This is to certify that the

thesis entitled

DETECTION OF VOLATILES EVAPORATING FROM
A PLASTIC CONTAINER AFTER BEING MICROWAVED

presented by

LYNN ELIZABETH DIXON

has been accepted towards fulfillment
of the requirements for

M.S. degree in PACKAGING

Bruce Harte
Dr. Bruce R. Harte
Major professor

Date July 23, 1987

O-7639

DETECTION OF VOLATILES EVAPORATING FROM A PLASTIC CONTAINER AFTER BEING MICROWAVED

By

Lynn Elizabeth Dixon

A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

School of Packaging

1987

ABSTRACT

DETECTION OF VOLATILES EVAPORATING FROM A PLASTIC CONTAINER AFTER BEING MICROWAVED.

By

Lynn Dixon

A coextruded plastic container was cut up and placed into a glass vial which was then sealed. The vial was suspended over the microwave floor and microwaved on full power for periods of time ranging from 3 to 7 minutes. The temperature the plastic surface experienced over the time range was approximated with the use of melting point standards waxes. Five major components were detected and quantified in the headspace of the vial using a gas chromatograph. The quantity of each component increased as the microwaving time increased. Using a mass spectrometer the five components were tentatively identified; four of the components were identified as hydrocarbons and the fifth component was positively identified as butylated hydroxytoluene.

To Gene, my mother and father for their support.

ACKNOWLEDGEMENTS

I would like to thank the many people which helped me with my thesis research. I sincerely appreciate the time and guidance that Dr. Bruce R. Harte gave to me throughout the graduate program. I would also like to thank Ruben Hernandez for his advice on the devising of the experimental design and the analysis of the results.

Many thanks to Kevin Slatkavitz from Pillsbury and Tom Cotton from Gerger for helping me with the Mass Spectrum interpretation. To Ball Plastics I extent an appreciation for donating the plastic containers used in this research.

Finally, I would like to express a deep appreaciation to my family and my friends for their support during this endeavor.

TABLE OF CONTENTS

Literature Review

Materials and Methods

TABLE OF TABLES

Appendix D

Appendix E.

LIST OF FIGURES

Appendix A

INTRODUCTION

Microwaving is becoming more commonplace in both the industrial and home sectors. Market penetration of microwave ovens in the home sector has reached about 60% and by 1990 it is estimated that 70 to 90% of all homes will have at least one microwave oven (Perry 1986). "In industry, microwave processes are slowly gaining acceptance by the food industry for various processing operations such as blanching, cooking, drying, pasteurizing, sterilizing and thawing of bulk food products. Perhaps the most widely accepted microwave food processes are the precooking of some meat products and the tempering or thawing of frozen foods" (Mudgett 1986).

There exists a large market potential for microwave products as people look for ways to save time in food preparation. The typical owner of a microwave is a woman who is well educated, sophisticated and leads an extremely active life. The microwave oven fits into her life by enabling her to prepare food quickly and easily (Nelson 1982).

Because of this market potential food companies are continuing to develop and market microwaveable products.

Therefore a real need exists for microwaveable packages. The packaging engineer must design packages which can overcome potential problems due to microwaving. Perry (1986) suggests that areas to be evaluated specific to microwave food preparation in a package are venting, arcing, uniform heating, handling, eruption, and indirect additives. Adequate venting must be provided to avoid possible steam burns. Arcing between the packaging structure and the oven should be avoided to prevent damage to the oven. Controlling the energy conversion is important to accomplish proper and uniform heating. Physical hazards may arise if eruptions occur as viscous materials reach their boiling point. Packages should be designed so that they may be handled when hot. Migration or evaporation of indirect food additives from the package might be a problem due to the elevated temperatures of the microwave environment.

Consumers have been warned never to place metal inside the microwave oven, because the metal could cause the reflection of energy that might damage the magnetron. Arcing may also occur, creating a fire hazard. However, microwaving of metal containers can be accomplished, as long as some precautions are taken. For example, the metal can be coated, left in its paperboard package if it has one, or the metal container can be placed in the center of the microwave away from the walls. Microwave ovens can also be designed to minimize damage to the magnetron.

Food manufacturers need to educate the public on usage of metal in the microwave oven, in order for consumers to accept products in metal containers.

Presently many manufacturers are making food containers out of plastic, paperboard, or a combination thereof. When using plastic containers there exists the potential problem associated with indirect food additives. Migrants can affect the quality of the food product; migrants may also have toxicological manifestations if the concentration exceeds an established safety level. Published information which describes the possibility of migration/ evaporation of indirect food additives from plastic containers to food as a result of microwaving in the container is limited.

This research investigation was made based upon the following hypothesis: during heating of a plastic container in the microwave oven indirect food additives may volatilize from the container. These volatiles may then be absorbed by the contained product. Absorption of indirect food additives by the product could create a potential legal problem if they exceed the level allowed by the FDA. Even if the indirect food additives are not a safety hazard, absorption by the product could have an adverse effect on product quality (i.e. odor and taste).

The objectives of this study were as follows:

1. To develop a method of detecting volatiles from plastic containers during microwave heating.

2. To determine the quantity of these components as a function of microwaving time.
3. To identify the major components.

LITERATURE REVIEW

2.0 A Brief Background on Electromagnetic Waves

Microwaves are a form of electromagnetic radiation. Electromagnetic waves travel at a velocity of 2.997×10^{8} meters per second in a vacuum; the waves are characterized by two parameters, frequency and wavelength. (See Figure (1) for the location of the microwaves in the electromagnetic wave spectrum.) The wavelength (λ) is related to velocity (c) and frequency (f) by:

$$\lambda = c / f \qquad (1)$$

The energy (E) in electromagnetic radiation varies with frequency according to:

$$E = h \times f \qquad (2)$$

where h is Plank's constant.

In a practical application such as in a microwave oven, a wave frequency of 2450 MHz is commonly used, which corresponds to a wavelength of 0.1223 meters and an energy of 1.624×10^{-24} J.

Electromagnetic waves consist of an electric field (El) and a magnetic field (H), which oscillate in a plane perpendicular to one another. The wave depicted in Figure 2 is described by:

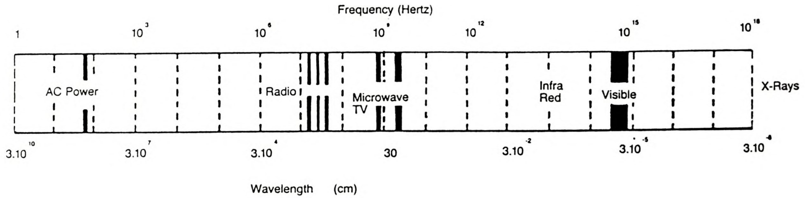


Figure 1. Electromagnetic Spectrum

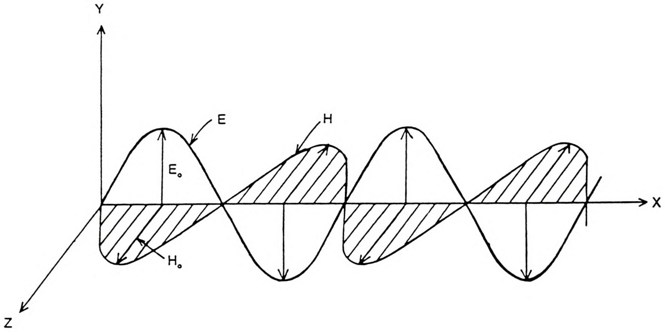


Figure 2. A Plane Monochromatic Electromagnetic Wave

$$El = El_0 \sin (wt-kx) \qquad (3)$$

$$H = H_0 \sin (wt-kx) \qquad (4)$$

where El and H are the amplitudes of the electric and magnetic fields respectively, and $w = 2\pi f$ and $k = 2\pi/\lambda$ (Vankoughnett 1973).

2.1 Explanation of a Dielectric Constant, Dielectric Loss and Loss Tangent

When materials are exposed to electromagnetic radiation the energy may be absorbed, reflected, or transmitted through the material body. The amount of microwave energy absorbed by a material depends upon its dielectric properties. Dielectric properties are defined by three parameters that are a function of the electromagnetic wave frequency. These parameters are the dielectric constant (ε')the dielectric loss factor (ε''), and the loss tangent ($\tan\delta$). "The dielectric constant relates the value of the electric field within the material to the value of the externally applied electrical field" (Tinga 1970). In more simplifed terms the dielectric constant is a measure of a material's ability to store electrical energy.

The loss factor, dielectric loss, (ε'') is a measure of a material's ability to dissipate electrical energy. The ratio of the dielectric loss to the dielectric constant defines the loss tangent.

$$\tan\delta = \varepsilon''/\varepsilon' \qquad (5)$$

The loss tangent is related to the material's ability to be

penetrated by an electrical field and to convert the electrical energy to heat (Mudgett 1986).

2.2 Description of Power Absorption, Depth Penetration and Change in Temperature

The dielectric constant and the dielectric loss determine the amount of energy absorbed, transmitted, and reflected by the material. Shiffmans (1986) states that power absorption by a material can be expressed by the following equation:

$$P = kfE^{2}\varepsilon' \qquad (6)$$

where P is the power developed in a volume of material; k is a constant in units of power and volume (watts/cm); f is the frequency of the microwave system; E is the electric field strength (volts/unit distance); and ε' is the dielectric loss factor for the material.

Increase in the temperature (ΔT) of a material when microwaved is expressed as:

$$\Delta T = \frac{k' f E \varepsilon''}{\rho C_p} \qquad (7)$$

Shiffmans (1986) defines the variables in the above equation as: ρ is the density of the material; C_p is the specific heat; k' is a constant used to express heating (C/minutes). Both the power absorption and the rate of increase in the temperature equations are based on an assumption that there exists a uniform electric field within a unit volume of the material.

Perry (1986) defines a penetration depth (d) as the

depth at which 1/e or 37% of the initial power remains as:

$$d = \frac{\lambda_0}{\pi}\left(\frac{1}{2\,\varepsilon'[(1 + \tan^2\delta\,)^{1/2} - 1]}\right)^{1/2} \quad (8)$$

where d is the penetration depth, λ_0 is the wavelength in free space (.1223 m in this case), ε' is the dielectric constant and tan δ is the loss tangent. At a depth d into the product, 63% of the incident power has been absorbed while 37% remains. For the next thickness into the product, 2d, only 23 % of the incident power will be absorbed. For a depth of 3d into the product, 9% of the incident power is absorbed. Because of the significant power difference in power absorption between depths, the outside surface will heat much faster.

2.3 Parameters Influencing the Dielectric Constant and Loss

Moisture content affects a material's dielectric constant since water has a very high dielectric value. At low moisture content levels, the water is bound and thus cannot freely rotate in the electromagnetic field. Beyond the critical moisture level the loss factor increases and the product is more receptive to microwave heating (Shiffman 1986).

The density of a material can affect its dielectric constant. Air is transparent to microwave radiation having a frequency of 2450 MHz. Therefore air spaces within a material reduce the dielectric constant below that of the

same material without air spaces. As the density of a material increases, the dielectric constant increases often in a linear fashion (Tinga 1970).

During heating of a material the dielectric loss changes with temperature, depending upon the moisture content of the material (Tinga 1970). For example, ice has a much lower dielectric constant than water; ice (3.2) is relatively transparent to microwaves where as water (80) absorbs microwave energy. In the frozen state the water molecules cannot move freely to orientate the dipoles when electromagnetic waves are applied.

2.4 Mechanisms of Heating via Microwave Absorption

Several mechanisms exist by which matter absorbs microwaves. The two most useful mechanisms are ionic conduction and dipole rotation (White 1973). In ionic conduction the ions are attracted by electric fields, because the ions are electrically charged. This movement constitutes a flow of current. The velocity of the ions represents kinetic energy as a result of their attraction to the electric field (White 1973). The ion particles (i.e. OH^-, Na^+, NH_4^+, NO_3^-, Cl^-, H_3O^+) collide with nonionized molecules such as H_2 and release their kinetic energy. Any particle may collide several thousands of times during microwaving. In each collision the kinetic enegy of the particles is converted into heat. The field may be either continous or intermittent; ionic conduction will occur in

either case. This heating process is not dependent to any great degree on either temperature or microwave frequency (White 1973).

In the mechanism of dipole rotation, the molecules will become oriented when an electric field is applied and will become random when the field is removed. In dipole rotation, the energy from the electrical field is stored as potential energy and then converted to random kinetic or thermal energy in the material. The energy conversion is efficient when the frequency coincides with the time required for the build up and decay of the induced order. As the temperature of the material increases, the faster orientation and randomization occurs; this process of buildup and decay is known as the relaxation frequency.

Dipole rotation has a large dependency upon the microwave frequency and temperature (White 1973). Also the molecule's size and shape influence the dipolar rotation. Since dipole molecules are not spherical in shape, they are not completely free to rotate; thus there exist a tendency for the orientation of the molecules to be random. Small molecules have a relaxation frequency which is higher than the microwave frequency. The relaxation frequency will increase as the temperature of the material increases, which slows down the conversion of energy into heat. Polymers, large molecules, have relaxation frequencies which are lower than the microwave frequency. Therefore as the temperature increases in the polymer, the energy

conversion into heat speeds up, which may result in a runaway heating phenomenon (White 1973). The runaway heating may result in hot spots and degradation of the polymer. Yet, polymers do not easily absorb the microwave energy as liquids and monomers do; hence, the chance of runaway heating of polymers is reduced.

2.5 Effects of the Magnetic Field

When a material is placed in a magnetic field, magnetic resonance occurs because energy is absorbed by the small magnetic particles in the material. VanKrevelen (1976) explains that two kinds of transitions may be responsible for magnetic resonance. The first transition involves the reorientation of the magnetic moment of the electons in the field. This transition is called electron spin resonance and is observed in the microwave region of 9 to 28 GHz. The second transition, nuclear magnetic resonance, occurs when the reorientation of the magnetic moment is in the nuclei of the material. The most widely studied nucleus is the proton. Nuclear magnetic resonance occurs in the radio frequency region of 10-300 MHz.

White (1973) suggests that the heating mechanisms induced by the magnetic field play a very minor role in heating microwave energy. As previously mentioned, ionic conduction and interface polarization have the greatest influence on microwave heating.

2.6 Influence of Electromagnetic Waves on Polymers

Polymer's influenced by electromagnetic waves will depend upon crystallinity. The density difference between the crystalline and amorphous regions affects the dieletric constant. The amorphous regions will allow more dipole rotation to occur. The dielectric constant and dielectric loss also depend more on unbalance or asymmetrical dipoles than on the presence of polar groups (Billmeyer 1984). Polyethylene has a lower ε' and ε'' , as compared to polyvinyl chloride. (See Table 1 for the dielectric constant of polymers and Figure 3 for how moisture can influence the ε' of some polymers (Vankrevelen 1976).)

Table 1. Dielectric Constant for Plastics

Polymer	ε' calculated
polyethylene	2.3
polypropylene	2.4
polystyrene	2.7
poly(o-chlorostyrene)	2.6
poly(vinly chloride)	2.8
poly(vinlyidene chloride)	2.9
poly(tetrafluoroethylene)	1.7
poly(chlorotrifluoroethylene)	2.2
poly(vinyl acetate)	2.8
poly(ethyl methacrylate)	2.7
polyacrylonitrile	3.7
poly(ethylene terephthalate)	2.9
polycarbonate	2.9
nylon 6,6	4.0

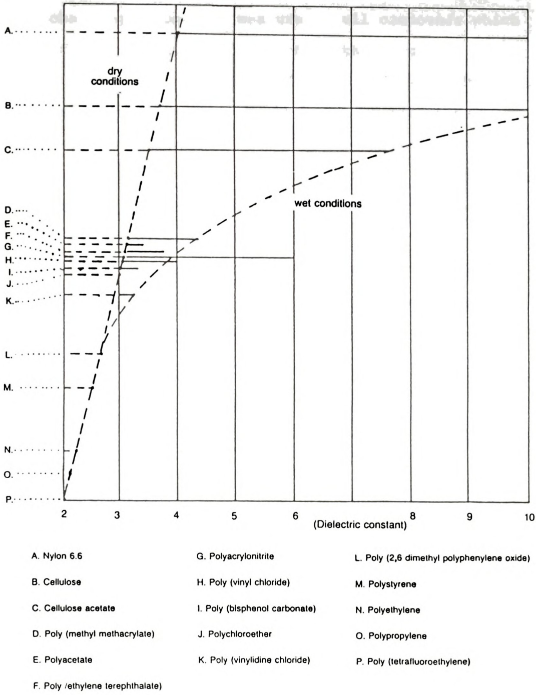


Figure 3. Triboelectric Series of Polymers

2.7 Theory of Migration of Indirect Food Additives

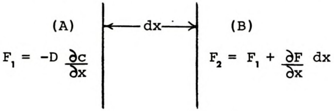
Global migration is a measure of all compounds which can transfer to a contained product, both toxic and nontoxic. The migration process depends partly on the diffusivity of the migrant. If the migrant interacts with the polymer then the process of migration is more complex. Diffusivity, or the diffusion coefficient (D), is defined as the tendency of a substance to diffuse through the polymer bulk phase (Giacin 1980).

The driving force for migration is due to a concentration gradient, where the dissolved species diffuses from a high concentration area to an area of lower concentration (Giacin 1980). The rate of diffusion is defined by Fick's law:

$$\frac{\partial m}{\partial t} = -DA \frac{\partial c}{\partial t} \qquad (9)$$

where m is the mass of the component transferred, t is time, c is the concentration, D is the diffusion constant and A is the area of the plane across which diffusion occurs (Crosby 1981). The negative sign in front of the diffusion constant arises from the fact that the concentration decreases as mass transfer occurs. The flux (F, quantity/unit time/unit area) is the rate of diffusion perpendicular to the concentration gradient; x is the direction of diffusion; c is the concentration per unit volume. Crosby (1981) illustrates the diffusion through the

first plane (A) and the second plane (B) as:



The change in the flux, with distance = $(-\partial c/\partial t)$, therefore is

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial x}\left(D \frac{\partial c}{\partial x}\right) \qquad (10)$$

Fick's second law defines D as constant and independent of the concentration:

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \qquad (11)$$

The effect of temperature on diffusion is often described by an Arrhenius type equation:

$$k = A\exp -(E/RT) \qquad (12)$$

where k is the rate constant; A is the frequency factor; E is the activation energy of the reaction; R is the gas constant; T is the absolute temperature. Crosby (1981) expresses diffusion in terms of the Arrhenius equation by:

$$D = D_0 \exp -(E_D/RT) \qquad (13)$$

where E_D is the energy of diffusion which is dependent upon the system.

Briston and Katon (1974) identify three packaging material/contact phase migration systems; (1) nonmigrating, (2) independently migrating, (3) leaching. For system 1,

nonmigrating, the transfer of the component occurs only from the package surface. In this case the diffusion coefficient approaches zero and cannot be measured. In system 2, the component evaporates from the surface of the polymer and is replaced by component diffusing through the material. The diffusion coefficient of the migrant is measurable under the specific time-temperature conditions. "The rate of migration and the amount of migrant transferred is dependent upon the contact phase volume, boundary layer resistance in the extracting phase and the time scale for desorption (Giacin 1980)." In leaching, system 3, the contact phase penetrates the polymer causing swelling, thus acting as a plasticizer so migration will occur (Giacin 1980).

Evaporation and diffusion of a component out of a material increases with temperature whereas the solubility decreases. Influence of temperature on migration of antioxidants out of films has been studied. Han (1984) profiles the migration of the antioxidant, butyl-4-methoxy phenol (BHA), out of high density polyethylene film at different temperatures. Little research is published on the influence of microwave heating on the migration of indirect food additives out of films. Heath and Reilly (1981) described the migration of acetyl-tributylcitrate (ATBC) from a plastic film into chicken during microwave cooking. ATBC was found to migrate out of the film and into the chicken. The rate of migration increased until the

chicken had been cooked for eight minutes. After eight minutes of cooking the rate of migration plateaus. The experiment had a cooking limit of ten minutes, so there was no data to indicate if the plateau represented the maximum absorption or if maximum extraction had been reached during the practical microwave cooking time for chicken products (Heath and Reilly, 1981). The experiment does indicate that the migration rate of ATBC is faster during the first part of the cooking period. Heath and Reilly (1981) show with their work that ATBC will migrated out of a film and into a chicken product after undergoing microwave cooking.

2.8 Packaging Materials for the Microwave Oven

When a containers is placed in a microwave oven, the container material may absorb the energy, be transparent to the energy, or reflect the energy. The material(s) chosen for the package will depend on the desired heating of the product.

A transparent material (i.e. most polymers currently used for packaging) will allow the microwaves to pass through with little absorption occurring. A transparent material is chosen when the entire product just needs to be heated. If the product has a large percentage of moisture then the package may experience temperatures around 100 ° C. Higher temperatures may occur as the product dries out or if the product contains a large percentage of fat or protein. The product may reach temperatures which could

melt the plastic material. Therefore, a plastic that has good thermal stability should be chosen for products containing a high percentage of fat or protein, or products having a long cooking time.

Paper products will absorb microwave energy if placed in a microwave with nothing else. Because paper products contain water in their structure as well as some mobile ions, paper products will heat in the microwave oven (Perry 1986). Usually the food product inside the paper container will absorb the microwave energy more readily than the paper container.

Metals are materials which reflect the microwave energy. Metals reflect the energy back into the microwave cavity and do not allow the product to heat. If water is placed into an aluminum can and then is microwaved, the water temperature inside the can will not increase.

Since these materials are good conductors of electricity, a large electrical potential energy may build up. If the material with the potential energy comes into contact with another piece of foil or a grounded surface an electrical discharge can occur. "This sparking (arcing) can occur between two packages, between layers of foil insulated from each other, across tears and wrinkles, and between foil packages and the oven walls (Perry 1986)." Arcing may be eliminated if precautions are taken such as centering a package in the oven or coating the foil with a non-conducting electric insulator.

A material which absorbs the microwave energy is called a susceptor. A susceptor is a metallized polyester laminated to a film, paper or paperboard (Perry 1986). The amount of metal on the polyester is so minute that arcing will rarely occur in the microwave oven. The construction of the susceptor causes localized heating to occur at the package/product contact surface. This localized heating causes higher temperatures which results in a drying and a browning effect on the food product surface.

2.9 Containers on the Market

In early microwave ovens the use of metal containers was discouraged because of the potential damage the reflected energy might do to the magnetron. Therefore alternative packaging materials to metal were developed. Crystallized polyester (CPET) is a common nonmetallic material used in today's microwave market. The CPET is thermoformed into a tray which may be used in both the microwave and conventional oven. Collier (1986) explains that residual compounds can often exist in the thermoformed tray (CPET) and be released during heating. These residual compounds can affect the odor and taste of foods.

As microwave ovens become more sophisticated and they are equipped with safeguards for protecting the magnetron, the use of modified metal containers becomes feasible. One method of modifying an aluminum tray is to coat the tray

with a polymeric coating, such as Alcoa's vinyl-matrix and epoxy coating, which enables the aluminum to absorb rather than reflect the energy (Aluminum Company of America). A plastic dome fits over the tray to provide extra protection against arcing between the sides of the tray and the sides of the microwave oven.

Food will not brown in the microwave oven as it will in a conventional oven. A special package using a susceptor has been developed to help aid in browning by causing localized heating (Perry 1986). The susceptor material can be found in the microwave pizza packages and some of the microwave popcorn bags.

Just as metal containers are being developed, plastic containers for microwave use are being improved. A new plastic container recently introduced into the market contains a shelf stable microwavable soup. The plastic bowl is constructed of a seven layer coextruded sheet composed of polypropylene / adhesive / polyvinylidene chloride / adhesive / polypropylene / adhesive / polypropylene copolymer. The lid material is an aluminum foil laminate composed of the following: oriented polyethylene terephthalate, adhesive, aluminum foil, adhesive, and polypropylene. The lid is designed to be removed before the soup is microwaved (Iezzi and Toner 1986).

As more people buy microwave ovens, more convenience type foods will appear on the market. These food products

will have packages which are microwavable; thus, there will be a growth in the development of new materials that can be used in the microwave oven.

MATERIALS AND METHODS

3.0 Description of Container

A local supplier donated the containers used in this work. The containers were made from a coextrusion of polypropylene / Saran^R / polypropylene. The manufacturer's specifications listed the thickness of the respective layers as follows, .010/.001/.010 inches. The manufacturer did not specify the composition of the two tie layers. The materials were converted into a cup style by thermoforming. The dimensions of the container were as follows; height 7.2 ± 0.1 cm, diameter at the top 6.5 ± 0.1 cm, diameter at the bottom 6.3 ± 0.1 cm. (See figure 4.)

3.1 Verification of the Container Materials

The material was separated into two layers by warming the material in a microwave oven for four minutes on full power. A Perkin-Elmer 1330 Infrared Spectrophotometer was used to qualitatively identify the two layers. The attenuated total reflectance (ATR) method was used, because of the thickness and opaqueness of the materials (See appendix F.) An ATR was run on both surfaces of each strip. The materials were identified by comparing the

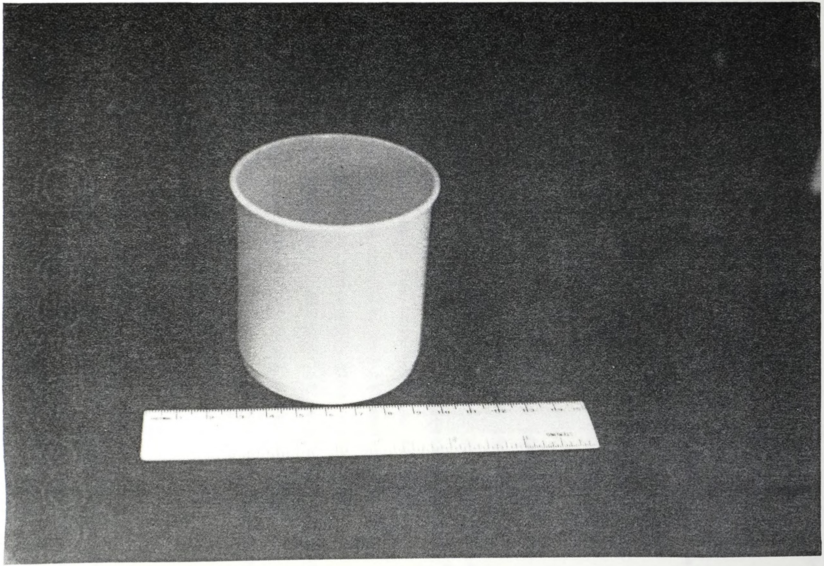


Figure 4. Coextruded Plastic Container

sample spectrums against known standard spectrums ("Identification and Analysis of Plastics," Haslam et al 1972).

Both strips of plastic were then soaked in Toluene for five days. By the end of five days, a thin piece of plastic film was visible and was easily separated from one of the plastic strips. A tranmission IR was run on the clear thin piece of plastic. Qualitative verification of the sample was made by using the source mentioned previously.

The thicknesses of the layers in the coextruded cup were checked by using an ocular microscope (American Optical Company). A piece of the container wall was placed into a freezer. The sample was held at approximately -15°C for one week. The sample was then mounted, so a cross-sectional view would be visible, and trimmed to fit the sample holder. Freezing the sample gave a sharp cut surface. Magnifications of 43X and 10X were used. The eye lense contained a micrometer having a scale of 1 unit equals 0.725 mils, which was used to measure the thickness of the different layers. (See figure 5 for a cross-sectional view.)

3.2 The Power Level of the Microwave Oven

An Amana Radarange (model RR1010) microwave oven was used for all microwave experiments. Power levels could be selected from 1 to 9, with level one being the lowest power output and 9 being the second highest. If no power level

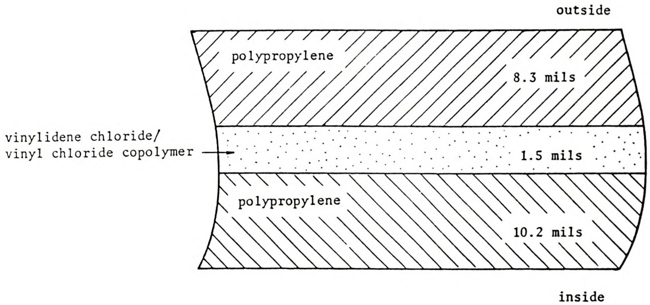


Figure 5. Container Cross-Sectional Area

was chosen then the microwave ran at full power. The power output of the mircowave was determined by placing a 500 ml beaker containing 300 mls of deionized water in the microwave oven. The water was heated for 60 seconds. The water temperature was measured before and after microwaving. Five different water samples were microwaved at each of the following levels; full power, 9, 8, 7 and 6. The power (P) equation: $\Delta tP = mC_p\Delta T$ was used to calculated the power of the microwave output (Fennema 1975). M was the mass of the water; t was the microwaving time; C_p was the specific heat of water; ΔT was the change in temperature the water experienced.

3.3 Temperature Correlation and Sample Preparation

The plastic samples were held in glass hypo-vials (Pierce) having a volume of 39.008 ± 0.148 ml with a standard deviation of 0.148. The volume of the vials was determined by weighing the vial with and without distilled water. The assumption was made that the density of the water used was .9982 gram/ml at 20°C. Ten vials were filled with distilled water and weighed. Teflon/silicone septums and aluminum crimp caps were used to seal the vials. All vials were cleaned and baked at 120°C prior to usage.

The plastic cup was cut into strips approximately 1 cm wide by 6.7 cm long. Five strips weighing a total of 2.00 ± 0.05 grams were selected (Mettler AE160 balance).

Melting point standard waxes, obtained from Omega, were used to determine the temperatures of the plastic strips during microwaving. The waxes came in liquid form and were applied by capillary action using pipets. The drops of waxes were no larger than 6 mm in diameter, which were significantly smaller in size by comparison to the plastic srtips. The waxes dried five minutes after the solvent evaporated. Five different drops of melting point standard liquid waxes were placed on the surface of three of the plastic strips. The melting temperatures of the waxes were as follows; 52°, 66°, 79°, 93°, 107° + .2°C. All five plastic strips were then cut in approximately half and put into a vial. The strips with the waxes were randomly oriented in the vials.

The microwave oven was preheated before runs and cooled between runs to maintain an oven temperature of approximately 27°C. The preheating was done by placing 2 grams of plastic in an unsealed vial and microwaving on high for 4.0 minutes. Eight minutes were allowed between each microwave run to permit the oven to cool down.

An apparatus was constructed to suspend the vials above the glass plate in the microwave oven. (figure 6 a & b) The apparatus was placed in the microwave oven so that each vial would hang over the center of the glass plate. The distance of the vial above the glass floor was maintained between 1/8 and 3/8 inch.

All experiments were run on full power. The vials

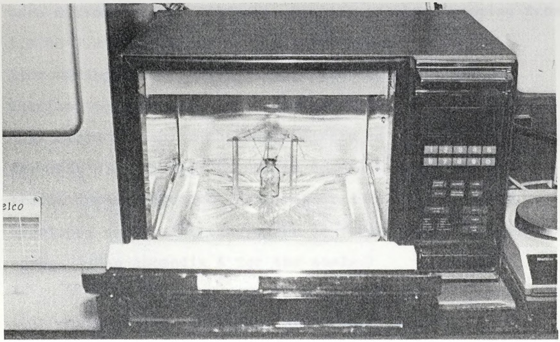


Figure 6.a. Apparatus in the Microwave Oven

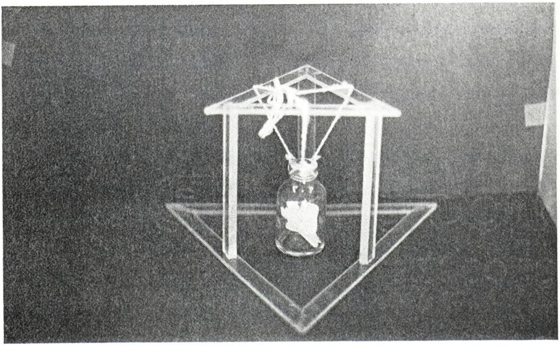


Figure 6.b. Apparatus for Suspending a Vial

were microwaved for different periods of time, ranging from 3.0 to 7.0 minutes in 30 second increments. After microwaving, the plastic strips with the waxes were examined to determine which ones melted. The waxes were considered melted if they were partly or completely glossy. The melt condition of the waxes for each different melt standard were recorded, as a yes or a no. A scale was developed to convert the yes or no recordings into temperatures. (See appendix A for the scale.)

3.4 Concentration vs Time Microwaved Correlation

To quantify the volatiles evaporating from the plastic containers, samples were prepared as followed, 2.00 ± 0.04 grams of the plastic container were weighed and placed in a clean vial. No waxes were applied to the plastic samples. The vials were flushed with nitrogen gas before sealing to rid the vials of all contaminants. The vials were sealed with teflon/silicone septums and aluminum crimp caps.

A variation of the hot jar technique was then used. Hamilton 0.5ml gas tight syringes were placed in a conventional oven maintained at 70°C and allowed to heat for 30 minutes before being used. The microwave oven was preheated as before. The vials were suspended from the same apparatus used in the temperature calibration section and kept a distance of 1/8 to 3/8 inches from the glass plate. The vials containing the plastic samples were

microwaved for time intervals ranging from 3 minutes to 7 minutes in 30 second increments. Ten runs were done for each time interval. Following microwaving, a 0.5 ml aliquot of the headspace was drawn from the vial using a hot syringe. The sample was injected into a Hewlett Packard 5890A gas chromatograph equipped with dual flame ionization detector (FID). Approximately 7 seconds was necessary to inject the sample, to prevent a surge of pressure in the capillary column.

The G.C. conditions were as follows: capillary column 60 m with an I.D. of .32 mm packed with SPB-5 (Supelco), 5% diphenyl / 94% dimethyl / 1% vinyl polysiloxane. To elute the sample, the G.C. was temperature programmed. The initial oven temperature was 75°C with an initial time of 5.0 minutes; the temperature was increased at the rate of 5° C per minute until the final temperature of 175°C was reached. The G.C. was then held at 175°C for 25 minutes. The injection port temperature was 200°C; column head pressure was 11 psi. A splitless injection port was used with the purge on after 0.70 minutes. The helium flow through the column was 1.02 cc/minutes. The flow rate was determined by injecting a sample of butane and recording the retention time (4.73 minutes). The following equation was used:

$$\text{flow rate} = \frac{(\text{I.D})^2 \times (\text{L})}{\text{rt of butane}} \times .785 \qquad (14)$$

where I.D. was the inner diameter of the column and L was

the length of the column.

3.5 Qualitative Anaylsis of Major Chromatograph Peaks

The samples of plastic used for the mass spectrometer were prepared as stated in the previous section. The vial was heated for 7 minutes in a microwave oven to produce a detectable concentration level. A 0.5 ml aliquot of headspace was taken with a hot syringe and injected into a (Jeol JMS-HX110) mass spectrometer. The column was a DB1 megabore (J & W Scientific) with a bonded stationary phase of methylsilicone having an I.D. of 0.53 mm. The temperature program started at 70°C for 5.0 minutes and increased at a rate of 5°C/minute until the final temperature of 175°C was reached. The flow rate through the column was 5 cc/minute.

The chromatograph of the mass spectrum was obtained for each of the peaks broken down into mass fragments. The reference text, EPA/NIH Mass Spectral Data Base, was used to identify the peaks of the mass chromatograph.

3.6 Verification of the Mass Spectrometer Identification

Using the above source, one peak from the mass spectrum was identified as Butylated Hydroxytoluene (BHT). To verify the BHT, a pure sample of BHT was placed in a vial and heated to 50°C. A headspace aliquot of .5 mls was injected into the gas chromatograph. (See section 3.4 for the G.C. conditions).

The BHT peak was found to have the same retention time as the retention time from the microwave chromatograph. Following the matching identification, a standard curve for BHT was constructed. 0.104 grams of BHT was weighed and placed into a 100 ml volumeric flask which had been partially filled with petroleum ether. Petroleum ether was added to bring the level to the 100 ml mark. A series of dilutions were made using the 100 ml stock solution: 2 mls into a 25 ml volumeric flask, 4 mls into a 10 ml volumeric flask, 2mls into a 50 ml volumeric flask and 2 mls into a 10ml volumeric flask. The volumes of the above series were brought up to the correct heights by adding petroleum ether to individual flasks. From the different dilutions 0.5 µl aliquots were taken and injected into the gas chromatograph. The conditions of the G.C. were the same as used for section (3.4). The area response units for each dilution were recorded.

RESULTS AND DISCUSSION

4.0 Verification of container

Using the ocular microscope technique three layers in the container wall were distinguishable. (See figure 6 for the depiction of the cross sectional area.) The thick-ness of the layers starting from the outside and moving inward were 8.3/1.5/10.2 mils, as compared to the manufacturer's specfication of 10/1/10 mils.

Analysis of the infrared spectrums using the reference "Identification and Analysis of Plastics" showed that the outside and inside layers of the container were polypropylene (figure 7). The spectrum of the container's middle layer appeared to be vinylidene chloride/vinyl chloride copolymer (figure 8). The percentage of each component remains unknown; the two most common ratios of vinyldene chloride/vinyl chloride are 80/20 and 90/10. An adhesive layer was found on both of the polyproplene layers which did not show up when the ocular microscope technique was used. The two adhesive layers had the same IR spectrums as ethylene vinyl acetate. (See figure 9 for the IR spectrum.)

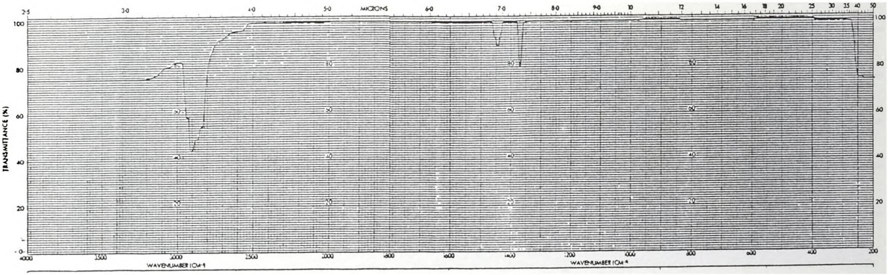


Figure 7. Infrared Spectrum of Polypropylene

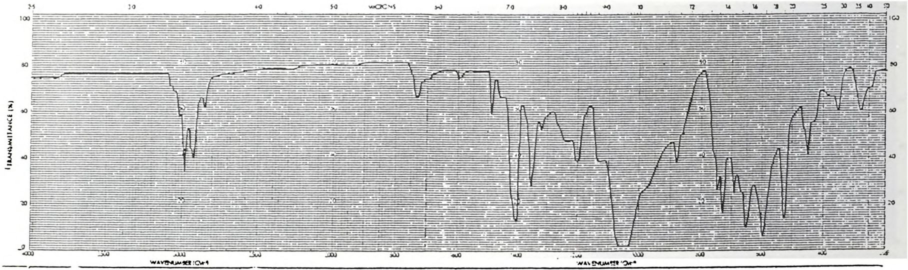


Figure 8. Infrared Spectrum of Vinylidene Chloride/ Vinyl Chloride Copolymer

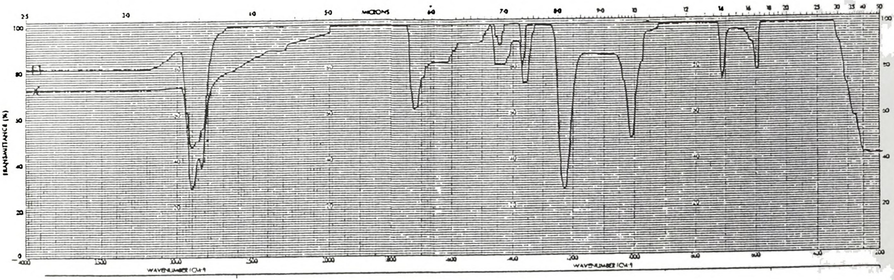


Figure 9. Infrared Spectrum: x - Ethylene Vinyl Acetate, o - Polypropylene

4.1 Power Output of the Microwave Oven

To understand the difference in the level settings of the microwave oven, the power output at several of levels were calculated by using the equation stated in the experimental section 3.2. The calculated results are listed in table 2 and plotted in figure 10.

Table 2. Power Output of the Microwave Oven

Level Setting	Power (J/s)	Standard Deviation	Coeff. of Variance
10	424.2	33.4	.079
9	379.3	17.8	.047
8	349.0	9.3	.027
7	305.1	15.0	.049
6	258.0	12.6	.049

The level setting of 10 was chosen to ensure the plastic would receive the most power the microwave oven could put out in the shortest period of time.

4.2 Microwaving Time vs. Temperature Correlation

The microwaving time was measured via the digital screen on the microwave oven, which can measure to the nearest second. From observing the microwave oven, a cycling of power on and off could be seen. An assumption was made that the cycling always started from the beginning each time the microwave was turned on. Therefore this cycling was not measured.

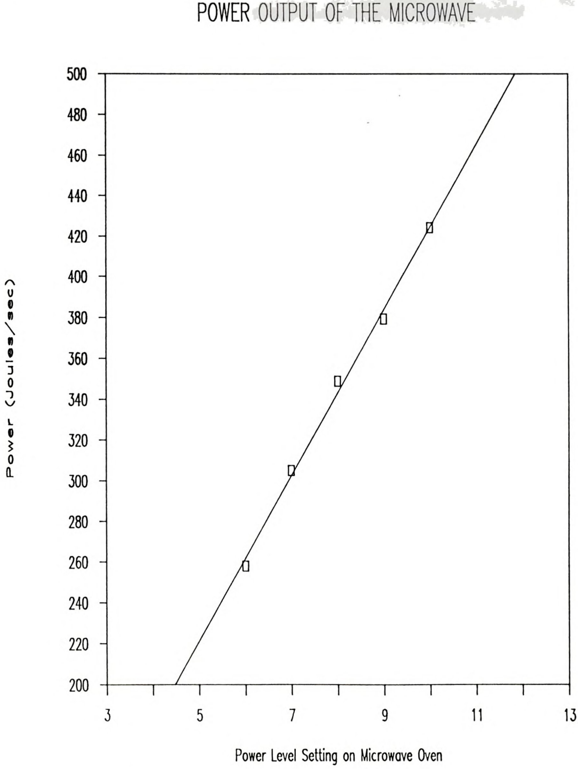


Figure 10. Power Output vs. Microwave Oven Level Settings

A scale was constructed to convert the 'yes' and 'no' results from the melting of the waxes into temperatures. (See appendix A.) From this data a curve was constructed to show the correlation between microwaving time and the temperature the plastic experienced. The relationship appears to be linear (see figure 11) for the range tested (3.0 to 7.0 minutes). Standard deviations are plotted on the same curve in order to show the temperature precision of the waxes. The largest deviation occured at 6 minutes 30 seconds and was 6.0°C. The time period from 0 to 2 minutes 59 seconds was not tested because the melting point standards have a lower temperature limit of 52°C. In this time period the temperature was not sufficient to cause melting of the waxes.

4.3 Heat Transfer via Conduction

The glass tray in the bottom of the microwave is made with materials which are capable of absorbing the electromagnetic waves. The glass tray will absorb the energy when the microwave oven is run with nothing in the cavity, or with a material with a low dielectic constant. Polypropylene has a low dielectric constant (2.2) compared to water (80), therefore the polypropylene will not absorb microwave energy easily. The glass tray will absorb part of the energy and will become hot.

The vial containing the plastic had to be suspended off the glass tray so there would be no conduction from the

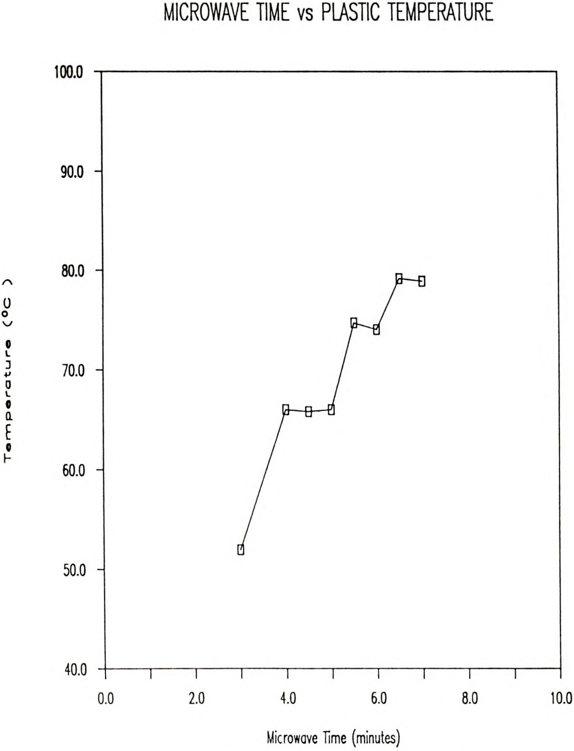


Figure 11. Microwave Time vs Temperature Experienced by the Plastic Strips

glass tray to the glass vial. The glass vial would get hot as the microwaving time increased. The plastic strips were placed into a vial randomly to determine whether the waxes were melting due to conduction from the glass. The waxes on the strips melted at random, which appeared to indicate that conduction from the glass vial did not influence the melting of the waxes. The waxes melt via the conduction of heat from the plastic.

4.4 Microwaving Time and Presence of Volatiles

Several major peaks were found using the gas chromatograph technique. (See appendix B for a sample chromatograph.) Five components were chosen for further examination in this study. These five components had high enough concentrations so they could be detected by the mass spectrometer and the resolution on the G.C. was good. The components had retention times of 7.98, 15.28, 15.50, 22.87 and 30.86 minutes. The average concentration area units for each of the five major peaks was plotted against microwaving time. (See figures 12 a,b,c,d,e.) The concentration of volatiles in the headspace increased as the plastic was exposed to longer periods of microwaving. The plots of the concentration of the components at the retention times 7.98 and 15.28 minutes appear to be of an exponential function. The components at the retention times 15.50, 22.86, and 30.87 minutes increased at a slower rate over time compared to the components at 7.98 and 15.28

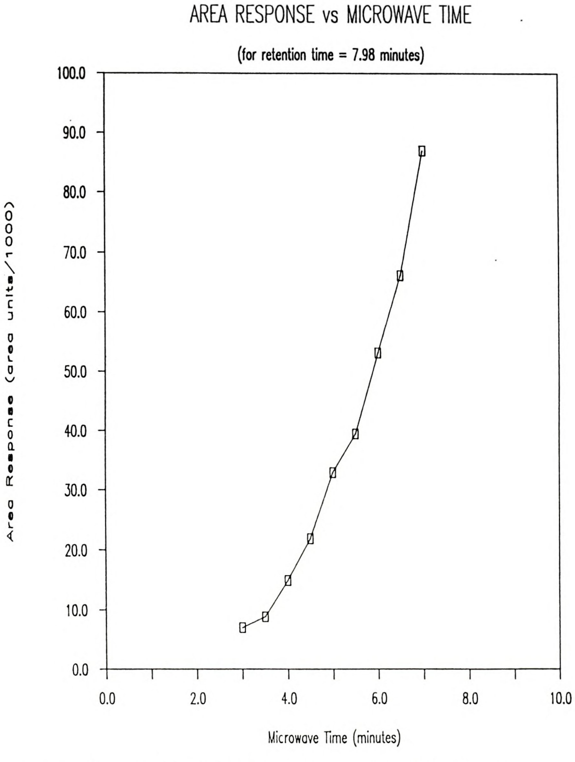


Figure 12.a. Microwaving Time vs Concentration for a Component with a Retention Time of 7.98 Minutes

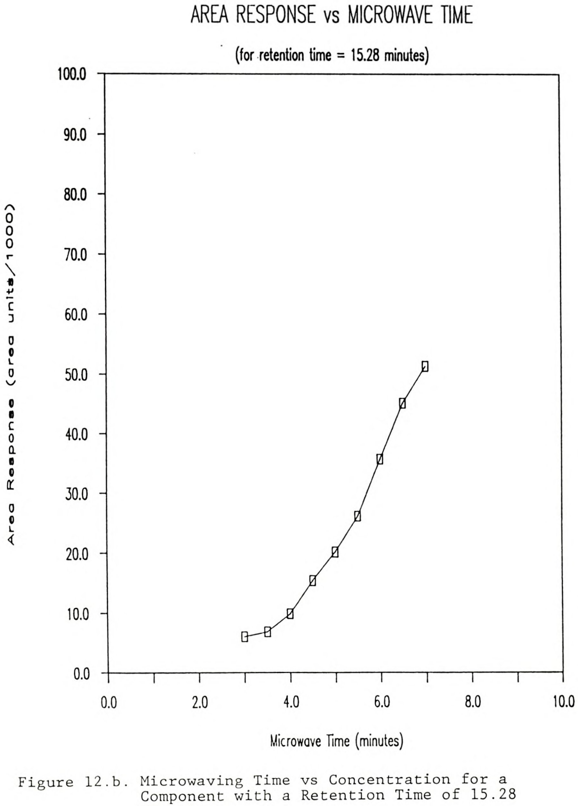


Figure 12.b. Microwaving Time vs Concentration for a Component with a Retention Time of 15.28 Minutes

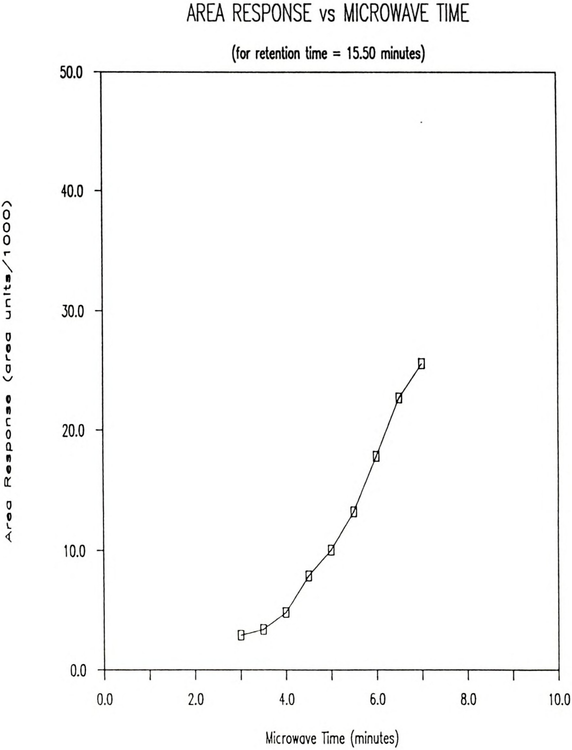


Figure 12.c. Microwaving Time vs Concentration for a Component with a Retention Time of 15.50 Minutes

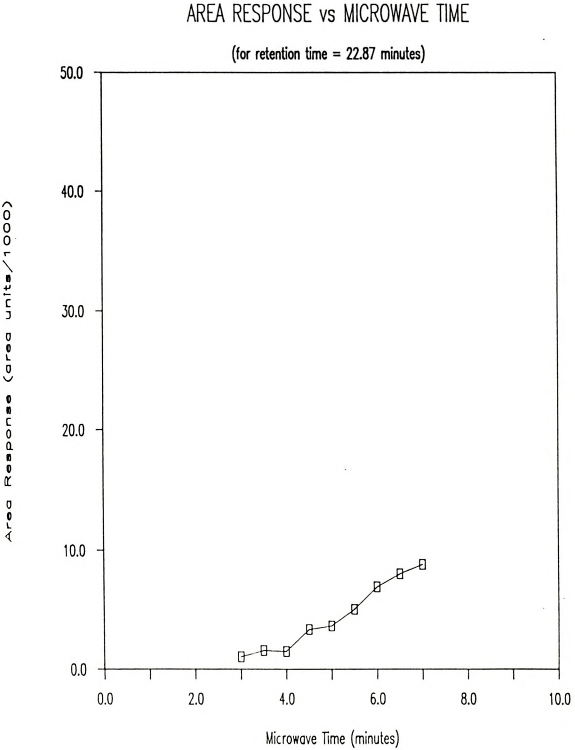


Figure 12.d. Microwaving Time vs Concentration for a Component with a Retention Time of 22.87 Minutes

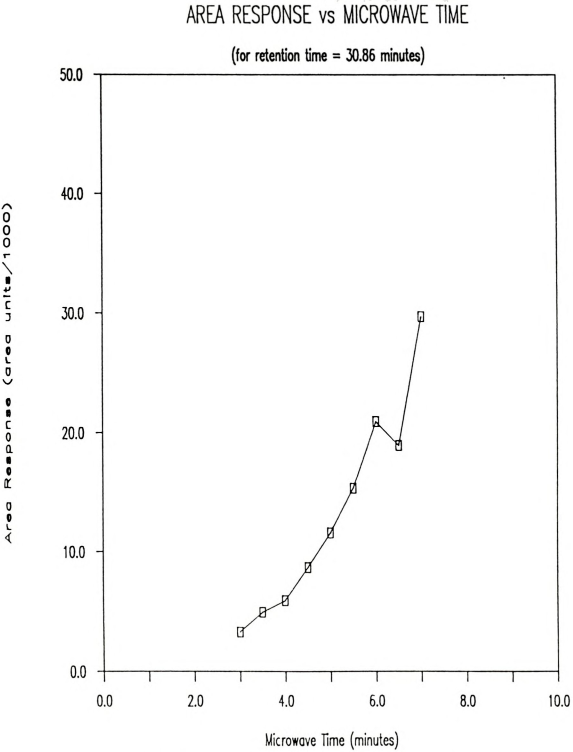


Figure 12.e. Microwaving Time vs Concentration for a Component with a Retention Time of 30.86 Minutes

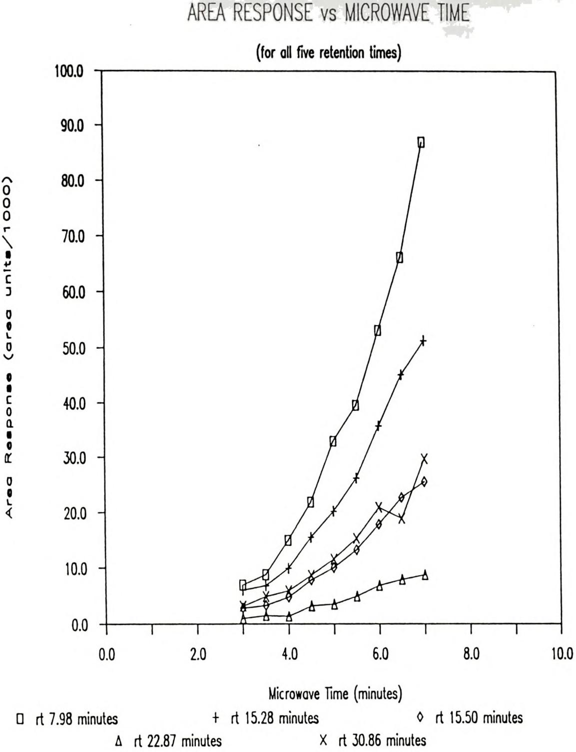


Figure 13. Relationship between the Concentrations of all Five Components vs Microwaving Time

minutes. Figure (13) depicts the concentration relationship between all five components. A correlation may exist between the size of the molecule and the length of the retention time. The longer the retention, time the larger the molecule. This correlation maybe important in identifying the components and in understanding the mechanism of transfer out of the plastic.

4.5 Mass Spectrometer Identification

The spectra obtained from the mass spectrometer were compared to standard spectra in the EPA/NIH Mass Spectral Data Base. (See figure 14 for the mass spectrum. See figures 15 a,b,c,d,e,f,g for the individual mass fragmentation schemes.) The spectrums are tentatively identified in table 3.

Table 3. Summary of Tentative Identification of Mass Spectra

Mass Spec retention time (minutes)	compounds	Molecular Weight
1.13	2,4 dimethyl Heptane	128
3.19	2,5-dimethyl Nonane	156
5.00	2 methyl undecane	170
5.48	4,8 dimethyl undecane	184
6.00	5 ethyl-5 methyl-decane	184
12.45	* -------------	212
17.58	Butylated Hydroxytoluene	220

* Not able to identify the hydrocarbon.

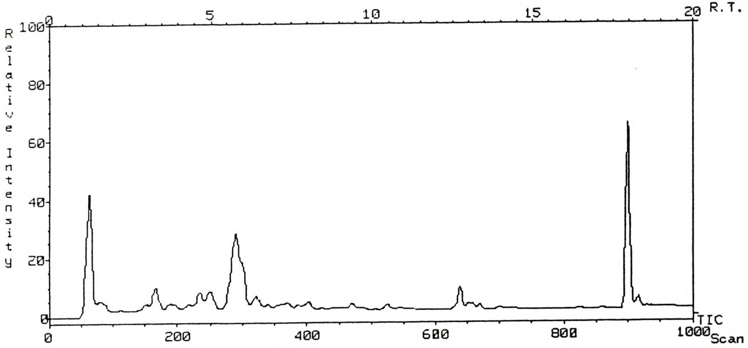


Figure 14. Mass Spectrum

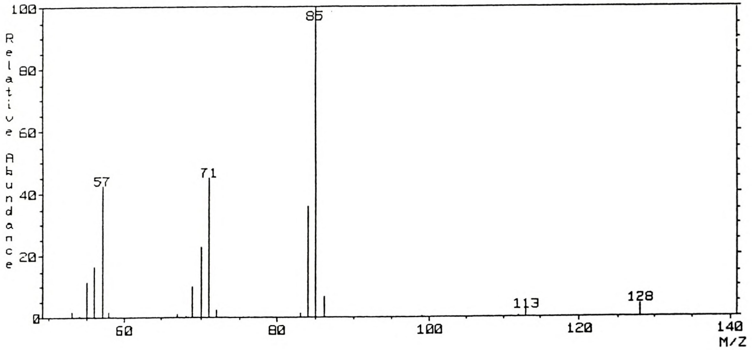


Figure 15.a. Mass Fragmentation for Retention Time of 1.13 Minutes

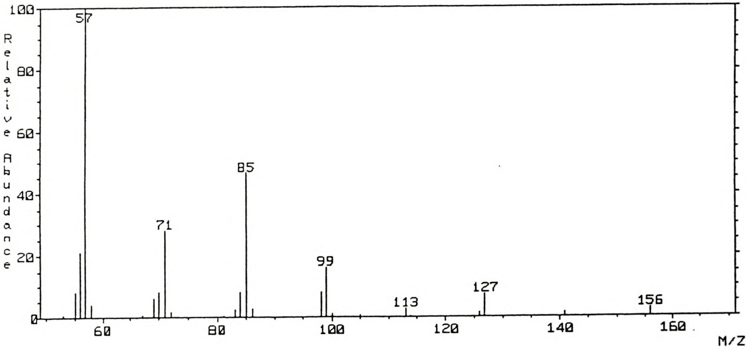


Figure 15.b. Mass Fragmentation for Retention Time of 3.19 Minutes

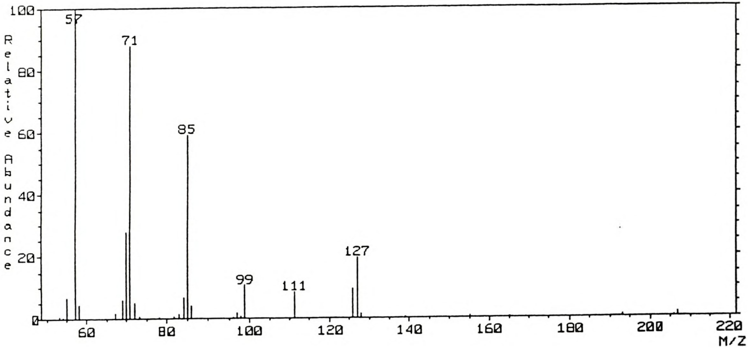


Figure 15.c. Mass Fragmentation for Retention Time of 5.00 Minutes

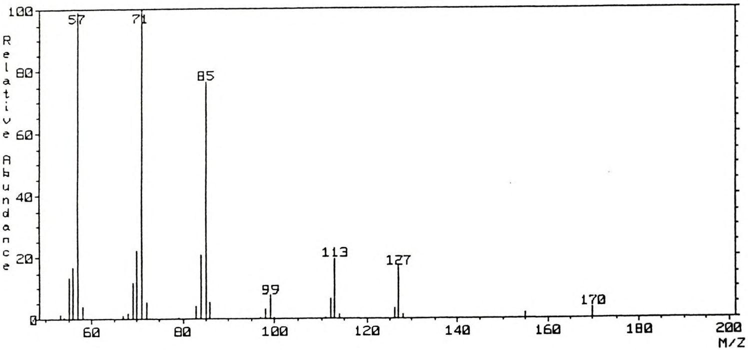


Figure 15.d. Mass Fragmentation for Retention Time of 5.48 Minutes

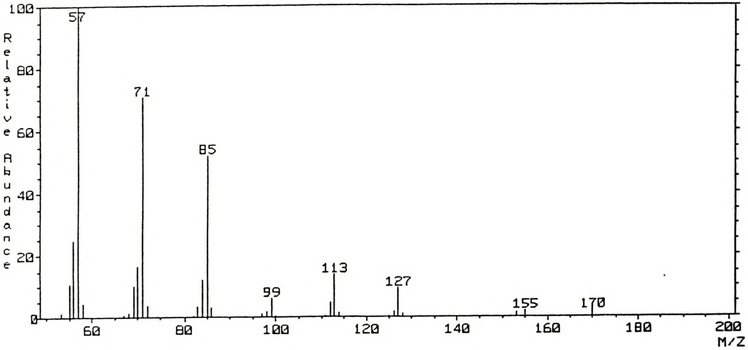


Figure 15.e. Mass Fragmentation for Retention Time of 6.00 Minutes

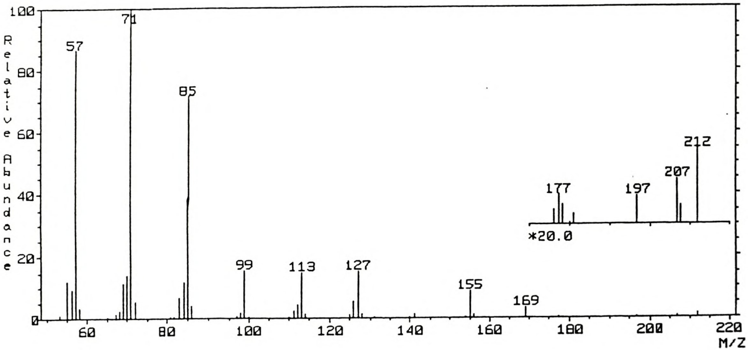


Figure 15.f. Mass Fragmentation for Retention Time of 12.45 Minutes

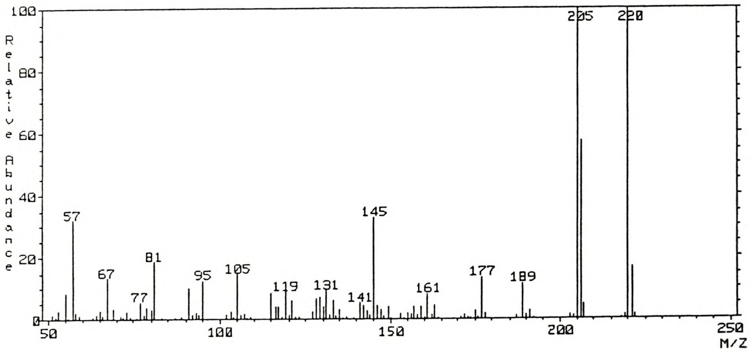


Figure 15.g. Mass Fragmentation for Retention Time of 17.58 Minutes

Positive identification of any the compounds listed above would have to be done by obtaining a pure sample of the compound and injecting a sample into the gas chromatograph. If the retention times were the same as previously found for the G.C. work then the pure compound should be evaluated by mass spectrometer. The two mass spectra could then be compared to see if the mass fragmentations were the same.

The peaks with the retention times 1'13" through 12'45" appear to be hydrocarbons. As retention time increases so does the molecular weight of the hydrocarbon. The identity of these hydrocarbons was not verified using gas chromatography. Therefore no direct correlation was made between the G.C. and the mass spectrometer. The speculation made was based upon the order of retention times, the amount of area response units from the G.C., and peak heights from the mass spectrometer. The results of the speculation are listed in table 4.

Table 4 Speculation on the Correlation between the Mass Spectrometer and the Gas Chromatograph.

Mass Spectrometer retention time (minutes)	Gas Chromatograph retention time (minutes)
3'19"	7.98
5'48"	15.28
6'00"	15.50
12'45"	22.87
17'58"	30.86

Butylated Hydroxytoluene was verified by injecting a headspace sample of gaseous BHT into the gas chromatograph. The G.C.peak from the BHT headspace sample matched the G.C. peak from the microwave sample. A standard calibration curve for BHT was then constructed. (See Figure 16.) The slope of the curve was found by using linear regression. Using the standard curve, the area response units from figure 16 were converted to grams of BHT per gram of plastic. (See appendix C for a sample calculation used for the conversion. See figure 17.) The data were then plotted against microwaving time. This curve has the same fit as when the data was plotted against area response units.

4.6 Origin of the Hydrocarbons and the BHT

The hydrocarbons could be coming from either scission of the polymeric chains or residuals remaining from the polymerization process. To determine whether the electromagnetic waves could cause scission a plastic sample was placed into a conventional oven. A headspace aliquot was injected into the G.C. to see if the retention times from the conventional oven method compared to those of the microwave method. The chromatographs were the same. Therefore the hydrocarbons were not produced because of the electromagnetic waves but probably due to a thermal phnenomenon. The hydrocarbons were most likely residuals from the processing of the plastic cup because the energy

required to break a C-C bond is 347 Kj/mol. The temperature to induce C-C bonds to break is higher than the melting temperature of polyproplyene (165°C). The plastic strips only obtained a maxium temperature of 90°C during microwave heating.

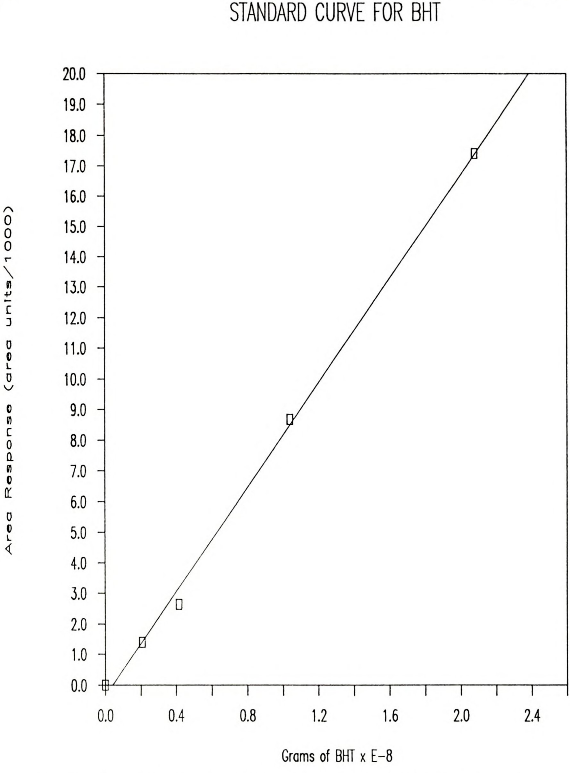


Figure 16. Standard Calibration Curve for Butylated Hydroxytoluene (BHT)

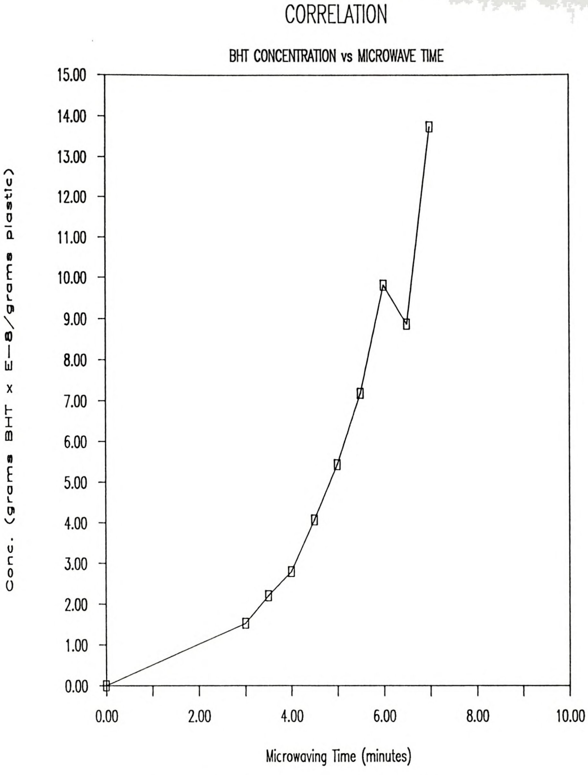


Figure 17. Grams of BHT per Gram of Plastic vs. Microwaving Time

CONCLUSION

Consumers have been taught never to place metal in a microwave oven and never to run the microwave oven empty. Since consumers shy away from foil packages, plastic containers have become the alternative packaging solution. How good are plastic containers?

Plastics in general have low dielectric constants if compared to water. However, a plastic container can absorb microwave energy, if no other substances are placed in the microwave oven along with the plastic container. A plastic container, composed primarily of polypropylene, reached an approximate temperature of 88°C after 7.0 minutes of microwaving. If an empty plastic container can reach a temperature of 88°C in 7 minutes then what temperature would the container experience if filled with a food product?

Food products will readily absorb more energy than the plastic container, because of their high water content. Liquid water within the product absorbs microwave energy and converts the energy into heat. The heat produced will be conducted to the remaining mass of the food product and the plastic container. The plastic container in this case

is heated indirectly. An incorrect assumption would be that the temperature of the container would never exceed 100°C. A plastic container may experience temperatures higher than 100°C, the boiling point of water, if the product contains sugars, oils, or proteins. Sugars and oils also absorb microwave energy but not as readily as water. With temperatures higher than 100°C more indirect food additives may evaporate and migrate into the food product.

The food additive regulations do not list microwave heating in the "Conditions of Use" set forth in Table 2 of 21 C.F.R. 176.170 (c). Many polymeric packaging materials are limited with respect to the temperature under which the materials may be used. The regulations do not make clear which of these materials are approved for use in packaging partially precooked foods intended to be microwaved.

Components will evaporate from a plastic container which has undergone microwave heating. One of the components identified as evaporating from the plastic container was butylated hydroxytoluene. After 7 minutes of microwaving the plastic container, 13.734 x 10 grams of BHT/ gram of plastic was detected in the headspace of a sealed vial. The FDA permits .02 % of BHT by weight to migrate into a food product.

The other components detected in the experiment were hydrocarbons. As microwaving time increased so did the concentration of these components in the headspace of the sealed vial. The FDA has no restriction on the amount of

hydrocarbons allowed to migrate into a food substance. If any work on the migration of hydrocarbons has been done it is very limited. Therefore little is known as to whether hydrocarbons influence the quality or safely of the food product.

This experiment should be done using a different contact phase such as water or oil. The experiment could determine how the temperture of the container is influenced by the contact. The higher temperatures may induce more components from the plastic container to migrate into the contact phase. As to whether these migrating components would alter the food quality or exceed the regulation levels must also be determined.

APPENDICES

APPENDIX A

Temperature Scale

YNN, YYN = 52°C

YYY, NNN, YNN, YYN = 66°C

YYY, NNN, YNN, YYN = 79°C

YYY, NNN, YNN, YYN = 93°C

YYY, NNN, YNN, YYN = 107°C

APPENDIX B

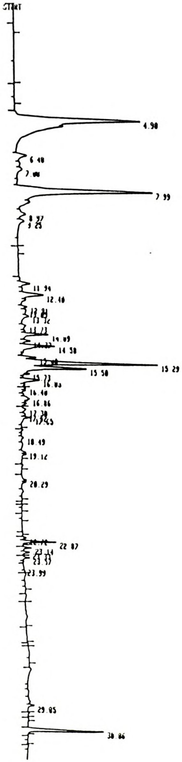


Figure 18. Sample Gas Chromatograph

APPENDIX C

Sample Calculation Used for Converting Area Response Units into Grams of BHT per Grams of Plastic

$$\left(\frac{1}{S}\right)\left(\frac{V \cdot R}{A \cdot P}\right) = \frac{\text{grams of BHT}}{\text{grams of polymer}}$$

$$\frac{1}{S} = \frac{.1172 \text{ grams BHT}}{\text{Area Response from Standard BHT Curve}}$$

V = Volume of the Vial (40mls)

A = Aliquot Size (.5mls)

R = Area Response from figure 12.e. for a given Microwave Time

P = Plastic Sample Size (2 grams)

APPENDIX D

Gas Chromatograph Raw Data for Five Different Retention Times vs. Microwaving Time.

Table 5. Gas Chromatograph Data for Microwaving 3.0 Minutes

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/3.0	8432	7866	3862	1496	5128
B/3.0	7668	6357	2768	1404	2786
C/3.0	9762	7184	3270	1025	4283
D/3.0	6030	5031	2196	622	2056
E/3.0	6015	5263	1876	748	2934
F/3.0	6263	5131	2322	589	2345
G/3.0	5617	4459	2313	856	2927
H/3.0	3809	5680	3987	1876	2679
I/3.0	7070	6015	2907	333	2852
J/3.0	9282	7767	3581	1079	4674

Table 6. Gas Chromatograph Data for Microwaving 3.5 Minute

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.87 minutes
A/3.5	11836	9711	4685	3897	4267
B/3.5	12870	9984	4886	1443	6207
C/3.5	4902	4740	2574	907	2748
D/3.5	11196	8855	4727	1296	6147
E/3.5	5186	3912	1825	650	6927
F/3.5	7218	5703	2463	739	3357
G/3.5	5015	5194	2444	1329	6027
H/3.5	8046	6195	2984	2336	7393
I/3.5	9175	5932	3109	----	4765
J/3.5	12868	8646	4219	1167	1945

APPENDIX D (continued)

Table 7. Gas Chromatograph Data for Microwaving 4.0 Minutes

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/4.0	19051	12394	5992	1120	5659
B/4.0	16693	13705	6511	2104	9881
C/4.0	16146	10289	4968	1198	5792
D/4.0	9414	5963	2763	790	3307
E/4.0	11195	6950	3367	980	5597
F/4.0	21272	11956	5645	1323	7166
G/4.0	10941	6519	3428	2105	4516
H/4.0	12463	9764	4988	1581	6077
I/4.0	16031	10188	4971	1361	6030
J/4.0	16571	11945	5860	1876	5594

Table 8. Gas Chromatograph for Microwaving 4.5 Minutes

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/4.5	10881	10270	5185	2031	10094
B/4.5	19010	14245	7133	2565	9369
C/4.5	28833	20887	10584	3855	14270
D/4.5	23388	16275	8028	2929	11849
E/4.5	20904	15186	8000	3165	8172
F/4.5	34389	22710	11682	4511	10275
G/4.5	42160	26887	13861	4279	8222
H/4.5	11397	8073	4152	6290	4971
I/4.5	14381	9802	4952	1752	3968
J/4.5	13791	10528	5406	1611	5773

Note rt = retention time

APPENDIX D (continued)

Table 9. Gas Chromatograph Data for Microwaving 5.0 minute

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/5.0	30633	22754	11498	3974	14253
B/5.0	34900	20890	10380	2861	14609
C/5.0	47394	27085	14227	5800	13981
D/5.0	26901	15102	7288	2270	9614
E/5.0	27748	19045	9346	3691	11555
F/5.0	57355	30484	14942	3652	10907
G/5.0	19890	13700	6778	3025	10118
H/5.0	26160	16887	8408	4238	12393
I/5.0	34367	22478	11020	4295	11860
J/5.0	23466	14031	6696	2222	6669

Table 10. Gas Chromatograph Data for Microwaving 5.5 Minutes

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/5.5	66027	39819	19940	6745	15621
B/5.5	31102	21803	11650	4786	14466
C/5.5	30897	21909	11162	4514	11996
D/5.5	60266	28223	14274	4075	17452
E/5.5	26013	18739	9827	4038	11972
F/5.5	29713	20480	9804	4627	12908
G/5.5	51728	39606	19779	7285	22435
H/5.5	66109	46653	23601	8255	22907
I/5.5	31487	22315	11011	3780	10440
J/5.5	22604	16175	8123	5327	25099
K/5.5	26530	18208	8997	3506	11765
L/5.5	32698	22077	11044	4417	14536
M/5.5	38767	24779	12963	3942	7775

Note rt = retention time

APPENDIX D (continued)

Table 11. Gas Chromatograph Data for Microwaving 6.0 Minutes

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/6.0	73815	50211	24417	8924	24353
B/6.0	70109	44507	21935	8624	23121
C/6.0	75146	52358	26525	9639	27658
D/6.0	49404	36599	18098	8048	30484
E/6.0	65704	46115	22611	9068	29127
F/6.0	57010	39872	20207	7353	24157
G/6.0	37214	25807	13298	5171	17956
H/6.0	42501	29644	14575	6442	24647
I/6.0	55233	30857	16308	5543	11641
J/6.0	31629	20312	10306	4124	9445
K/6.0	26644	16979	8415	3250	7673

Table 12. Gas Chromatograph Data for Microwaving 6.5 Minutes

Area Response (area units)

Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/6.5	65519	45541	22445	7641	23121
B/6.5	111180	77076	38755	14016	29932
C/6.5	73810	50961	26038	7866	15427
D/6.5	58616	40688	20395	6246	11204
E/6.5	32987	22879	11702	4066	8499
F/6.5	71049	49291	23886	8989	17170
G/6.5	61446	39503	19446	7837	20273
H/6.5	50568	32633	16432	6207	15761
I/6.5	62849	41309	20688	7689	17006
J/6.5	93920	66559	34604	11781	33402
K/6.5	46000	29990	15307	5791	16387

Note rt = retention time

APPENDIX D (continued)

Table 13. Gas Chromatograph Data for Microwaving 7.0 Minutes

	Area Response (area units)				
Vial #	rt 7.98 minutes	rt 15.28 minutes	rt 15.50 minutes	rt 22.87 minutes	rt 30.86 minutes
A/7.0	55062	37463	18304	6825	23277
B/7.0	75146	42054	20345	5496	16732
C/7.0	108390	69836	35672	16337	25003
D/7.0	65196	36628	17919	5592	18842
E/7.0	76269	42969	21415	5903	28005
F/7.0	66897	38148	18945	5627	19638
G/7.0	133800	77545	38863	14764	54058
H/7.0	121190	69869	34955	11709	46984
I/7.0	72152	40590	19899	6092	22743
J/7.0	95838	58133	29519	9771	41680

Note rt = retention time

APPENDIX E

Temperature Data for Different Microwave Times

Table 14. Temperature Data for 3.0 Minutes

Sample Number	Temperature (°C)
180a	52
180b	52
180c	52
180d	52
180e	52
180f	52
180g	52
180h	52

Table 15. Temperature Data for 4.0 Minutes

Sample Number	Temperature (°C)
240a	66
240b	66
240c	66
240d	66
240e	66
240f	66
240g	66
240h	66

APPENDIX E (continued)

Table 16. Temperature Data for 4.5 Minutes

Sample Number	Temperature (°C)
270a	66
270b	52
270c	66
270d	66
270e	66
270f	79
270g	66
270h	66

Table 17. Temperature Data for 5.0 Minutes

Sample Number	Temperature (°C)
300a	66
300b	66
300c	66
300d	66
300e	66
300f	66
300g	66
300h	66
300i	66

APPENDIX E (continued)

Table 18. Temperature Data for 5.5 Minutes

Sample Number	Temperature (°C)
330a	79
330b	66
330c	66
330d	79
330e	79
330f	66
330g	79
330h	79
330i	79
330j	66

Table 19. Temperature Data for 6.0 Minutes

Sample Number	Temperature (°C)
360a	79
360b	66
360c	66
360d	79
360e	79
360f	66
360g	79
360h	79
360i	79

APPENDIX E (continued)

Table 20. Temperature Data for 6.5 Minutes

Sample Number	Temperature (°C)
390a	79
390b	79
390c	66
390d	79
390e	66
390f	79
390g	79
390h	93
390i	79
390j	93

Table 21. Temperature Data for 7.0 Minutes

Sample Number	Temperature (°C)
420a	79
420b	93
420c	93
420d	79
420e	93
420f	93
420g	79
420h	79
420i	93
420j	93

APPENDIX F

Instructions for Attenuated Total Reflectance

The following instructions for the ATR method were modified from Perkin-Elmer.

1. Remove the clamping plate and clamping block from the sample holder assembly afer removing the three knurled-head screws from the assembly.

2. To insert the crystal (KBS-5) in the crystal holder assembly:
   a. Turn the knurled-head crystal release screw on top of the crystal holder assembly, to open the cell.
   b. Slide the crystal between the grooves of the three 'V' blocks on the holder until the crystal is seated against the limiting adjustment pin.
   c. Turn the crystal release screw counterclockwise until spring tension holds the crysal in place.

3. Place one of the pressure pads on the clamping plate and place the film sample over the pad. (The sample must be as long as the pad. The pressure pad must not be in direct contact with the crystal.)

4. Place the crystal holder and crystal down on the plate so that the holes in the crystal holder are centered over the holes in the plate.

5. Place the sample and then a pressure pad in the cutout of the crystal holder.

6. Place the clamping block over the gasket.

7. Insert the three knurled screws, removed in step 1, in the clamping block and screw them through the clamping plate until resistance is felt.

APPENDIX F (continued)

8. Tighten each screw a small amount as evenly as possoble until each screw is finger-tight.

9. Mount the crystal holder assembly on the accessory base so that the crystal holder is at a 45 degree angle from the third mirror. To clamp the holder onto the mounting pin, tighten the 1/8 inch Allen clamping screw on the side of the holder.

10. Adjust mirror 2 with the adjustment setscrew until the exit face of the crystal appears the brightest.

11. Adjust mirror 3 with the adjustment setscrew until maximum upscale deflection of the recorder pen is obtained.

12. Use a reference beam attenuator to bring the maximum transmittance level to 80-90 %T.

13. Operate the spectrophotometer according to the instrument instruction manual.

BIBLIOGRAPHY

BIBLIOGRAPHY

1 Agoos, A., "Serving Up a Better Package for Foods," Chemical Week, October 1985, pp 82-83.

2 Aluminum Company of America, Alcoa Opens the Door to America's First Microwave-Compatible Aluminum Tray: Wave'r Bake, Pittsburgh, Pa, 1986.

3 Billmeyer, F.W., Textbook of Polymer Science; 3rd Edition, New York, John Wiley & Sons Inc., 1984, pp 344 - 348.

4 Bishop, C.S. and Dye, A., "Micowave Heating Enhances the Migration of Plasticizers Out of Plastics," Journal of Environmental Health, March/April 1982.

5 Briston, J.H. and Katan, L.L., Plastics in Contact with Food, London, Food Trade Press LTD, 1974, pp 136-150.

6 Collier, J.C., "Practical Considerations on the Use of PET and Food Containers," Packaging Technology, May/June 1986.

7 Copson, D.A., Microwave Heating; 2nd Edition, Westport, Connecticut, AVI Publishing, 1975, pp 1 - 28.

8 Cotton, T.S., Personal Consultant, Gerber Products Company, Fremont, Mi, 1986.

9 Crosby, N.T., Food Packaging Materials; Aspects of Analysis and Migration of Contaminants, London, Applied Science Publishers LTD, 1981, pp 106 - 122.

10 Fennema, O.R., Karel, M. and Lund, D.B., Principles of Dood Science Part II; Physical Principles of Food Preservation, New York and Basel, Marcel Dekker Inc., 1975.

11 Giacin, J.R., "Evaluation of Plastics Packaging Materials for Food Packaging Applications: Food Safety Considerations," Journal of Food Safety, 1980 Vol. 2:4.

12 Gilbert, S.G., Miltz. J., and Giacin, J.R., "Transport Considerations of Potential Migrants from Food Packaging Materials," Journal of Food Processing and Preservation, 4(1980), 27-49.

13 Han, J.K., "Evalutionof Loss of 2-Tertiary Butyl-4-Methoxy Phenol (BHA) from High Density Polyethylene Film," Thesis for M.S., Michigan State University, 1984.

14 Hancock, G., "New Microwavable Packaging System Could Revolutionize Frozen Food Business," Packaging Technology, May/June 1986, pp 23-25.

15 Haslam, Willis, Squirrell, Identification and Analysis of Plastics; 2nd Edition, England, Hazell, Watson & Viney Ltd., 1972.

16 Heath, J.L. and Reilly, M., "Migration of Acetyl-Tributylcitrare from Plastic Film into Poultry Products During Microwave Cooking," Poultry Science, 1982, pp 2258-2264.

17 Iezzi, R.A. and Toner, D.F., "Plastics Packaging Thrusts at Campell Soup Company," Packaging Institue Conferenc, Changing Patterns in Domestic and International Packaging, Illinois, September 1986.

18 Karel, M., Fennema, O.R. and Lund, D.B., Principles of Food Science; Physical Principles of Food Preservation, New York, N.Y., Marcel Dekker Inc., 1975.

19 Kashtock, M.E., Giacin, J.R. and Gilbert, S.G.,"Migration of Indirect Food Additives: A Physical Chemical Approach," Journal of Food Science, Vol.45, 1980, pp 1008-1011.

20 Mudgett, R.E., "Microwave Properties and Heating Characteristics of Foods," Food Technology, June 1986, pp 84-93.

21 Nelson, E.B., "The Microwave Consumer : A Life Style Profile," Journal of Microwave Power, 17(4), 1982.

22 NSRDS-NBS 63, EPA/NIH Mass Spectral Data Base, Washington, D.C., U.S. Government Printing Office, 1978.

23 Perry, M.R., "Packaging for the Microwave Oven," 1986, Food Product Packaging Compatability Seminar, Michigan State University, July 1986.

24 Rice, J., "New Mexican-Style Snack Products in Aseptic, Microwavable Packaging," Food Processing, September 1986, pp 82-83.

25 Schiffmann, R.F., "Food Product Development for Microwave Processing," Food Technology, June 1986, pp 94-98.

26 Shapiro, R.G. and Bayne, J.F., "Microwave Heating of Glass Containers," Food Technology, February 1986, pp 46-48.

27 Slatkavitz, K.J., Personal Consultant, Pillsbury Company, Minneapolis, Mn, 1986.

28 Tinga, W.R., "Interactions of Microwaves with Materials," 1970, Proceedings of IMPI Short Course, Microwave Processing in the Food Industry, Chicago, 1979.

29 VanKoughnett, A.L., "Fundamentals of Microwave Heating," 1973, Proceedings of IMPI Short Course, Microwave Processing in the Food Industry, Chicago, 1979.

30 VanKrevelen, D.W., Properties of Polymers; Their estimation and Correlation with Chemical Structure, Amsterdam, Elsevier Scientific Publishing, 1976 pp 233-256.

31 White, J.R., "Why Materials Heat," 1973, Proceedings of IMPI Short Course, Microwave Processing in the Food Industry, Chicago, 1979.