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OFF-FLAVOR DEVELOPMENT IN MILK PACKAGED IN

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By

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ABSTRACT

OFF-FLAVOR DEVELOPMENT IN MILK PACKAGED IN POLYETHYLENE-COATED PAPERBOARD CARTONS

By

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Development of a "packaging flavor" in milk packaged in half-pint polyethylene (PE)-coated paperboard cartons was investigated to determine if this type of flavor occurs during storage. Whole, 2% fat, and skim milk and water were evaluated by a 10-member trained panel using a paired comparison test method after 1, 3, and 6 days storage at 2.2°C. Extent of lipid oxidation in whole milk was determined using the thiobarbituric acid (TBA) test.

"Packaging flavor" developed in milk and water packaged in half-pint cartons after one day storage. There was no significant increase in the off-flavor intensity in milk after 3 days. Milk packaged in half-pint cartons was significantly more off-flavored than milk in quart and half-gallon cartons after 6 days. Off-flavor development was not due to lipid oxidation or excessively high heat sealing temperatures.

A 91-member consumer panel did not find differences between milk in half-pint cartons and glass containers after 3 days storage at 2.2°C and indicated no preference among the samples.

This thesis is dedicated to my parents

for their love, support, and understanding.

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INTRODUCTION

The first package used as a sanitary container for distributing milk to the consumer was the glass bottle, which was introduced in 1878 (Prucha and Tracy, 1943). As retail store sales of milk increased, there was demand for an inexpensive single service container. Consumers preferred to buy milk in a safe, convenient, one-trip package. To meet this need paper milk containers were developed.

The paper milk container was invented by G. W. Maxwell of San Francisco and first used by dairymen in Los Angeles (Winslow, 1909). These early containers resembled an ordinary drinking glass and were sterilized and waterproofed by dipping into paraffin at 220°F, then filled and capped by a machine especially designed for that purpose. The paper containers had several advantages, which included nonbreakable, light weight, easier handling, and nonreturnable, which eliminated bottle collecting, washing, and sterilizing.

Although the first paper milk container was invented in 1906, the glass bottle remained almost universal until 1929 (Prucha and Tracy, 1943), when a cone-shaped paper container was introduced by Sheffield Farms in the Bronx, New York (Anonymous, 1929). Borden Company and National Dairy

Products Corporation began using paper bottles extensively at this time (Anonymous, 1930). Since then the paper container has become widely used. By 1937, it was generally accepted by both industry and milk sanitarians (Sanborn, 1942).

There were in general three types of paper containers used: those that were prefabricated and filled on a regular glass bottle filling machine; those that were prefabricated and requiring a special filling machine; and those formed and paraffined in the dairy immediately before filling, requiring of course a special machine (Tracy, 1938).

While about 30 different varieties of paper milk containers have been developed, only 5 were in every day use in the late 1930's. Containers commonly used were generally of two shapes, rectangular or round. The former had either a flat or gable top. These were the Canco® and Pure-Pak® containers. There were three round types, two of which were nearly identical, cone-shaped and were known as the Sealright® and Pure-cone® (formerly Purity®) containers. The other round container, the Reed®, was cylindrical with a folded star-shaped top, and sealed with a metal fastener (Sanborn, 1942).

Paraffin wax was soon replaced by plastic vinyl and later by polyethylene (PE) as a coating for paperboard. Important properties of PE included chemical inertness, toughness, flexibility, water resistance, and heat sealability. Plastic-coated paperboard cartons were first introduced commercially in 1932 (Milk Industry Foundation, 1989). In 1936 Ex-Cell-O Corporation developed the first gable-top carton. The square, plastic-coated paper cartons, were referred to as sealking containers and were developed by Sealright Company, Inc. of Fulton, New York. They were available to the market nationwide in 1949 (Anonymous, 1949). Quart PE-coated milk cartons made their debut in 1959 after a 10-year experience with vinyl coating (Anonymous, 1960). The PE-coated Pure-Pak® cartons currently in use were first introduced in the U.S. in 1963 and in the U.K. four years later (Anonymous, 1967). These plastic-coated cartons gained consumer acceptance.

With the exception of half gallons, there are more half pints sold than any other size of container (U.S.D.A., 1990). Consumption of half pints grew as the institutional market, including schools, expanded. More than 5.5 billion half pints of milk are served to 43 million U.S. school children each year. In 1975, Bandler *et al.* reported that this amounted to over 10% of the fluid milk market. On the national average this was 0.7 of a half pint per pupil per day. Across the nation, if each child drank one half pint per day, it would mean a potential sales gain of 2.2 billion half pints of milk a year.

In 1987, 331 million pounds of fluid milk were sold in half-pint cartons (U.S.D.A., 1990). Approximately 7% of fluid milk sales in 1988 were delivered to schools and 9% of milk sold in half-pint containers (Milk Industry Foundation, 1989).

Numerous complaints from children and food service people concerning off-flavored milk have been reported (Partridge, 1988). A complaint frequently heard is that the milk tastes "like the package." This off-flavor in milk may discourage milk consumption in children, who are traditionally heavy milk drinkers. Lower consumption may result because of the low quality of milk served in schools.

In this study, the "packaging flavor" problems in milk packaged in half-pint PE-coated paperboard cartons were investigated. The specific objectives of this study were to determine the presence or absence of "packaging flavor" in milk of various fat contents and the relationship between storage time, size of container, heat sealing of the package, and off-flavor.

REVIEW OF LITERATURE

Light Protection

Some of the paper milk bottles and containers used during the 1930's were not opaque and, therefore, transmitted light rays which adversely affected the flavor of the milk. The effect of sunlight on the flavor of milk packaged in single service paperboard containers has been studied extensively.

Doan and Myers (1936) studied the influence of sunlight on milk packaged in Sealcone® containers. They concluded that paper milk bottles protected skim milk, whole milk, and buttermilk from burnt flavors but provided no protection to homogenized and nonhomogenized whole milk and cream against the tallowy flavors caused by sunlight. Blue and green colored paper bottles retarded the development of tallowiness and burnt flavor in skim milk, whole milk, buttermilk and cream.

Tracy (1938) reported "sunshine flavor" in milk packaged in another type of paper bottle (Pure-Pak®). Guthrie *et al.* (1939) also observed that paper bottles, to some extent, protected milk against the development of burnt, oily, or sunlight flavor.

Dahle and Palmer (1937) treated the inner surface of paper bottles to prevent or delay the development of oxidized flavor in milk when exposed to sunlight. The results showed that paraffin containing 25% oat flour prevented off-flavor in milk exposed to sunlight for one hour. It was later shown by Dahle and Josephson (1939) and Garrett (1940) that oat flour sized paper bottles prevented or delayed the oxidized flavor in milk after 30 and 60 min exposure. England and Wiedemer (1941) found that the treated paper bottles offered protection to milk against oxidized flavor when the period of exposure to sunlight exceeded 10 minutes. Roadhouse and Henderson (1941) reported that the flavor of milk in treated and untreated containers exposed to sunlight for 30 min was only slightly impaired.

Henderson *et al.* (1940) studied the effects of sunlight on milk in different types of paraffined paperboard containers. Milk packaged in cartons made of white bleached paper had a slight "sunshine" flavor after one hour of direct sunlight exposure. Milk in cartons of multiple layers of bleached white outer plies and unbleached light brown inner plies, was not affected while milk in cream colored paper had a slightly different flavor. Wildbrett (1960) reported that dark red and dark brown cartons protected milk much better than white cartons. Koenen (1967) stated that brown and dark red cartons remarkably reduced the detrimental effect of light.

Prucha and Tracy (1943) exposed Pure-Pak® containers made from heavy- and light-weight papers (basis weight) and filled with nonhomogenized and homogenized milk to direct sunlight to determine the extent of light penetration and its effect on the flavor of milk. Milk in the heavy-weight paper containers was less affected by the sunlight. Homogenized milk acquired the burnt flavor more readily than nonhomogenized milk under the same conditions.

Barnes (1960) reported that plastic-coated cartons did not prevent light-induced off-flavor when exposed to direct sunlight for 2 hours. Nordlund *et al.* (1970) concluded that PE-coated cartons effectively prevented deterioration due to sunlight after 1, 2, and 3 hr exposure.

In addition to sunlight, fluorescent light can cause oxidized flavor in milk. Numerous studies on this subject are available in the literature. Radema (1956) observed different degrees of oxidation in unhomogenized milk packaged in three different types of cartons exposed to diffuse daylight, incandescent light, and fluorescent light. Birdsall *et al.* (1958) showed that a quart foil-laminated waxed carton was more effective than a waxed carton in preventing undesirable flavor change in milk exposed to fluorescent light.

Dunkley *et al.* (1962) reported that the minimum time required to induce detectable light flavor was 1 - 14 hr for several different unprinted fiberboard milk cartons coated with paraffin or PE. Cartons with large areas printed with inks (yellow, red, orange, and brown) that absorb light associated with the shorter wavelengths gave the best protection.

Hendrickx and Moor (1962) exposed pasteurized whole and skim milk in clear and colored waxed cartons and "light protected" Tetra Paks® (black inner coating) to fluorescent light of various light intensities. Early development of light-induced flavor occurred in uncolored cartons. Tetra Paks® afforded almost complete protection against off-flavor development, closely followed by brown, yellow, and red colored containers. Blue colored containers on the other hand, offered only moderate protection.

Bradfield and Duthie (1965 and 1966) conducted a series of experiments in which various milk cartons were evaluated against fluorescent light. Preliminary studies showed that wax-coated cartons provided less protection than plastic-coated cartons and cartons with aluminum foil laminated between two layers of paper.

In the first experiment (Bradfield and Duthie, 1965 and 1966) PE-coated cartons in solid colors (plain, red, blue, black, and green) were tested. Green cartons afforded the greatest amount of protection and gave the lowest light transmission values. Light transmission through all cartons correlated very closely with the time required to develop oxidized flavor.

A second experiment was conducted using PE-coated cartons and the top portion of the container varied. Milk in the container with the aluminum foil top was the most effective and the container with the plain top was the least effective in preventing the development of oxidized flavor.

In the last experiment in the series, various carton color and coating combinations were tested. The container with the aluminum foil top gave the best protection. Half-gallon cartons provided better protection for the milk than the quarts due to lower light transmission and less surface area in relation to volume.

Several investigators have confirmed these findings. Sattar and deMan (1973) reported off-flavor development in milk in quart cartons after a 12-hr exposure of 100 footcandles (ftc) of light. Dimick (1973) reported that milk in half-gallons afforded protection for up to 48 hours. The flavor of milk in one-gallons was not significantly different from that of the control milk after 72 hr (Hoskin and Dimick, 1979). Average light transmissions at 800 nm for quart, half-gallon, and gallon paperboard cartons were 4.5, 3, and 3%, respectively (Nelson and Cathcart, 1983).

Storgårds and Lembke (1966) examined various types and grades of paper with different coatings for light transmission and gas permeability. Materials which were completely light impermeable were aluminum-laminated paper, black PE-coated paper, and the paperboard impregnated with paraffin. Koenen (1967) and Barnard (1972) recommended using an inner layer of plastic-coated aluminum foil for cartons.

Shield (1972) exposed milk packaged in quart PE-coated paperboard cartons to fluorescent light. Slight activated flavor was detected after 96 hr of high intensity exposure (900 ftc). No activated flavor was detected in the low intensity exposure (25 ftc). Flückiger (1972) compared raw and uperised [ultra-high-temperature (UHT)] milk in PE-coated cartons exposed for 6 hr to various light intensities, and found that uperisation and PE-coated carton offered sufficient protection against light.

Coleman *et al.* (1976) exposed milk in various colored half-gallon paperboard containers to 100 ftc and found that unpigmented, yellow, and red colors offered less protection to light-induced flavor changes than the other colors (black, blue, green, orange, purple, and brown) investigated. deMan (1978) studied the effect of light on milk in Pure-Pak® cartons and cartons with brown printing. Milk in the latter containers did not change in flavor after 48 hr of exposure.

Bradley (1980) summarized the available literature on light-activated flavor development in milk packaged in paperboard containers, and stated that paperboard containers with a large printed area of dark ink or made from aluminum foil laminate provided the best protection.

Schröder *et al.* (1985) studied the flavor of milk packaged in PE-coated cartons. Milk in quart, white Pure-Pak® cartons overprinted with blue which was stored under white fluorescent light developed an off-flavor after 17.5 hr of exposure.

O_2 Permeability

In addition to light permeability, the O_2 permeability of the containers used for milk packaging has been studied. The aluminum-laminated paper examined by Storgårds and Lembke (1966) was gas as well as light impermeable. Black PE-coated paper and paperboard impregnated with paraffin were not gas impermeable, the latter had significantly much higher permeability.

Permeability of the package to O_2, among other factors, can affect both the concentration and consumption of O_2 in milk. Flückiger and Heuscher (1966) investigated the O_2 permeability of milk packages. There were only slight differences in the O_2 permeability among PE-coated papers (Tetra Pak®, Zupack®, and Pure-Pak®) while paraffin-coated material (Perga®) was significantly more permeable. However, after 24 hr the O_2 content was highest in milk packaged in Zupack® and lowest in Perga®. The O_2 uptake in water was significantly higher than in milk, possibly due to O_2 consumption by bacteria. In the aluminum-lined Tetra Pak® the O_2 content remained low (0.4 ppm) whereas, in the normal Tetra Pak® (without aluminum) it increased relatively rapidly to about 8 ppm. This was later confirmed by Flückiger (1972). The O_2 level remained constant at about 1 ppm in PE-coated aluminum foil cartons. Milk in plain cartons was saturated with O_2 after 48 hours.

Nordlund *et al.* (1970) reported that O_2 content in 1-L PE-coated cartons increased from 43.3 to 55.0% after 24 hr and was the same after 48 hours. Hansson (1975) studied the O_2 permeability of various milk containers filled with water. Loss of O_2 pressure inside the Pure-Pak®, Pure-Pak® with aluminum foil, and Tetra Brik® after 24 hr were 26, 11, and 35%, respectively. The corresponding loss constants expressed as percentage loss of O_2 pressure in 24 hr were 107, 0.7, and 99% . Mehta and Bassette (1978) found that aluminum foil-lined cartons were less permeable to gases than were PE-lined cartons.

Dejmek and Ånäs (1977) stated that O_2 in the package headspace exerted little influence on the O_2 content of milk in 1-L Pure-Pak® cartons due to the slow diffusion of O_2 through milk and relatively small contact surface between milk and headspace O_2. The authors concluded that oxygen transfer was limited by the carton walls and conditions inside the package. Small temperature differences between the inside and outside of the package wall gave rise to convection currents which increased O_2 transfer 3-fold and shaking a carton with headspace further increased it to 5-fold. Schröder *et al.* (1985) reported that the dissolved O_2 concentration of milk packaged in quart Pure-Pak® cartons increased slightly when stored in the dark and dropped marginally when exposed to fluorescent light during a 4-day period.

Off-flavor

While numerous reports describe light-induced off-flavor in milk packaged in paperboard containers, research describing the effect of the plastic-coated cartons on the flavor of milk and other dairy products is meager.

Burgess (1950) concluded that PE caused less organoleptic and chemical changes in food products than other plastic coating materials. Barnes (1960) and Hedrick *et al.* (1961) reported that no off-flavor attributable to the PE could be detected in whole milk, cultured buttermilk, and orange drink packaged in quart PE-coated cartons during storage or after freezing, storing, and defrosting.

Nordlund *et al.* (1970) evaluated the quality of milk packaged in 1-L PE-coated cartons and concluded that the package did not affect the organoleptic properties of the milk.

Flavor problems in sterilized milk in quart paperboard cartons made from various materials were investigated by Feldstein *et al.* (1977). The four types of paperboard were: (a) rosin (sizing) paperboard; (b) rosin paperboard with foil lining; (c) cyanasize (sizing) juice paperboard; and (d) cyanasize-juice paperboard with foil lining. No significant differences in flavor were found between day 0 milk and milk stored for up to nine weeks in cartons or in glass bottles. When milk was stored for up to eight weeks in cartons and glass bottles, there was no significant difference between these and the nonstored pasteurized milk.

Hoskin and Dimick (1979) found that milk in 1-gal unprinted fiberboard (Pure-Pak®) containers was not significantly different in flavor from the control during a 72-hr period. Similarly, Janzen *et al.* (1981) reported no significant differences in flavor between milk packaged in fiberboard containers and plastic jugs. Milk in fiberboard containers had a satisfactory shelf-life of 11 days at 4.5°C and 9 days at 7.0°C. The flavor of pasteurized, nonhomogenized, full cream milk packaged in quart PE-coated Pure-Pak® cartons remained good during 4 days storage (Schröder *et al.*, 1985).

Although the previously cited studies did not find any off-flavor problems, the following studies describe off-flavors in milk packaged in PE bags and PE-coated cartons. Most of this work has been done by researchers outside of the U.S.A.

During direct contact between plastics and food products, interactions among the two may occur. Low molecular weight components may migrate from plastics and can adversely affect the organoleptic properties of the food products (Wildbrett, 1968). Pasteurized milk packaged in PE pouches, 100 and 50 μm thick developed a slight heated flavor after 24 and 48 hours (Yurin, 1966).

Kiermeier and Stroh (1969) observed that ketocarbonyl and carboxyl containing compounds in oxidized PE exchanged their weakly acidic protons with metallic cations in milk. This autoxidation produced C_{14}, C_{19-21}, C_{24-26}, C_{38-42}

unbranched carboxylic acids in water, which could be responsible for the "plastic flavor" sometimes found in milk packaged in PE. Methane, ethane, propane, n-butane, and 1-butene can result from the radical decomposition of PE and have been detected in water and milk in contact with PE.

Kringlebotn (1971) conducted an investigation of a PE bag used for packaging milk, in which an unpleasant off-flavor described as "graphite flavor" was found. It was established that the origin was 4-4′-thio-bis (3-methyl-6-tertiarybutyl-m-phenol), a heat stabilizer and antioxidant used in the PE. Very low concentrations caused perceptible aftertaste in milk and the threshold value was found to be 0.6 ppm.

Badings (1971) concluded from a number of experiments that the "plastic flavor" in milk packaged in PE pouches was more easily detected as the fat content of the milk was lowered. Milk and dairy products interacted with packaging materials mainly in the water phase; perhaps in the protein phase but not in the fat phase (Figge and Baustian, 1983). It is possible, however, that it could interact with the fat phase.

Migration of constituents from PE was observed to occur by Chuchlowa and Sikora (1976). A slight bitter off-flavor was noticeable in milk packaged in PE pouches after 3 days storage.

Regular (2.7% fat) milk and skim milk packaged in PE bags and stored in a refrigerator developed an off-flavor after two days of storage (Peled and Mannheim, 1977). Bags in a closed container stored in the same refrigerator did not develop any off-flavor during six days of storage. This was because of the permeability of the film to off-flavors in the refrigerator. When PE granules were extracted with milk and water, development of off-flavor occurred. No extractable matter in water was obtained due to the low concentration of the constituents which caused the off-flavors.

Radema (1973) stated that milk packaged in air permeable paper or plastic material sometimes develops an off-flavor, which is probably caused by oxidation. Sour cream buttermilk and aseptically packaged sterilized milk packaged in these materials may show oxidation defects. Low pasteurized milk was assessed organoleptically for aroma and flavor, particularly oxidized flavor and flavor due to packaging. Results showed that neither oxidation rate nor organoleptic quality was significantly affected by the air permeable bags. A tallowy flavor defect in buttermilk packaged in plastic-coated cartons was found (Anonymous, 1969).

Nilsson and Lodin (1964) compared milk packaged in PE-coated Tetra Pak® and wax-coated Pure-Pak® containers as well as glass bottles. No significant differences were observed between flavor and aroma scores of milk in 3 different types of container. When the containers were stored together with highly aromatic substances, milk in

Tetra Paks® received a markedly lower score than milk in Pure-Paks®.

Studies on pasteurized milk packaged in Tetra Pak®, Polipack®, and Bloc-Pak® containers were made by Leali (1968) and Quaroni (1968). The former found organoleptic changes after 240, 192, and 192 hr storage, respectively, while the latter reported that changes occurred after 192 - 288 hours.

Bockelmann (1972) compared two types of packages, Tetrahedron® and Tetra Brik®, both of which have no headspace, and Tetra Rex® and Pure-Pak®, which are gable-top cartons with headspace. Milk packaged in completely filled cartons was significantly better in flavor than milk in gable-top cartons.

Schönborn *et al.* (1975 and 1976) evaluated the flavor of milk in Tetra Brik®, Pure-Pak®, and Bloc-Pak®. The results concurred with those of Bockelmann (1972). Flavor of the milk packaged in Tetra Brik® was significantly superior to that of Pure-Pak® and Bloc-Pak®. The difference occurred after one day and did not increase after prolonged storage. This was attributed to the headspace, which provided for O_2 for microbial growth.

A comparison between uperised milk stored in 1-L cartons with and without aluminum foil was made by Flückiger (1972). The oxidative flavor of the milk in the plain packages increased. Milk in plain packages was much more affected by storage time and temperature than milk in aluminum foil packages. The difference in flavor after three days was due to the different O_2 permeabilities of the two materials. The results were in agreement with those obtained by Ashton (1965). Srivastava and Rawat (1978) studied the organoleptic quality of milk and physical characteristics of plastic pouches for in-package processing of milk and reported that the PE in the paper/aluminum/PE laminates imparted pronounced plastic flavor to milk.

Mehta and Bassette (1978 and 1980) investigated the effect of carton materials on flavor of UHT sterilized milk. Milk in aluminum foil-lined pint cartons retained desirable flavor characteristics longer than did milk stored in PE-lined cartons. The authors proposed that stale flavor was caused by volatile carbonyl compounds produced from, among other sources, one or more of the carton layers. Wrapping cartons with Saran™ and aluminum foil was detrimental to flavor in all instances. Differences in browning were observed between UHT sterilized and reference (freshly pasteurized) milk and between 2- and 12-day-old UHT sterilized milk.

Mannheim *et al.* (1987) also found greater browning in citrus juice aseptically packaged in laminated cartons than in glass. The authors theorized that this was due to contact with the PE surface layer. They also pointed out that some manufacturers use elevated temperatures during coating operations, which may result in excessive surface oxidation and thus adversely affect products. In a review article recommendations were provided by Goldenberg and Matheson (1975) which included careful control of additives in plastics and avoidance of overheating of PE.

Bojkow *et al.* (1976c) studied flavor problems in dairy products induced by PE film and PE-coated paper. An off-flavor, confirmed by IR spectroscopy, was caused by oxidative changes on the PE surface. Attempts were made by Bojkow *et al.* (1979) to identify the off-flavor causing substances in PE-coated papers. The flavor components migrating from the internal PE coating were found to be largely soluble in water and quite volatile. Unsaturated aliphatics, free fatty acids, and alcoholic compounds occurred more frequently in materials causing the off-flavor.

The origin of off-flavors in products stored in PE bottles and PE-coated cartons was investigated by Berg (1980). Off-flavor producing compounds were present in PE granulates. Converting/extrusion processes were responsible for only a small increase in the amount of off-flavor volatiles. The amount of volatiles decreased up to 90% when the final materials were aired for 24 hours.

Bandler *et al.* (1975) conducted a study on "acceptability of school milk" to determine causes of complaints from school children and to pinpoint problems that limit 100% acceptance of school milk. Surveys at 693 schools in New York State showed a high incidence of off-flavored milk and excessively high storage and serving temperatures.

One hundred two complaints of off-flavor, among others, were reported by cafeteria managers. Flavor analysis of 969 school milk samples showed that by far the most widespread objection was the cowy/barny/unclean off-flavor, followed by feed and rancid. A direct correlation was shown between the flavor and consumption of milk by school age children. The authors concluded that generally, the problem was not being caused by schools.

Another study on the quality of milk served in schools was conducted by Hankin *et al.* (1980). A total of 271 schools in the state of Connecticut were studied. Sixty three complaints (about 16%) by cafeteria personnel were about off-flavored milk. Flavor analysis of 401 milk samples collected at schools indicated that the major criticisms were feed, lacks freshness, and burnt paper or plastic. A burnt flavor was a result of excessive heating of paper cartons during sealing.

Sensory Evaluation

Sensory evaluation is a useful method for studying undesirable organoleptic changes in foods and beverages, such as off-flavors in milk. Peled and Mannheim (1977) concluded that organoleptic tests are the most reliable tests for determination of off-flavors originating from a package. Sensory evaluation is an important tool used to identify causes of flavor defects. This often involves use of experienced judges or a trained panel. Hoskin and Dimick

(1979) conducted three training sessions prior to beginning a study. The number of panelists employed for sensory evaluation of off-flavors in milk varied from 2 (Janzen *et al.*, 1981; Bradfield and Duthie, 1966) to 15 - 20 (Peled and Mannheim, 1977). Both expert and trained panels were used by Dimick (1973). In addition to a trained panel, Mehta and Bassette (1980) used a 24-member untrained consumer panel consisting of students and faculty.

Little information is available in the literature dealing with preparation and presentation of milk samples. Hoskin and Dimick (1979) mixed milk samples by inverting the containers. The samples were then transferred to 30-mL cups in dim light and presented to the panel members within 15 min. Dimick (1973) utilized 30-mL cups. Dunkley *et al.* (1962) presented the milk samples in randomized order in coded 50-mL beakers. Fenton (1957) stated that samples should be warmed to about 75°F to 80°F (24°C to 27°C) prior to examination. A cold sample (below 50°F (10°C)) is difficult to distinguish certain flavors. Bockelmann (1972) warmed milk samples to 17°C prior to presentation. Schönborn (1975) heated milk samples to 17°C and held at this temperature for 3 hr prior to distribution in 10-mL beakers. Peled and Mannheim (1977) conducted sensory tests in a well ventilated room with dim illumination. Schröder *et al.* (1985) warmed the samples to 20°C and presented in randomly coded beakers and under red/blue light. In a study by Mehta and Bassette (1980) coded milk samples were judged by a trained panel at room temperature whereas samples tasted by a consumer panel were tested cold (4°C).

Various standard methods have been used for measuring the quality of milk. Four studies (Henderson *et al.*, 1940; Prucha and Tracy, 1943; Feldstein *et al.*, 1977; Janzen *et al.*, 1981) utilized the American Dairy Science Association (A.D.S.A.) scorecard. To evaluate the flavor of milk served in schools, the A.D.S.A. scoring systems as modified by the TriState Milk Flavor Program (Bandler *et al.*, 1975) and by the Connecticut Milk Flavor Improvement Program (Hankin *et al.*, 1980) were used.

Dunkley *et al.* (1962) used a combination of paired comparison and scoring. The sample with the greater intensity of off-flavor was selected. All samples were scored for off-flavor intensity using the following scale: 0, none; 1, questionable; 2, slight; 3, distinct, objectionable; and 4, strong, very objectionable. To approximate the point where off-flavor development could be detected by the consumer, Bradfield and Duthie (1966) referred to samples that were definitely oxidized as positive (oxidized flavors present) and slight off-flavor samples as negative (little or no oxidized flavor present).

Badings (1971) examined milk samples using duo-trio and preference tests. Aroma and flavor were rated on a scale of 3 to 8 (3 = very poor, 4 = poor, 5 = unsatisfactory, 6 = satisfactory, 7 = good, and 8 = very good) and off-flavor intensity on a scale of 0 to 4 (0 = none, 0.5 = small, 1 =

slight, 2 = moderate, 3 = strong, 4 = very strong). Radema (1973) evaluated the aroma and flavor of milk and oxidative and plastic flavor defects using scales similar to Badings (1971).

Flückiger (1972) used a triangle test to determine differences in flavor and asked panelists to answer the following two questions: (1) Which two of three samples are identical? (2) Which sample(s) is(are) better with respect to taste? Organoleptic difference was rated on the following scale: 0 = no deviation from excellent taste, 1 = slight deviation, 2 = noticeable deviation, 3 = strong deviation, and 4 = very strong deviation.

Bockelmann (1972) rated appearance, consistency, aroma, and flavor of milk samples on a 5-point scale: 5, in accordance with product specification; 4, good agreement; 3, agreement; 2, minor deviations; and 1, major deviations. Schönborn *et al.* (1975) initially examined flavor differences using both duo-trio and triangle tests. Appearance, aroma, and flavor were then assessed using a 5-point scale (Bockelmann, 1972). A duo-trio test was used by Sattar and deMan (1973) to examine flavor changes in milk. A triangle test was performed by Peled and Mannheim (1977) to compare milk packaged in PE bags and glass bottles and Berg (1980) to compare water stored in PE-coated cartons and glass containers.

Dimick (1973) and Coleman *et al.* (1976) did preference evaluation using a 9-point hedonic scale (1, dislike extremely; 9, like extremely) and a multiple comparison test where the control sample was the reference. In the latter test (Coleman *et al.*, 1976), the mean was based on 5 as equal to the reference. Numbers less than 5 indicated dislike, and numbers greater than 5 were liked better than the reference.

In addition to preference evaluation using a 9-point hedonic scale, Hoskin and Dimick (1979) employed a magnitude estimation scale technique to quantify the off-flavor intensity. In this procedure the panel member was instructed to assign values in proportion to the perceived intensity of the off-flavor. The reference (control) stimulus was assigned an arbitrary value of 10 and all judgements of intensity were based on this control. For example, if the off-flavor intensity was perceived as twice that of the reference its score would be 20. Unlike previous investigations, Schröder *et al.* (1985) used a 8-point hedonic scale to evaluate the flavor of cartoned milk.

Bojkow *et al.* (1976a) conducted sensory tests according to recommendations from the Institute of Food Technology and Packaging in Munich. The samples were judged independently using a triangle test. Off-flavor intensity was rated on a scale of 0 to 4 (Dunkley *et al.*, 1962). Magnitude estimation was also used to quantify intensity of the off-flavor caused by extrusion coating operations.

Mehta and Bassette (1978 and 1980) used a modification of the National Collegiate Student Judging Contest procedure to evaluate the flavor of cartoned milk. Trained panelists were asked to assign intensity of stale and cooked flavors a score on a scale of 0 to 9 (0 = none and 9 = very pronounced). The untrained consumer panel on the other hand, graded the samples using a 7-point hedonic scale ranging in descriptive terms from like extremely to dislike extremely.

MATERIALS AND METHODS

Preliminary Studies

Preliminary sensory studies were conducted to determine the effect of storage temperatures on "packaging flavor" development in whole and 2% fat milk packaged in half-pint gable-top PE-coated paperboard cartons. Milk packaged in cartons was obtained from a local dairy immediately after processing and evaluated by 3 individuals experienced in the flavor quality of fluid milk after 0, 1, 2, 5, 7, 9, and 12 days storage at 2.2°C, 5.6°C, and 11.1°C. Results indicated the presence of off-flavor in both whole and 2% fat milk stored at all three temperatures. At higher temperatures (5.6°C and 11.1°C) flavor deterioration of the milk due to microbial growth was accelerated. Thus, the storage temperature selected for this study was 2.2°C.

Initial Conditions and Set-up

Fresh, pasteurized, homogenized milk packaged in commercial paperboard and blow-molded plastic containers was transferred on the day of packaging from a local dairy in insulated styrofoam containers by car (approximately 15 min) to a dark walk-in refrigerator (Chrysler & Koppin Company, Detroit, MI) maintained at 2.2°C ± 1°C. Milk packaged in half-pint, quart, and half-gallon gable-top PE-coated paperboard cartons was used in the sensory analysis. The controls maintained in 4000-mL pyrex Erlenmeyer flasks (Corning Glass Works, Corning, NY) and half-pint cartons without heat seal closures required milk to be transferred from one-gallon blow-molded high density polyethylene (HDPE) containers. Half-pint knocked-down carton blanks and heat sealed empty cartons were obtained from the dairy.

Trained Panel

Preparation of Samples

Five cartons of milk from each treatment variable were removed from the walk-in refrigerator an hour prior to sensory analysis. Milk was transferred from half-pint cartons to a 2000-mL pyrex Erlenmeyer flask and from quart and half-gallon cartons to 12-qt stainless steel containers. Both the control, stored in a 4000-mL pyrex Erlenmeyer flask and cartoned milk samples (30 mL ± 5 mL) were dispensed into 2 fl. oz. clear polystyrene containers with lids (Sweetheart® US2 and LUS2, Maryland Cup Corporation, Owings Mills, MA), which had been previously coded with a 3-digit random number. Sample codes were changed each time the sensory evaluation was conducted. The milk samples were served to judges at 15.6°C ± 2°C. Each judge was presented in a randomized order (Appendix A) a tray with 3 pairs of milk samples and a cup of double distilled water for oral rinsing between samples.

Panel Training

Sensory evaluation was conducted to determine if a panel trained to detect "packaging flavor" could differentiate between milk stored in cartons and glass containers. A panel of 21 subjects participated in the training which lasted 10 weeks. The participants were prescreened for milk consumption habits, health, and interest.

During the first few weeks subjects were tested for ability to detect difference among the samples using triangle test in two sessions. The samples consisted of 2% fat milk packaged in half-pint Eco Pak® cartons for 2 and 7 days (15.6°C ± 2°C) and the control stored in a glass container. An initial meeting was subsequently held to explain the objectives of the study and give instructions which was followed by testing of the panelists.

A duo-trio test was then used to monitor panelists for ability to discriminate difference between samples when given a reference sample. Six sessions were conducted during the next three weeks of training. A ranking test was used to measure panelists ability to perceive different off-flavor intensities in three sessions during the last three weeks of training. Milk samples of different intensities of "packaging flavor" were prepared using the following dilutions of 6-day glass-stored milk and cartoned milk: 4:0, 2:2, 1:3, and 0:4.

Sensory Evaluation

At the conclusion of the training sessions, about half of the potential judges successfully passed the tests. A 10-member panel consisting of 3 faculty and 7 students at Michigan State University, which were made up of 3 males and 7 females between the ages of 20 - 40, was assembled and the actual testing initiated. Selection criteria included ability to discriminate and reproduce results, prior testing experience, and availability for testing. Judges evaluated the samples in individual partitioned booths under white fluorescent light in a quiet sensory evaluation laboratory maintained at 22°C ± 1°C. Tasting sessions were conducted mid-mornings and/or mid-afternoons.

All samples were evaluated at 15.6°C ± 2°C using paired comparison tests (Meilgaard *et al.*, 1987a). For testing of all 2% fat milk and water three samples were evaluated, in which each sample was compared with each of the two other samples. For skim and whole milk two samples were evaluated, one of which was the carton sample compared to the control. Three replicate analyses of all samples were performed. An example of the evaluation sheet is shown in Appendix B.

Experimental Variables

Fat Content

The relationship between fat content in the milk and "packaging flavor" in milk packaged in half-pint cartons was studied. Two percent fat milk packaged in Eco Pak® and standard cross-section cartons was compared as well as to 2% fat milk in a glass container after 1, 3, and 6 days storage at 2.2°C ± 1°C. Whole milk in Eco Pak® cartons and skim milk in standard cross-section cartons were compared to the respective milk in a glass container.

Storage Time

The relationship between duration of storage and "packaging flavor" intensity in milk packaged in half-pint cartons was determined. Two percent fat milk which was stored in Eco Pak® cartons for 1, 3, and 6 days at 2.2°C ± 1°C was compared for change in the off-flavor intensity during storage. Three- and 6-day-old milk were compared as well as to 1-day-old milk. The test was designed such that milk samples representing the different storage times were evaluated on the same day.

Size of Container

The influence of container size on intensity of the "packaging flavor" in milk was examined. Off-flavor development in 2% fat milk packaged in quart and half-gallon cartons was compared as well as to milk packaged in half-pint Eco Pak® cartons after 1, 3, and 6 days storage at 2.2°C ± 1°C.

Heat Sealing

The effect of heat sealing of the package on "packaging flavor" development in milk was determined. Non-heat seal cartons were constructed from knocked-down carton blanks and closed 1.4 cm from the bottom with a circular piece of glass, 7.0 cm in diameter and 1.0 cm thick, with an O-ring (The glass pieces were made by Department of Chemistry, Michigan State University, East Lansing, MI) as shown in Figure 1. To prevent leakage, the glass pieces were sealed on the bottom with sanitary lubricant Petrol-Gel (McGlaughlin Oil Company, Columbus, OH), which was not in direct contact with the milk. The cartons were filled with 2% fat milk transferred from a one-gallon HDPE container and the top closed with labeling tape (TimeMed Labeling Systems, Inc., Burr Ridge, IL). These cartons were then placed in the dark walk-in refrigerator until sensory analysis. Two percent fat milk packaged in half-pint Eco Pak® with and without heat seal closures was compared as well as to milk in a glass container after 1 and 3 days storage at 2.2°C ± 1°C.

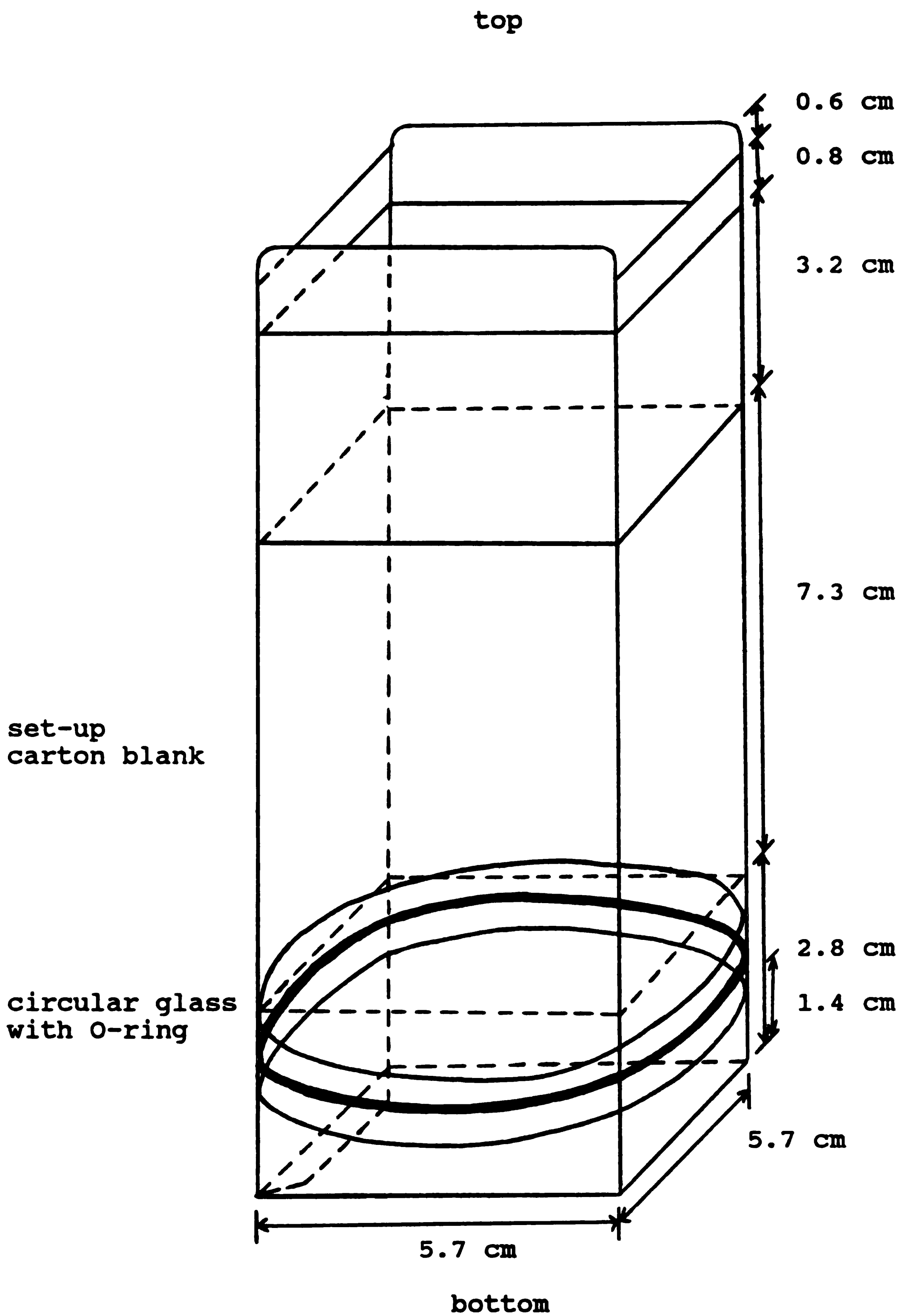

Figure 1. Schematic diagram of half-pint Eco Pak® carton without heat seal closures

Water

A study of "packaging flavor" development in water packaged in milk cartons with and without heat seal closures was also undertaken. Heat sealed empty half-pint Eco Pak® cartons were filled with double distilled water using a sterile, disposable 60 cc syringe with a Luer-Lok tip (Becton Dickinson & Company, Rutherford, NJ). The syringe was inserted through the middle of the heat sealed top so that it was positioned in the middle of the gable top. Closure of non-heat seal cartons was accomplished as previously described. Double distilled water packaged in half-pint Eco Pak® cartons with and without heat seal closures was compared as well as to water in a glass container after one day storage at 2.2°C ± 1°C.

Statistical Analysis

Two statistical analyses were employed. A pairwise ranking test (Meilgaard *et al.*, 1987a) was used for all sensory evaluation involving 2% fat milk and water. Chi-square was used to determine significance of difference between paired samples. When significance was observed (minimum $\alpha = 0.05$) rank sums were compared using Tukey's HSD test (minimum $\alpha = 0.05$). A one-sided paired comparison test (Meilgaard *et al.*, 1987a) was used for skim and whole milk. A critical numbers table, as reported by Meilgaard *et al.* (1987b) was utilized to determine if any significant difference existed between carton samples and controls.

The pairwise ranking test with Friedman analysis was chosen because several samples were compared for a single attribute, in this case, off-flavor. The Friedman approach also arranges the samples on a scale of intensity of the attribute and provides a numerical indication of the differences between samples and the significance of such differences. The one-sided paired comparison test was chosen because two samples were compared, one of which had a higher off-flavor intensity.

Consumer Panel

Preparation of Samples

The initial conditions and preparation of samples were similar to those for the trained panel. Two percent fat milk packaged in half-pint Eco Pak® cartons and one-gallon blow-molded HDPE containers was used for the sensory analysis. Milk in one-gallon plastic containers was transferred to glass containers and served as the control. Milk in cartons and glass containers was removed from the dark walk-in refrigerator after 3 days of storage at 2.2°C ± 1°C.

Following preparation, milk samples were maintained at 10°C ± 1°C in another dark walk-in refrigerator until presentation to the panelists. Each panelist was presented in a randomized and balanced order (Appendix D) with a set consisting of 3 milk samples and a set of 2 samples and a cup of double distilled water for oral rinsing between samples. The set of 3 samples was made up of either two glass controls and one carton sample or one control and two carton samples. The other set of 2 samples was made up of a control and a carton sample. Three individuals experienced in the flavor quality of fluid milk screened the milk samples and did not find a flavor defect in the control sample but did find a typical and definite "packaging flavor" in the sample packaged in cartons.

Sensory Evaluation

A 91-member consumer panel consisting of faculty, staff, and students at Michigan State University was assembled. Milk samples were evaluated using a triangle test and a paired preference test (Meilgaard *et al.*, 1987a). Examples of the evaluation sheets and a short demographic questionnaire are shown in Appendices E and F. A triangle test was chosen to determine if an untrained panel could detect a difference between the two samples (control and carton) and a paired preference test to determine their taste preference for samples of a control and a sample with "packaging flavor."

Statistical Analysis

For a paired preference test, a two-sided paired comparison test was applied (Meilgaard *et al.*, 1987a).

Critical numbers tables, as reported by Meilgaard *et al.* (1987b) were utilized to determine levels of significant difference in the triangle and paired preference tests.

Thiobarbituric Acid (TBA) Test

The TBA method of King (1962) was performed on whole milk to determine whether "packaging flavor" off-flavor in milk packaged in half-pint cartons was due to lipid oxidation. Milk was transferred from one-gallon blow-molded HDPE containers into 4000-mL Erlenmeyer flasks, one of which served as a control and the other exposed to 5 hr of direct sunlight between 9 a.m. - 2 p.m. prior to storage to induce lipid oxidation. The flask was gently swirled and rotated 1/4 turn every hour during exposure. Five half-pint Eco Pak® cartons were used in each analysis. All milk samples were evaluated by an individual experienced in the flavor quality of fluid milk prior to analysis after 0, 1, 3, and 6 days storage at 2.2°C ± 1°C. No initial flavor defect was found. Milk subjected to sunlight had a pronounced light-induced off-flavor immediately following exposure. TBA values of each sample were measured three times for each of the three replications.

Statistical Analysis

The TBA test was designed as a randomized complete block model for factor A (day) with factor B (treatment) a split plot on A. Means, standard errors, sums of squares, and mean squares were calculated using the MSTAT-C microcomputer statistical program (Michigan State University, 1988). Tukey's test was then used to determine significance of mean differrences.

Purge and Trap Procedure

A dynamic headspace analysis method (Konczal, 1989), which utilized a purge and trap system, was modified and used to examine for presence of compounds responsible for the "packaging flavor" in water packaged in milk cartons. As used, sodium chloride was omitted and Tenax was transferred directly from the trap into a splitless glass insert without first being extracted with isopentane after 5 hours of purging. Double distlled water packaged in half-pint Eco Pak® cartons (five) with and without heat seal closures as well as water in a glass container was analyzed after one day storage at 2.2°C ± 1°C. Duplicate test runs were conducted.

The purge and trap equipment was based on a design outlined by Konczal (1989). The purge and trap apparatus (Figure 2) was constructed in-house (Department of Chemistry, Michigan State University, East Lansing, MI) and consisted of 2 pieces of equipment: 1) Traps used for collecting volatiles were glass tubes 10 cm in length and 5 mm ID packed with 60-80 mesh Tenax-GC® (Tekmar Company, Cincinnati, OH) and fitted with a 12/5 standard taper ball joint on one end to facilitate attachment to the purging equipment. Silicanized glass wool plugs on both ends held

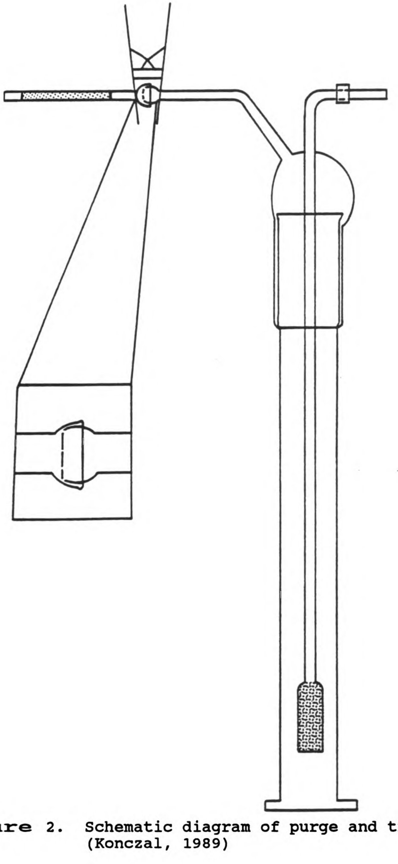

Figure 2. Schematic diagram of purge and trap apparatus (Konczal, 1989)

the absorbent in the trap. 2) Three gas bubbling towers were used to purge volatiles from three different sample solutions. The tower was constructed from a 50-mL graduated cylinder affixed on the top with a 29/42 standard taper gas washing bottle fixture. The inner tube, attached to fritted glass, was extended to the 5-mL mark of the graduated cylinder to insure efficient purging. The trap was clamped to the gas outlet port of the tower and a nitrogen line attached to the gas inlet port of the tower.

All traps were rinsed with distilled acetone and conditioned at 110°C for 24 hr prior to use. Carrier gas was allowed to flow through the traps during conditioning. A 40-mL water sample was purged with nitrogen for 5 hours at a flow rate of 25 mL/min in a 65°C water bath. Nitrogen flow through the trap was controlled using a flow meter (Cole Parmer, Chicago, IL). Following purging, Tenax was transferred from the trap into a splitless glass insert using a glass funnel. Introduction of the glass insert into the gas chromatograph (GC) and GC conditions were the same as described in the following section.

Diffusion Trapping Procedure

The water extracts were also analyzed using a diffusion trapping procedure (Booker and Friese, 1989) modified for use in this analysis. Tenax-GC®, 35-60 mesh (Alltech Associates Inc., Deerfield, IL) was previously conditioned by placement into a 20-m glass column. The column was then connected to the injection port of a Hewlett Packard 5830A GC equipped with a flame ionization detector (FID) (Hewlett-Packard Company, Avondale, PA).

The GC conditions were as follows: injection port 175°C, initial oven temperature 50°C, initial time 30 min, temperature rate 1°C/min to a final temperature of 240°C, and a final time of 600 min, and carrier gas helium flow of 43 mL/min. After conditioning, Tenax was poured into a 40-mL glass vial, which was closed with a screw cap and stored at room temperature for future use.

A 10-μL water sample was placed into a 40-mL glass vial fitted with a Teflon-lined screw cap (Pierce Chemical Company, Rockford, IL) using a 10-μL gas-tight glass syringe (Hamilton Company, Reno, NV). A 12 mm x 75 mm disposable borosilicate glass culture tube containing approximately 0.03 g conditioned Tenax was inserted into the vial. The vial was sealed with a screw cap and then placed in an oven maintained at 90°C for 60 min, removed from the oven, and allowed to sit overnight. The Tenax was transferred immediately into a splitless glass insert and held in place with glass wool plugs on both ends of the insert.

A Hewlett Packard 5890A GC, equipped with a FID and a splitless injection port, and a Hewlett Packard 3390A integrator were used to analyze water extracts. To load the sample, the carrier gas flow was terminated and the glass insert containing Tenax was introduced into the injection port as quickly as possible. The gas flow was then resumed.

All separations were performed using a 30 m by 0.32 mm ID fused silica capillary column packed with Supelcowax™ 10 (Supelco, Inc., Bellefonte, PA).

The GC analytical parameters were as follows: injection port 220°C, initial oven temperature 40°C, initial time 5 min, temperature rate 3°C/min to a final temperature of 165°C for 5 min, and a carrier gas helium flow rate of 2.21 mL/min.

The diffusion trapping procedure was also employed to examine for migration of compounds from the package into the water. Half-pint Eco Pak® cartons with and without heat seal closures, which had previously contained water for one day, empty cartons with heat seal closures, and knocked-down carton blanks were analyzed. The paperboard specimens (10 mm in diameter) were prepared using a cork borer. Specimens were secured from unprinted areas in the center of a front panel, back panel, and the bottom. The sample was weighed and placed with inner PE coating upward in the bottom of the glass vial. After heating the vial, Tenax in the glass tube was transferred to the injection port of the GC as previously described. Diffusion trapping analysis on the water extracts and the carton samples were performed in duplicate.

RESULTS AND DISCUSSION

Trained Panel

Fat Content

"Packaging flavor" development was observed in milk of various fat contents (whole, 2% fat, and skim), which were packaged in half-pint cartons during 6 days storage at 2.2°C as shown in Tables 1, 2, and 3. Whole milk in Eco Pak® cartons and skim milk in standard cross-section cartons were significantly more off-flavored than the controls in glass containers after 1, 3, and 6 days ($\alpha = 0.001$). "Packaging flavor" was more easily detected in skim milk than whole milk. Two percent fat milk in both Eco Pak® and standard cross-section cartons was significantly more off-flavored than the control after 1, 3, and 6 days. With the exception of standard cross-section cartons on day 1 ($\alpha = 0.05$), the level of difference was significant at $\alpha = 0.01$.

The results of this study concur with those of Badings (1971), which showed that "plastic flavor" in milk (3.2%, 1.7%, and 0.25% fat) packaged in PE pouches was more easily detected with decreasing fat content. Thus, milk fat played an important role in masking or diluting this flavor defect.

Bockelmann (1972) and Schönborn *et al.* (1975) reported

Table 1. Paired comparisons evaluation[1] of whole milk samples packaged in half-pint cartons during 6 days storage at 2.2°C

		No. of samples	
Day	Type of package	Total	"Packaging"[2]
1	Eco Pak® vs. glass	27	26[a]
3	Eco Pak® vs. glass	30	28[a]
6	Eco Pak® vs. glass	30	27[a]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[a] significant at $\alpha = 0.001$

Table 2. Paired comparisons evaluation[1] of skim milk samples packaged in half-pint cartons during 6 days storage at 2.2°C

		No. of samples	
Day	Type of package	Total	"Packaging"[2]
1	Std x-sect[3] vs. glass	30	30[a]
3	Std x-sect vs. glass	30	30[a]
6	Std x-sect vs. glass	30	30[a]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[3] std x-sect = standard cross-section
[a] significant at $\alpha = 0.001$

Table 3. Paired comparisons evaluation[1] of 2% fat milk samples packaged in half-pint cartons during 6 days storage at 2.2°C

Day	Type of package		No. of samples: Total	No. of samples: "Packaging"[2]
1	Std x-sect[3]	vs. glass	21	16[c]
	Eco Pak®	vs. glass	21	17[b]
	Eco Pak®	vs. std x-sect	21	13[d]
3	Std x-sect	vs. glass	18	14[b]
	Eco Pak®	vs. glass	18	16[b]
	Eco Pak®	vs. std x-sect	18	10[d]
6	Std x-sect	vs. glass	27	20[b]
	Eco Pak®	vs. glass	27	21[b]
	Eco Pak®	vs. std x-sect	27	14[d]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[3] std x-sect = standard cross-section
[b] significant at $\alpha = 0.01$ by HSD
[c] significant at $\alpha = 0.05$ by HSD
[d] not significant

that milk flavor was superior in Tetra Briks® than in Pure-Paks®. A flavor difference after one day storage was observed (Schönborn *et al.*, 1975). Flückiger (1972) noted increasing oxidative flavor of uperised milk packaged in PE-coated cartons. Milk packaged in plain PE-coated cartons (without aluminum) was organoleptically different from milk in aluminum foil cartons after 3 days. Bojkow *et al.* (1976b) examined 32 PE-coated papers and paperboards and found that 8 produced critical and 2 unacceptable flavor changes in milk (3% fat).

The off-flavor was due to migration of low molecular weight components from the plastic (Wildbrett, 1968; Chuchlowa and Sikora, 1976) as a result of surface oxidation of PE caused by elevated temperatures used during coating operations (Bojkow *et al.*, 1976b; Mannheim *et al.*, 1987).

Storage Time

"Packaging flavor" intensity in 2% fat milk packaged in half-pint Eco Pak® cartons increased during 6 days of storage at 2.2°C (Table 4). Both 3- and 6-day-old milk were significantly more off-flavored than 1-day-old milk ($\alpha = 0.01$). Off-flavor was more easily detected in 6-day-old than 3-day-old milk in comparison with 1-day-old milk. No significant difference in off-flavor intensity was observed when 3- and 6-day-old milk was compared.

Most organoleptic changes occurred during the first 3 days of storage. Flückiger (1972) made a similar

Table 4. Paired comparisons evaluation[1] of 2% fat milk samples packaged in half-pint cartons at several storage times during 6 days storage at 2.2°C

Day of storage	No. of samples Total	"Packaging"[2]
3 vs. 1	30	23[b]
6 vs. 1	30	27[b]
6 vs. 3	30	19[d]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[b] significant at $\alpha = 0.01$ by HSD
[d] not significant

observation with uperised milk packaged in plain PE-coated cartons (without aluminum). The difference in flavor of milk packaged in Pure-Pak® and Tetra Brik® did not increase after prolonged storage (Schönborn *et al.*, 1975). No significant change in off-flavor intensity in milk packaged in PE-coated cartons was observed during one-week storage (Bojkow *et al.*, 1976a).

Size of Container

Container size played an important role in "packaging flavor" development. The extent of off-flavor development was indistinguishable in 2% fat milk packaged in half-pint Eco Pak® and standard cross-section cartons (Table 3). Two percent fat milk packaged in half-pint Eco Pak® cartons exhibited significantly more off-flavor than in quart and half-gallon containers after 6 days ($\alpha = 0.01$) (Table 5). Milk was not significantly more off-flavored in quarts than half gallons.

Half-pint Eco Pak® cartons had a slightly higher surface area in contact with the product than the standard cross-section cartons and much higher contact surface area/volume ratio than quart and half-gallon cartons (Table 6). Flavor problems will increase with the decrease in container size as a result of the increase in surface to volume ratio. This was supported by Mannheim *et al.* (1987).

Table 5. Paired comparisons evaluation[1] of 2% fat milk samples packaged in half-pint (HPT), quart (QT), and half-gallon (HGL) cartons during 6 days storage at 2.2°C

		No. of samples	
Day	Type of package	Total	"Packaging"[2]
1	HPT vs. QT	27	14[d]
	HPT vs. HGL	27	19[c]
	QT vs. HGL	27	17[d]
3	HPT vs. QT	27	18[c]
	HPT vs. HGL	27	17[d]
	QT vs. HGL	27	10[d]
6	HPT vs. QT	27	26[b]
	HPT vs. HGL	27	25[b]
	QT vs. HGL	27	12[d]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[b] significant at $\alpha = 0.01$ by HSD
[c] significant at $\alpha = 0.05$ by HSD
[d] not significant

Table 6. Contact surface area/volume ratios for the polyethylene-coated paperboard cartons

Type of package	Volume (mL)	Area (cm^2)	Area/volume (cm^2/mL)
Half-pint			
Eco Pak®	236	199	0.84
Standard cross-section	236	186	0.72
Quart	946	556	0.59
Half-gallon	1892	845	0.45

Heat Sealing

To isolate the effect of the packaging material from possible contribution of heat sealing of the package, the bottom of carton blanks were closed with a circular glass piece and the top sealed with tape. Significantly more "packaging flavor" development occurred in 2% fat milk packaged in half-pint Eco Pak® cartons with and without heat seal closures than in glass containers during 3 days storage ($\alpha = 0.01$) (Table 7). There was no significant difference in the off-flavor intensity in cartons with and without heat seal closures. Because of the results obtained, no sensory test was conducted on day 6. This off-flavor was, therefore, not caused by excessively high heat sealing temperatures during fabrication of the carton.

Water

Water was used to evaluate the effect of packaging material in contact with a product which did not contain fat on the organoleptic quality of the product. Similar results were obtained for water as for 2% fat milk packaged in both heat sealed and non-heat sealed cartons (Table 8). Water was significantly more off-flavored in cartons than in glass containers after one day storage ($\alpha = 0.01$). No significant difference in flavor was observed in heat sealed and non-heat sealed cartons. Similar results were reported by Berg (1980) who also observed significant differences in

Table 7. Paired comparisons evaluation[1] of 2% fat milk samples packaged in half-pint cartons with and without heat seal closures during 3 days storage at 2.2°C

		No. of samples	
Day	Type of package	Total	"Packaging"[2]
1	Heat seal vs. glass	27	24[b]
	Non-heat seal vs. glass	27	22[b]
	Heat seal vs. non-heat seal	27	18[d]
3	Heat seal vs. glass	27	23[b]
	Non-heat seal vs. glass	27	27[b]
	Heat seal vs. non-heat seal	27	16[d]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[b] significant at $\alpha = 0.01$ by HSD
[d] not significant

Table 8. Paired comparisons evaluation[1] of water samples packaged in half-pint cartons with and without heat seal closures after 1 day storage at 2.2°C

Day	Type of package	No. of samples	
		Total	"Packaging"[2]
1	Heat seal vs. glass	27	24[b]
	Non-heat seal vs. glass	27	25[b]
	Heat seal vs. non-heat seal	27	18[d]

[1] evaluated by 10-member trained panel
[2] the first has more "packaging flavor" than the second
[b] significant at $\alpha = 0.01$ by HSD
[d] not significant

water stored in glass containers and PE-coated cartons.

Kiermeier and Stroh (1969) reported that autoxidation of PE produced unbranched carboxylic acids in water, which could be responsible for the "plastic flavor" in milk packaged in PE bags. Methane, ethane, propane, n-butane, and 1-butene were detected in water in contact with PE. Water developed off-flavor when in contact with PE granules (Peled and Mannheim, 1977) and PE strips (Mannheim *et al.*, 1987). The migrants from the plastic were mostly water soluble (Bojkow *et al.*, 1979).

Bojkow *et al.* (1976a) reported that no significant change in off-flavor intensity in water occurred during one-week storage. Since the water acquired a pronounced off-flavor after one day storage, no further sensory testing was conducted. The results confirmed previous findings in this study, which showed that the off-flavor found in milk was neither due to lipid oxidation nor was it caused by excessively high heat sealing temperatures during package fabrication and closure.

Consumer Panel

A consumer panel was assembled which consisted of 91 members, of which 44 were males and 47 females, between the ages of 18 - 50 (Appendix G). A triangle test was employed to determine if the panelists could detect a difference between milk stored in half-pint Eco Pak® cartons and glass containers after 3 days storage at 2.2°C. Thirty four panelists correctly identified the odd sample. Preference evaluation was also obtained using a paired preference test. Fifty panelists preferred the control milk sample. Contrary to results obtained using a trained panel, no significant difference and/or preference were observed ($p = 0.05$) (Appendix H).

On the other hand, school age children may be able to detect "packaging flavor" in milk and may find this unacceptable. A direct correlation was shown between the flavor and consumption of milk by school age children (Bandler *et al.*, 1975). Milk drinking habits are established during early school years. Adult consumers, however, may be conditioned to off-flavor(s) since milk purchased at the retail level is usually 3 - 4 days old and the majority of panelists buy milk packaged in one-gallon or half-gallon blow-molded HDPE containers (Appendix G). Milk frequently develops an undesirable off-flavor when stored in PE bottles and PE-coated cartons. Consumers often describe this off-flavor as "unpleasant plastic" (Berg, 1980).

TBA Test

The results of the TBA test are shown in Table 10. The TBA values of whole milk samples in cartons and glass containers did not significantly increase during 6 days storage at 2.2°C ($p < 0.05$). "Packaging flavor" was found in cartoned milk during this storage period. Milk, which was exposed to sunlight for 5 hr to induce lipid oxidation,

Table 9. Analysis of variance for TBA values of whole milk samples packaged in half-pint cartons during 6 days storage at 2.2°C

Source of variation	Degree of freedom	Sum of squares	Mean square	F value	Probability
Replication (R)	2	0.00025	0.00013	2.99	> 0.05
Day (D)	3	0.00056	0.00019	4.47	< 0.05
Treatment (T)	2	0.00030	0.00015	3.62	< 0.05
DT	6	0.00052	0.00009	2.07	> 0.05
Error	22	0.00092	0.00004		
Total	35				

Table 10. TBA values of whole milk samples packaged in half-pint cartons during 6 days storage at 2.2°C

	Optical density at 532 μm[1,2]		
Day	Control	Carton	Sunlight[3]
0	0.012a	0.014a	0.014b
1	0.015a	0.009a	0.016b
3	0.012a	0.016a	0.033a
6	0.022a	0.021a	0.023ab

[1] all values represent the average of three replicated experiments

[2] means within each column represented by the same letter are not significantly different at $p < 0.05$ by Tukey's test

[3] milk in glass containers exposed to 5 hr sunlight prior to storage

exhibited a pronounced light-induced off-flavor immediately following exposure and throughout the storage period. A significant increase in TBA value was observed on day 3. Thus, the TBA test was able to monitor lipid oxidation. Therefore, this confirmed the sensory findings in that the "packaging flavor" associated with cartoned milk was different from flavors caused by lipid oxidation.

Analysis of variance (ANOVA) of the TBA values was performed based on a randomized complete block model with a split plot. Since the values of mean square errors were relatively close, the data were analyzed using a two factor randomized complete block model. The ANOVA (Table 9) of the results showed significant effects ($p < 0.05$) of days (storage times) and treatments.

Analytical Methods

A modification of a purge and trap procedure (Konczal, 1989) was employed to look for compounds which could be responsible for the "packaging flavor" in water packaged in cartons after one day storage at 2.2°C. There were no marked differences in retention times between glass-stored water and water packaged in heat sealed and non-heat sealed cartons after one day storage (Appendix J). Differences in peak heights were observed. Due to variation in the results, no attempt was made to identify the components. A modification of a diffusion trapping procedure (Booker and Friese, 1989) was then utilized to analyze water extracts of the cartons and paperboard samples to determine compounds migrating from the package. Results obtained using this method were likewise inconsistent (Appendices J and K).

Bojkow *et al.* (1976c) reported that IR spectroscopy seemed to be the most useful tool for differentiation of satisfactory PE films and those which induced off-flavor. Direct identification of flavor inducing substances by gas chromatography and thin layer chromatography was unsuccessful. An IR spectrum of PE film with "critical" organoleptic quality showed a distinct C=O band at 1720 cm^{-1} (5.8 μm).

Bojkow *et al.* (1979) found that the flavor components migrating from the internal PE coating of the package were largely soluble in water and quite volatile. Even in successive sections of a continuously processed sheeting of packaging material or in successive blanks within a shipping box these components may be present in widely different concentrations. The total release of volatile substances and the total residue in the water extract correlated with the area of gas liquid chromatographic peaks of the ether soluble components of the water extract but not with the intensity of the off-flavor. Gas liquid chromatography/mass spectroscopy (GLC/MS) of headspace volatiles and the ether soluble part of the water extract as well as hexane extracts resulted in separation of 120 components in the ppb range. The following organoleptically suspect classes of substances were isolated: unsaturated aliphatics, free fatty acids, and alcoholic compounds. These occurred frequently in materials which were responsible for the off-flavor. For the GLC/MS analysis of these substances the ether soluble part of the water extract was the most suitable.

Peled and Mannheim (1977) found no extractable matter from 7 types of PE granules in water, although the water had considerable off-flavor. This was probably due to the low concentration of the constituents which caused the off-flavors. Injection of the water extract in the GLC gave inconclusive results.

Berg (1980) investigated the origin of off-flavors from PE-coated cartons using a headspace GC technique, combined with "on-line" sniffing and identification of components by MS. Almost every volatile component found in PE-coated bleached paperboards was present in the PE granules. Intense plastic odor was also observed in both PE-coated paperboards and the granulates but not in uncoated paperboards. The dominant plastic off-flavor peaks had retention times starting at 20 minutes. The component which gave the very intense plastic off-flavor was (C4) alkenylbenzene.

SUMMARY AND CONCLUSIONS

"Packaging flavor" developed in whole, 2% fat, and skim milk and water packaged in half-pint PE-coated paperboard containers after 1 day storage at 2.2°C. Off-flavor was more easily detected with decreasing fat content. This was confirmed by results of the TBA test, which indicate that off-flavor was not caused by lipid oxidation. No significant increase in the off-flavor intensity was observed in 2% fat milk packaged in half-pint Eco Pak® cartons after 3 days.

There was no significant difference in the off-flavor in 2% fat milk packaged in half-pint Eco Pak® and standard cross-section cartons. Two percent fat milk in the Eco Pak® cartons exhibited significantly more off-flavor than in quart and half-gallon cartons after 6 days.

No significant difference in the off-flavor was observed in 2% fat milk or water packaged in half-pint Eco Pak® cartons with and without heat seal closures. This off-flavor was, therefore, not caused by excessively high heat sealing temperatures.

The consumer panel did not find differences among the milk samples stored in half-pint Eco Pak® cartons and glass containers and showed no preferences between the samples.

Results obtained in analysis of water extracts and PE-coated paperboard materials using a modified purge and trap and diffusion trapping procedures were inconsistent.

RECOMMENDATIONS

The following are recommendations for future research:

1. A survey of school children should be conducted at schools throughout Michigan and neighboring states to determine whether "packaging flavor" is a consumer perceived problem.

2. Identification of the component(s) of the package responsible for the "packaging flavor" development.

3. Following identification of the off-flavor components, methods and/or materials which will reduce or eliminate "packaging flavor" should be developed.

APPENDICES

APPENDIX A

Codesheet for Trained Panel

Date:

Type of samples: 2% fat milk after 1 day storage at 2.2°C

Type of test: Paired comparisons

Sample identification			Code
793	418	(1/2 pt standard cross-section)	A
369	927	(1/2 pt Eco Pak®)	B
143	681	(glass)	C

Code serving containers as follows:

Panelist No.	Order of Presentation			Panelist No.	Order of Presentation		
1	A B	A C	B C	2	B A	C A	C B
3	A C	A B	B C	4	C A	B A	C B
5	A C	B C	A B	6	C A	C B	B A
7	B C	A C	A B	8	C B	C A	B A
9	B C	A B	A C	10	C B	B A	C A

APPENDIX B

Sensory Evaluation Sheet for Trained Panel

Taste panelist no.

Sensory Evaluation Sheet

for

2% Fat Milk

Name ______________________________ Date ____________________

Please taste the samples in the order listed below.

Determine which of the two samples is more "cardboardy" and indicate by placing an X next to the code.

Sample	More "cardboardy"[1]	Comments
______	__________	__________________________
______	__________	__________________________
______	__________	__________________________
______	__________	__________________________
______	__________	__________________________
______	__________	__________________________

[1] the term "cardboardy" was changed to "packaging flavor" (American Society for Testing and Materials, 1984) at the conclusion of the study

APPENDIX C

Statistical Analysis of Trained Panel Data

Type of samples: 2% fat milk after 1 day storage

Type of sensory test: Paired comparisons

Test objective: To compare the intensity of "packaging flavor" in milk packaged in Eco Pak® and standard cross-section cartons and milk in a glass container (control)

Type of statistical test: Pairwise Ranking Test - Friedman Analysis

Analysis of results:

The table below shows the number of times (out of 21) each "row" sample was chosen as being more off-flavored than each "column" sample.

		Column samples (less off-flavored)		
		A	B	C
Row samples	A	-	8	16
(more	B	13	-	17
off-flavored)	C	5	4	-

A = 1/2 pt standard cross-section
B = 1/2 pt Eco Pak®
C = glass

To compute the rank sum for each sample, the rank sum of one is assigned to the more off-flavored and the rank of two to the less off-flavored sample. The rank sums are then obtained by adding the sum of the row frequencies to twice the sum of the column frequencies, e.g., for sample A, (8 + 16) + 2(13 + 5) = 60:

Sample	A	B	C
Rank sum	60	54	75

The test statistic, Friedman's T, is computed as follows:

$$T = (4/pt)\sum_{i=1}^{t} R^2 - (9p[t-1]^2)$$

$$= [4/(21)(3)][60^2 + 54^2 + 75^2] - [9(21)(2^2)] = 14.86$$

where p = the number of times the basic design is repeated
t = the number of treatments
R_i = the rank sum for the i'th treatment
ΣR^2 = sum of all Rs squared, from R_1 to R_t

The value of T is compared to the critical value of χ^2 with (t-1) degrees of freedom (Table T5). The critical Ts are

Level of significance, α	0.10	0.05	0.01
Critical T	4.61	5.99	9.21

The results can be shown on a rank sum scale of more off-flavored vs. less off-flavored:

```
    more      -+-----6+0---------70----+----80-     less
off-flavored B      A                C        off-flavored
```

On the same scale, the HSD value for comparing two rank sums (α = 0.05) is:

$$HSD = q_{\alpha,t,\infty} \sqrt{pt/4} = 3.31\sqrt{(21)(3)/4} = 13.14$$

(where the value $q_{\alpha,t,\infty}$ is found in Table T14).
The difference between A and C is larger than 13.14 and that between B and C is much larger than 13.14. However, the difference between A and B is much smaller than 13.14.

Thus, A and B are significantly more off-flavored than C, however, B is not significantly more off-flavored than A.

(Meilgaard *et al.*, 1987b)

Statistical Analysis of Trained Panel Data (cont'd)

Type of samples: Whole milk after 1, 3, and 6 days storage

Type of sensory test: One-sided paired comparisons

Test objective: To compare the intensity of "packaging flavor" in milk packaged in Eco Pak® cartons and milk in a glass container (control)

Type of statistical test: One-sided paired comparisons

Analysis of results:

The table below shows the number of milk samples packaged in cartons chosen as being more off-flavored:

Day	Total	"Packaging flavor"
1	27	26
3	30	28
6	30	27

The number of correct responses on day 1 is compared to the critical number of correct answers with n = 27 (Table T8). The critical numbers are

Level of significance, α	0.05	0.01	0.001
Critical number	19	20	22

The numbers of correct responses on day 3 and 6 are compared to the critical number of correct answers with n = 30 (Table T8). The critical numbers are

Level of significance, α	0.05	0.01	0.001
Critical number	20	22	24

The difference in "packaging flavor" was perceived at $\alpha = 0.001$.

(Meilgaard *et al.*, 1987b)

APPENDIX D

Codesheet for Consumer Panel

Date:

Type of samples: 2% fat milk after 3 days storage

Type of test: Triangle and pair preference tests

Sample identification			Code
467	813	(glass)	A
168	793	(1/2 pt Eco Pak®)	B
587		(glass)	C
973		(1/2 pt Eco Pak®)	D

Code serving containers as follows:

Panelist No.	Order of Presentation		Panelist No.	Order of Presentation	
1	A B B	C D	2	B B A	D C
3	B A B	C D	4	B A B	D C
5	B B A	C D	6	A B B	D C
7	A A B	C D	8	B A A	D C
9	A B A	C D	10	A B A	D C
11	B A A	C D	12	A A B	D C
13	C D	A B B	14	D C	B B A
15	C D	B A B	16	D C	B A B
17	C D	B B A	18	D C	A B B
19	C D	A A B	20	D C	B A A
21	C D	A B A	22	D C	A B A
23	C D	B A A	24	D C	A A B

APPENDIX E

Sensory Evaluation Sheets for Consumer Panel

Taste panelist no.

Sensory Evaluation Sheet
for
2% Fat Milk

Please taste the three coded samples in the order listed below. Rinse your mouth with the water provided after you have finished with each sample. Two samples are identical; one is different. Select the **odd/different** sample and indicate by placing an X next to the code of the odd sample.

Sample	Odd sample	Comments
______	__________	____________________________
______	__________	____________________________
______	__________	____________________________

Please continue on the next page --->

Taste panelist no.

Sensory Evaluation Sheet

for

2% Fat Milk

Please taste the two coded samples in the order listed below. Rinse your mouth with the water provided after you have finished with each sample. Place an X next to the code of the sample that you prefer.

Sample	Preference	Comments
______	__________	____________________________
______	__________	____________________________

Please continue on the next page --->

APPENDIX F

Demographic Questionnaire Sheet for Consumer Panel

PLEASE CIRCLE THE CORRECT INFORMATION BELOW ABOUT YOURSELF.

SEX:	male	female		
AGE RANGE:	18-25	26-35	36-50	over 50
STATUS:	faculty	staff	student	

others (please specify) ____________________

TYPE(S) OF MILK REGULARLY CONSUMED:

whole	2% fat	1/2% fat	skim

others (please specify) ____________________

TYPE(S) OF CONTAINER(S) OF MILK REGULARLY PURCHASED:

	plastic bottle	paperboard carton	others (please specify)
			____________________
SIZE(S):	gallon		____________________
	half-gallon	half-gallon	
	quart	quart	
	pint	pint	
	half-pint	half-pint	

THANK YOU FOR PARTICIPATING IN THIS TASTE PANEL.

ENJOY YOUR TREAT!

APPENDIX G

Demographic Data of Consumer Panel

NUMBER OF PANELISTS: 91

SEX:	male	female		
	44	47		
AGE RANGE:	18-25	26-35	36-50	over 50
	44	23	18	6
STATUS:	faculty	staff	student	others
	18	14	55	4

TYPE(S) OF MILK REGULARLY CONSUMED:

whole	2% fat	1/2% fat	skim
12	54	24	19

TYPE(S) AND SIZE(S) OF CONTAINER(S) OF MILK REGULARLY PURCHASED:

	plastic bag	plastic bottle	paperboard carton
gallon		53	
half-gallon	3	30	8
quart		3	7
pint		1	1
half-pint			

APPENDIX H

Statistical Analysis of Consumer Panel Data

No. of panelists correctly identified the odd sample 34 n.s.
No. of panelists preferred milk stored in glass 50 n.s.

n.s. = not significant

TRIANGLE TEST (Table T7)

No. of respondents	Minimum no. of responses required for significance 5%	1%	0.1%
90	38	42	45

TWO-SIDED PAIRED COMPARISON TEST (Table T9)

No. of respondents	Minimum no. of responses required for significance 5%	1%	0.1%
92	56	59	63

(Meilgaard *et al.*, 1987b)

APPENDIX I

Data File for Statistical Analysis of TBA Values

Data for TBA test was entered into the computer under the following format:

Case No.	Variable 1	2	3	4	5
1	1	1	1	0.014	1.400

Key:

Case No.: data points 1 - 36

Variable: 1 = day
2 = treatment
3 = replicate
4 = TBA value
5 = TBA value * 100

Day: 1 = 0 day
2 = 1 day
3 = 3 days
4 = 6 days

Treatment: 1 = control
2 = carton
3 = 5 hr sunlight exposure

Replicate: 1 = 1
2 = 2
3 = 3

TBA value: optical density at 532 μm

The complete data file is represented on the following page.

Case No.	Variable 1	2	3	4	5
1	1	1	1	0.014	1.400
2	1	1	2	0.015	1.500
3	1	1	3	0.006	0.600
4	1	2	1	0.015	1.500
5	1	2	2	0.021	2.100
6	1	2	3	0.007	0.700
7	1	3	1	0.017	1.700
8	1	3	2	0.014	1.400
9	1	3	3	0.010	1.000
10	2	1	1	0.025	2.500
11	2	1	2	0.019	1.900
12	2	1	3	0.002	0.200
13	2	2	1	0.018	1.800
14	2	2	2	0.001	0.100
15	2	2	3	0.009	0.900
16	2	3	1	0.012	1.200
17	2	3	2	0.021	2.100
18	2	3	3	0.015	1.500
19	3	1	1	0.014	1.400
20	3	1	2	0.012	1.200
21	3	1	3	0.011	1.100
22	3	2	1	0.024	2.400
23	3	2	2	0.013	1.300
24	3	2	3	0.011	1.100
25	3	3	1	0.035	3.500
26	3	3	2	0.034	3.400
27	3	3	3	0.030	3.000
28	4	1	1	0.028	2.800
29	4	1	2	0.024	2.400
30	4	1	3	0.013	1.300
31	4	2	1	0.023	2.300
32	4	2	2	0.009	0.900
33	4	2	3	0.032	3.200
34	4	3	1	0.024	2.400
35	4	3	2	0.019	1.900
36	4	3	3	0.026	2.600

Note: Variable 5 was used in the analysis.
Sums of squares and mean squares in the ANOVA table were corrected by dividing the values by 10,000.

APPENDIX J

Chromatograms of Water Extracts

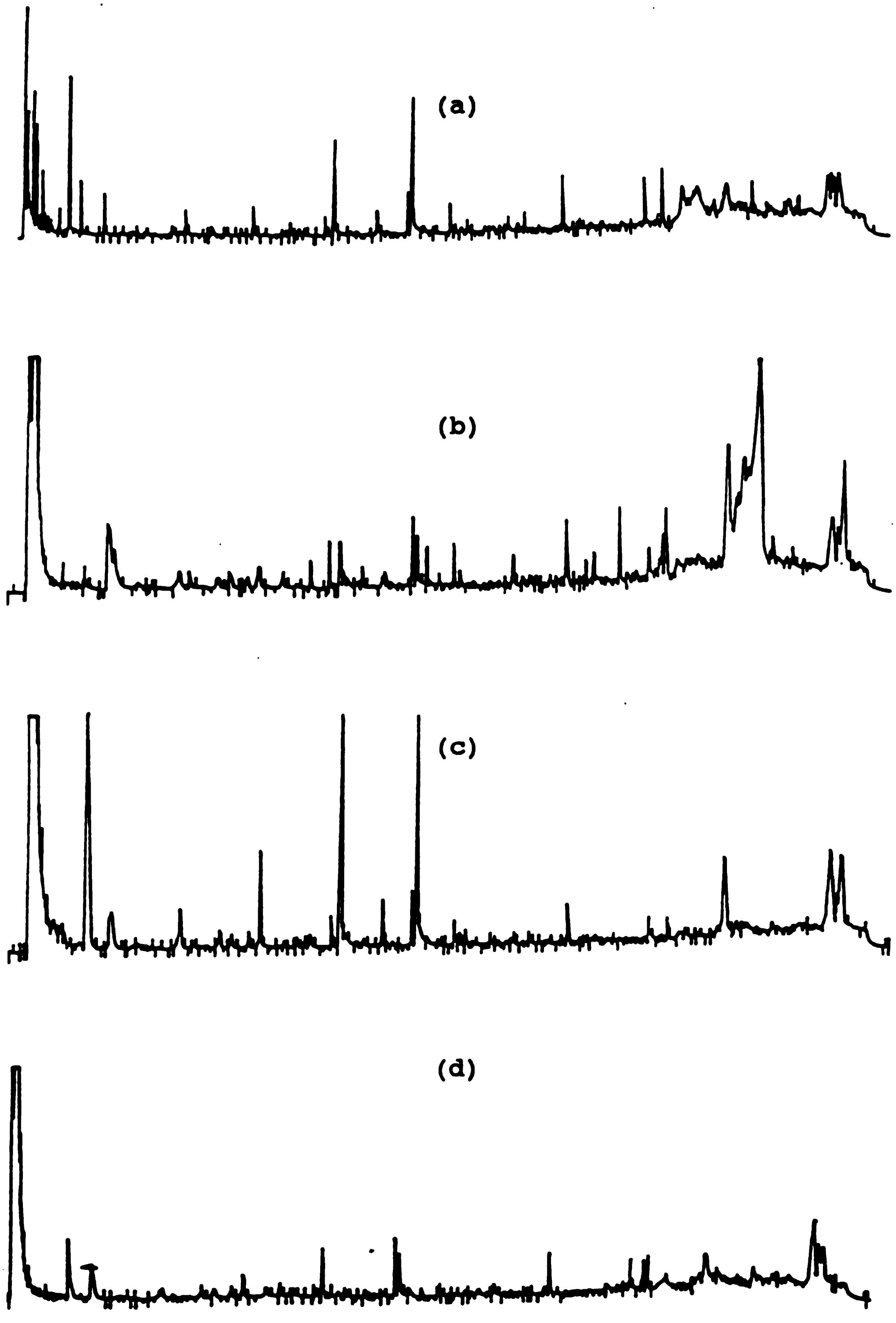

Figure 3. Water packaged in (a) glass containers, (b) cartons with heat seal closures, and (c) and (d) cartons without heat seal closures using purge and trap procedure

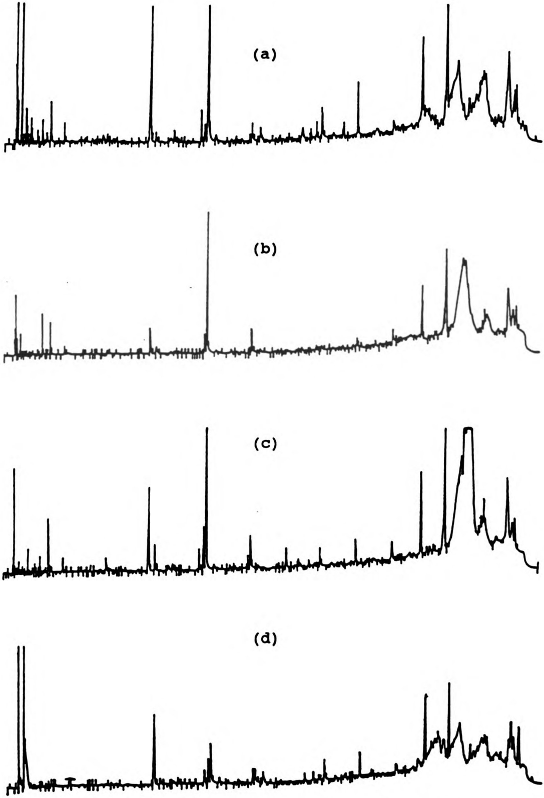

Figure 4. Water packaged in (a) glass containers, (b) cartons with heat seal closures, and (c) and (d) cartons without heat seal closures using diffusion trapping procedure

APPENDIX K

Chromatograms of Paperboard Specimens

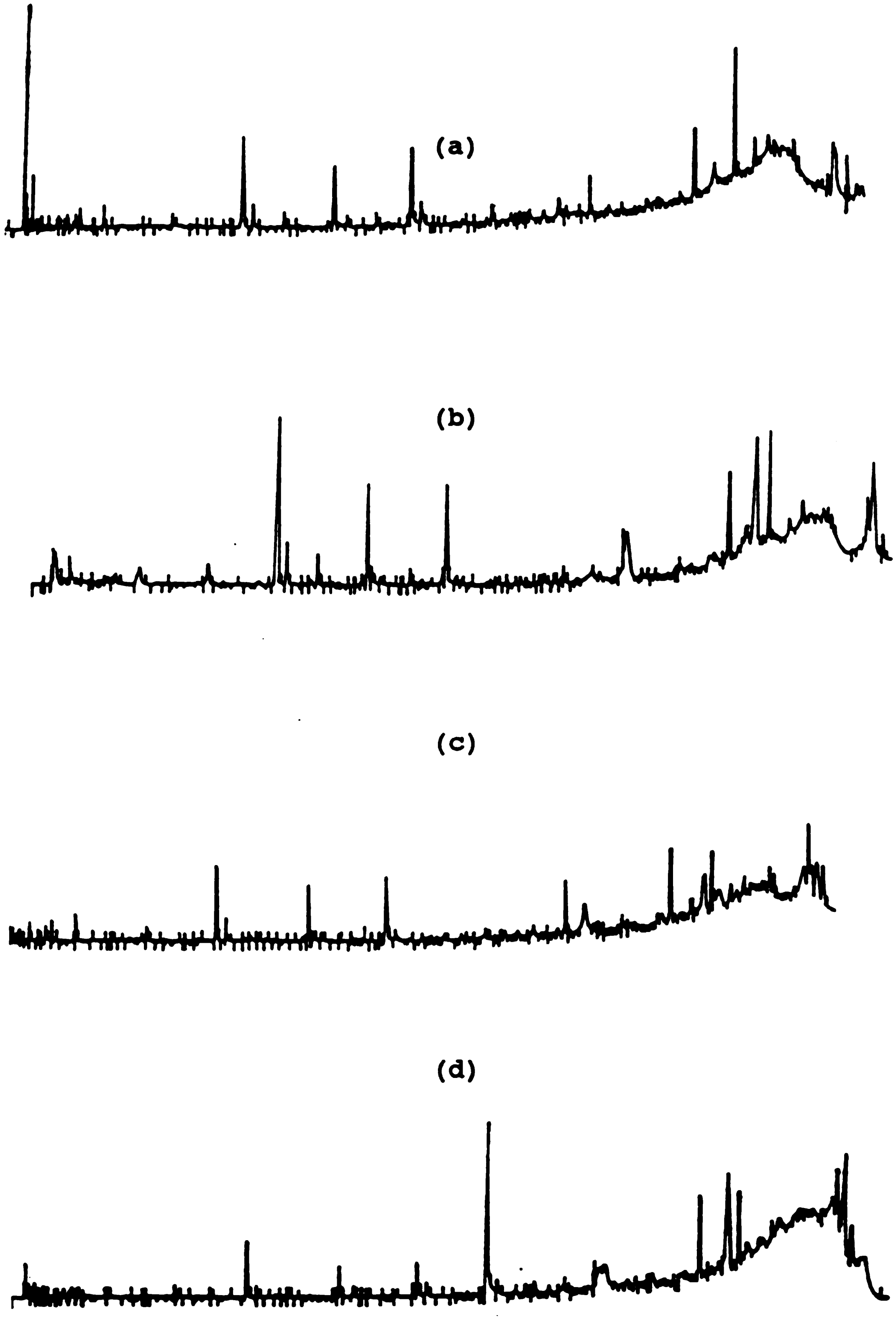

Figure 5. Unprinted paperboard specimens from
(a) front panel, (b) back panel, and
(c) and (d) bottom of knocked-down carton blanks

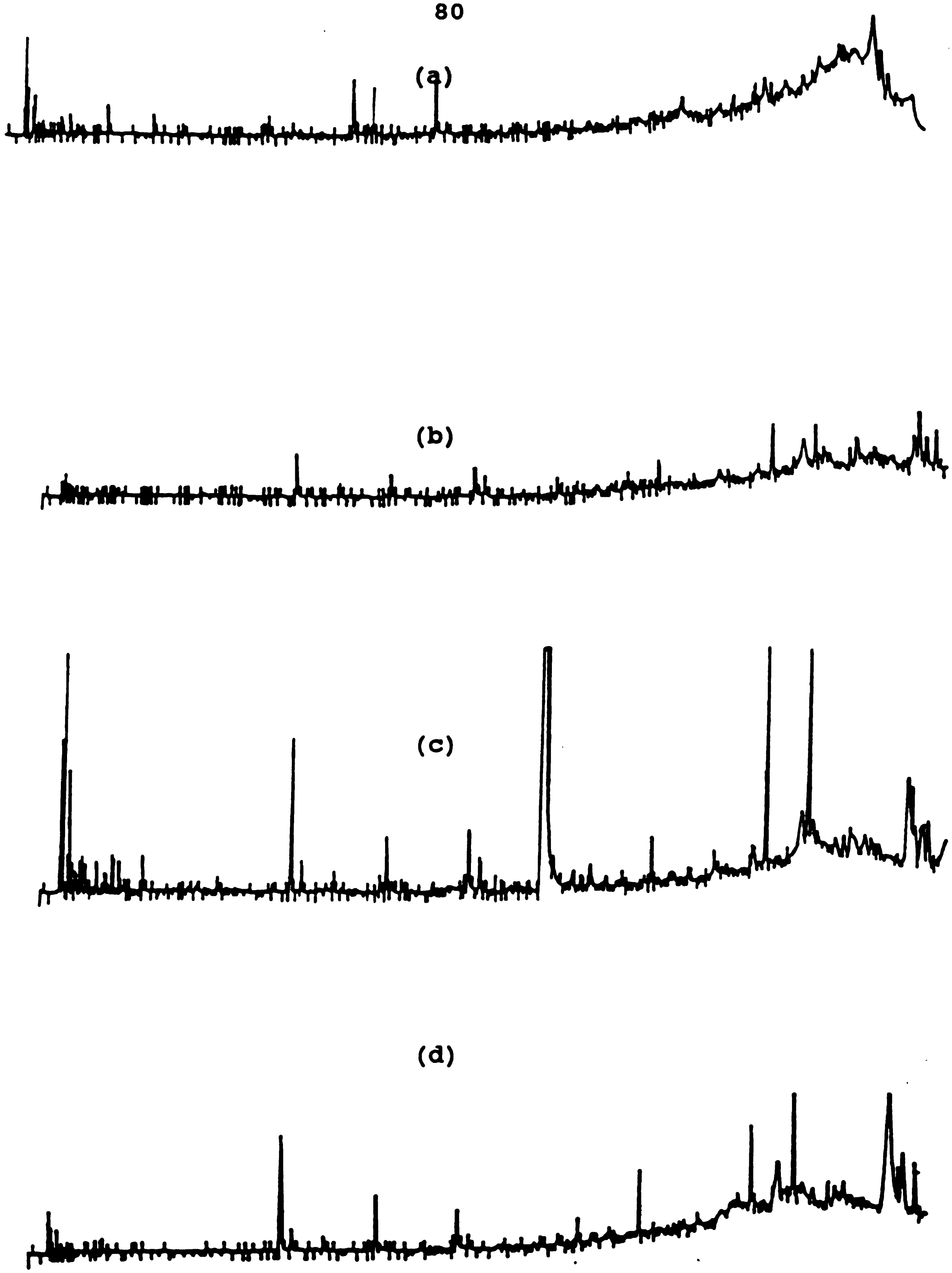

Figure 6. Unprinted paperboard specimens from (a) front panel, (b) back panel, and (c) and (d) bottom of cartons with heat seal closures

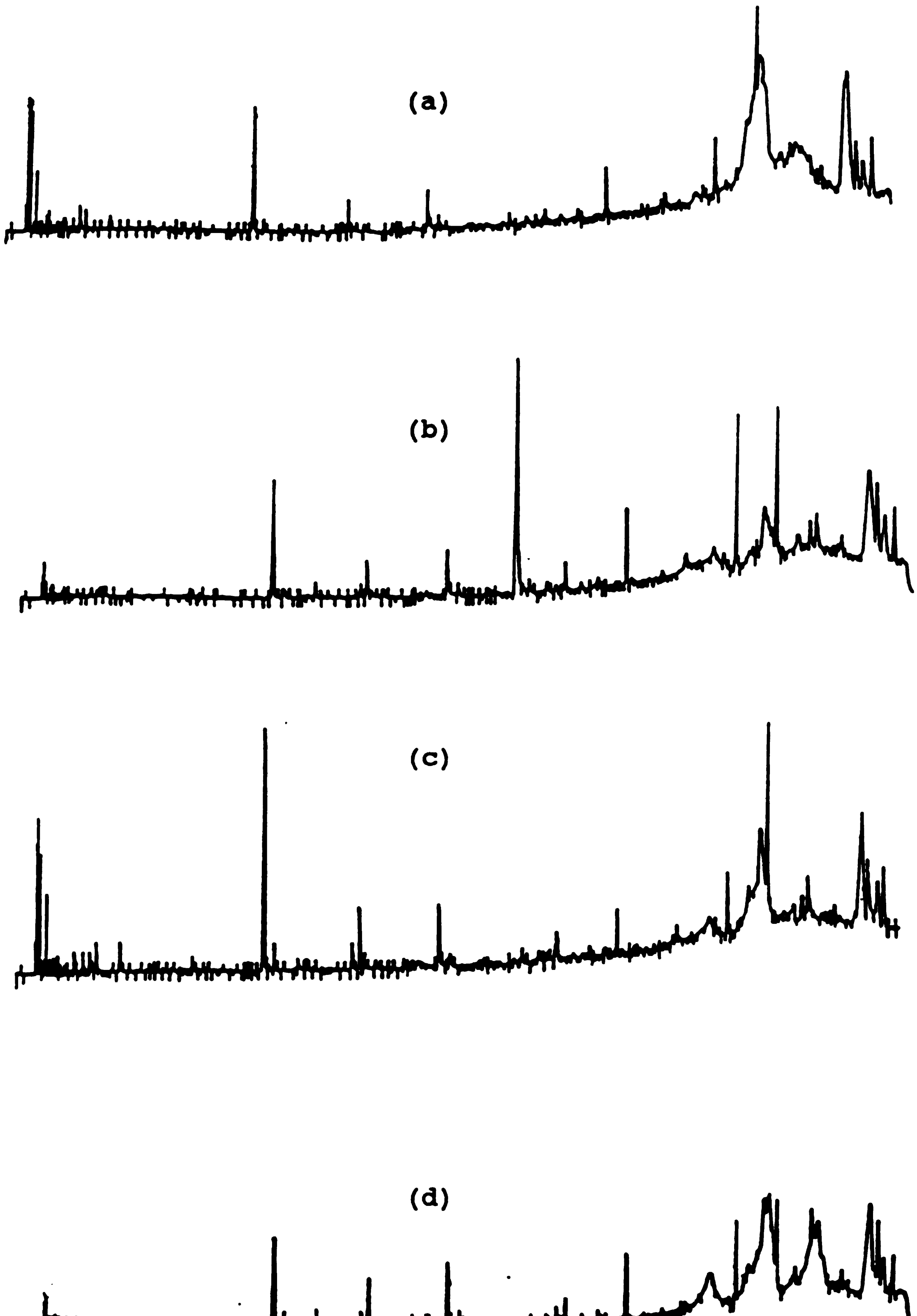

Figure 7. Unprinted paperboard specimens from (a) front panel, (b) back panel, and (c) and (d) bottom of cartons with heat seal closures previously contained water

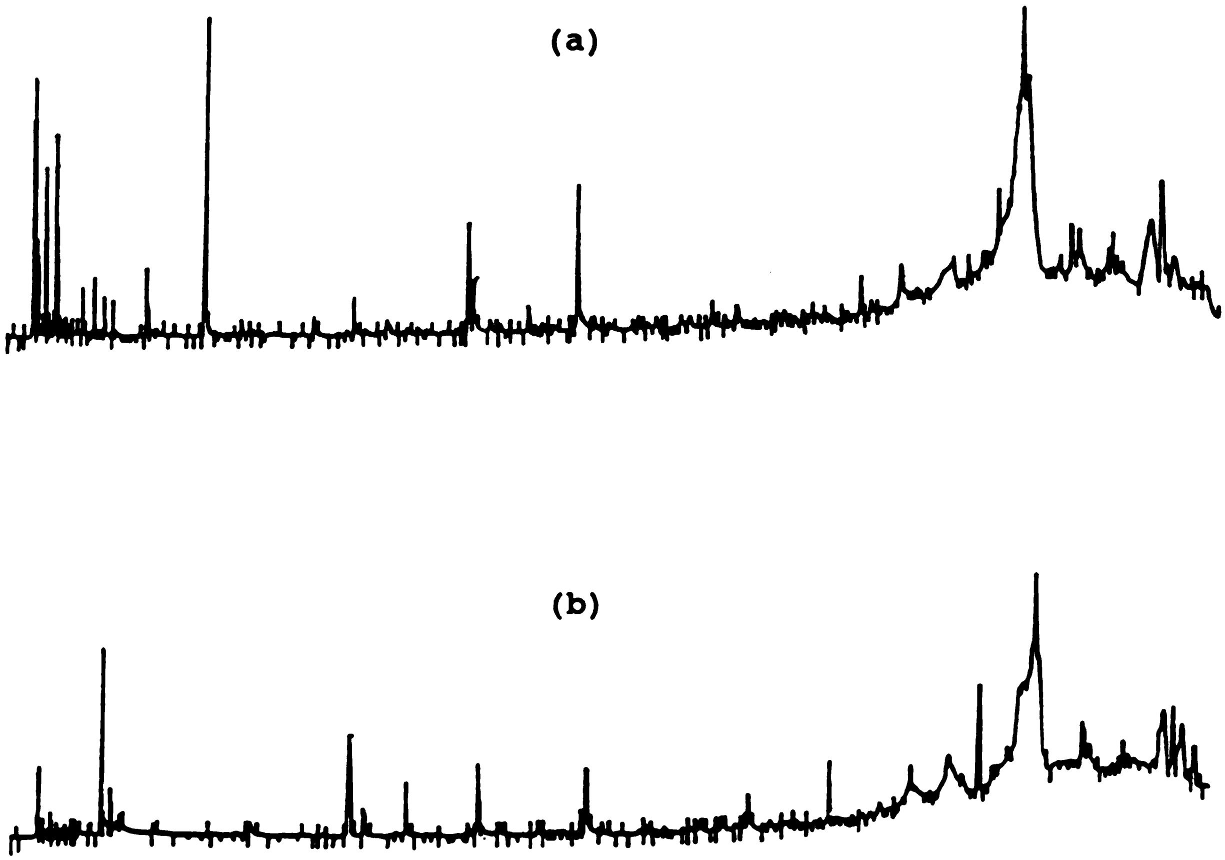

Figure 8. Unprinted paperboard specimens from (a) front panel and (b) back panel of cartons without heat seal closures previously contained water

APPENDIX L

Calculations for Contact Surface Area of PE-coated Paperboard Cartons

Half-pint Eco Pak® carton

Contact surface area, A_C, is

$$A_C = A_l + A_b$$

where A_l = lateral area
= area of four rectangles
= 4 x bh
= 4 x (5.7)(7.3)
= 4 x 41.61
= 166.44 cm^2

A_b = area of the base
= b^2
= $(5.7)^2$
= 32.49 cm^2

$$A_C = 166.44 + 32.49$$
$$= 198.93 \text{ cm}^2$$

Half-pint standard cross-section carton

Contact surface area, A_C, is

$$A_C = A_l + A_b$$

where A_l = lateral area
= area of four rectangles
= 4 x bh
= 4 x (7.0)(4.9)
= 4 x 34.30
= 137.20 cm^2

A_b = area of the base
= b^2
= $(7.0)^2$
= 49.00 cm^2

$$A_C = 54.60 + 137.20 + 49.00$$
$$= 186.20 \text{ cm}^2$$

Quart carton

Contact surface area, A_C, is

$$A_C = A_l + A_b$$

where A_l = lateral area
= area of four rectangles
= 4 x bh
b = base (cm)
h = height (cm)
= 4 x (7.1)(17.8)
= 4 x 126.38
= 505.52 cm^2

A_b = area of the base
= b^2
= $(7.1)^2$
= 50.41 cm^2

$$A_C = 505.52 + 50.41 = 555.93 \text{ cm}^2$$

Half-gallon carton

Contact surface area, A_C, is

$$A_C = A_l + A_b$$

where A_l = lateral area
= area of four rectangles
= 4 x bh
b = base (cm)
h = height (cm)
= 4 x (9.6)(19.6)
= 4 x 188.16
= 752.64 cm^2

A_b = area of the base
= b^2
= $(9.6)^2$
= 92.16 cm^2

$$A_C = 752.64 + 92.16 = 844.80 \text{ cm}^2$$

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