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Date December 17, 1989

SENSORY AND CHEMICAL EVALUATION OF BROWNED FLAVOR IN CONVENTIONAL AND MICROWAVE BAKED COOKIES

CRANES CREST

A Thesis

Presented for the

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Susan Gail Varner

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DEDICATION

To my parents, Charles and Sue Varner

because I will always cherish their love and support

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ABSTRACT

The flavor of 4 different cookies was investigated by sensory and chemical analyses. Cookies were made from 2 different formulas, a conventional chocolate chip cookie base (CON) and a cookie formula optimized for spread and texture during microwave baking (MW). The cookies from the CON and MW formula were both baked in a conventional oven (CON CON and MW CON, respectively), and the cookies from the MW formula were baked in the microwave oven (MW MW). Also, commercial color and flavoring additives (ADD) were added to the MW formula before baking in the microwave oven (MW MW ADD). Two paired-comparison tests using selected pairs of cookies were conducted. Panelists were blindfolded and asked if there were a flavor difference and to describe any detectable difference. Test 1 consisted of the following pairs of cookies: CON CON/MW MW, CON CON/MW CON and MW CON/MW MW and test 2, MW MW ADD/CON CON, MW MW ADD/MW MW and MW CON/MW MW. Each of the 4 cookies also was analyzed for acceptability (8-point scale were 8 = like extremely and 1 = dislike extremely) by a consumer panel of 92 people. Head-space volatile compounds from each of the 4 cookies (repli-cated twice) and the color-flavoring additive were trapped

on Tenax GC precolumns and analyzed by gas chromatograph-mass spectroscopy (GC-MS).

Results of test 1 indicated flavor differences ($P < .05$) between members of the CON CON/MW CON and CON CON/MW MW cookie pairs. The difference in flavor in test 1 was attributed primarily to a difference in perceived sweetness with the CON CON cookie being sweeter. Results from test 2 showed flavor differences ($P < .05$) between members of the MW MW ADD/MW CON and the MW MW ADD/MW MW cookie pairs. The flavor difference in test 2 was due to a burnt sugar, overbaked, and burnt marshmallow flavor in the MW MW ADD cookies compared with bland, flour-like, flavorless MW MW or MW CON cookies. Consumers liked ($P < .05$) the CON CON cookies more than the MW CON, MW MW and the MW MW ADD cookies. They also liked ($P < .05$) the MW CON cookies better than the MW MW and MW MW ADD cookies. Twenty-four volatiles were isolated form the headspace of the cookies, 13 of which were identified. Seven volatiles (6 identified) were isolated from ADD and were also found in the MW MW ADD cookies. Two of the volatiles, diacetyl and furfural, in ADD were also isolated from the CON CON and MW CON cookies. In general, except for these 7 volatiles, cookies baked in a conventional oven had more volatiles and a higher concentration of those volatiles than cookies baked in a microwave

oven. The majority of these volatiles were formed by nonenzymatic browning reactions and contributed to consumers liking the CON CON and MW CON cookies more than the MW MW and MW MW ADD cookies.

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CHAPTER 1

INTRODUCTION

For baked products, raw materials and processing conditions affect the chemistry of the browning reaction which produces the most significant flavor-contributing compounds associated with browned flavor and odor. Prior to World War II, little attention was paid to Louis-Camille Maillard who determined that heat treatment of sugars and amino acids causes these reactants to condense and emit an aroma similar to that of freshly baked bread. Caramel, buttery, burnt sugar and toasty flavor notes are contributed to baked products by volatile compounds produced from nonenzymatic browning. These compounds were identified by early researchers interested in the chemistry of bread flavor. These researchers studied the oven vapors of dough during baking and the extracts of browned crusts by paper chromatography, and to some extent, gas chromatography. Unpleasant and even repulsive aromas were reported by researchers studying the volatile compounds emitted from heating model amino acid-sugar systems. The type of amino acid and sugar reacted under different heat, moisture and pH levels determines which of the many classes of volatile compounds

are formed during carbonyl-amine browning reactions. Sensory evaluation of the aroma emitted from model browning systems indicates that a description of the browned flavor developed in foods can be as elaborate and complex as the reactions from which it is produced. The carbonyl-amine condensation may follow 7 different reaction pathways.

Advanced technology concerning instrumental analysis has provided flavor chemists the ability to identify and quantitate trace volatile compounds from browned foods and model browning systems. The gas chromatograph interfaced with a mass spectrometer has become the instrument of choice because the instrument permits separation of complex mixtures and positive identification of volatile compounds. Investigations by researchers concerned with the reactants, their precursors and conditions of the browning reaction provide the necessary information to simulate or duplicate browning to obtain these flavor volatiles. Methods of using the browning reaction to produce, generate or enhance browned flavor in foods are being applied in the field of flavor technology. Natural process flavors can be generated and isolated by combining specific reactants under their optimum reaction conditions. Compounded flavors can be produced from a wide variety of materials and chemicals according to their concentration and flavor contribution as

determined by instrumental and sensory analysis. Also, precursor flavor systems can be incorporated into a product formula and allowed to release the desired flavors during heat processing.

The flavor and aroma compounds manufactured by using Maillard technology can be used to rebalance or replace flavor after heat processing or to mask undesirable and accent the preferred flavor. The overall flavor quality of a baked product depends upon the nature of flavor components produced from carbonyl-amine reactions during heat processing.

The popularity of microwave ovens does not compensate for the lack of browning in microwave baked foods. Microwave baking produces a finished product with obvious color, texture and flavor differences compared to the same products baked conventionally. The microwave oven does not provide within the product a high enough temperature to produce the surface dehydration required before browning will occur. The addition of compounds which give a browned flavor to the microwavable formula could improve the quality of these products and promote new microwave product development.

The chemical composition and concentration of volatile compounds influencing flavor and aroma attributes of bread have been extensively studied. No other baked product has

received as much attention nor were any published reports found of investigations of volatile compounds responsible for the browned flavor and aroma of cookies. Evaluation of the effects of microwave heating versus conventional heating upon the development of browned flavor compounds can provide important information for determining the differences in flavor profiles between conventionally and microwave baked cookies.

The objectives of this study were (1) to determine volatile flavor compounds in and sensory differences between conventional and microwavable cookies baked using a chocolate chip cookie base formula and (2) to reduce sensory differences between the conventional and microwavable cookies by addition of food color and flavorings.

CHAPTER 2

REVIEW OF THE LITERATURE

A. BROWNED FLAVOR

Significance. Flavor has always been a dominant factor influencing the acceptance and development of the available food supply. United States food processors recently concluded that product "taste" would continue to compel the industry to develop new food products into and beyond the 1990s (Osnabrugge, 1989). Among the most appreciated food flavors are those developed by the heat treatment of otherwise bland and even repulsive-tasting ingredients. Within this product category are baked goods such as breads, cookies and cakes that are all based on flour, mixed as necessary with sugar, eggs, milk, fat and leavening agents. The caramel, buttery and burnt sugar flavors of baked products plus the savory browned surfaces of fried and roasted meats are typical browned flavor notes. The appreciation of these flavors did not have to await advances in technology because application of heat to food has occurred for nearly 400,000 years (Mauron, 1981).

Browned flavors are categorized as "process flavors" because they are produced by conventional processes such as baking and roasting. Browned flavor components are found in a wide range of concentrations and contribute a variety of odors to flavor in foods (Hall and Merwin, 1981).

The variety of browned flavors developed during baking is primarily attributable to the large number of volatile compounds produced by nonenzymatic browning reactions involving an amino group of amino acids and an aldehyde group of aldoses. Evidence that browning in cookies is due to carbonyl groups of sugars was provided in research reported by Johnson and Miller (1961). The researchers used methylated derivatives of dextrose to replace sucrose in a cookie formula. The methyl substitution prevented browning by blocking the aldehyde group and the number 2 carbon atom of the carbonyl reactant.

Evidence implicating amino acids as browning compound precursors is abundant. In model systems, browning is more dependent upon amino acid content than on sugar content, and the rate of browning decreases faster with a decrease in amino acid content than sugar (Spark, 1969). For baked products, raw materials and processing conditions affect the chemistry of nonenzymatic browning by which nearly two-thirds of the more than 3000 volatile flavor compounds

identified in foods are formed (Heath and Reineccius, 1986). Also termed the Maillard reaction or more descriptively carbonyl-amine browning, nonenzymatic browning is the most significant chemical reaction producing a browned flavor. In addition to the production of a browned flavor in food by conventional heating, the reaction may produce an undesireable browned color or flavor which requires inhibition (Williams, 1976). Also, the use of the browning reaction as a tool for the intended production of artificial or engineered food flavors is gaining popularity (Buckholz, 1988).

Major objectives of flavor research and development are to expand usage of existing flavors by increasing acceptability of present foods or creating interesting new food products, to utilize new ingredients and to meet new consumer needs (Hall, 1986). Since browned flavor is such an important part of food flavor, a better understanding of its chemistry is needed. Knowledge of the elaborate pathways, reactants and intermediate and final products of the carbonyl-amine browning reaction was gained by researchers who studied simple model browning systems. Researchers used simple model browning systems as a tool to study the chemistry of flavor compound formation from the reaction because in heat treated foods, flavor compound formation is complex and accompanied by the presence of nonvolatile and color

contributing compounds (Hodge, 1953). Model system studies and instrumental analyses of browned food products have helped to describe the chemical composition of browned flavor. Sensory evaluation of browned food products and the aromas emitted from model systems also has shown the important contribution made by volatile flavor constituents to the odor and flavor of heat processed food. The chemical and sensory data generated have encouraged the use of single chemical compounds as additive flavorants and the development of new flavoring ingredients which impart browned odor and flavor by combining such compounds.

The Society of Flavor Chemists (1969) defined flavor as

> . . . a substance which may be a single chemical entity, or a blend of chemicals of natural or synthetic origin whose primary purpose is to provide all or part of the particular flavor effect to any food or other product taken into the mouth.

Volatile browned flavor compounds are best classified as "aromatic chemicals" according to Hall (1986) because the category "chemicals" are limited to compounds important for taste (i.e., saccharin as sweet) and those that contribute some odor but primarily mouth-feel sensations of warmth, coolness or astringency. The category "aromatic chemicals" is composed of compounds which contribute nearly 100% of the odor to the flavor of food.

Advances in browning technology are also critical to the development of microwavable food systems to provide enhanced aroma, flavor and color needed to duplicate baking by conventional methods (Buckholz, 1988). Processed foods accounted for nearly 75% of the American family food budget in 1979 according to Mabrouk indicating that the demand for convenience foods is not a new trend. However, the need for microwave-only and dual-ovenable products for consumers who demand quality convenience foods is rapidly growing (Risch, 1989). Browning flavor technology can be applied to formulation of microwavable bakery products in a number of ways to realize flavor research objectives by supplying the number and type of products consumers need and desire. Application of nonenzymatic browning reactions and methods to generate, maintain or enhance specific browned flavor formation is significant to the baked goods and baking mix industry because the quality of these products depends upon the nature of flavor components developed during baking. Baked goods and baking mixes are among 18 food categories used in a product classification by the National Academy of Sciences in which flavor has a major "characterizing" role (Hall and Merwin, 1981).

Production. Flavor compound production in baked products proceeds mainly as pure Maillard reactions between the acyclic carbonyl function of an aldehyde, a ketone or a reducing sugar (including sucrose upon hydrolysis) and the free amino group of an amino acid, amine, peptide or protein. The complete reaction can be divided into three stages: early including chemically well-defined steps, advanced leading to a multitude of reactions producing volatile and heterocyclic compounds which contribute a host of flavors and odors to food and a final stage developing insoluble, brown polymeric pigments from intermediates formed during early and advanced stages (Mauron, 1981). The advanced stage and its products are the focus of this review of the contribution of nonenzymatic browning to the flavor of baked products.

The formation of the Amadori rearrangement product in the early stage begins with the spontaneous nucleophilic addition of the amino group to the carbon-oxygen double bond. The addition product condenses to form a Schiff base which undergoes cyclization to the corresponding N-substituted glycosylamine (or ketosylamine). Glycosylamine formation is favored in alkaline pH because the less protonated amino group maximizes electron transfer (Labuza and Schmidl, 1986) and is dependent on the moisture content of food. At pH values greater than 6 and low moisture levels, browning

is accelerated with a maximum rate at 0.6-0.85a_w (Warmbier et al., 1976). The flavor of cakes, cookies and other baked goods is influenced by the pH of the dough or batter subjected to heat processing (Crocker, 1945). An elevated pH produces a "coarse-brown bread taste" in white bread while a very low pH may create a "sourish astringency" for the same baking time attributed to the familiar mild, browned flavor.

Glycosylamines, particularly those derived from amino acids, immediately isomerize to a more stable 1-deoxy-1-amino-2-ketose (or 2-amino-2-deoxyaldose) by the Amadori (or Heyns) proton transferring rearrangement (Baltes, 1982). The well-defined chemical nature of early Maillard reactions and synthesis of the Amadori product were discussed and proposed by Danehy and Pigman (1951) and the advanced and final stages by Hodge (1953).

Monitoring physical (Haugaard et al., 1951) and chemical (Wolfrom et al., 1953) aspects of the Amadori compound and its formation in model carbonyl-amine systems contributed significantly to identification of the advanced stage reaction pathways (Mauron, 1981) and conditions (Saltmarch and Labuza, 1982). From the Amadori compound, sugar dehydration, sugar fragmentation and the Strecker degradation are the three most important pathways that produce flavor-contributing, volatile compounds in baked

products. The sugar fraction of the Amadori compound undergoes dehydration and fragmentation because the incorporated nitrogen molecule reduces its stability resulting in eneaminol and enediol formation as shown by Baltes (1982).

In dehydration, 1,2-enolization ultimately yields 5-hydroxymethyl-2-furaldehyde from hexoses and furfuryl derivatives from pentoses (Heath and Reineccius, 1986). Fragmentation via 2,3-enolization yields a wider range of compounds by liberating the amino group and forming primarily methyl-alpha-dicarbonyl intermediates (Hodge, 1967). Whether enolization of the Amadori compound involves the C-1 or the C-3 carbon atom depends upon the protonated state of the amino group. Enolization of the 1,2-type (complete protonation) occurs in strong acidic conditions and the 2,3-type at a more alkaline pH corresponding to the dissociation constant (pKa) of the Amadori compound (Feather, 1982). Hodge (1967) published an excellent scheme depicting both pathways. In addition to the carbonyls, dicarbonyls, volatile acids and the liberated amino groups generated during fragmentation, reductones and dehydroreductones are formed (Heath and Reineccius, 1986). Reductones and dehydroreductones decompose further to yield cyclopentenes, pyranones, di-acetyls, furan derivatives and 2,5-dimethyl-3-2(H)-4-hydroxy-furanone (predominately from 6-deoxy sugars)

(Baltes, 1982). In both enolization pathways, the reactions which follow primary intermediate formation are complex and little understood but result in the formation of heterocyclic nitrogen compounds such as pyrroles, imidazoles and pyridines (Mauron, 1981).

In the third pathway, Strecker degradation, amino acids (including those liberated from paths one and two) are oxidatively degradated by the alpha- and other conjugated dicarbonyls produced by the breakdown of the Amadori compound and unreacted reducing sugars in paths one and two. A thorough review of Strecker degradation chemistry was published by Schonberg and Moubacher (1952). The net results of the Strecker reaction are the transamination, liberation of carbon dioxide and formation of aldehydes and new nitrogen-containing heterocyclics. The isobutyric, isovaleric and phenylacetic aldehydes produced by the Strecker degradation are major contributors to the aroma of heat processed foods (deMan, 1980). Linko et al. (1962) reviewed studies in which other Strecker carbonyls: pyruvaldehyde, acetaldehydes, crotonaldehyde, formaldehyde, 2-methylbutanal and methylglyoxal were identified as responsible for flavor and aroma in white bread.

Sulfur-containing amino acids are particularly important flavor precursors. Methionine upon entering

Strecker degradation yields methional which decomposes to yield methanethiol (methyl mercaptan) and disulfides (dimethyldisulfide). The thermal degradation of cysteine yields acetaldehyde which further reacts with acetoin from sugar fragmentation to form thiazole compounds (Lindsay, 1985).

The aroma produced by the Strecker degradation is dependent upon the amino acid reacting to give a specific aldehyde. Hodge (1967) tabulated volatile Strecker aldehydes from common alpha amino acids and their corresponding aromas as described by sensory panels.

Volatiles produced from reactions involving Strecker aldehydes, amino compounds and intermediates of both enolization pathways include a host of thiazolines, pyridines, imidazoles, oxazoles and pyrazines (Nursten, 1981). Pyrazines have been extensively studied as flavor contributors to a variety of foods. Furia and Bellanca (1975) discussed carbon and nitrogen precursors for pyrazines and listed more than 100 pyrazines identified in a variety of foods by 16 specific formation pathways associated with the browning reaction. The "olfactive" properties of pyrazines were reviewed by Masuda and Mihara (1988) and Fors and Olofsson (1985, 1986). Pyrazines are important to the quality of heat treated and toasted foods because they contribute a wide variety of odors and flavors (Maga and Sizer, 1973).

Additional compounds which have been identified in the volatile flavor components of processed foods include alcohols, ethers, ketones, esters and aromatic lactones (Mabrouk, 1979).

Application. Nonenzymatic browning reactions that produce flavor components in food can be used to synthesize process flavors. Buckholz (1988) outlined three methods of flavor creation using browning technology. First, natural process flavors can be produced by physically processing the same precursors required by carbonyl-amine browning within foods. Secondly, compounded flavors can be formulated from artificial or natural materials to reconstruct a flavor profile of a browned food. The third method involves incorporating a flavor precursor system into the matrix of a food product to produce additional flavor volatiles during processing, thus enhancing the browned flavor.

Investigations concerning the reactants, their precursors and conditions of the browning reaction provide information about the ingredients necessary to simulate or duplicate browning in order to obtain the desired flavor. Hodge (1953) demonstrated the importance of aqueous model systems for determining the nature of the volatile compounds which arise from heating combinations of amino acids and

sugars in a review of nearly 200 model systems pertaining to nonenzymatically produced browned flavor. Reynolds (1963, 1965) published a comprehensive review of the chemistry of the browning reaction which described the occurrence of specific reactants. The reactants selected by many researchers to build model systems concur with those chemical compounds described in this review. Rohan (1970) reported that sugars and amino acids are the precursors of browned flavor compounds and emphasized the inclusion of "precursor chemistry" in any study attempting to isolate flavor compounds from natural sources. Ellis (1959) reported that a variety of conditions affected the browning reaction. Lane and Nursten (1983) investigated the influence of a few of these conditions upon the odors produced from amino acid-sugar model systems. The latter investigators assessed aromatic variations caused by change in heating temperature, relative humidity, type of sugar and presence of a third ingredient, such as soluble starch.

The identity and aromatic character of the volatile compounds via the browning reaction under specific conditions must also be known before the compounds can be used in an applied situation. Much of this information is already available. Fors (1983) reported sensory descriptors for nearly 450 browning reaction products indicating not only an

assessment of their odor but also the recognition that specific browning compounds are important aroma and flavor substances in a wide variety of foods. Danehy (1986) reported a variety of aromas produced in model systems composed of each of 19 different L-amino acids with a specific sugar during heat treatment.

Although a browned flavor is the result of heat treating raw materials, it can be developed as a concentrated natural process flavor when the reactants and conditions are recreated as found in model systems, and more importantly, in normal food processing. Established principles are adequately available to support the isolation of natural process flavors from browning reactions and the use of these flavors as agents giving browned flavor notes in foods. A major advantage of these naturally developed flavorants is a perceivable mouthfeel because they are developed from a nonvolatile base with generated volatile compounds (Buckholz, 1988). Artificial flavorants are usually developed from aromatic materials and do not contribute any mouthfeel attributes to food products.

Using naturally produced browning flavors to flavor foods is very expensive. However, the flavor industry has isolated many of these flavors and often uses them to boost the freshly baked aroma and taste of bakery products. The

consumer then experiences a fresh flavor even after a baked product has been packaged and transported to the market (Osnabrugge, 1989).

Compounded flavors also are formulated in an attempt to reproduce the flavor of browned foods. These flavors may contain volatile compounds that have been identified in browned food products and/or contain volatile compounds with aromas similar to those determined in the product by sensorial analyses (Thulin and Grimm, 1960). These compounded flavors are usually artifical or nature-identical flavors containing synthetic substances or chemicals that are not always derived from natural sources. Artificial flavors have occupied an increasingly important niche in the industrial production of food since the 1950s when as part of a changing pattern of living, Americans began to use more prepared food products. The food industry accepted many of the food preparation duties and tried to meet and stimulate the demand for convenience foods with creative products. The industry soon discovered consumers would not pay for products that did not deliver satisfactory flavor. There is not an abundance of natural browning flavoring substances for flavoring foods which creates an immediate demand for synthetic flavors (Bennion, 1980).

The blending of materials to create characteristic, functional and acceptable flavorings is a highly skilled process and an art. Ingredients that individually may possess irrelevant or dissimilar characteristics to the target flavor are combined in specific concentrations to create an easily manufactured food flavorant (Hall, 1986). Sound knowledge of the physiochemical and sensory properties of foods and the variety of materials recognized as safe for use in these flavors is essential to their production.

Improved instrumental methods, particularly gas chromatography and mass spectrometry (GC/MS), have stimulated investigations into the fundamental chemistry of the browning reaction and the flavor compounds produced in foods. Studies of browned food products or model system mixtures using GC/MS ultimately lead to identification and quantification of volatile components that have developed during heat treatment and may contribute browned flavor. Concurrent sensory studies that determine the number and type of analytically identifiable compounds important to the desired flavor profile are indispensable. Few, if any,foods have a single flavor component sensorially predominant (Heath and Reineccius, 1986).

Identification of volatile compounds responsible for bread flavor and aroma has received considerable attention.

Forty aromatic compounds were identified in the bread making process (pre-ferment, dough, oven vapor and bread) between 1930 and 1961 (Wiseblatt, 1961). Linko and Johnson (1963) investigated the reactions within bread doughs baked with various carbohydrates and free amino acids which produce volatile aldehydes. The researchers used a less sophisticated separation and identification technique (paper chromatography) than GC/MS to determine a significant relationship between concentrations of furfural, hydroxymethylfurfural and a browned crust. In comprehensive reviews, Hodge (1967) and Coffman (1967) summarized the accomplishments of early researchers who attempted to elucidate bread flavor and aroma.

Folkes and Gramshaw (1981) reported a unique example of information GC/MS and sensory techniques can provide about the nature of browned flavors. These researchers identified volatile compounds from white bread crust extracts and evaluated their odors by sniffing the gas chromatographic effluent. A list of browning compounds positively identified and sensorially validated as important contributors to the browned flavor of white bread crust was established. This list provides a menu for flavor chemists who select chemicals and materials to produce compounded flavors. Use of the compound(s) on such a list to flavor other foods validates the importance of the compounds to food flavor.

Methylhydroxy furanone, on the list of Folkes and Gramshaw (1981), has been identified by Osnabrugge (1989) as a compound capable of contributing a caramelized (browned) flavor to foods. Another compound, dimethylhydroxy furanone, mentioned by Osnabrugge (1989) also contributes the same flavor to foods.

An abundance of information from GC/MS and sensory analyses of flavors has been collected by flavor companies but has not been published. An artificial cake crust flavor (Anon, 1989), probably developed from such information, recently has been introduced to the market. The flavorant was designed to deliver browned flavor notes to microwavable bakery products (Fritzsche Dodge & Olcott^{TM}). Information that provides new flavor compounds or new ideas for known compounds and materials to simulate or replace desirable flavors in foods is essential to the production of compounded food flavors.

A third method to improve or impart browned flavor to foods is the use of flavor enhancers. This method uses a "precursor flavor system" included in the formulation of a food to supplement flavor compound formation during heat processing. Identifying such food flavor precursors, however, is complex because the possible number of flavor components produced per precursor is large. Theoretically, this

number may be as high as 150, but usually only 40 or fewer flavor compounds are isolated per precursor (Mabrouk, 1979).

The precursors of bread flavor have been studied since the isolation of maltol and isomaltol in 1910 (Coffman, 1967). Danehy (1986) reviewed reports of investigations in which the effects of adding different sugars and amino acids to the dough was studied on bread aroma. They reported that an important precursor of bread aroma constituents was L-proline. The ability to alter or enhance bread aroma and flavor by the intended addition of amino acids and/or sugars to the formula has been known for some time (Danehy, 1986). Linko and Johnson (1963) reported studies demonstrating that leucine, when added to an instant bread mix, enhanced bread aroma. Leucine and xylose added to bread dough by Linko et al. (1962) improved bread flavor by increasing the amount of isovaleraldehyde developed in the crust during baking.

The reactants required in nonenzymatic browning have been studied extensively since Hodge (1953) reported the browning reaction theories. The occurrence of the Amadori rearrangement product and production of flavor and color compounds from its thermal degradation are well accepted (Williams, 1976; deMan, 1980). Also, the significant role of carbohydrates and their thermal degradation products in the development of browned flavor is well defined (McWeeny,

1973; Houminer, 1973). The Amadori rearrangement product is an essential browned aroma precursor (Hodge and Rist, 1953; Mills, 1979) that produces different intensities of browning usually proportional to the browning ability by the parent sugar (Hashiba, 1982). Doornbos and coworkers (1981) synthesized Amadori compounds from 6-deoxy sugars, heated them under various reaction conditions and concluded that they specifically generated at least 8 different classes of compounds important to the flavor of browned foods. Feather (1982) reported finding many of the same furanone derivatives from browning model systems that Doornbos et al. (1981) found. However, Feather (1982) crystallized most of the compounds from different Amadori compounds. Hashiba et al. (1981) synthesized 18 Amadori products, tasted them and concluded that their flavor was similar to, yet milder than, the taste of each constituent amino acid.

Investigations concerning browning compound precursors must also include studies of single sugars. Walter and Fagerson (1968) utilized gas chromatographic and mass spectrometric analyses to identify 11 of the 100 compounds they isolated from heated glucose. Pomeranz et al. (1962) studied model sugar-amine systems and determined that pentoses, in the order of ribose>xylose>arabinose were the most reactive sugars in promoting a brown color and flavor.

Researchers have concluded that sugars, amino acids and heat are the minimum requirements to initiate the browning reaction. The character of the browned flavor formed in a food is dependent, in part, on the amino acids and sugars present in the food before baking. Researchers can alter, enhance or add browned flavor notes to the finished product by addition of specific amino acids and/or sugars during product formulation. The application of such precursors is feasible and acceptable because these products naturally contain amino acids (Bushuk, 1986) and carbohydrates, such as sugars or starches, as sweeteners.

Problems associated with the use of "precursor flavor systems" are the lack of available natural precursor materials (most are very unstable), high cost and determination of the physical parameters required by the precursor to generate the desired flavor compound (Buckholz, 1988). Often the chemicals or materials added as flavor precursors to product formulations are so dispersed they never meet, and therefore, the desired reaction never occurs. This is a primary concern for microwavable food formulas because the heating time and intensity is limited.

Several inventions to limit the problems with "precursor flavor systems" have recently been reported (Osnabrugge, 1989). "Dynamic flavors" are precursor systems rich in

Amadori rearrangement products that will break down more quickly than reducing sugars and amino acids can react and at lower temperatures. Encapsulation of flavor precursors such as the Amadori rearrangement products might also help solve some of the problems (Dziezak, 1988). The inclusion of flavor precursors in the formulation of microwave-baked products, however, will not contribute positively to the browned flavor or color of the product unless the precursors react at or just below 100°.

B. BROWNED FLAVOR ANALYSIS

Headspace technique: isolation and concentration. Foods possess flavor attributes beyond the basic taste of component ingredients whenever an odor is emitted from the food surface or an aroma is perceived during consumption. Determination of the odorous compounds in the vapor phase above food is of considerable interest because it represents the mixture of chemical stimulants sensorially perceived as aroma. Headspace analysis of food volatiles has consequently become a valuable and popular method in flavor research because the volatile compounds cause the aromas we perceive during consumption and thus determine the quality of the food flavor. The task is to isolate from the vapors above

food a sample of volatile components concentrated enough to allow their individual separation, measurement and identification (Teranishi et al., 1971). Isolation of the volatiles may be accomplished directly or indirectly depending upon how they are collected.

Direct headspace technique involves equilibration of a food sample in a closed container at a given temperature or time and then sampling the headspace volatiles by drawing the vapor into a syringe. The problems with this simplistic approach are that trace volatiles cannot be detected by instrumental analysis and only those volatiles present at concentrations exceeding 10^{-5}g/L (headspace) can be adequately identified using mass spectrometry (Heath and Reineccius, 1986). Land (1985) reported that direct headspace techniques were limited during the early 1970s to highly volatile flavor compounds that could be analyzed by direct syringe injection onto the packed column of a gas chromatograph. Instrumental analysis of the drawn vapors often could not be performed with maximum sensitivity because large quantities of water were present in the isolate. Direct headspace technique is a rapid and efficient method for analyzing only the most abundant volatiles present in a food sample since concentration of many of the volatiles is limited (Heath and Reineccius, 1986).

Analysis of abundant and trace volatile compounds can be achieved by indirect headspace techniques. In this method, the equilibrated vapors above a food placed in a closed container are purged with an inert gas and the quantities of volatiles present are concentrated by trapping them on adsorbent materials (Heath and Reineccius, 1986). By 1978, porous polymer beads were developed and are now widely used adsorbents in purge and trap systems. The synthetic polymer beads are popular adsorbents because they limit co-condensation of water with the odorous volatiles present in foods (Land, 1985). Tenax GC™ (Akzo Research Laboratories) has become the adsorbent of choice. Tenax GC™ has a high affinity for a wide range of organic compounds, permits excellent recovery of adsorbed volatiles, is relatively hydrophobic and yields a characteristic flavor isolate by aroma judgements (Heath and Reineccius, 1986).

Breakthrough of very volatile components may occur during the use of porous polymer adsorbents in purge and trap systems. Breakthrough is usually a result of adsorbent saturation but may be related to the relatively large volume of sweeping gas needed to displace the odorous volatiles onto the trap. The volume of gas is large when compared to the amount of headspace vapor emitted from food and its use requires that the trap is vented to the air. Adsorbents may

also retain compounds during heat desorption or cause artifact formation. Artifact formation occurs because the adsorbent reacts with the volatiles upon contact or because it provides a surface for reactions among volatiles isolated (Charpentier et al., 1985). Tenax GC™, however, has been shown not to form artifacts during heat desorption (Heath and Reineccius, 1986).

Volatile flavor and aroma components in food usually are present from parts per billion (ppb) to parts per million (ppm). Emberger (1985) reported a concentration range of 6 to 10 ppm for the total flavor compounds in bread. The vapor pressure of each volatile affects the concentration isolated. The actual headspace concentration of volatile flavor compounds above food depends upon Henry's law which states the partial pressure of a solute above a solution is proportional to its molar concentration in the liquid (Nawar, 1971). The relationship between the headspace concentration of a volatile compound and actual concentration of that compound in the food matrix also depends upon the extent of interactions between the component and the nonvolatile macroconstituents in food (Solms, 1986). Nawar (1971) studied the effect of sucrose on the volatility of carbonyls and determined that the headspace volatility of heptanone and heptanal decreased by two-thirds in a 60% sucrose

solution when compared to 100% water. The sample temperature is also a factor which influences headspace composition and concentration because the volatility of flavor and odor compounds is based upon their respective boiling points.

One published study in which the purge and trap technique was used to isolate and concentrate browned flavor compounds present in baked goods was found. Gilbert and Hatzidimitrio (1986) used helium to purge the odorous compounds present in the headspace vapor above cookies onto Tenax GC. The trapped volatiles were prepared for GC/MS analysis by heat desorption (200°C) onto the gas chromatograph column. The researchers investigated the quantitative changes in nonenzymatic browning reaction products in the cookies caused by different temperature and packaging conditions during storage.

Isolation and concentration of flavor volatiles found in the headspace vapor of baked products is more meaningful than a total volatile compound analysis. Total volatile analysis of foods usually requires multistage distillations or solvent extractions. Headspace methods are more meaningful because they do not require intense heat which browns the sample further. Headspace methods also limit sample manipulation, which reduces artifact formation, and omit the need for solvents. The flavor attributes of the product are

better preserved, and therefore, headspace volatile analyses are more representative of freshly baked quality.

Gas chromatography and mass spectrometry: separation and identification. Adsorption of aroma volatiles onto porous solids has become an established procedure for preparing food samples for flavor analysis by gas chromatography and mass spectrometry. The GC/MS is an ideal instrument for food flavor analysis. The GC/MS coupling permits efficient separation of the minute quantities of isolated volatiles and structural identification of those compounds after molecular fragmentation by moderate energy electrons. Capillary gas chromatography is the most frequently used separation method for complex mixtures of food flavor and odor components. Highly refined instruments equipped with diverse injection techniques, fused silica glass columns and sensitive yet versatile ionization detectors have integrated separating power with reliability (Land, 1985).

Chemists interested in volatile food components during the early 1950s were among the first researchers to recognize the relatively simple correlation between mass spectra and organic compound structure (Issenberg, 1969). The sampling requirements of the mass spectrometer are compatible with the fast release of effluent from the gas

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chromatograph. Any loss of volatile material caused by the GC/MS interface must be avoided because at that point very small amounts of single substances are involved. Mass spectral data provide the mass weights of the molecule, ions from the molecule and their relative abundances. A standard procedure for interpreting these data was published by McLafferty (1967), and GC/MS manufacturers often include compound qualification and quantitation techniques.

Buckholz (1988) reported that 27 studies were published during the years between 1953 and 1982 which referenced the volatile compounds that investigators had found in white bread. The majority of these studies identified volatiles in bread pre-ferments and doughs using column and paper chromatography. Many researchers interested in browning compounds evaluated aqueous condensates of oven gases evolved during bread baking. The composition of the condensates was usually identified by gas-liquid chromatography (Coffman, 1967). Most of the reported studies concerned with identifying browned flavor volatiles have used white bread extracts and have determined those browned flavor constituents that are abundant or the easiest to obtain. GC/MS identification techniques could yield not only abundant but also trace volatile component elucidation. Folkes and Gramshaw (1981) published a unique study in which they

identified browned flavor volatiles from white bread crust extracts. The researchers identified over 190 volatile compounds using a three-channel combination of sulfur, nitrogen and flame ionization GC detectors in tandem with a mass spectrometer. Although many browning compounds have been identified, the present challenge of browned flavor analysis is to determine those compounds sensorially predominant in and responsible for the flavor of baked goods.

Sensory evaluation: validation. Whatever may be the chemical identity and concentration of volatile food components, conclusions cannot be drawn about flavor without considering human perceptions of food quality. Flavor researchers must establish a relationship between chemical composition of flavor volatiles and their sensory importance to the food flavor. The contribution of specific volatiles to food flavor is affected by the food matrix. Also, the flavor response results from the aroma and taste of food taken into the mouth with all senses contributing to its appreciation (Hall, 1968).

Objective flavor profiles are most frequently determined by nasal appraisal of the effluent from gas chromatographs equipped with sniff ports (Chang, 1973).

Fractions of the isolated volatiles that possess an odor characteristic of the food receive special attention during chromatogram interpretation. Folkes and Gramshaw (1981) used this technique to attribute bread-like odors to 12 areas of the total GC/MS chromatogram from which they identified 190 volatiles in white bread crust extract.

Once a volatile compound has been associated with the characteristic odor of food, its effect in the food requires evaluation. Hodge and Moser (1961) spiked bread and pastry formulas with compounds that had been previously isolated in trace amounts from baked cereals and breads. Sensory panelists were asked to describe odor and taste characteristics and state whether any difference in taste existed between the control and treated samples. The panelists also were asked their preference among the products. The flavor of yeast rolls spiked with maltol and isomaltol was described as similiar to yet stronger than that of the unspiked fresh rolls. The panelists described the flavor contributed by maltol and isomaltol as caramel-like and sometimes fruity. Pie crusts that contained galactosylisomaltol were preferred by tasters to the unspiked control. The compound was reported to have a bitter taste but in some pastries it splits into isomaltol and galactose by the action of heat and moisture which changes its taste.

Lorenz et al. (1973) determined that microwave-baked, whole wheat and rye breads were less preferred by sensory panelists than were conventionally-baked, whole wheat and rye breads. Gilbert and Hatzidimitrio (1986) investigated the effect storage had upon the headspace volatile composition of packaged cookies using GC/MS. The researchers employed a sensory panel to score the flavor of the cookies from "very poor, objectionable" to "excellent, pleasant" on a scale from one to five.

Sensory evaluations that include descriptors and measure differences of the flavor attributes are important tools for validation of the existence of chemical compounds in foods which contribute odor and taste. Unfortunately, knowledge of the relationship between the structure of a molecule and its perceived flavor is not great enough to predict which organic compounds and mixtures are useful as flavorants. Identification of character impact compounds that contribute browned flavor notes yet remain a mystery can be expected as other baked products in addition to breads are studied. Isolation, identification and validation of key compounds imparting the complex browned flavor may not be possible using methods currently available. These relatively unstable compounds are present in extremely

low concentrations and are generated by many heat induced reactions that are not well defined.

C. MICROWAVE BAKING

The problem. Microwave-baked cookies differ significantly in browned flavor quality from conventionally baked cookies because two principal factors responsible for browning, high temperature and relatively long baking time, are absent in microwave baking. The temperature in a microwave oven during baking is rarely above ambient temperature, and the surface temperature of foods rarely exceeds the boiling point of water. Because of this and the penetration ability of microwaves, evaporative cooling occurs at the food surface creating a negative temperature gradient. The temperature inside the food is higher than that of the food surface (Decareau, 1973). Conventionally baked cookies are prepared in a relatively high temperature environment which results in surface dehydration (Steinke et al., 1989) and slow, even heating.

For carbonyl-amine browning reactions to proceed, the surface of the food must be dry, the surrounding oven air cannot be saturated with moisture (Risch, 1989), and a rapid decrease of temperature at increasing product depth must

exist (Keefer, 1986). These conditions do not occur in microwave baking because the radiant energy of microwaves differs from that of infrared radiation in wavelength, frequency and distribution (Van Zante, 1973). The short microwave baking times and oscillating nature of the radiant energy heats the product unevenly and spontaneously. Nonenzymatic browning reactants do not receive the required activation energies (heat) to produce browning.

Microwave baking does not rely upon convection to heat food. Instead, microwaves penetrate deeply into food materials and produce heat by energy transfer to a dipolar material. Microwaves are attracted in differing degrees to water, fat and sugar molecules causing them to vibrate and create friction which develops the heating effect. The most common dipole in food is water which influences microwave absorption and penetration depth (Ohlsson, 1983). The amount of moisture in food has a direct effect on its rate of heating in a microwave oven and on the rate of the carbonyl-amine browning reaction. Browning reactions occur at a maximum rate in intermediate moisture foods (30-60%) and at a decreasing rate with increasing water content because the reactants are diluted (Eichner and Karel, 1972). A soft cookie contains moisture in the formula at the intermediate

level, but low temperatures aggravated by accelerated moisture loss during microwave heating prevents browning.

The cookie. The cookie has become a popular snack food since the Dutch introduced it to America during early settlements (Mariani, 1983). One of the most popular cookies today, the chocolate chip cookie, originated in the kitchen of Ruth Wakefield's Toll House Inn during the 1930s. The present day chocolate chip cookie entrepreneur, Wally Amos of Famous Amos™ cookies, reported the Ruth Wakefield story in a book (Amos and Robinson, 1983) documentating the success of his own chocolate chip cookie. Ruth cut chunks of Nestle's™ semisweet chocolate into "an old recipe". The chunks did not melt during baking and Ruth called the baked product a chocolate crunch cookie.

A soft cookie dough that is properly prepared so that it is easily manageable will produce cookies with acceptable appearance and flavor when baked in a conventional oven. Cookie dough baked by microwave energy should be slightly stiffer with more flour in proportion to fat than cookie dough intended for conventional baking. Also, cookies with high fat levels do not microwave well because the fat melts over the surface of the dough causing uneven heating. To assure uniform heating, drop cookies should not be

refrigerated or contain nuts or raisins before baking (Lorenz et al., 1973). Attempts to overcome the lack of browned color in microwave-baked chocolate chip cookies are expected to be successful because of the availability of caramel coloring. Compensation for the lack of browned flavor developement, however, requires information describing the differences in volatile flavor compounds between a cookie formulated for the microwave and one intended to be conventionally baked.

The objective of this study was to investigate the differences in flavor, both sensorially and chemically, between conventionally baked microwave-baked cookies. A secondary objective was to evaluate the flavor of a microwave-baked cookie containing flavor and color additives commercially available.

CHAPTER 3

MATERIALS AND METHODS

A. EXPERIMENTAL PLAN

Four variations of cookies were prepared. Cookies were baked from conventional (CON) and microwave (MW) cookie formulas.

Conventional formula cookies were conventionally baked (CON CON) and the microwave formula cookies were microwave-baked with (MW MW ADD) and without added flavor and color (MW MW) and conventionally baked (MW CON). A single replication consisted of the 4 cookies baked without vanilla flavoring and chocolate chips, and two replications were completed. A balanced paired-comparison sensory test was used to determine if a difference existed between selected pairs of cookie treatments. A consumer sensory panel evaluated each cookie baked with vanilla flavoring and chocolate chips on an 8-point hedonic scale. Each cookie treatment-replication combination was also analyzed for headspace volatile compounds. Headspace volatile compounds from combined flavor and color additives equivalent to that added to the MW MW ADD cookies were also analyzed.

B. COOKIE PRODUCTION

Formulas and mixing. The formulas (Table 1) used were a modified, conventional Toll House™ chocolate chip cookie (ANON, 1986) and an experimental cookie based upon conventional chocolate chip cookie ingredient ratios that were altered to produce a microwave-baked cookie with acceptable texture and spread (Hanger, 1988). The conventional Toll House™ cookie formula was modified by substituting shortening for butter. The conventional and microwave-baked cookies used for volatile analyses and paired-comparison sensory evaluations did not contain vanilla flavoring or chocolate chips in order to prevent their interference with flavor evaluations. However, cookie formulas included the vanilla and chips when baked for the consumer sensory panel because these flavorings are normally present.

The shortening, egg and nonfat dry milk were stored in a refrigerator (4°C), and all other ingredients were stored at room temperature before weighing. The doughs were blended in a Kitchen-Aid™ mixer (Model K45SS) using a multistage mixing technique that is widely used for drop-type cookies (Matz, 1972).

The conventional and microwave formulas had different mixing methods and speeds. The conventional cookie dough

Table 1--Cookie formulations

Ingredients	Bakers percent	
	Conventional	Microwave
Flour[a]	100	100
Sugar[b]	56.3	15.1
Brown sugar[c]	60.5	45.5
Shortening[d]	68.9	50.5
Baking soda[e]	1.5	1.0
Salt[f]	1.9	1.0
Fresh whole eggs[g]	36.4	32.3
Distilled water	12.6	23.2
Mono- and di-glycerides[h]	----	1.0
Nonfat dry milk[i]	----	3.0
Cream of tartar[j]	----	0.5
Pre-gelatinized starch[k]	----	1.0
Caramel color[l]	----	2.5
Artificial cake crust flavor[m]	----	6.0
Vanilla flavoring[n]	1.8	1.8
Chocolate chips[o]	57.5	50.5

[a]All-purpose, White Lily Foods Co., Knoxville, TN.
[b]Domino's Extra Fine Granulated, Amstar Corp, New York, NY.
[c]Kroger Light, Cincinnati, OH.
[d]Super Quick-Blend, Beatrice/Wesson, Fullerton, CA.
[e]Arm & Hammer, Princeton, NJ.
[f]Noniodized, Kroger, Cincinnati, OH.
[g]Kroger Large
[h]Dur-em 117 Beads, Durkee Industrial Foods Corp., Louisville, KY.
[i]Classic Extra Grade, Sysco, Houston, TX.
[j]McCormick & Co. Inc., Baltimore, MD.
[k]Tendergel 434 Instant, A.E. Staley Mfg. Co., Decatur, IL.
[l]RT #220, Sethness Products Co., Clinton, IA.
[m]Aromalok, Fritzsche Dodge & Olcott™, New York, NY.
[n]Kroger, Cincinnati, OH.
[o]Nestle Foods Corp., Purchase, NY.

was prepared by blending the shortening and the sugars at speed 3 for 1 min. The bowl was scraped with a plastic scraper, egg and water were added, and the mixture blended at speed 4 for 30 sec. Vanilla flavoring was added with the egg in cookies baked for the consumer sensory panel. The bowl was scraped, and the dry ingredients were added and blended at speed 2 for 30 sec and then speed 4 for 15 sec. The bowl was scraped again, and the mixture was blended at speed 4 for 15 sec. For cookies for the consumer sensory panel, chocolate chips then were added and mixed for 15 sec at stir speed.

The microwave cookie dough was prepared by blending shortening, sugars, mono- and diglycerides, cream of tartar and soda at speed 3 for 1 min. The artificial flavor and caramel color were added with the shortening for the microwave cookie with added flavor and color that was microwave-baked. The bowl was scraped and the mixture blended at speed 4 for 30 sec. A suspension of nonfat dry milk and 24 ml of the water was added and the mixture blended at speed 3 for 45 sec. The bowl was scraped, eggs were added and the mixture was blended at speed 3 for 45 sec. Vanilla flavoring was added with the egg to dough baked for the consumer sensory panel. Flour and starch were added, and the mixture blended at speed 2 for 30 sec. The bowl was scraped, the

remaining water added and the mixture blended at speed 2 for 30 sec. Chocolate chips then were added and mixed for 15 sec at stir speed in cookies baked for the consumer sensory panel.

Baking and storage. The conventional cookie dough was dropped by level tablespoons for the paired-comparison sensory evaluations and volatile analyses or teaspoons for the consumer sensory panel onto a 38.7 X 26.0 X 0.6-cm baking sheet. The cookies were baked for 10 min in a rotary Despatch oven preheated to 375°F. The cookies were allowed to cool on the pans placed on wire racks for 4 to 6 min.

The microwave cookie dough was dropped by level tablespoons for the paired-comparison sensory evaluations and volatile analyses or teaspoons for the consumer sensory panel onto a wax-coated paper plate. The plate was placed upon a Microware™ (Anchor Hocking, Mansfield, OH) rack and baked in a White-Westinghouse countertop microwave oven (Model KM540G and 750 watts). Total baking time was 2 min on a setting of 7 within a range of settings between 1 and 9 with a half rotation of the plate at 1 min. Five tablespoon dropped or 10 teaspoon dropped cookies were baked per plate and 1 plate was baked at a time. The baked cookies were left on the paper plate and placed upon a wire rack to cool

for 6 to 8 min. The microwave cookie dough also was baked conventionally as described previously for the conventional cookie dough.

For the paired-comparison sensory evaluations and volatile analyses, 200 g of cookies each were placed into 469-ml, wide-mouth mason jars (Ball Corp., Muncie, IN), flushed with nitrogen and closed with dome lids. The jars were wrapped with foil to prevent light-induced fat oxidation and then stored frozen (-18°C) for no more than 14 days before evaluation. Cookies for the consumer sensory panel were placed upon a wire rack before being packaged individually into reclosable plastic sandwich bags. The sealed bags were stored in a covered plastic container at ambient temperature for no more than 2 days before evaluation.

C. SENSORY EVALUATION

Paired-comparison tests. Twenty-four panelists (14 females and 10 males) consisting of students, faculty and staff at The University of Tennessee were blindfolded and asked to determine if a difference in flavor existed between cookie treatments that were presented as pairs. Samples were ground to minimize textural differences. The blindfolds were used as a method to mask the color differences

between samples. Two tests were conducted. Test 1 consisted of all cookies except for the microwave cookie with added flavor and color and baked in the microwave oven (MW MW ADD). Test 2 consisted of all cookies except the conventional cookie baked conventionally (CON CON).

Cookies were taken from the freezer approximately 20 min prior to grinding with a food processor (Shortcut™ model CFP10, Black and Decker, Shelton, CT) equipped with a serrated chopping blade. The 2-cup bowl was filled with cookies and processed for 20 sec. A 2-g cookie sample was immediately weighed into a plastic cup covered with a plastic lid and evaluated within 8 hr.

Panelists were asked to obtain a portion of the two samples from the cup with their tongue, taste the samples and respond yes or no to the question "Is there a difference in flavor between the two samples?". Whenever the panelist responded "yes", they were asked to describe that difference (Appendix A). Every sample in each test and replication was balanced so that each sample was paired with every other sample an equal number of times. The order of presentation was balanced for each pair across panelists. Each panelist received 3 pairs and rinsed with water between each pair. Each panelist was oriented with a practice session.

The results of the session were not analyzed.

Results from each replication within each test were pooled and analyzed as a two-tailed (p=1/2) test (Larmond, 1977) for the minimum number of responses required for significance at P<.05 (Roessler et al., 1978). Descriptions of flavor differences between members of pairs were summarized.

Consumer panel. Ninety-two panelists participated in a consumer panel that was conducted in the sensory evaluation laboratory (The University of Tennesse, Knoxville) to determine how they liked each of the 4 cookies. Panel demographics were obtained with a questionnaire indicating age, gender, ethnic orientation and including frequency of eating cookies and chocolate chip cookies (Appendix B).

Panelists were presented a sample of each cookie in a recloseable plastic sandwich bag, one at a time, for evaluation. Cookies baked for this evaluation were made with vanilla flavoring and chocolate chips. A scorecard with instructions and an 8-point hedonic scale where 8 = like extremely and 1 = dislike extremely (Appendix C) was presented with each sample. The panelists were instructed to rinse their mouths with water between samples and complete the questionnaire after the fourth sample. Samples were coded

with 3-digit random numbers. Order of sample presentation was balanced across panelists.

Results were coded and analyzed by ANOVA using a General Linear Model to test for differences in the hedonic score attributable to the main effects cookie treatment, age, gender, ethnic group and frequency of consumption of cookies and of chocolate chip cookies (SAS Institute Inc., 1985). All 2- and 3-way interactions were nonsignificant ($P>.05$), and therefore, dropped from the model.

D. HEADSPACE VOLATILE ANALYSIS

Headspace volatiles were isolated from a 200-g sample of each cookie replication combination and from an aqueous dispersion of the combined artificial cake crust flavor and caramel color additives. Before each isolation, however, a blank was run.

Volatile isolation. Volatiles were isolated using a 1000-ml jacketed purge and trap Wheaton apparatus (No. 9917800) purchased from Alltech, Inc., Deerfield, IL. This apparatus was equipped with a gas diffusion tube and a thermometer adapter.

Prior to isolation of any volatiles, the apparatus was thoroughly cleaned. All glassware were washed with chlorinated soap and hot water and rinsed successively with the following: deionized water, acetone, deionized water, 4% acetic acid solution, deionized water and Chromar^{TM} HPLC purified water (Mallinckrodt, Paris, KY). The bowl and blade of the food processor used to prepare the cookie samples for volatile isolation were washed with chlorinated soap and water and rinsed with deionized water then with Chromar^{TM} HPLC purified water.

Prior to the isolation of any volatiles, a precolumn for the collection was prepared and conditioned in the following manner. A 108 mm long X 4-mm ID glass precolumn was packed with 125 mg Tenax GC, 60/80 mesh (Alltech, Inc., Deerfield, IL) and heated under a helium flow of 50 ml min^{-1} in a 220°C gas chromatograph oven for 15 hr.

At the start of volatile isolation, a gas diffusion tube, the inlet of which was attached to a Tygon tube was inserted into the Wheaton apparatus. The apparatus was closed and brought to temperature by circulating 50°C water through the water jacket. After conditioning, the precolumn was attached to the thermometer adapter tube on the Wheaton apparatus and 400 ml min^{-1} of purified nitrogen via the diffusion tube was passed through the apparatus into the

precolumn. The nitrogen was purified by passing through a hydrocarbon trap No. 14633 (Alltech, Deerfield, IL) before entering the diffusion tube. Nitrogen was circulated for 30 min, the precolumn was removed and any volatiles collected on the precolumn were heat desorbed and analyzed by GC-MS as described later. The resulting GC-MS chromatogram was the analysis of the blank.

While the blank was being collected and analyzed, a 200-g sample of a cookie that was stored in a sealed, 469-ml Mason jar was removed from the freezer approximately 20 min prior to preparation for volatile analysis. The cookie sample was ground in the food processor equipped with a serrated chopping blade for 20 sec. The sample was placed into the 50°C Wheaton apparatus that had been used to collect the blank and the nitrogen diffusion tube was set approximately 1.25 cm above the sample surface. The Wheaton apparatus was sealed and allowed to equilibrate at 50°C for 30 min. After the precolumn had been desorbed and analyzed for the blank, it was reconnected to the Wheaton apparatus containing the sample. A 400 ml min^{-1} flow of the purified nitrogen was passed over the sample and through the precolumn. The volatiles from the sample were purged and trapped on the Tenax GC precolumn for 30 min. Volatiles were analyzed within 30 min of isolation.

The artificial flavor and color sample was prepared for volatile analysis in the following manner. The artificial flavor (1.47 g) and color (0.6 g) additives were blended with 500 ml of the HPLC purified water for 5 min in a Osterizer commercial blender. After a blank had been collected as previously described, the aqueous solution was poured into the Wheaton apparatus and stirred with a magnetic stir bar controlled by a Nuova II stirrer (Dubuque, IA) at a setting of 6. After the sample had equilibrated to 50°C the diffusion tube directing the purified nitrogen was submerged approximately 1.25 cm under the solution surface. The Tenax GC precolumn that had just been analyzed for the blank as previously described was attached to the apparatus, and the volatiles were isolated from the flavor and color sample for 30 min with a purified nitrogen flow of 400 ml min^{-1}. The amount of flavor and color additives analyzed corresponded to the amount added to a 200-g sample of cookies.

Volatile analyses. A QP-1000 GC/MS interfaced with a GC-9A gas chromatograph and a SPL-7 moving precolumn injector (Shimadzu Corp., Kyoto, Japan) was used to desorb and analyze volatiles trapped on the Tenax GC precolumn including blanks, cookies and the flavor and color additives. The blank traps were desorbed and analyzed for the presence of

volatile compounds arising from the isolation apparatus, other glassware used in the procedure or from the precolumn Tenax GC trap.

The precolumn containing volatiles was attached to the SPL-7 moving precolumn injector holder. An internal standard (1 mg of methyl palmitate) was added to the precolumn through the syringe injector port and allowed to penetrate for 30 sec. The injector was lowered into the GC oven and headspace volatiles were desorbed at 250°C for 10 min onto a 30 m by 0.25 mm ID fused silica SP-2330 capillary column (Supelco Inc., Bellafonte, PA) held at 0°C. The GC-MS analysis was begun at the time desorption started in order to establish a consistent starting time for all samples and blanks. At the end of desorption, the GC temperature program was raised from 0°C to 25°C at 8°C/min; held at 25°C for 5 min; increased to 50°C at 3°C/min; and then raised to a final temperature of 220°C at 5°C/min. GC-MS parameters for the analysis were as follows: ion source temperature 220°C, ion source energy 70 ev, separator temperature 250°C, injector temperature 250°C, He carrier gas flow rate 1.5 ml min^{-1} and a gain set at 4.

The concentration of each headspace volatile was quantified in milligram equivalents of methyl palmitate/200 g cookies by dividing the corrected (peak height minus back-

ground) volatile peak area (measured as total ion intensity) by the corrected peak area of methyl palmitate. The relative response ratio of each headspace volatile to methyl palmitate was assumed to by 1.00. The identity of each headspace volatile was tentatively determined by matching its mass spectrum with that of a known volatile compound via the library search system LSS-20 on the GC-MS computer. Positive identification of each headspace volatile was determined by matching the retention time and mass spectrum of an unknown volatile with those of a known standard volatile analyzed under exactly the same conditions as the samples.

Each headspace volatile was analyzed by ANOVA using a General Linear Model to test for differences in concentrations attributable to the main effect cookie treatment. The mean concentrations of cookie volatiles were analyzed using Student Newman-Keuls test for significant differences in concentrations of each volatile found in more than one cookie treatment (SAS Institute Inc., 1985). The mean concentrations of cookie volatiles for conventional formula conventionally baked and the microwave formula conventionally baked and microwave-baked with and without added flavor and color were compared orthogonally for differences in concentration attributable to different pairs of cookie

treatments. The mean concentrations of cookie volatiles from two different combinations of cookies were averaged and also compared orthogonaly for differences in mean concentration.

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CHAPTER 4

RESULTS AND DISCUSSION

A. SENSORY EVALUATION

Paired-comparison tests. The numbers of panelists who indicated a difference in flavor between cookies of varying treatments are shown in Table 2. In test 1 the flavor of conventionally formulated cookies baked in a conventional oven (CON CON) differed from that of the cookies from the microwave formula baked in the microwave oven (MW MW). They (CON CON) also differed from microwave formula cookies baked in a conventional oven (MW CON).

In test 2 microwave-formulated and -baked cookies with flavor and color additives (MW MW ADD) differed in flavor from the cookies from the microwave formula without additives regardless of the baking method. Baking method used did not effect differences in flavor of microwave-formulated cookies in either test 1 or test 2.

The differences in flavor between the conventional cookie and the microwave cookie baked either in microwave or conventional ovens were primarily attributable to differences in sweetness. Most of the panelists (63%) in test 1

Table 2--Number of panelists indicating a flavor difference between cookies from paired-comparison sensory evaluations

Cookie pair[a]	Yes response[b]
Test 1 (N=48)	
CON CON/MW MW	44[b]
CON CON/MW CON	40[b]
MW CON/MW MW	17
Test 2 (N=47)	
MW MW ADD/MW CON	35[b]
MW MW ADD/MW MW	33[b]
MW CON/MW MW	23

[a]CON CON=conventional cookie formula baked conventionally; MW MW= microwave cookie formula microwave-baked; MW CON=microwave cookie formula baked conventionally; MW MW ADD=microwave cookie formula microwave-baked with added flavor and color.

[b]Indicates significant difference in flavor between samples $P<.05$ (Roessler et al., 1978).

who detected a difference between the conventional cookie and the microwave cookie microwave-baked (CON CON/MW MW) said the flavor difference was in the degree of sweetness with the CON CON cookie being sweeter. Seventy-five percent of the panelists in the same test who found a difference between the conventional cookie and the microwave cookie both conventionally baked (CON CON/MW CON) also attributed the flavor difference to a degree in sweetness. The conventional cookie formula was also described as "burnt", "buttery", "more flavorful" and vanilla-like".

Since the main difference between the conventional and the microwave cookie formulas was the level of sugar (Table 1, p. 40) and paired-comparison test results indicated a difference in perceived sweetness between the cookies, efforts were made to increase the sweetness of the microwave cookie without additional sugar. Artificial cake crust flavor and caramel color were added to the microwave cookie and another paired-comparison test was completed (test 2) as shown in Table 2. The addition of flavor and color to the microwave cookie did not seem to significantly increase the perceived sweetness. Only 9% of the panelists in test 2 who detected a flavor difference between the microwave cookie with and without added flavor and color (MW MW ADD/MW CON and MW MW ADD/MW MW) reported that degree of sweetness was

responsible. A variety of other descriptors were used by these panelists to describe the flavor difference between microwave cookies with and without added flavor and color. Descriptors for the cookie with added flavor and color included "burnt sugar", "overbaked" and "burnt marshmallows". The cookie from the microwave formula without added flavor and color was described as "bland", "flavorless" and "flour-like".

Consumer panel. Although panelists could detect a difference between cookies produced from conventional and microwave formulas, the degree of desirability needed to be established for each cookie treatment. Cookies tested in the paired-comparison tests were evaluated by a consumer panel. The results of this test are given in Table 3. Panelists liked the conventional cookie formula baked in the conventional oven (CON CON) best of all cookies followed by the cookie produced by the microwave formula baked in the conventional oven (MW CON). The difference between the mean scores of these two cookies was significant. Panelists liked the CON CON cookie very much, and liked the MW CON cookie slightly to moderately. Panelists liked the microwave cookies, with and without added flavor and color, baked in the microwave oven (MW MW ADD and MW MW) least of

Table 3--Mean hedonic scores of cookies

Cookie[a]	Mean score[bc]
CON CON	6.9 ± 0.2 A
MW CON	5.5 ± 0.2 B
MW MW ADD	4.8 ± 0.2 C
MW MW	4.6 ± 0.2 C

[a]CON CON=conventional cookie formula conventionally baked; MW CON= microwave cookie formula conventionally baked; MW MW ADD=microwave cookie formula microwave-baked with added flavor and color; MW MW= microwave cookie formula microwave-baked.

[b]Least-squares means (N=92) in a column followed by different letters are significantly different $P<.05$.

[c]8=like extremely, 7=like very much, 6=like moderately, 5=like slightly, 4=dislike slightly, 3=dislike moderately, 2=dislike very much and 1=dislike extremely.

all cookies ($P < .05$). Panelists slightly disliked to slightly liked the MW MW ADD and MW MW cookies.

The mean hedonic scores of cookies analyzed as a function of panelist age and gender are shown in Table 4. The mean hedonic score across all age and gender groups indicates that all cookies were liked slightly to moderately. Panelists, age 45 and over, preferred the cookies ($P < .05$) more than panelists, age 35 to 44, but not more ($P < .05$) than panelists younger than 35. Panelists less than age 35 preferred ($P < .05$) the cookies as well as all age groups. Females, however, liked ($P < .05$) the cookies less than males.

The mean hedonic scores of cookies analyzed as a function of frequency of eating chocolate chip cookies are shown in Table 5. Panelists who reported they ate chocolate chip cookies every day to several times a week liked ($P < .05$) the cookies more than those who ate chocolate chip cookies several times a month or a year. The former panelists liked the cookies moderately. There was no difference ($P < .05$) in hedonic scores as assigned by panelists who ate chocolate chip cookies several times a month and those who ate them several times a year. These panelists liked the cookies slightly.

Table 4--Mean hedonic scores of cookies by panelist age and gender groupings

Group	Mean hedonic score[ab]
Age	
45 and over (N=11)	5.7 ± 0.2 A
35-44 (N=11)	5.4 ± 0.2 B
25-34 (N=32)	5.6 ± 0.2 AB
Less than 18-24 (N=38)	5.1 ± 0.2 AB
Gender	
Males (N=57)	5.7 ± 0.1 A
Females (N=35)	5.2 ± 0.2 B

[a]Least-squares means represent values for 4 cookies per panelist; means in a column within age or gender followed by different letters are significantly different P<.05.

[b]8=like extremely, 7=like very much, 6=like moderately, 5=like slightly, 4=dislike slightyl, 3=dislike moderately, 2=dislike very much and 1=dislike extremely.

Table 5--Mean hedonic scores of cookies by frequency of eating chocolate chip cookies

Frequency	Mean hedonic score[a]
Every day to several times a week (N=18)	6.0 ± 0.03 A
Several times a month (N=48)	5.4 ± 0.02 B
Several times a year (N=25)	5.0 ± 0.02 B

[a]Least-squares means (N=91) followed by different letters are significantly different P<.05.

[b]8=like extremely, 7=like very much, 6=like moderately, 5=like slightly, 4=dislike slightly, 3=dislike moderately, 2=dislike very much and 1=dislike extremely.

B. HEADSPACE VOLATILE ANALYSES

The results of the analyses of variance for concentrations of cookie volatile compounds analyzed as a function of cookie treatment are shown in Tables 6-8. The treatment sums of squares for Peak 4, ethyl benzene, Peak 13, 2-acetyl pyridine and methyl furfural were significant at $P < .10$ when the cookie treatments were considered as a group. However, when the cookie treatment sums of squares were partitioned by orthogonal comparisons, significant differences ($P < .10$) between specific treatments were found for additional volatile compounds: isovaleraldehyde, diacetyl, 2,3-pentadione, 2-ethyl toluene, Peak 9, Peak 10, Peak 12, Peak 15, benzaldehyde, Peak 19, Peak 20, 5-methyl furfural and benzyl alcohol. In addition to the differences significant at the $P < .10$ level, differences between specific treatments at $.10 > P < .15$ were found for Peak 2, Peak 6, 2-ethyl toluene, Peak 12, Peak 16, furfural, Peak 20 and p-cresyl acetate. Considering only 8 observations were statistically analyzed, this range in level of significance shows trends that probably would have a higher level of significance if more samples had been analyzed.

The mean concentration of each cookie volatile compound for each treatment is given in Table 9. The CON CON cookie

Table 6--Sums of squares from analyses of variance for concentrations of volatiles isolated from more than one cookie treatment and for orthogonol comparisons of cookie treatments--Part 1

Source	Degrees of freedom	Isovaler-aldehyde	Peak 2	Diacetyl	Peak 4	2-3-Penta-dione	Peak 6	Ethyl benzene	2-Ethyl toluene
Treatment	3	.4698	.1812	1.1670	.2014**	.1003	.0452	.0034**	.0021
CC-MMA[a]	1	.3501*	.1338	.5794*	.1816**	.0752**	.0333	.0000	.0000
MC-MM	1	.0233	.0062	.0033	.0001	.0098	.0072	.0023**	.0014*
CC/MMA-MC/MM	1	.0964	.0412	.5842*	.0197	.0153	.0048	.0011*	.0007
Error	4	.2433	.1280	.5140	.0412	.0416	.0395	.0006	.0008
Total	7	.7131	.3092	1.6809	.2426	.1419	.0847	.0040	.0029

[a]CC=conventional cookie formula baked conventionally; MMA=microwave cookie formula microwave-baked with added color and flavor; MC=microwave cookie formula baked conventionally; MM=microwave cookie formula microwave-baked.

*P<.10
**P<.05

Table 7--Sums of squares from analyses of variance for concentrations of volatiles isolated from more than one cookie treatment and for orthogonol comparisons of cookie treatments--Part 2

Source	Degrees of freedom	Peak 9	Peak 10	Peak 11	Peak 12	Acetol	Acetoin	Peak 15	Peak 16
Treatment	3	.0133	.0205	.0023	.0101	2.7626*	.0095	.0322	.0040
CC-MMA[a]	1	.0090*	.0151**	.0014	.0000	1.8946**	.0060	.0230*	.0005
MC-MM	1	.0038	.0032	.0009	.0067*	.0038	.0033	.0070	.0030
CC/MMA-MC/MM	1	.0006	.0022	.0000	.0034	.8642*	.0002	.0023	.0005
Error	4	.0072	.0078	.0046	.0039	.6368	.0126	.0192	.0033
Total	7	.0205	.0284	.0069	.0140	3.3999	.0222	.0515	.0073

[a]CC=conventional cookie formula baked conventionally; MMA=microwave cookie formula microwave-baked with added color and flavor; MC=microwave cookie formula baked conventionally; MM=microwave cookie formula microwave-baked.

*P<.10
**P<.05

Table 8--Sums of squares from analyses of variance for concentrations of volatiles isolated from more than one cookie treatment and for orthogonol comparisons of cookie treatments--Part 3

Source	Degrees of freedom	Furfural	Benzal-dehyde	Peak 19	Peak 20	2-Acetyl pyridine	Methyl furfural	p-Cresyl acetate	Benzyl alcohol
Treatment	3	7.0360	.0186	.0190	.0932	.0683**	.0912	.4470*	4.1830
CC-MMA[a]	1	4.0929	.0159**	.0143*	.0621*	.0473**	.0088	.2980**	2.7886*
MC-MM	1	.0248	.0002	.0016	.0000	.0002	.0745*	.0000	.0000
CC/MMA-MC/MM	1	2.9183	.0025	.0031	.0311	.0208**	.0079	.1490	1.3943
Error	4	4.5186	.0131	.0099	.0375	.0058	.0675	.1452	2.3023
Total	7	11.5546	.0318	.0289	.1307	.0742	.1587	.5923	6.4854

[a]CC=conventional cookie formula baked conventionally; MMA=microwave cookie formula microwave-baked with added color and flavor; MC=microwave cookie formula baked conventionally; MM=microwave cookie formula microwave-baked.

*P<.10
**P<.05

Table 9--Mean concentrations (μg equivalents methyl palmitate/200 g cookies) of volatile compounds isolated from more than one cookie treatment

Compound	Mean retention time (min)[a]	Cookie treatment[b]			
		CON CON	MW MW ADD	MW CON	MW MW
Isovaleraldehyde[e]	13.4	.592A[c]	.000B[c]	.153	.000
Peak 2	14.4	.366	.000	.079	.000
Diacetyl	15.2	.950A	.189B	.058	.000
Peak 4	16.5	.427A	.000B	.110	.118
2,3-pentadione	19.8	.274A	.000B	.099	.000
Peak 6	20.9	.182	.000	.085	.000
Ethyl benzene[e]	22.4	.000	.000	.047X[d]	.000Y[d]
2-ethyl toluene[e]	24.5	.000	.000	.038X	.000Y
Peak 9	25.2	.095A	.000B	.061	.000
Peak 10	27.4	.123A	.000B	.056	.000
Peak 11	28.6	.038	.000	.030	.000
Peak 12	28.8	.000	.000	.000X	.082Y
Acetol[e]	29.9	1.376A	.000B	.062	.000
Acetoin	30.3	.000	.078	.058	.000
Peak 15	32.2	.152A	.000B	.083	.000
Peak 16	33.4	.022	.000	.055	.000
Furfural	35.0	.275	2.298	.157	.000
Benzaldehyde	36.7	.000A	.126B	.035	.020
Peak 19	37.2	.119A	.000B	.041	.000
Peak 20	37.6	.000A	.249B	.000	.000
2-acetyl pyridine	38.1	.000A	.218B	.014	.000
Methyl furfural	39.1	.121	.027	.273X	.000Y
p-cresyl acetate[e]	42.5	.000A	.546B	.000	.000
Benzyl alcohol	44.7	.000A	1.670B	.000	.000

[a]GC-MS analyses.

[b]CON CON=conventional cookie formula conventionally baked; MW CON= microwave cookie formula conventionally baked; MW MW ADD=microwave cookie formula microwave-baked with added flavor and color; MW MW= microwave cookie formula microwave-baked.

[c]Means(N=2) for CON CON and MW MW ADD cookies followed by an A or a B are significantly different (P<.10).

[d]Means (N=2) for MW CON and MW MW cookies followed by an X or a Y are significantly different (p<.10).

[e]Tentative identification.

had a significantly higher level of isovaleraldehyde, diacetyl, Peak 4, 2,3-pentadione, Peak 9, Peak 10, acetol, Peak 15 and Peak 19 and a significantly lower level of benzaldehyde, Peak 20, 2-acetyl pyridine, p-cresyl acetate and benzyl alcohol than did the MW MW ADD cookie ($P < .10$). Although not significant at the $P < .10$ level, differences at $.10 > P < .15$ were found between the CON CON cookie and the MW MW ADD cookie for Peak 2, Peak 6 and furfural.

The MW CON cookie had a significantly higher level of ethyl benzene, 2-ethyl toluene and 5-methyl furfural and a significantly lower level of Peak 12 than did the MW MW cookie ($P < .10$). The MW CON cookie also tended ($.10 > P < .15$) to have a higher level of Peak 16 than the MW MW cookie.

The mean concentration of each cookie volatile compound averaged across CON CON plus MW MW ADD and MW CON plus MW MW is given in Table 10. The CON CON and MW MW ADD cookie had significantly higher concentrations of diacetyl, acetol and 2-acetyl pyridine and significantly lower concentrations of ethyl benzene than the MW CON plus MW MW cookie combination ($P < .10$). The CON CON plus MW MW ADD cookie combination also tended ($.10 > P < .15$) to have higher concentrations of Peak 20 and p-cresyl acetate and lower concentrations of Peak 8 and Peak 12 than the MW CON plus MW MW cookie combination. Concentrations and retention times of the headspace

Table 10--Mean concentrations (μg equivalents methyl palmitate/200 g cookies) of volatile compounds isolated from averaged orthogonal comparisons one and two

Compound	Mean retention time (min)[a]	Comparison[b]	
		One	Two
Isovaleraldehyde[d]	13.4	.296	.076
Peak 2	14.4	.183	.039
Diacetyl	15.2	.569A[c]	.029B[c]
Peak 4	16.5	.213	.114
2,3-pentadione[d]	19.8	.137	.049
Peak 6	20.9	.091	.042
Ethyl benzene[d]	22.4	.000A	.024B
2-ethyl toluene[d]	24.5	.000	.019
Peak 9	25.2	.047	.031
Peak 10	27.4	.062	.028
Peak 11	28.6	.019	.015
Peak 12	28.8	.000	.041
Acetol[d]	29.9	.688A	.031B
Acetoin	30.3	.039	.029
Peak 15	32.2	.076	.042
Peak 16	33.4	.011	.027
Furfural	35.0	1.287	.079
Benzaldehyde	36.7	.063	.028
Peak 19	37.2	.060	.020
Peak 20	37.6	.125	.000
2-acetyl pyridine	38.1	.109A	.007B
Methyl furfural[d]	39.1	.074	.136
p-cresyl acetate[d]	42.5	.273	.000
Benzyl alcohol	44.7	.835	.000

[a]GC-MS analyses.

[b]One=conventional cookie formula baked conventionally + microwave cookie formula microwave-baked with added flavor and color/2.

Two=microwave cookie formula baked conventionally + microwave cookie formula microwave-baked/2.

[c]Means (N=4) in a row followed by an A or a B are significantly different (P<.10).

[d]Tentative identification.

volatile compounds isolated and analyzed from an aqueous solution the artificial cake crust flavor and caramel color additives are given in Table 11.

The unidentified compounds shown in Tables 9 and 10 either were not identified by library search of private or EPA-NIH Mass Spectra Libraries or the compound identity given by the library search was not confirmed by actual GC-MS analysis of the standard compounds. In addition, the mass spectrum obtained was from low resolution mass spectroscopy, and the molecular formula of the unknown compounds could not be deduced from relative isotope abundance (McLafferty, 1967). Therefore, only the types of compounds that are compatible with the spectrum of each unknown volatile are discussed.

Compounds represented by Peaks 2, 6, 9, 10, 11, 15, 16 and 19 were found in measurable amounts only in cookies that were baked conventionally (Table 9). This indicates that these compound were most likely formed by the nonenzymatic browning reaction. Why only the MW MW cookie contained Peak 12 compound is not known at the present time. The Peak 20 compound that was present only in the MW MW ADD cookie (Table 9) was in added flavor and color (Table 10).

The mass spectra for compounds represented by Peaks 2, 4, 6, 9, 10, 11, 12, 15, 16, 19 and 20 are shown in Appendices D-1 through D-11, respectively. Peak 2 compound had a

Table 11--Concentrations of volatile compounds isolated from artificial cake crust flavor and caramel color

Compound	Mean retention time (min)[a]	µg equivalents methyl palmitate per equivalent flavor and color added to 200 g cookies
Diacetyl	15.3	.566
Furfural	35.5	4.178
Benzaldehyde	37.0	2.335
Peak 20	37.9	.727
2-acetyl pyridine	38.1	.561
p-cresyl acetate[b]	42.8	7.909
Benzyl alcohol	44.5	.904

[a]GC-MS analyses.

[b]Tentative identification.

base peak (most abundant fragment ion) of mass weight (m/e) 41 (Appendix D-2). Possible identity of this m/e is $C_3H_5^+$ (Williams and Fleming, 1980), and it can be formed from alkyls, alkenes, primary aliphatic alcohols and aliphatic nitriles (Beynon et al., 1968). These compounds can be formed by NEB.

Peak 4 compound had a base peak at m/e 43 (Appendix D-3) which can have the chemical composition of $CH3CO^+$ or $C3H7^+$ (Williams and Fleming, 1980). The highest m/e of the fragment ion in the spectrum of Peak 4 was 86 which may indicate that the compound is a propyl ketone unbranched on the alpha carbon (Beynon et al., 1968). The whole spectrum of Peak 4 is compatible with that of an alkyl ketone. In contrast to Peak 4, Peak 6 had a base peak at m/e 68 (Appendix D-4) with possible identities of cycloalkane, cycloalkene or an alkyl nitrile (McLafferty, 1967).

Peak 9 compound had a spectrum with a base peak at m/e 105 (Appendix D-4) which is indicative of alkyl benzenes or aromatic alcohols (Beynon et al., 1968). The spectrum of Peak 10 compound (Appendix D-5) also shows presence of an aromatic ring with the fragment ions at m/e of 91 and 105 (Beynon et al., 1968). The rest of the spectrum for Peak 10, however, is not consistent with an alkyl benzene. The base peak of the spectrum at m/e 42 in combination with the

fragment ion at m/e 55 are compatible with cycloalkanes or alkenes (Beynon et al., 1968). Because of the complexity of the Peak 10 spectrum it is probable that more than one compound eluted within the peak.

The spectra for compounds in Peaks 11, 15 and 19 have base peaks at m/e 43 (Appendices D-6, D-7 and D-9, respectively). Close examination of the spectra, however, reveals that Peak 15 spectrum is dissimilar to the other two spectra. In addition to that at m/e 43, the spectra for Peaks 11 and 19 have several common fragment ions, those at m/e of 39, 40, 41, 42, 56, 84, 96 and 106. The relative abundance of each m/e ion in the two spectra is similar also. The base peak at 43 m/e is common to several compounds: alkanes, methyl ketones, acetates, vinyl alkyl ethers, cyclic ethers, aliphatic nitriles and isothiocyanates. However, the m/e of the other fragment ions and their relative abundance are not characteristic of any of these compounds. In contrast, the spectrum of the Peak 15 compound is compatible with an alkane (McLafferty, 1967).

The spectrum for the compound in Peak 12 (Appendix D-7) has a base peak at m/e 44. The chemical formula for m/e 44 can be $C_2H_6N^+$, CO_2^+, $C_3H_8^+$, $C_2H_3O^+$ or NH_2CO^+. The ion of m/e 44 is formed from aliphatic amines, primary amides, aldehydes, ethers or alcohols (Beynon et al., 1968).

The m/e 57 base peak of Peak 16 compound (Appendix D-9) is either $C_2H_5CO^+$ or $C_4H_9^+$ (Williams and Fleming, 1980), and may fragment from cycloalkanes, cycloalkenes or cycloalkanols (Beynon et al., 1968). The spectrum of the compound responsible for Peak 20 (Appendix D-11) has a base peak at 81, which in combination with the presence of the ion at m/e 53, is compatible with the spectra of acetylenes and dienes (Beynon et al., 1968).

Isovaleraldehyde, diacetyl, 2,3-pentanedione, acetol, acetoin, furfural, 2-acetyl pyridine, benzaldehyde and methyl furfural identified in the cookie volatiles (Table 9, p. 66), have been reported as volatiles formed from monenzymatic browning reactions (Heath and Reineccius, 1986). Furfural and 2-methyl furfural are formed by dehydration of sugar molecules. Diacetyl, 2,3-pentanedione, acetol and acetoin are formed by fission of reductones and dehydroreductones. Benzaldehyde can be formed from phenylalanine through the Strecker degradation reaction, and isovaleraldehyde can be formed from leucine through the same reaction. Pyridines, such as 2-acetyl pyridine, also are formed via the browning reaction, but the mechanisms of their formation are poorly characterized.

The aromatic compounds such as ethyl benzene and 2-ethyl toluene which were also found in the cookie volatiles

(Table 9, p. 66) may be derived from carotenoid pigments found in the shortening and wheat flour (Table 1, p. 40). Trans-beta-carotene has been shown to fragment into this type of products when heated at a high temperature (Fennema, 1985).

P-cresyl acetate and benzyl alcohol were only found in the cookies made from the microwave formula with added flavor and color (Table 9). These two compounds were not formed in measurable amounts in other cookies, but were identified and measured in the flavor and color sample (Table 11, p. 70).

The flavor resulting from the browning reaction is not due to any single volatile compound but to several compounds (Heath and Reineccius, 1985). The concentration of each volatile in relation to the others also plays an important role in determining the overall flavor quality of any browned food.

The odor and/or flavor of several of the volatile compounds identified in the cookies (Table 9) and formed from the browning reaction have been characterized in this study. Diacetyl and 2,3-pentanedione are reported to be oily and butter-like with 2,3-pentanedione being less sharp than diacetyl (Coffman, 1967; Fors, 1983). Acetoin was reported to be flavorless by Heath and Reineccius (1985),

but Furia and Bellanca (1971) reported that it had a pleasant butter-like odor and flavor. Furfural has been reported to have a fragrant, aromatic, woody, resinous and vanilla-like aroma (Dravnieks, 1985) compared to a sweet, spicy, caramel-like and warm odor for 5-methyl furfural (Furia and Bellancia, 1971). It would seem, therefore, that the characteristic flavor notes of each of these compounds are pleasant.

In contrast, if smelled alone, the aroma of isovaleraldehyde found in the CON CON and the MW CON cookie (Table 9) is not pleasant. Dravnieks (1985) reported that the odor of isovaleraldehyde was sickening, putrid, foul, decayed, rancid, sweaty, sharp and pungent. The possibility exists, however, that this volatile compound, in the right concentration and combination with other browning volatiles contributed to a desirable browning flavor. It was not present in the cookies made from the microwave formula and microwave-baked, but it was present in the cookies made from the conventional or microwave formulas and baked conventionally (Table 9). Consumers liked the conventionally baked cookies made from the conventional or microwave formulas significantly more than either of the microwave-baked cookies (Table 3, p. 57).

Addition of the flavor and color to the MW MW cookie

resulted in greater concentrations of 4 identified volatile compounds than was present in the CON CON or the MW CON cookie (Table 9). Two of the compounds, p-cresyl acetate and benzyl alcohol, were not found in the CON CON or the MW CON cookie. The aroma and/or flavor characteristics of each of these compounds have been reported. Benzaldehyde is almond flavor, but Dravnieks (1985) reported that it also has fragrant, aromatic, honey and vanilla aroma notes. Benzyl alcohol has a characteristic pleasant, slightly pungent, fruity odor that tends to become similar to the aroma of benzaldehyde as it ages (Furia and Bellancia, 1971). The aroma of p-cresyl acetate is fragrant and aromatic with sweet, vanilla-like characteristics, but it also has a strong anise or licorice aroma note (Dravnieks, 1985). In contrast to the sweet, aromatic aromas of these compounds, the aroma of 2-acetyl pyridine is almost bland. Fors (1983) reported that 2-acetyl pyridine was like popcorn with bready, cracker-like notes. Although the artifical flavor and color added to the microwave cookie contained these volatiles (Table 11), its addition to the MW MW cookie did not significantly improve the flavor of the cookie from a consumer standpoint (Table 3).

The results of this study show that more research is needed to produce a microwave-baked cookie with a flavor as

well liked as that of a conventionally baked cookie. The only commercial flavoring that was found for adding browned flavor notes to microwave-baked, sweet products failed to improve the flavor likeability (Table 3) of the cookie made from a microwave formula and then baked in a microwave oven. Baking the microwave cookie formula in a conventional oven, however, significantly improved the cookie likeability (Table 3,).

In the present study, the presence of the flavor volatiles, isovaleraldehyde, 2,3-pentanedione, acetol and nine unidentified compounds, in conventionally baked cookies compared with the microwave-baked cookies, undoubtedly contributed to a more desirable cookie flavor (Table 9). In contrast, the less desirable flavor in the microwave-baked cookies may be due to the lack of volatiles in the cookie without added flavor and color and to higher concentrations of benzaldehyde, 2-acetyl pyridine, p-cresyl acetate and benzyl alcohol in the microwave cookie with added flavor and color (Table 9).

CHAPTER 5

SUMMARY

In this study, the flavor of four different cookies was investigated by sensory and chemical analyses. Cookies were made from two different formulas, a conventional chocolate chip cookie base (CON) and a cookie formula optimized for spread and texture during microwave baking (MW). The cookies from the CON and MW formula were both baked in a conventional oven (CON CON and MW CON, respectively), and the cookies from the MW formula were baked in the microwave oven (MW MW). Also, artificial cake crust flavor and caramel color additives (ADD) were added to the MW formula before baking in the microwave oven (MW MW ADD). Two paired-comparison tests using selected pairs of cookies were conducted in which the panelists were blindfolded and asked if there were a flavor difference and to describe any detectable difference. Test 1 consisted of the following pairs of cookies: CON CON /MW MW, CON CON/MW CON and MW CON/MW MW and test 2, MW MW ADD/CON CON, MW MW ADD/MW MW and MW CON/MW MW. Each of the four cookies was also analyzed for acceptability (8-point scale where 8 = like extremely and 1 = dislike extremely) by a consumer panel (n=92).

In addition, headspace volatile compounds from 200-g samples of each of the four cookies (replicated twice) and an equivalent amount of the coloring and flavoring added to the MW MW ADD cookies were trapped in a Tenax GC precolumn and analyzed by GC-MS. An internal standard, methyl palmitate, in concentrations of 1 mg per 200 g cookies was added to each sample prior to volatile trapping. The concentration of each volatile isolated was calculated in mg equivalents of methyl palmitate/200 g cookies. In addition, every effort was made to identify as many of the volatiles as possible.

In paired-comparison test 1 panelists could detect a flavor difference ($P < .05$) between the CON CON cookies and the MW CON or the MW MW cookies (Table 2, p. 55). The difference in flavor was attributable to the difference in perceived sweetness.

To increase the perceived sweetness and simulate the browned flavor in the CON CON cookies, artificial cake crust flavor and caramel color were added to the microwave cookie formula before baking in the microwave oven, and test 2 was done. The results of this test indicated a flavor difference ($P < .05$) between the MW MW ADD cookies and the MW MW or MW CON cookies (Table 2). As in test 1, however,

panelists did not detect a flavor difference between MW CON and MW MW cookies ($P < .05$).

Although panelists did not detect a flavor difference between the MW CON and MW MW cookies, consumer panelists liked the MW CON cookies better ($P < .05$) than the MW MW cookies or the MW MW ADD cookies (Table 3, p. 57). The consumer panelists, however, liked ($P < .05$) the CON CON cookies best of all. These results show that ADD failed to improve the flavor of the MW MW cookies as much as baking the MW cookies in a conventional oven (MW CON).

Consumer panelists, age 45 and over, preferred the cookies ($P < .05$) more than panelists, age 35 to 44, but not more ($P < .05$) than panelists younger than 35 (Table 4, p. 59). Panelists less than age 35 preferred ($P < .05$) the cookies slightly to moderately as did all age groups. Females, however, liked ($P < .05$) the cookies less than males (Table 4). Panelists who reported they ate chocolate chip cookies every day to several times a week liked ($P < .05$) the cookies more than those who ate chocolate chip cookies several times a month or a year (Table 5, p. 60).

A total of 24 volatiles was found in the headspace of the cookies, 13 of which have been identified at the present time (Table 9, p. 66). Seven of these volatiles were isolated from ADD, 5 were identified (Table 11 p. 70). Five of

the volatiles from ADD were found only in the MW MW ADD cookies (Table 9). Two of the volatiles found in ADD, diacetyl and furfural, also were formed in cookies that were conventionally baked (CON CON and MW CON). Four of the volatiles from ADDS, benzaldehyde, 2-acetyl pyridine, p-cresyl acetate and benzyl alcohol, were found in higher concentrations ($P < .10$) in the MW MW ADD cookies than in the CON CON cookies (Table 9). With the exception of 2-acetyl pyridine, the other three volatiles are known to impart sweet and fragrant flavor notes. The compound, 2-acetyl pyridine, imparts a bland flavor note.

The CON CON cookies had the highest number (15) of measurable volatiles of all cookies and had higher concentrations ($P < .05$) of isovaleraldehyde, diacetyl, 2,3-pentanedione, acetol and 5 unidentified volatiles than did the MW MW ADD cookies (Table 9). The MW CON cookies also had higher levels of ethyl benzene, 2-ethyl toluene and methyl furfural than the MW MW cookies while the MW MW cookies only had a higher concentration of one unknown volatile than the MW CON (Table 9). The volatiles present in the CON CON cookies were formed, most likely, through the nonenzymatic browning reaction, and their presence contributed to the CON CON cookies being the most desirable to consumers.

The previously listed compounds in the CON CON cookies were also present in measurable amounts in the MW CON cookies but not in the MW MW or MW MW ADD cookies (Table 9). This fact combined with the higher levels of methyl furfural in the MW CON cookies compared with the MW MW cookies may be the reason that the MW CON cookies also were liked better than the MW MW or MW MW ADD cookies.

The failure of ADD (commercially available additives for baked products to replace browning flavor and color in baked products) to improve the likeability of the MW MW cookies shows that more research is neccessary to provide a desirable flavoring for microwave baked cookies. Results of the present study show that the presence of isovaleraldehyde, diacetyl, 2,3-pentanedione, acetol, acetoin, and methyl furfural in combination with five other unidentified volatiles in specific concentration in the baked cookies needs to be considered in developing this flavoring.

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APPENDICES

APPENDIX A

PAIRED-DIFFERENCE SENSORY EVALUATION

RESPONSE RECORDING SHEET

SCORECARD

BLINDFOLD
FIRST SAMPLE IN RIGHT HAND

DATE ____________
REPLICATION ____________

IS THERE A DIFFERENCE?
DESCRIBE DIFFERENCE

PANELIST #	PAIR	DIFFERENCE	DESCRIPTION
____	____	____	____________
	____	____	____________
	____	____	____________
____	____	____	____________
	____	____	____________
	____	____	____________
____	____	____	____________
	____	____	____________
	____	____	____________
____	____	____	____________
	____	____	____________
	____	____	____________
____	____	____	____________
	____	____	____________
	____	____	____________
____	____	____	____________
	____	____	____________
	____	____	____________

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APPENDIX B

CONSUMER SENSORY PANEL

DEMOGRAPHIC AND FREQUENCY QUESTIONNAIRE

1. Male _____ Female _____

2. Age _____less than 18
   _____ 18-24
   _____ 25-34
   _____ 35-44
   _____ 45-54
   _____ 55-64
   _____ 65 and over

3. How would you best describe yourself?
   _____Black _____White _____Asian
   _____ American Indian _____ Hispanic _____Other

4. How often do you eat cookies?
   _____every day
   _____several times a week
   _____several times a month
   _____several times a year
   _____never

5. How often do you eat chocolate chip cookies?
   _____every day
   _____several times a week
   _____several times a month
   _____several times a year
   _____never

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APPENDIX C

CONSUMER SENSORY PANEL

HEDONIC SCORECARD

1. Please indicate how well you like or dislike each sample by checking the term that best describes your feeling toward that sample. Rinse your mouth with water before testing the next sample.

Like extremely	_____
Like very much	_____
Like moderately	_____
Like slightly	_____
Dislike slightly	_____
Dislike moderately	_____
Dislike very much	_____
Dislike extremely	_____

COMMENTS:

2. When completed with sample, return scorecard with paper plate and close hatch toward you.

3. After your fourth sample you will receive a short questionaire to complete.

Thank you.

APPENDIX D

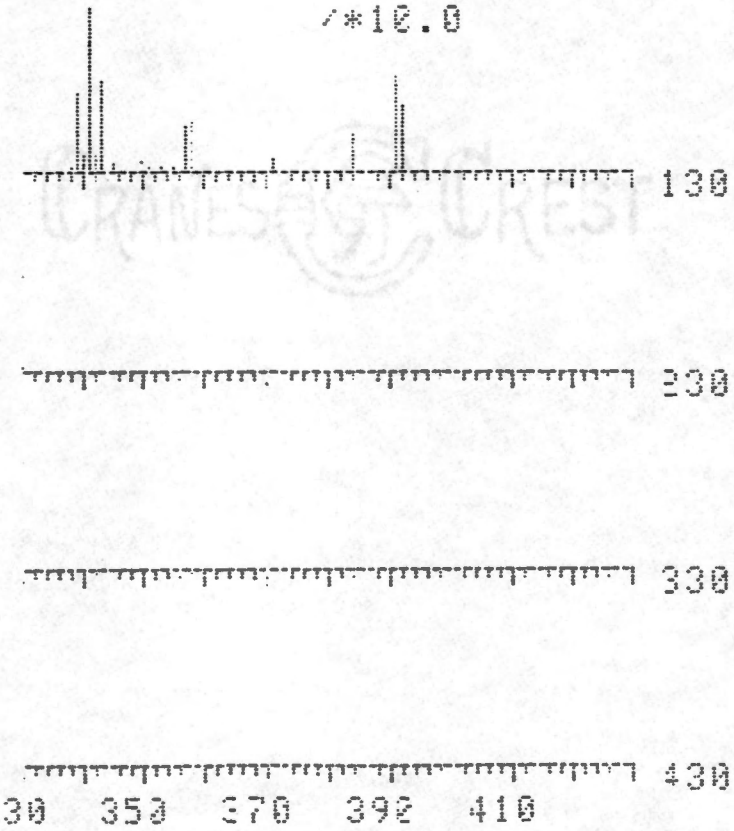
HEADSPACE VOLATILE ANALYSES

UNIDENTIFIED VOLATILES

Peak 2

FILENAME:RICKE .31
COMMENT:BGS INPUT:RICKE .01

DATA	R.T.	PEAK	BASE	PEAK (*100)	TOTAL (*100)
1	14.3	177	465(41)		1754
		27	465(41)		1641

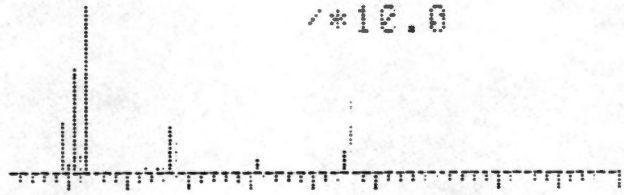


CRANES CREST

Peak 4

```
FILENAME:RICKE  .08
COMMENT:BGS   INPUT:RICKE  .01
                        (*100)      (*100)
DATA   R.T.  PEAK    BASE  PEAK    TOTAL
   1   16.2   173    521(   43)    1595
              17    521(   43)    1495

                    /*10.0
```



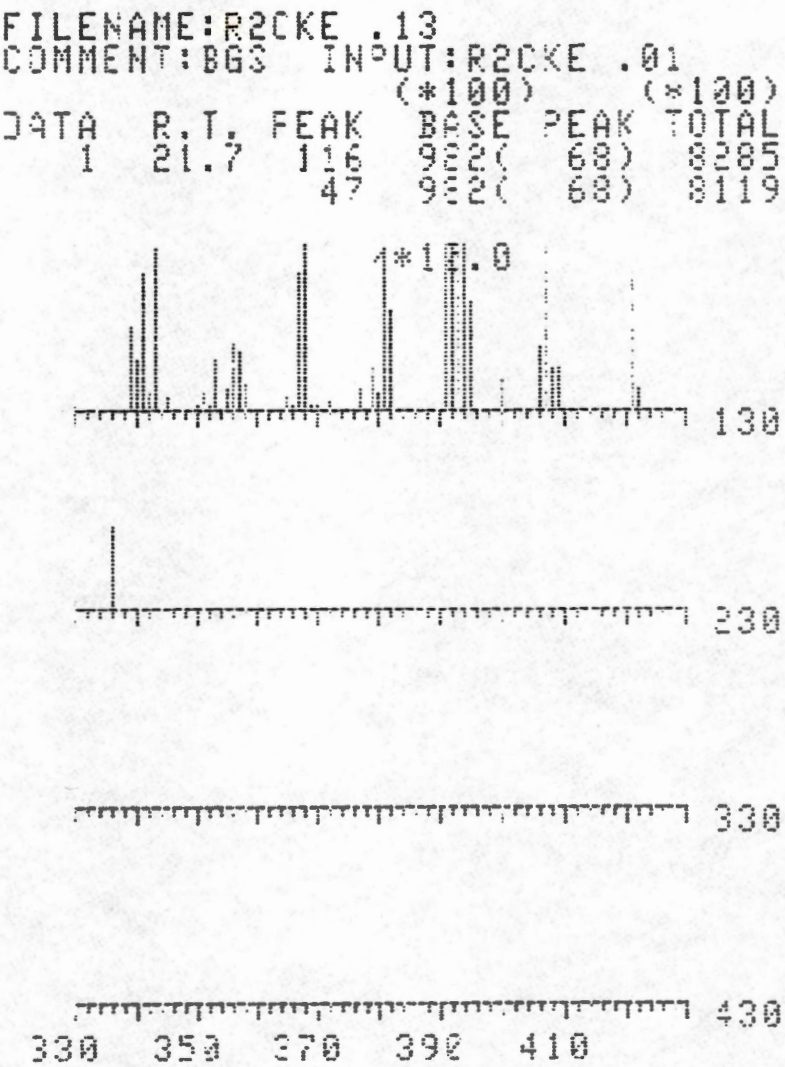
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130

230

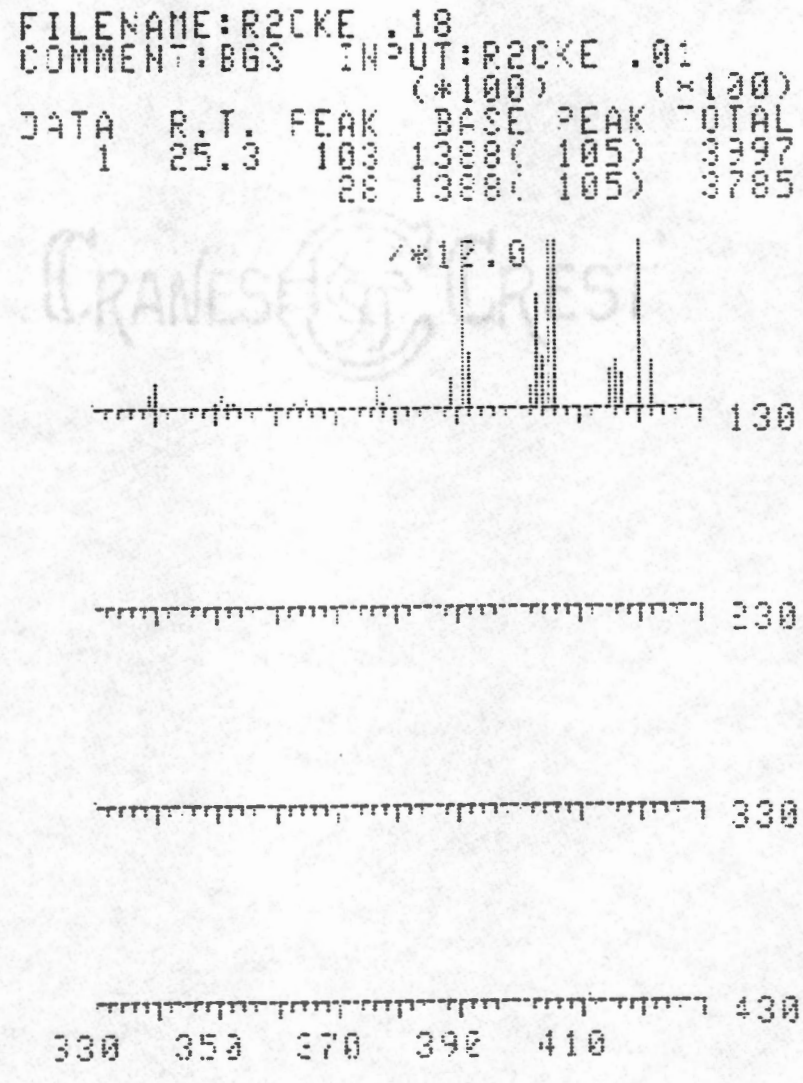
330

430
330   350   370   390   410
```

Peak 6


FILENAME:R2CKE .13
COMMENT:BGS INPUT:R2CKE .01
(*100) (*100)
DATA R.T. PEAK BASE PEAK TOTAL
1 21.7 116 982(68) 8285
47 982(68) 8119
1*10.0
130
230
330
430
330 350 370 390 410

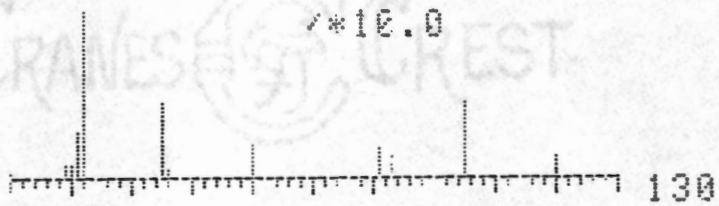
Peak 9

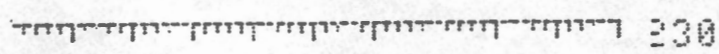

FILENAME:R2CKE .18
COMMENT:BGS INPUT:R2CKE .01
(*100) (*100)
DATA R.T. PEAK BASE PEAK TOTAL
1 25.3 103 1388(105) 3997
28 1388(105) 3785
/*12.0
130
230
330
430
330 350 370 390 410

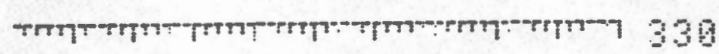
Peak 10

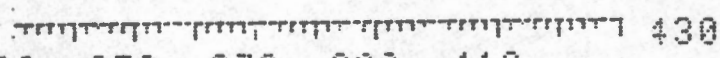
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FILENAME:R2CKE  .20
COMMENT:BGS   INPUT:R2CKE  .01
                      (*100)      (*100)
DATA   R.T.  PEAK   BASE  PEAK  TOTAL
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                  1 25130(   42)   5790

                    /*10.0
```



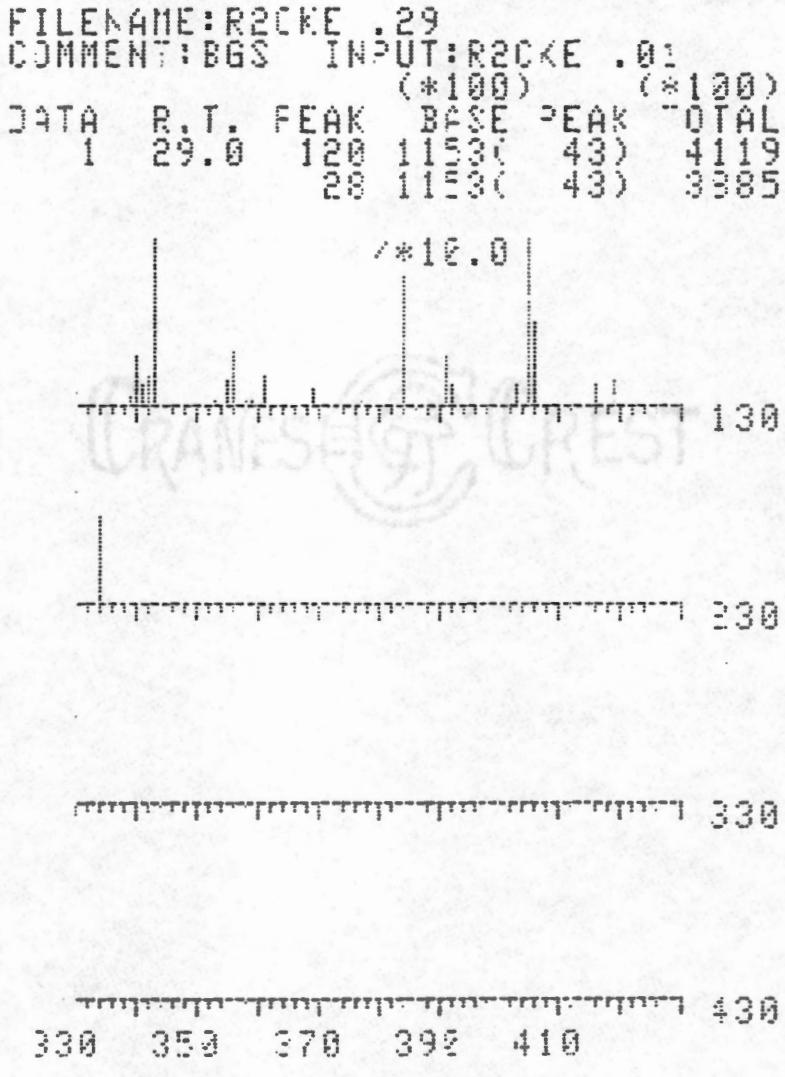




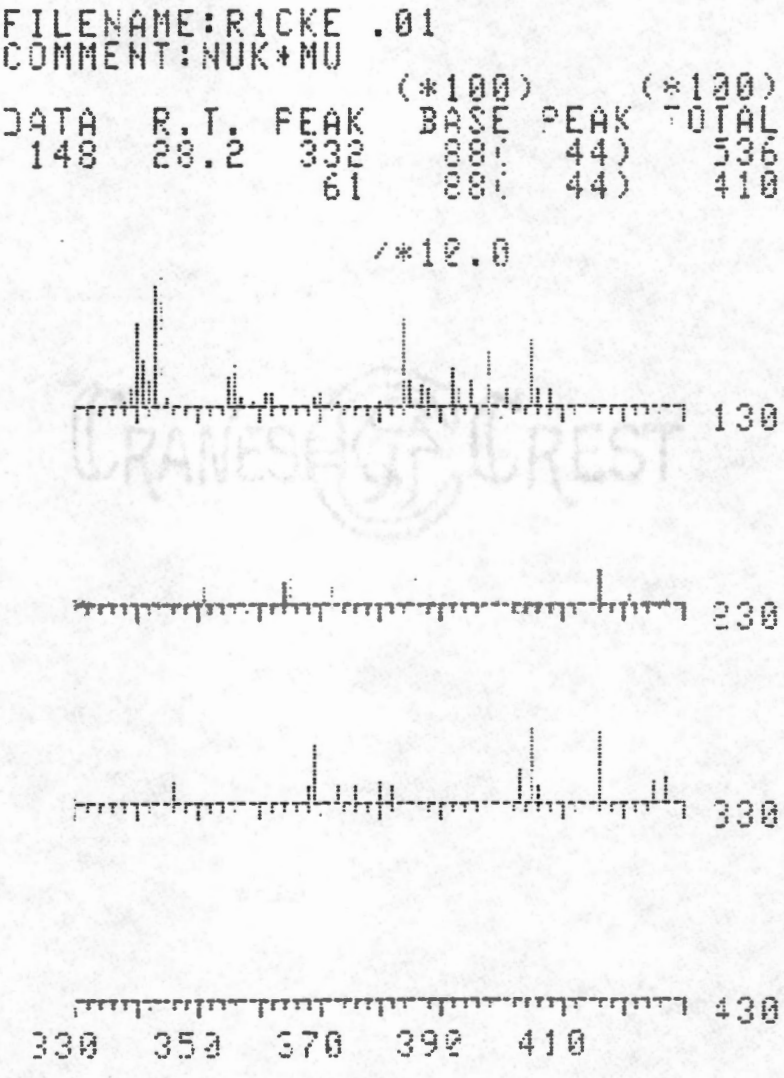



```
330   350   370   390   410
```

Peak 11


FILENAME:R2CKE .29
COMMENT:BGS INPUT:R2CKE .01
(*100) (*100)
DATA R.T. PEAK BASE PEAK TOTAL
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28 1153(43) 3385
/*10.0
130
230
330
430
330 350 370 390 410

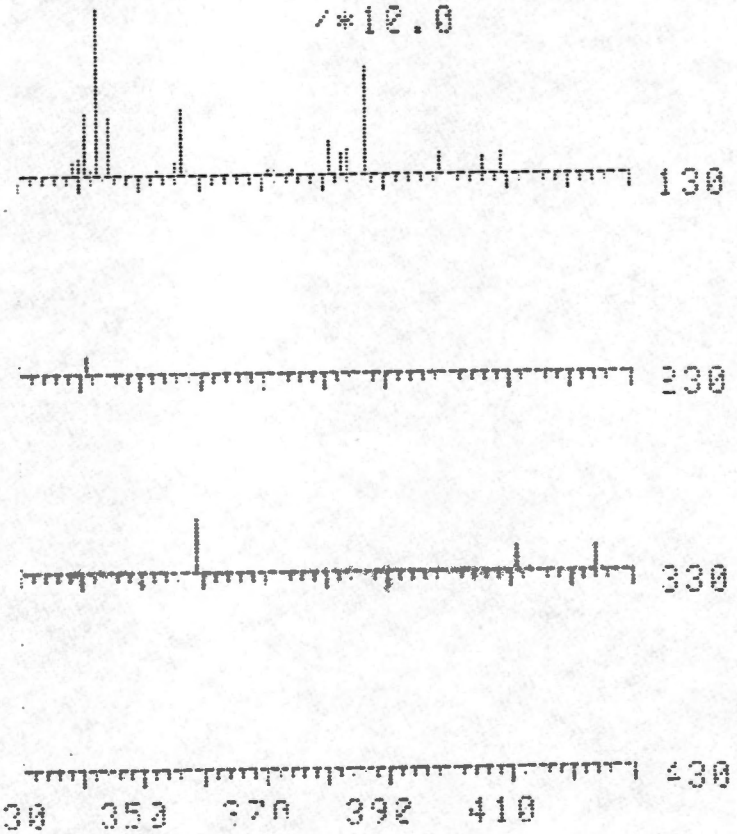
Peak 12


FILENAME:R1CKE .01
COMMENT:NUK*MU
(*100) (*100)
DATA R.T. PEAK BASE PEAK TOTAL
148 28.2 332 88(44) 536
61 88(44) 410
/*10.0
130
230
330
430
330 350 370 390 410

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Peak 15

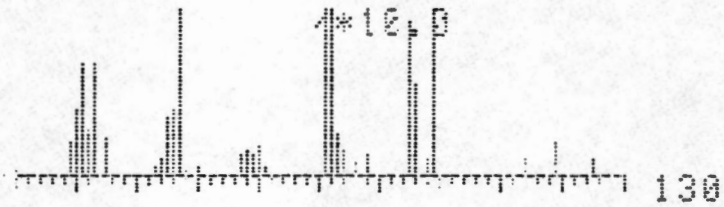
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FILENAME:RICKE  .18
COMMENT:BGS   INPUT:RICKE  .01
                  (*100)        (*100)
DATA   R.T.  PEAK   BASE  PEAK   TOTAL
   1   31.9   276   191(   43)     657
              31   191(   43)     563
```



CRANFIELD CREST

Peak 16

```
FILENAME:R2CKE  .38
COMMENT:BGS   INPUT:R2CKE  .01
                      (*100)      (*100)
DATA   R.T.  PEAK   BASE  PEAK   TOTAL
   1  33.9   128   9960(  57)   5186
             38   9960(  57)   5986
```




```
130
230
330
430
330   350   370   390   410
```

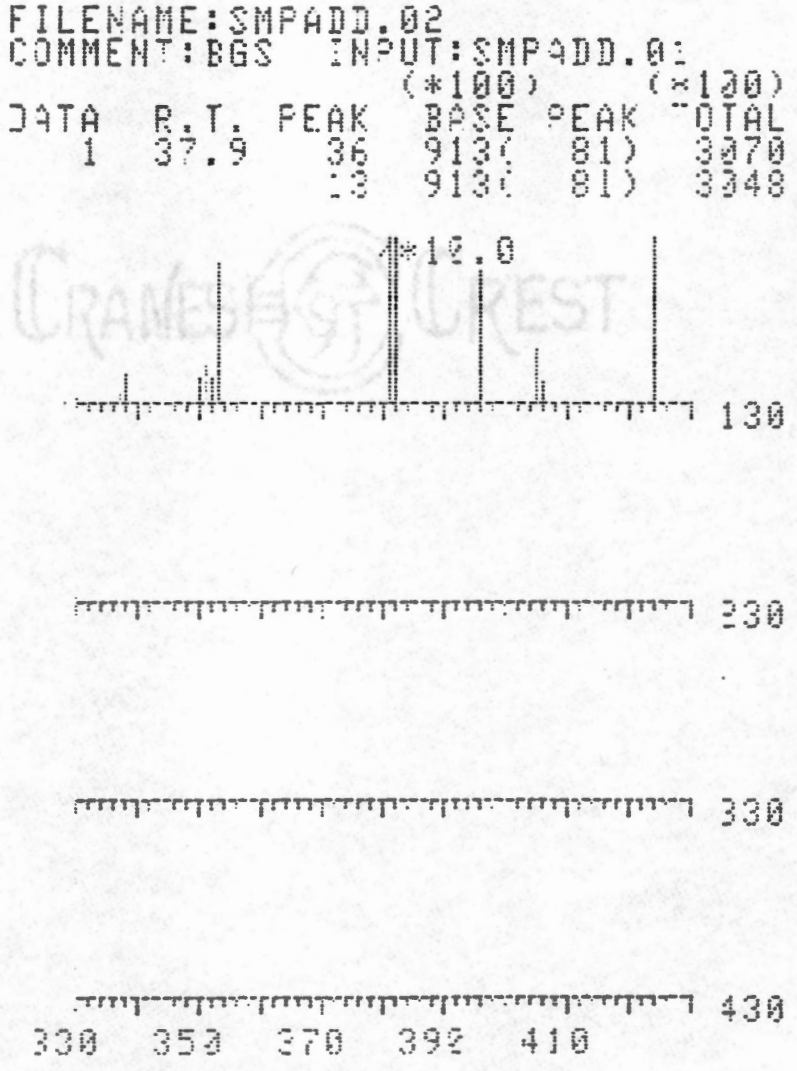
Peak 19

FILENAME:R1CKE .21
COMMENT:BGS INPUT:R1CKE .01

DATA	R.T.	PEAK (*100)	BASE	PEAK (*100)	TOTAL
1	36.9	300	195(	43)	528
		35	195(	43)	401



Peak 20


FILENAME:SMPADD.02
COMMENT:BGS INPUT:SMPADD.01
(*100) (*100)
DATA R.T. PEAK BASE PEAK TOTAL
1 37.9 36 9130 81) 3070
13 9130 81) 3048
*10.0
130
230
330
430
330 350 370 390 410

VITA

Susan Gail Varner was born in Chattanooga, Tennessee, December 17, 1961. After graduating from Hixson High School in 1979 with honors bestowed upon her by fellow students as Best All Around and Most Versatile, she enrolled at the University of Tennessee, Knoxville in the College of Home Economics.

During her undergraduate studies she participated in the 1982 World's Fair in food service management. Pursuing food service experience, she traveled to Lexington, Kentucky, and attended the University of Kentucky, Lexington. Before returning to the University of Tennessee, Knoxville, she traveled to New Orleans, Louisiana where she participated in the 1984 World's Fair in food service management and attended the University of New Orleans.

She received a Bachelor of Science degree with honors in Nutrition and Food Science March 1987 from the University of Tennessee, Knoxville. In the fall of 1987 she was accepted as a Graduate Research Assistant in the Food Technology and Science department in the Institute of Agriculture at The University of Tennessee, Knoxville, and was awarded the Master of Science degree in May 1990.

The author is a member of the Institute of Food Technologists and Gamma Sigma Delta agricultural honor society. She resides and is employed in Nashville, Tennessee.

CRANES CREST