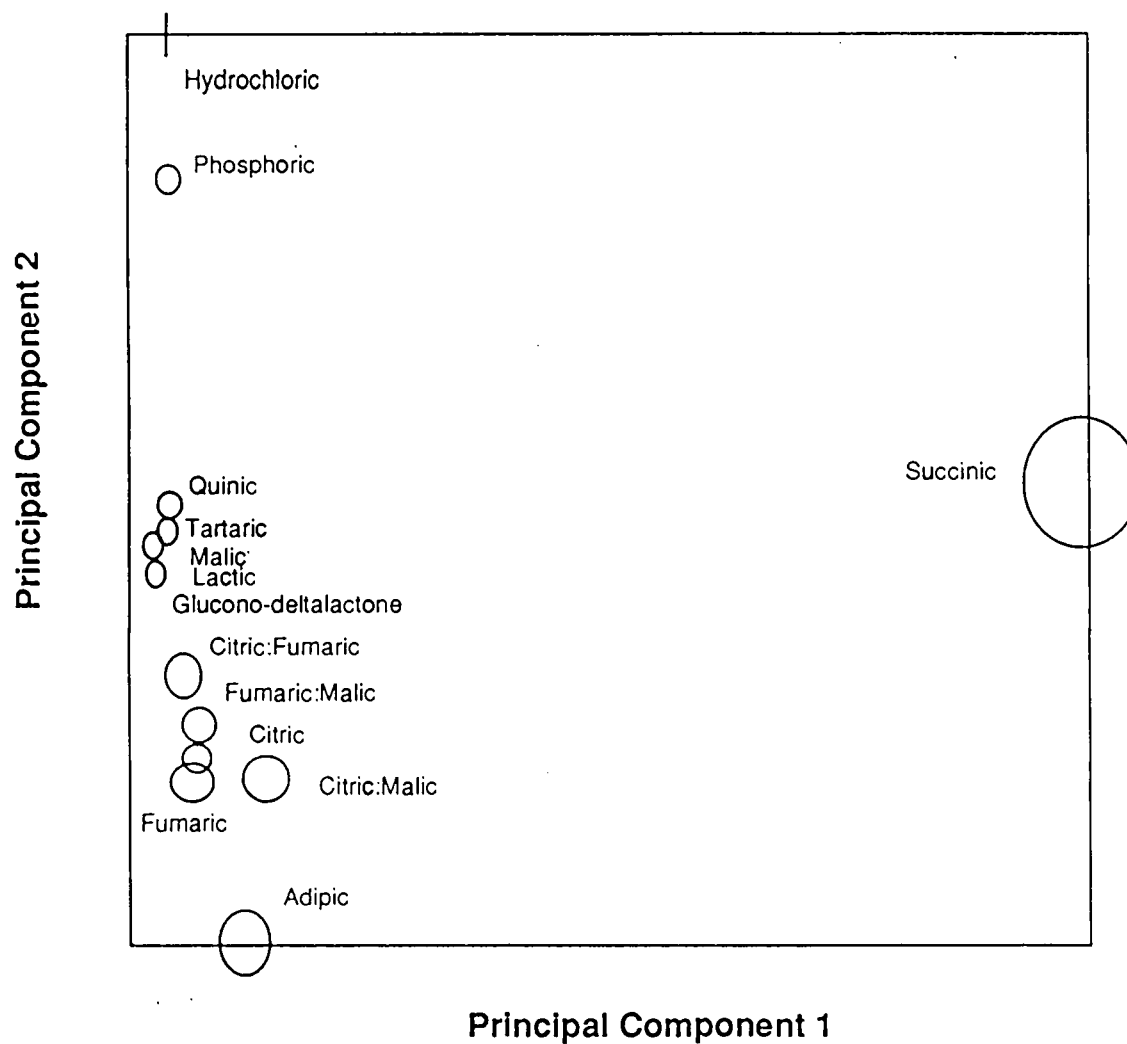


Figure 4.6. Overlay plot of saltiness of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (_).

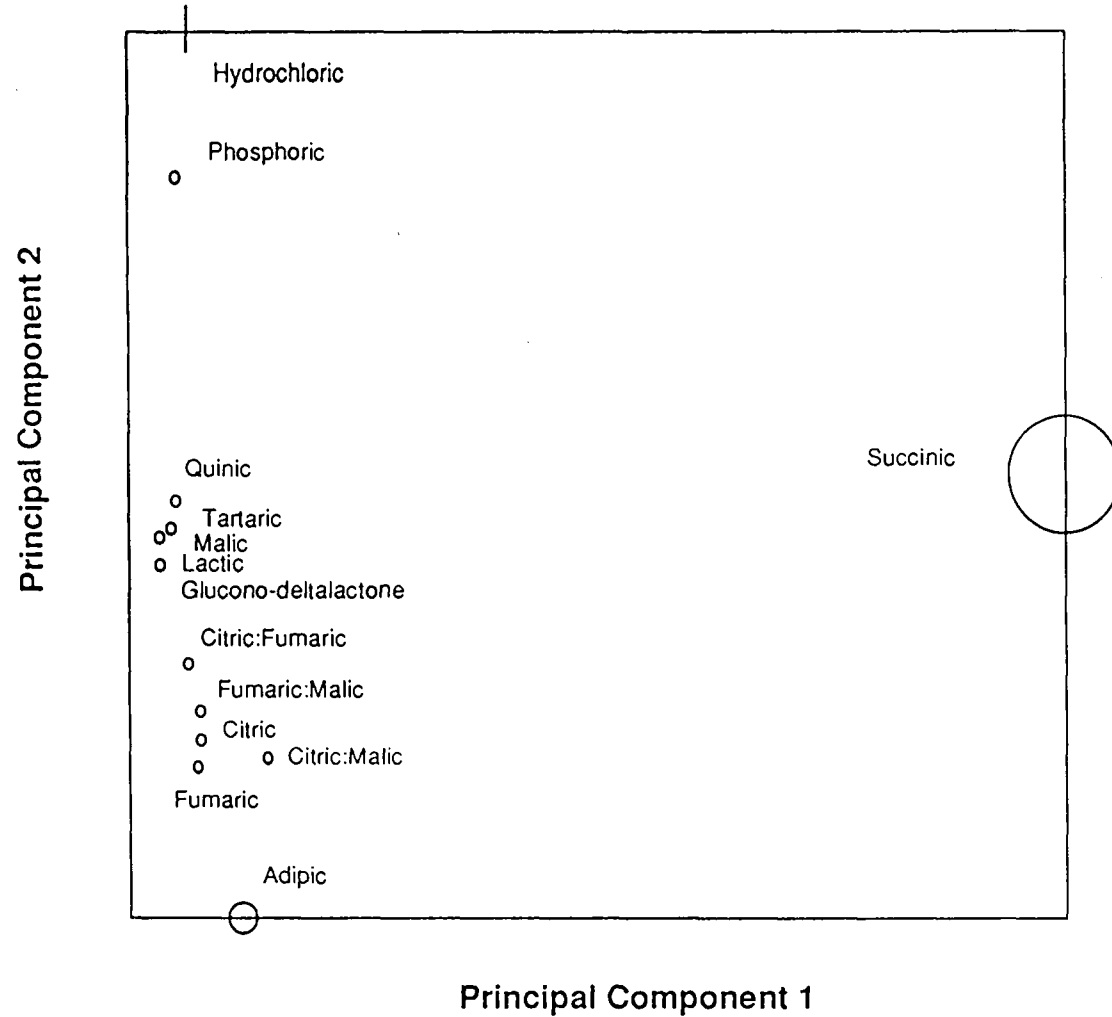


Figure 4.7. Overlay plot of MSG of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (_).

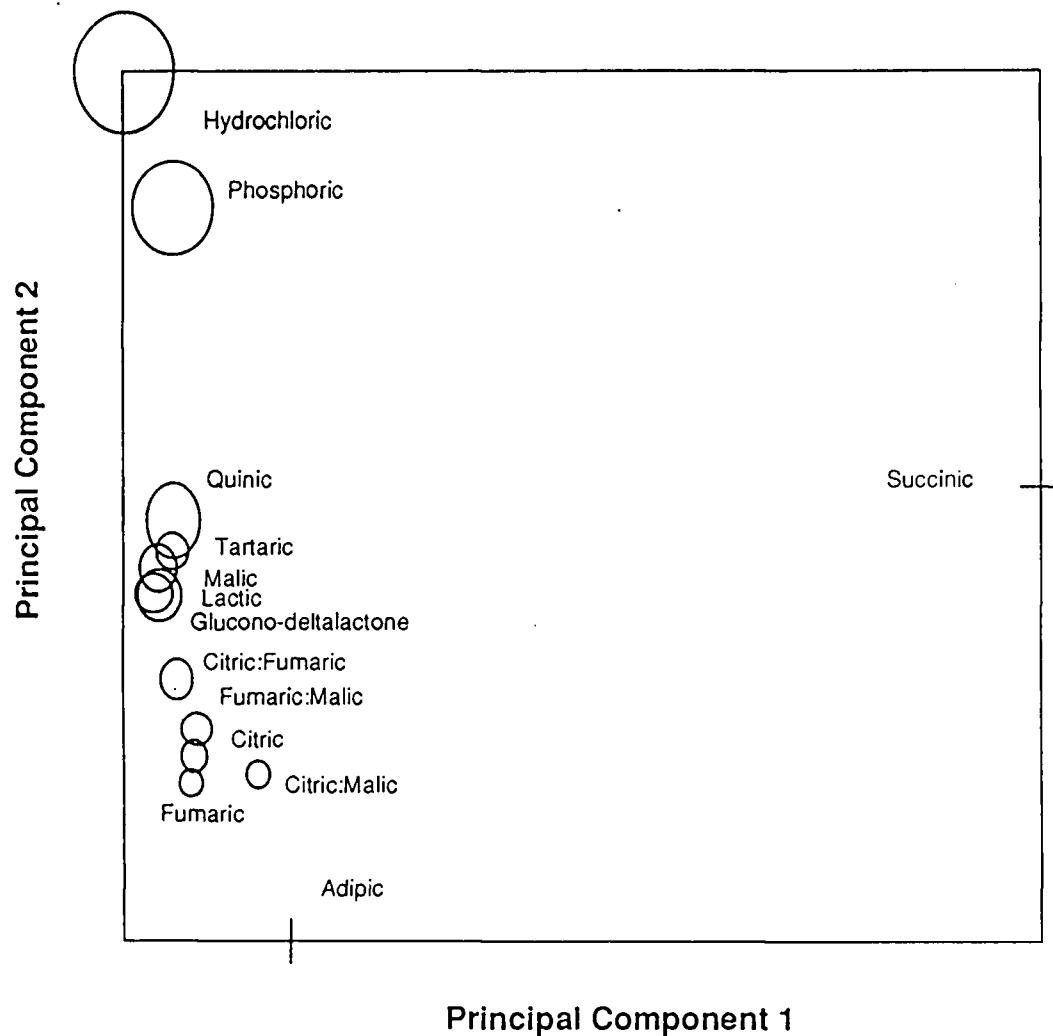


Figure 4.8. Overlay plot of astringency of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (_).

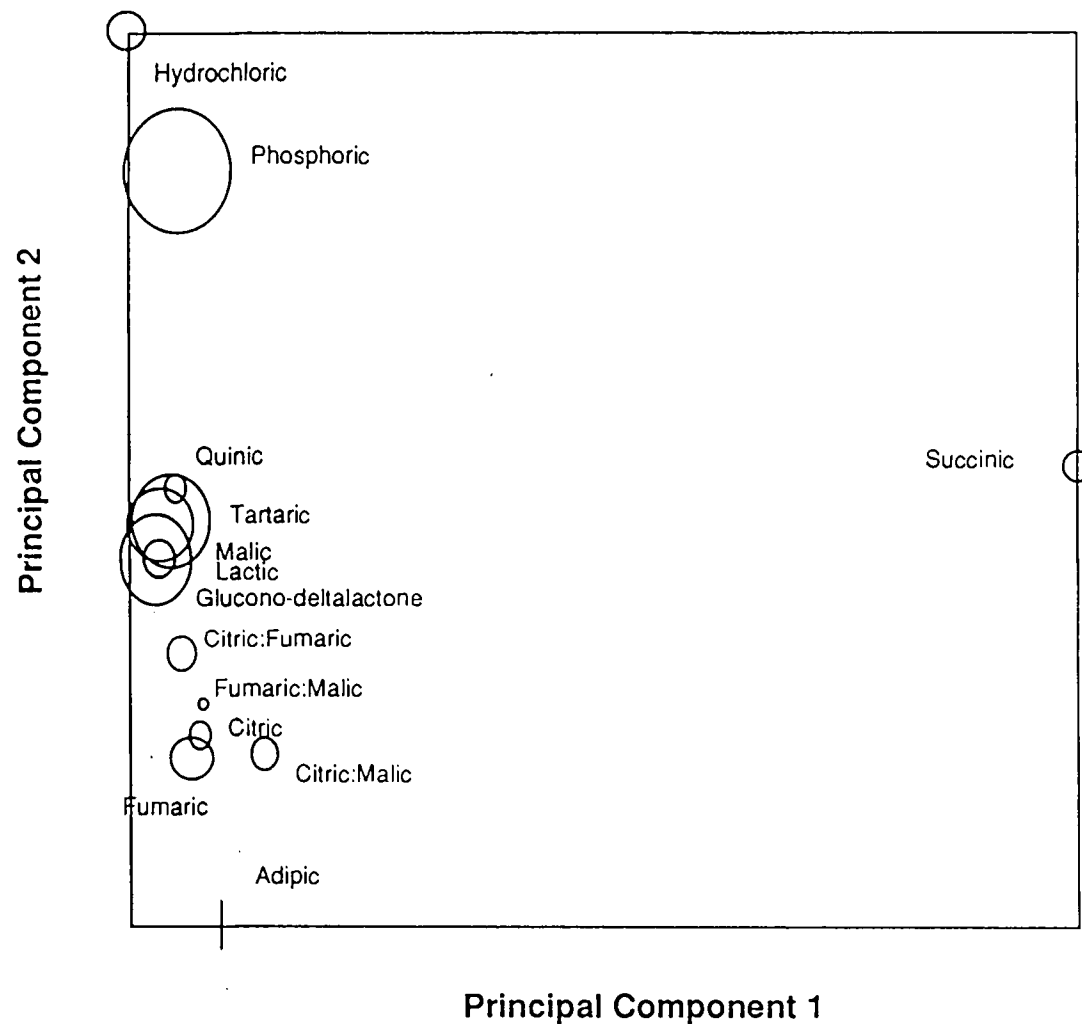


Figure 4.9. Overlay plot of chalkiness aftertaste of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (_).

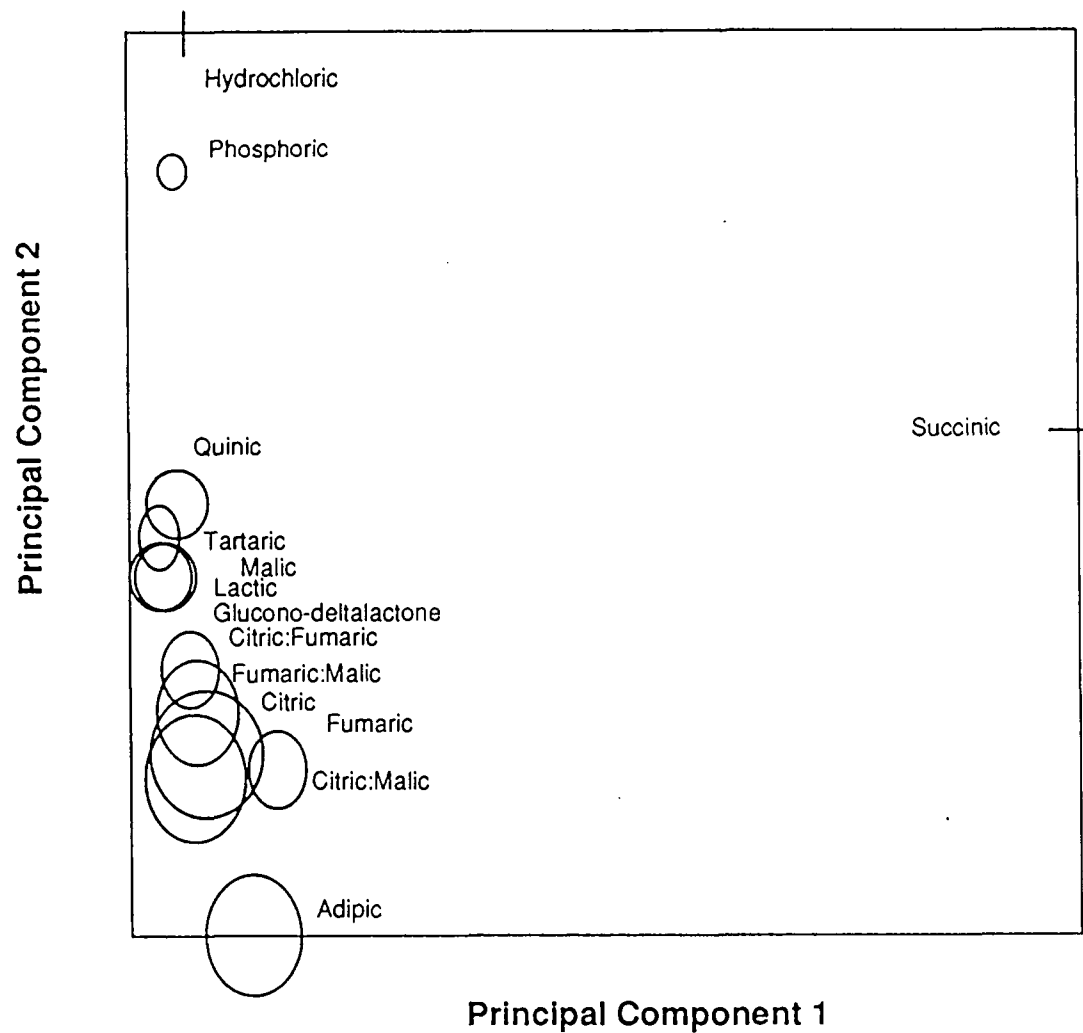


Figure 4.10. Overlay plot of sourness aftertaste of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher intensity of the attribute. Absence or very low intensity of an attribute is symbolized by a line (_).

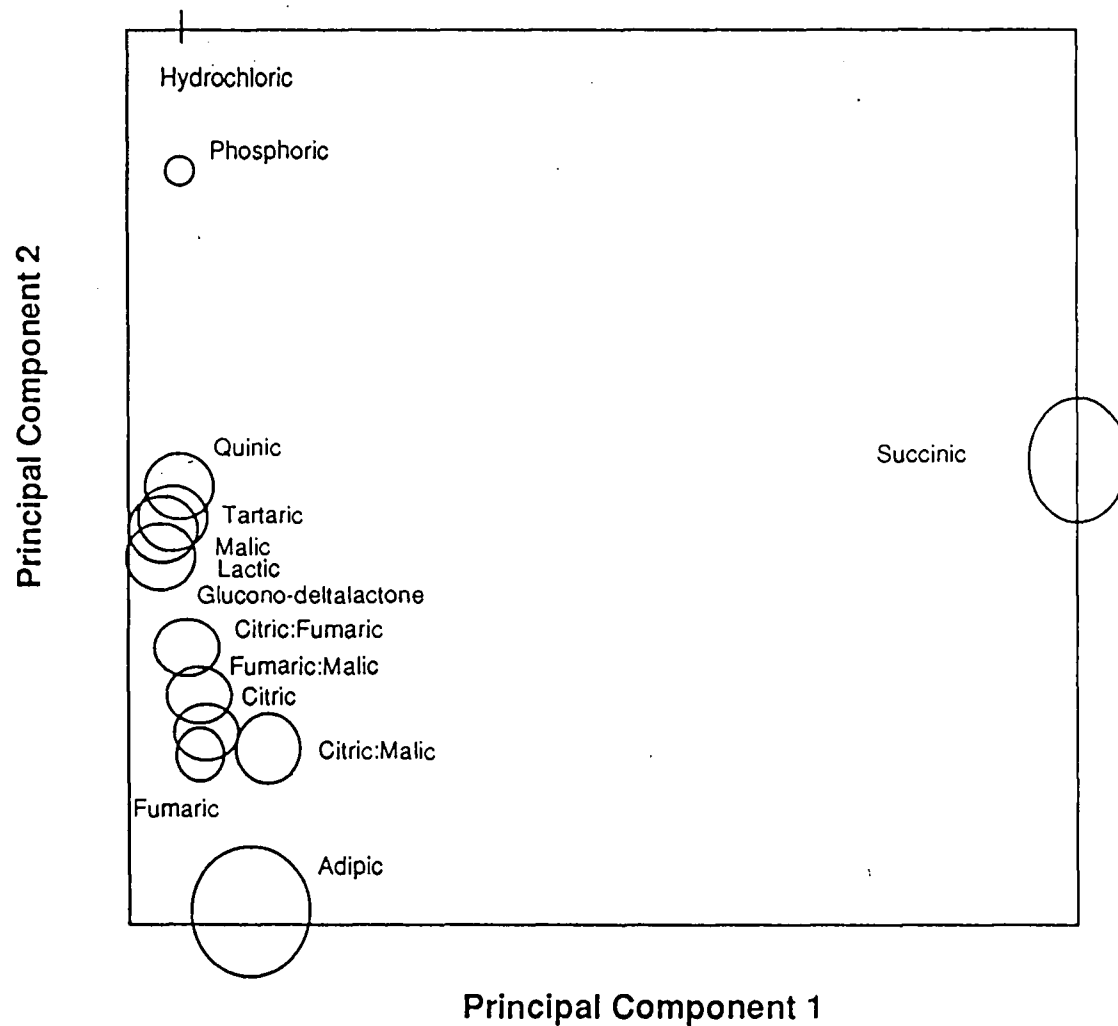


Figure 4.11. Overlay plot of pH of the 14 acid samples on the first two principal axes following PCA. The bigger bubble size (O) represents higher pH and a line () represents very low pH.

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