

Analysis of Aroma Release during Microwave Heating

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A device to measure aroma compounds released during microwave heating was developed which allows time profiles of release to be determined. During microwave heating of food in an enclosed glass vessel, gas was displaced from the vessel due to water in the food turning to vapor. The released aroma compounds were trapped by a series of cold traps, all designed with serpentine flow to maximize surface and time contacts. The first cold trap used liquid nitrogen to trap the aroma compounds released prior to the food attaining 100 °C. The second trap was a modified condenser and trapped the aroma compounds released with the water, i.e., at 100 °C. Multiple fractions over time were collected. Aroma compounds in each water fraction were quantified by solid phase microextraction (SPME) and GC-FID. The sample analyzed was frozen spaghetti with added aroma compounds. Total losses of aroma compounds during heating were between 30 and 50%. The aroma compounds eluted at different times, and their air–water partition coefficients predicted this order.

Keywords: *Microwave; flavor release; partition coefficient; kinetics*

INTRODUCTION

Food products designed for microwave heating are popular, mainly because of their quick preparation time. However, they often have less desirable sensory characteristics than products cooked in a conventional oven. This is due to different heating mechanisms. For example, without special microwave susceptors, the maximum temperature of microwave heated foods is just under 100 °C as compared to surface temperatures approaching 230 °C in conventional cooking. Also as opposed to conventional cooking, steam is released during microwave heating as water migrates from the center to the surface (Reineccius and Whorton, 1990). This lower temperature, combined with the high water content at the surface, limits the Maillard reaction, and thus the amount of aroma compounds produced during heating (van Eijk, 1994). Furthermore, aroma compounds are lost from the food during heating.

In attempting to understand aroma release in the microwave, a theory called the delta T theory was developed from experiments with pure flavor molecules to predict the release of compounds from their heat capacity and dielectric properties (Shaath and Azzo, 1989). However, when tested with cakes, no relationship was found between delta T and flavor volatilization (Li et al., 1994). Likewise, with flavor compounds in fat or water, compounds with higher microwave absorption and higher microwave heating rates were not preferentially volatilized by microwave heating as compared to a heated bath (Lindstrom and Parliment, 1994; Stanford and McGorin, 1994). This theory is not applicable to flavor compounds in foods because the properties of the food and not the flavor compound influence heating characteristics.

Properties of the flavor compound that have been shown to influence aroma release during microwave heating include Henry's law constant and air–water partition coefficient. Henry's law constants gave good predictions for the losses of aroma compounds in water

(Lindstrom and Parliment, 1994), and a model based on partition coefficients and resistance to mass transfer predicted losses from a cake (de Roos and Graf, 1995).

The methods by which researchers have studied microwave effects on flavor have involved an analysis of the aroma before and after heating either by extraction of the food (de Roos and Graf, 1995; Yeo and Shibamoto, 1991), analysis of the headspace (Lindstrom and Parliment, 1994; Stanford and McGorin, 1994), or analysis of the compounds remaining in the cooking water by HPLC (Steinke et al., 1989). The approach to measure the amount remaining in a food sample often involves a time-consuming volatile isolation step. This paper describes a device and method to trap directly and quantitatively the aroma compounds released during microwave heating which followed the time profiles of release/loss. This information, in conjunction with the food temperature and thermodynamic compound parameters, added further understanding to aroma release.

MATERIALS AND METHODS

Sample Preparation. Buitoni brand spaghetti (1700 g) broken up into 7 cm pieces were immersed in 11.8 L of boiling water and cooked for 8 min. After draining and rinsing with cold water for 2 min, the final weight was 3992 g.

The aroma mixture contained diacetyl, dimethyl trisulfide, ethyl 2-methylbutyrate, 1-octen-3-ol, 2,3-diethyl-5-methylpyrazine, and (2*E*)-nonenal at 3 g/kg dissolved in medium-chain triglycerides (MCT), containing approximately 60% caprylic and 40% capric fatty acids. The aroma compounds were obtained from commercial sources.

The aroma mixture (0.285 g) was dispersed on top of 200 g of spaghetti in a plastic container and sealed. Samples were frozen at –25 °C until microwave oven analysis. Eight samples were analyzed whose frozen storage times were 2, 3, 5, 11, 13, 14, 16, and 17 days. This gave a final concentration of 4.275 mg/kg in the spaghetti.

Temperature of Meal. The heating profile of the spaghetti dish was obtained by placing fiber-optic probes at different places in the spaghetti and measuring the temperature during microwave heating, using the same microwave as in the release analysis. Eight measurements were taken from different places in the meal.

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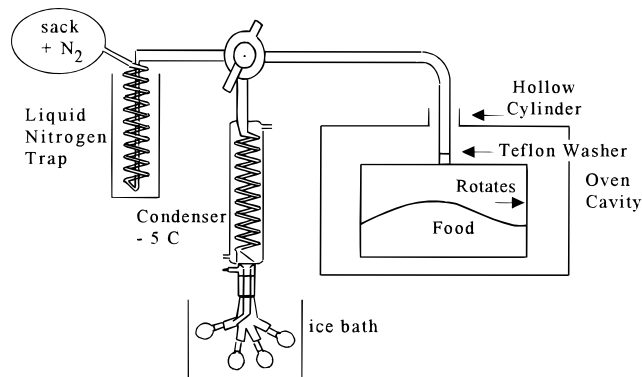


Figure 1. Apparatus to analyze aroma release during microwave heating.

Measurement of Partition Coefficients. Air–water partition coefficients were determined for all of the aroma compounds studied. The method determined the partition coefficient independent of the concentration of the reference and without adding a standard (Chaintreau et al., 1995). It involved a calculation based on the difference in slope between 1 and 2 mL solutions as GC area is plotted against concentration. The aroma compounds were analyzed simultaneously at three different concentrations that were soluble in water.

Device To Analyze Aroma Released in Microwave. A Panasonic Genius NN 5852 microwave oven with turntable, 23 L volume, 1420 W required power, and 100–900 W output power was used. The actual power output at full power was found to be 711 W, measured by heating 1 kg of water (28 °C) for 1 min and measuring the increase in temperature (Buffler, 1993).

The microwave oven (Figure 1) was modified by placing a hollow cylinder in the center of the top cover whose dimensions prevent leakage of microwave energy (Mingos and Baghurst, 1991). Specially designed glass vessels, as shown in Figure 1, allowed the trapping of aroma released in the microwave. All glassware was silanized with Sylon CT (5% dimethyldichlorosilane in toluene, Supelco) to avoid adsorption of aroma compounds. The food vessel (19 cm in diameter by 5 cm high) was glued to the center of the turntable to allow reproducible placement of the food. A Teflon washer with Viton o-rings, which connected the food vessel to glass tubing, allowed the food vessel, but not the glass tubing, to turn during microwave heating. The glass tubing was connected to a three-way valve that allowed the air stream to be directed to either the liquid nitrogen trap or the condenser. The liquid nitrogen trap was placed in this configuration, instead of after the condenser, to avoid a partial trapping of the initial aromas on the condenser. The glass trap was placed in a Dewar of liquid nitrogen and its exit was connected to a deflated plastic bag which prevented oxygen in laboratory air from condensing in the trap. The liquid nitrogen trap and the condenser incorporated serpentine designs which maximized surface area and time for contact of the air flow with the cold surface. The condenser was kept at –5 °C and the flasks, in ice, were rotated to allow multiple fraction collections.

Microwave Analysis Procedure. Immediately before analysis, liquid nitrogen was poured into the liquid nitrogen trap and alcohol coolant began to circulate in the condenser. The entire system was filled with nitrogen gas to prevent oxygen condensation in the liquid nitrogen trap. The sample was placed in the food vessel and sealed. The sample was heated for a total of 255 s. During the first 165 s, when the temperature was less than 100 °C, the air stream which came off was collected in the liquid nitrogen trap. Then, water began to elute and the air stream was diverted into the condenser. Two consecutive samples were collected (165–225 s, 225–255 s). The liquid nitrogen trap was rinsed with 5 mL of water to recover the aroma trapped. All fractions, which were aroma solutions in water, were weighed. The amounts of aroma compounds in the solutions were determined by SPME. The experiment was repeated eight times in its entirety with separate spaghetti samples.

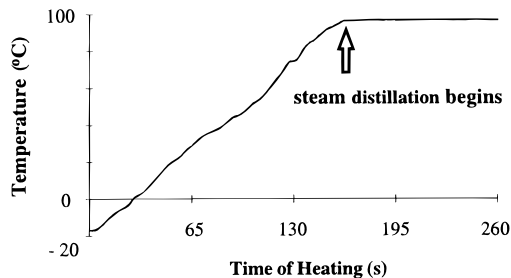


Figure 2. Average temperature profile of spaghetti during microwave heating.

Solid Phase Microextraction (SPME): GC/FID Quantification. Six external standard curves for quantitation were produced using standard solutions in water, one for each molecule of interest. The most concentrated solution was prepared by dissolving the aroma compounds in water with continued agitation until complete solubilization of the compounds. This solution was diluted to produce five different concentration levels, which included the concentration range of the samples. These ranges were (in mg/L) 1-octen-3-ol (0–16), diacetyl (0–28), ethyl-2-methylbutyrate (0–13), 2,3-diethyl-5-methylpyrazine (0–19), and (2*E*)-nonenal (0–17). As most of the curves were reproducibly slightly curvilinear, a quadratic function was used for regression with R^2 values of at least 0.999.

The standard samples and the microwave elution samples (all flavor solutions in water) were analyzed in the same manner using the following procedure: 3 mL was placed in a 4 mL septum-closed vial and stirred with a stir bar (800 rpm). A Carbowax/divinylbenzene fiber (Supelco) was immersed for 10 min. A trial with 10% sodium chloride addition was also performed. The fiber was then desorbed for 5 min at 200 °C in the GC injection port containing a 0.75 mm i.d. liner. During the first three minutes of desorption, the purge was off. The last two minutes with purge on further cleaned the fiber. The GC/FID contained a DBWAX column: 30 m, 0.32 mm, 0.25 μ m film thickness, 10 psi, and it was programmed to begin at 50 °C for 3 min, heat to 140 °C at 8 °C/min, and then to 220 °C at 25 °C/min.

RESULTS AND DISCUSSION

Description of Meal during Heating. Figure 2 shows the temperature profile of the spaghetti sample during microwave heating. This is the average curve obtained from eight measurements. As opposed to conventional oven cooking, the microwave oven and surrounding air are cooler than the product, resulting in a lower maximum temperature. Also, the surface of the food during microwave heating remains wet due to the constant migration of water from the food interior. The temperature of the meal changed from frozen temperature of –18 °C to a maximum of about 97 °C, where it remained until the end of heating. During this time at 97 °C, water evaporated off the meal at a flow rate of about 10 mL/min.

Frozen food is particularly susceptible to hot spots where different regions will heat much faster than other regions. This may affect aroma release where the rate of release may depend, in part, on how quickly the surrounding food heats. This can also contribute to the variation seen between samples.

Apparatus Design Considerations. For the release analysis from food systems, which normally contain large percentages of water, several specific parameters must be considered. One was the initial air flow out of food in the enclosed vessel (>500 mL/min). This air flow did not occur when only air was heated in the enclosed vessel. It was attributed to volume expansion as water from the food changed from a liquid to a gas.

Thus, because of the inherent air flow rate, an air purge of the food in the apparatus was not necessary. As the food temperature approached 100 °C, large amounts of water began to elute. Due to the high flow rate and the large amount of water collected, analysis of the eluted aroma compounds using a liquid nitrogen trap and condenser was chosen over using a trap such as Tenax. Similar designs for the aroma collection have been reported where microwave ovens with attached reflux systems extracted essential oil constituents from plant materials (Chen and Spiro, 1994; Mingos and Baghurst, 1991; Yeo and Shibamoto, 1991).

To analyze the aroma compounds eluting during the first minutes of microwave heating, a trap was chosen which could function during high flow rates. A glass trap where air flowed through a serpentine path was designed to maximize surface area and time of contact with the coolant. Liquid nitrogen (−196 °C) was chosen as the coolant because it showed the best trapping of highly volatile compounds. However, at −196 °C, oxygen (bp −183 °C) from the air was also trapped and it vaporized during rinsing of the trap, resulting in some simultaneous loss of aroma compounds. Steps were taken to reduce the amount of oxygen that condensed, including closing off the liquid nitrogen trap to room air and running nitrogen gas through the entire system before commencing the analysis.

After the water began to elute, a condenser with an alcohol coolant was used. In the design of the condenser, the temperature of the condensed sample was measured and a low temperature was desired to reduce aroma loss. At a high rate of condensation, simple condensers operating at below 0 °C could not cool the condensed sample to below 15 °C without the formation of ice in the condenser. Thus, a large condenser was designed which maximized air-coolant contact as air was directed into a serpentine trap. The fractions, eluting with a temperature of about 5–10 °C, were cooled in an ice bath as they were collected. Aroma losses were measured by connecting a liquid nitrogen trap after the condenser and found to contain minimal amounts, <1%, of condensed aroma. To see the kinetics in more detail, multiple fractions could easily be collected.

Use of SPME for Quantification of Flavor Solutions. SPME has been shown to be a sensitive, reproducible, and quick method for analyzing volatile compounds (Arthur and Pawliszyn, 1990; Steffen and Pawliszyn, 1996), especially for aqueous samples (Nilsson et al., 1995). The fibers show a good adsorption of nonpolar, but not of polar, compounds. In our studies, poly(dimethylsiloxane) was not sensitive to polar compounds such as diacetyl and could not detect acetic acid at 1000 ppm. Likewise, poly(dimethylsiloxane) was found to be less suitable for analysis of orange juice volatiles due to its lower compound extraction (Steffen and Pawliszyn, 1996). Three fibers, polyacrylate, poly(dimethylsiloxane/divinylbenzene), and Carbowax/divinylbenzene showed the best sensitivity to diacetyl (60-fold increases) with minor differences in sensitivity to the other compounds. Carbowax/divinylbenzene was chosen because it showed a better separation of the more volatile compounds during chromatography, most likely due to the desorption rate.

The fibers' differential adsorption of compounds makes an absolute quantitation of aroma compounds difficult unless the natural matrix can be duplicated (Miller et al., 1996). However, in this study, the natural matrix

Table 1. Effects of SPME Parameters on Adsorption from a Poly(dimethylsiloxane) Fiber^a

compound	agitation	10% NaCl addition
ethanol	4	240
ethyl 2-methylbutyrate	470	210
dimethyl trisulfide	2200	30
1-octen-3-ol	130	860
2,3-diethyl-5-methylpyrazine	170	430
(2 <i>E</i>)-nonenal	1600	−10

^a Values are % increase in amount from base condition of no agitation and no salt addition after 10 min of extraction.

can be easily duplicated because all of the samples for analysis are distilled water solutions containing aroma compounds. Thus, an external standard curve was made with standard aroma solutions in water where the aroma compounds were dissolved directly in water without the use of a solvent. In this manner, the quantification of the compound amounts in the samples was accomplished.

The fiber can be immersed in the solution or rest in the solution's headspace. For many non-water applications, the headspace is the most appropriate method because of interferences from nonvolatiles in the sample (Field et al., 1996). For these aqueous samples, fiber immersion and headspace analysis showed an overall similar sensitivity for the compounds analyzed. Less volatile 2,3-diethyl-5-methylpyrazine was detected with more sensitivity using the immersion technique while more volatile (2*E*)-nonenal and dimethyl trisulfide were better detected with the headspace technique. Previous comparisons showed similar sensitivities but different equilibration times of the two methods (Nilsson et al., 1995).

Different sampling methods using agitation and salt addition were also compared. Agitation was found to increase adsorption between 4 and 2200% for a range of different compounds by increasing the adsorption kinetics (Table 1). Salt addition changed the adsorption between −10 and +860% for different compounds. It was not used in this study, however, because it did not visibly increase adsorption of diacetyl, the least adsorbed compound.

A column focusing method was not necessary, even with an initial GC temperature of 50 °C. Several factors were modified to produce good GC resolution. The GC injection liner was changed to 0.75 mm i.d. (from 2.0), and a column with an internal diameter of 0.32 mm was employed (instead of 0.20). With these changes, the transfer of compounds to the column was sufficiently rapid (less volume and larger flow rate).

Each fiber lasted for approximately 30 sample analyses. This number is the amount of samples we could analyze after the production of the standard curve and also taking into account cleaning desorptions. The fibers were able to easily detect most compounds in water at 100 ppb; however, the thresholds for detection of polar compounds were higher (i.e., 1 ppm for diacetyl with Carbowax/divinylbenzene). Reproducibility was demonstrated by the production of standard curves with high correlations ($R^2 > 0.999$) which gave the correct values when the same samples were analyzed at 0- and 10-fold dilutions. Ease of use, sensitivity, and reproducibility make this technique an appropriate choice for many applications.

The total method of microwave release, trapping, and SPME quantitation was very sensitive. Preliminary studies showed that water solutions containing 0.6 mg/L of aroma compounds were easily measured. Its rela-

Table 2. Percent Released^a during Microwave Heating of Frozen Spaghetti

	0–165 s	165–225 s	225–255 s	total
diacetyl	3.7 (2.5)	22.1 (4.4)	15.0 (3)	41
ethyl 2-methylbutyrate	15.3 (5.7) ^b	2.7 (1.2)	8.8 (1.7)	27
dimethyl trisulfide	21.1 (3.3) ^b	3.1 (2)	2.4 (0.7)	27
1-octen-3-ol	11.0 (3.3)	21.9 (6)	12.8 (3.8)	46
2,3-diethyl-5-methylpyrazine	2.8 (0.6)	26.0 (7.3)	15.2 (3)	44
(2E)-nonenal	10.0 (3.4)	13.4 (4.2)	9.2 (2.7)	33

^a 100 = total amount added to spaghetti before freezing, average of eight analyses. Standard deviations in parentheses. ^b Average of four analyses.

tively high CV of 30%, average value from Table 2, for analysis of frozen spaghetti samples is primarily due to the nonuniform heating in the microwave. Hot spots in the meal which heat much more rapidly than the overall meal can induce faster aroma release. Losses are also possible during the liquid nitrogen trapping procedure if oxygen is simultaneously trapped.

Mechanism of Aroma Release. Table 2 shows the percent released as related to the amount added. Even if the aroma compounds added to spaghetti were dissolved in MCT, their vapor pressure was high enough for a total amount released ranging from 26 to 46%. This gives an indication of the large amount of aroma compounds that are lost when food is heated in the microwave. These losses most likely came from heating in the microwave and not during frozen storage. No effect of frozen storage time (up to 17 days) was found for diacetyl, 2,3-diethyl-5-methylpyrazine, (2E)-nonenal, or 1-octen-3-ol. For the first time fraction only, a relationship between storage time and amount released was found for dimethyl trisulfide ($R^2 = 0.77$) and for ethyl 2-methylbutyrate ($R^2 = 0.4$) where less was released after a longer storage time; thus, values of storage over 11 days were not included. Likewise, in a study of aroma release from frozen pancakes, losses of 10–56% were found due to microwave reheating and frozen storage (Li et al., 1994). Food products designed for microwave use may need to change their flavor systems to compensate for losses. Knowledge of which

compounds are lost to greater degrees and a method for prediction of these losses can guide aroma formulation changes. As noted earlier, the liquid nitrogen trap (trap 1) showed a loss of aroma compounds. This trap contained high amounts of dimethyl trisulfide and ethyl 2-methylbutyrate. Thus, the values for total percent released for these compounds may be underestimated. The most volatile compound in water, dimethyl trisulfide, showed a large release during the beginning of heating and less at the end which indicates that the majority was released.

Figure 3 shows that the aroma compounds were released at different times during microwave heating. The order of release corresponds exactly with the order of the compounds air–water partition coefficients. Dimethyl trisulfide and ethyl 2-methylbutyrate were released to the largest extent early in the heating of the frozen spaghetti, in the first 165 s. These are the most volatile compounds in water (largest K_{aw}). The average temperature in the meal was below 100 °C at this point. The other compounds showed their largest release after 165 s, when the meal reached the maximum temperature of just under 100 °C. (2E)-Nonenal was the next compound to elute and also exhibited the next lowest K_{aw} . 1-Octen-3-ol followed in time and also had the next lowest K_{aw} . And lastly, diacetyl and 2,3-diethyl-5-methylpyrazine eluted primarily at the end of heating and had the lowest K_{aw} . Thus, for a meal that has low fat content, the K_{aw} was found to be an excellent approximation of the microwave elution kinetics. As expected, boiling point of the aroma compound does not give a good approximation. Diacetyl has the lowest boiling point (88 °C) but is one of the last to be released due to its low volatility in water. The spaghetti with added aroma compounds in MCT contained approximately 72% water, 3.4% protein, 0.6% fat, and 23% carbohydrates. This corresponds with results that show a correlation between the air–product partition coefficient and the % flavor loss during microwave baking of cakes with fat contents of 4, 18, and 26% fat (de Roos and Graf, 1995). After the addition of the aroma mixture and certainly during heating as water vapor

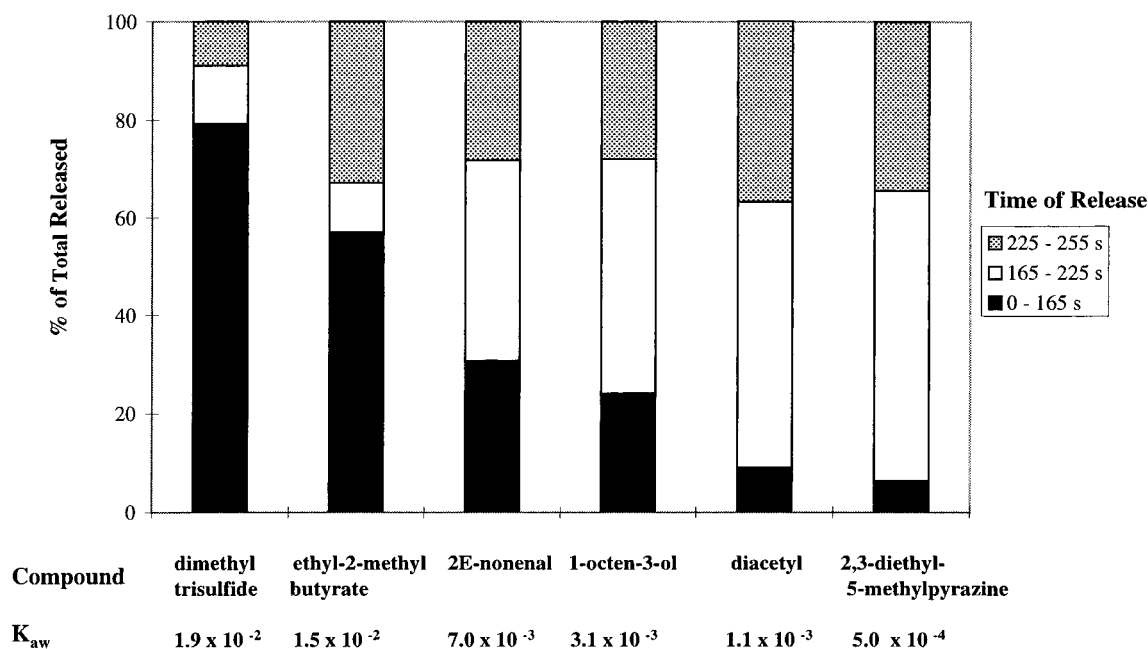


Figure 3. Differences in the kinetics of aroma compound release during microwave heating showing the dependence on the air–water partition coefficient (K_{aw}).

migrated to the food surface, the aroma compounds were exposed to the large amount of water in the spaghetti. Thus, although the aroma compounds originated in an oil phase, they would have moved into the much larger water phase, according to their lipophilicity. This may be why K_{aw} showed a good prediction with this low-fat food.

In addition to knowledge of the aroma compound amounts lost, an understanding of the loss kinetics show how the perceived aroma of the meal would change during microwave heating. The earlier release of dimethyl trisulfide, as compared to other compounds, would impart a sulfur, cabbage-like aroma at the beginning of heating. Depending on the product, this could give a negative impression. The ability to use aroma compound properties to predict release kinetics can allow a product's aroma to be better controlled.

Conclusions. A method was developed that was able to quantitate the amount of aroma compounds eluting at different times from food as it is heated in the microwave. The method was readily able to measure aroma compounds present at 4 mg/kg in the meal and is sensitive to aroma compounds at the levels they are present in food. The trap and condenser glassware were designed to accommodate the high air and water flow rates, in effect using the distillant nature of microwave heating. In addition, SPME showed high sensitivity to nonpolar compounds and was an effective method to quantitate aroma compounds in the distilled solutions.

When analyzing actual food, aroma compounds were found to elute at different times during heating. The release order corresponded directly with the order of the compounds' air-water partition coefficients. Thus, predictions for which types of compounds will be more readily lost can be made and aroma formulations for microwaveable food can be modified accordingly.

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