Analysis of Volatile Flavor Components in Roasted Peanuts Using Supercritical Fluid Extraction and Gas Chromatography−Mass Spectrometry

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A method for extraction and identification of volatile flavor components in roasted peanuts is described. The method is based on supercritical fluid extraction (SFE), with identification using gas chromatography−mass spectroscopy (GC−MS) in scan mode. Appropriate choice of supercritical fluid density (0.35 g/mL of CO_2) and extraction temperature (50 °C), at a pressure of 96 bar, results in selective extraction of compounds associated with roasted flavor rather than nonvolatile lipid material. The compounds examined in this study were hexanol, hexanal, methylpyrrole, benzene acetaldehyde, methylpyrazine, 2,6-dimethylpyrazine, ethylpyrazine, 2,3-dimethylpyrazine, 2,3,5-trimethylpyrazine, 2-furancarboxaldehyde, 2-ethyl-5-methyl- and 2-ethyl-6-methylpyrazine, and 3-ethyl-2,5-dimethylpyrazine. Careful grinding of frozen samples and alternate layering with silanized glass wool in the extraction thimble allows good recovery of the volatiles (>85%) in a single extraction step. Comparison of chromatograms for samples produced over a range of roasting conditions and sensory panel results shows that this method can be used to relate roasting conditions and consumer acceptance of roast quality.

Keywords: *Supercritical fluid extraction; peanuts; roasting; GC−MS*

INTRODUCTION

Supercritical fluid extraction (SFE) utilizes the unique solvent power of a medium at conditions above its critical temperature and pressure. Supercritical fluids have physical and transport properties characteristic of gases and chemical or solvent properties comparable to liquids. Compared to liquid solvents, supercritical fluids have lower viscosities and higher diffusivities, thus allowing more efficient mass transfer of solutes from sample matrices (McHugh et al., 1986). A major advantage of these fluids is that the solvating power can be adjusted through mechanical compression. Small changes in pressure can result in a substantial change in density and solvent power, thereby permitting selective extraction of target analytes with recoveries up to 100% (Magard et al., 1995). Sequential adjustment of SFE conditions can be used to separate different fractions from samples. Following extraction, the supercritical fluid is returned to a gaseous state leaving only the extract on the SFE trap and generally saving on solvent disposal costs.

Supercritical fluid extraction as a sample preparation technique in food analysis is gaining in popularity in recent years. Its ability to selectively extract the analytes of interest, without the presence of any contaminating solvent or other undesirable compounds, has made SFE a useful research tool. To establish a range of extraction conditions selective for flavor components rather than nonvolatile lipids, previously established methodology was reviewed. Since 1989, SFE has been used for the extraction of lipids in peanuts and muscle foods (Santerre et al., 1994; Bailey et al., 1993) and of oils in seeds (Taylor et al., 1993). In addition, SFE methods have also been utilized for the extraction of fatty acids in whole-wheat flour, soybeans (Arts and Sauer, 1992; Nikolov et al., 1992), and soybean oil (King, 1989) and of cholesterol in egg yolks (Ong et al., 1990).

Carbon dioxide is often used for SFE work due to the fact that it is nonflammable, odorless, chemically inert, easily disposed, and available in good purity at a relatively low cost. The low critical temperature (31.1 °C) permits extraction of thermally labile compounds. Supercritical critical CO_2 is a low polarity solvent that requires the addition of cosolvents or modifiers to dissolve polar compounds. For the purposes of this research, carbon dioxide was used alone as the analytes of interest, volatile and semivolatile flavor compounds in roasted peanuts, are low to moderately polar.

Several methods for the removal of volatile compounds associated with flavor from food samples have been reported: headspace analysis (Chang et al., 1977; Dickens et al., 1987; Young and Hovis, 1990), the use of impinging methodology with an external closed inlet device (ECID) (Vercellotti et al., 1992), and nitrogen purge-and-trap (NPT) (Ramarathnam et al., 1993a,b). The principle of headspace analysis is the removal and subsequent condensation of the volatile compounds in the headspace of a sample. To ensure that all of the compounds are captured in the headspace, the samples are heated at temperatures ranging from 55 °C under vacuum (Braddock et al., 1995) for roasted high oleic acid peanuts to as high as 150 °C (Young and Hovis, 1990) for raw and roasted peanuts. Due to the combination of high temperature and time required for headspace analysis, it was not acceptable for studying the kinetics of a thermal process such as peanut roasting.

The initial approach in our research was to strip volatile compounds from peanuts using inert gas flow through an ECID and to recover the stripped compounds by impinging the gas stream in a solvent trap. The

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temperatures used for stripping were 120−180 °C for the valve and 100−120 °C for the ECID inlet. GC−MS analysis of compounds stripped from raw peanuts showed that roasting reactions occurred during stripping at these temperatures, and this was unacceptable. Furthermore, the impinging solvent had to be evaporated to concentrate the extract for GC−MS analysis, and not surprisingly, substantial losses of the volatile flavor compounds occurred, even under moderate evaporation conditions.

Nitrogen purge-and-trap (NPT) was also examined as a suitable method for the removal of the peanut volatiles. However, there were problems creating reference standards. Preliminary tests run with standard compounds injected onto a solid adsorbent polymer Tenax GC column (*p*-2,6-diphenylene oxide) demonstrated that recovery of the volatiles from the Tenax decreased with an increase in standard concentration.

In this paper, we present a selective, efficient, and rapid SFE method for extraction of flavor compounds from roasted peanuts. GC−MS analysis is used to identify, at quantitative and qualitative levels, a group of extracted compounds known to relate to roasted flavor (Johnson et al., 1971a,b). The chemical profiles are correlated with sensory evaluation of roasted flavor and consumer judgment of quality. This knowledge of how roasting conditions affect flavor profiles and consumer acceptance will ultimately be used to control roasting conditions.

MATERIALS AND METHODS

Peanut Sample Preparation. Peanuts were roasted under controlled conditions (a range of air temperatures from 145 to 170 °C and roasting times from 3 to 20 min) and stored in sealed containers in a freezer (−10 °C). Just prior to extraction, frozen samples were finely ground in a coffee mill, Model KSM2 (Braun).

Supercritical Fluid Extraction. Extractions were carried out using a Hewlett-Packard Model 7680T supercritical fluid extractor (Mississauga, ON) controlled by Windows-based, HP Chemstation software (Hewlett-Packard). The extraction medium was carbon dioxide with a purity of 99.995% (BOC Gases, Guelph, ON). Carbon dioxide (extra-dry grade, BOC Gases) was also used for cryogenic cooling of various zones in the SFE apparatus.

A portion of ground peanut sample (2.00−2.50 g) was weighed and loaded into the extraction thimble (7 mL, Hewlett-Packard) in alternating layers of heat-treated, silanized glass wool followed by half of the ground peanut sample. A plug of glass wool was placed on top of the peanut sample.

A range of supercritical conditions and extraction procedures was tested. Highest recoveries were obtained using a 10-min static equilibration step, followed by a 10-min dynamic extraction period. Both stages were performed at a pressure of 96 bar with a chamber temperature of 50 °C, resulting in a CO_2 density of 0.35 g/mL. Hewlett-Packard 7-mL sample thimbles were used, and 7.5 thimble volumes were swept. Extractions were performed with a chemically active diol trap (Hewlett-Packard), consisting of silica particles with a hydrophilic coating. Trap and nozzle temperatures were set at −5 and 45 °C, respectively, for the extraction. When the extraction was complete, the analyte trap containing the peanut volatiles was rinsed into 1.5-mL collection vials with approximately 1.0 mL of methylene chloride. The SFE collection vials were weighed before extraction and after rinsing to determine the exact weight of methylene chloride to provide a basis for concentration calculations. During the rinse stage, a trap temperature of 5 °C was used to help dissolve and transport the collected analytes to the collection point, and the nozzle temperature was lowered to 10 °C to limit volatilization of the methylene chloride.

Gas Chromatography−Mass Spectrometry (GC−MS). GC−MS analysis of the SFE extract in methylene chloride was performed on a Hewlett-Packard 5890 Series II gas chromatograph interfaced to a Hewlett-Packard 5971A mass spectrometer. The gas chromatograph was equipped with a RTX-200 capillary column 60 m × 0.32 mm coated with a 1.0-μm film of trifluoropropylmethyl polysiloxane (Restek Corp, Bellefonte, PA). The carrier gas was UHP-grade helium with a purity of 99.999% (BOC Gases) and delivery pressure was set to 280 kPa. The injector was used in splitless mode for 2 min and set at 250 °C. Following an initial isothermal period of 1.0 min at 30 °C, the temperature was raised to 190 °C at a rate of 7 °C/min and held there for 10 min. The mass detector temperature was set at 280 °C, and the detector was turned on after the first 10 min of separation. The mass spectrometer was used in scan mode, monitoring the abundance at every 0.1 amu over a scan range of masses from 25 to 250 at a rate of 1.4 scans/s. Earlier literature (Johnson et al., 1971a,b) has established that compounds associated with roasted peanut flavor are smaller than the upper mass limit of 250 amu.

Identification and Quantitation of Individual Compounds. Identification of the compounds in the chromatograms was based on a probability-based matching algorithm library using all of the ion fragments rather than a specific set of target ions. A user-created library was also developed from the spectra of standards obtained on the same instrument and at the same operating conditions as the sample spectra. The search of both libraries gave a higher degree of confidence in identification of the compounds reported.

In addition to the mass spectral identification, a mixed external standard, prepared at a suitable range of concentrations, was used to determine retention times and to develop standard curves for quantitation of the peanut volatiles. Individual standards were purchased from Aldrich (Milwaukee, WI). Methylene chloride, Optima grade, was purchased from Fisher Scientific (Nepean, ON).

Sensory Analysis. Sensory analysis was performed by a group of eight panelists, who evaluated aroma, flavor and color of the roasted peanut samples over several tasting sessions. At the beginning of each session, four reference samples were presented to the panelists. The sensory evaluation form listed linguistic terms to describe different levels of sensory perception (e.g., strong bitterness, weak roasted flavor) and judgment of overall quality (excellent, acceptable, marginal and unacceptable). Panelists indicated a judgment for each linguistic term on a five-value scale ranging from definitely false to definitely true (Sun, 1996). These linguistic truth values were converted to fuzzy values between 0.0 and 1.0 and then aggregated over all of the panelists for each sample attribute (i.e., all opinions combined as a fuzzy value). The center of area of each aggregated truth value was calculated to represent the overall opinion of the panelists (Driankov et al., 1993). A value close to 1.0 indicated that the panelists considered the linguistic term to be a true value for the sensory or quality attribute.

RESULTS AND DISCUSSION

Supercritical Fluid Extraction. Traditional flavor extraction techniques, such as purge-and-trap, headspace, and distillation, are usually labor-intensive, time-consuming, and prone to losses when a concentration of a liquid solvent phase is necessary. A SFE method was developed to provide a simple and rapid extraction (20 min) technique for peanut samples. The extract was free of nonvolatile lipid material and was dissolved in methylene chloride for GC−MS analysis.

The initial extraction experiments were conducted to define supercritical extraction conditions that removed roasted flavor components from the peanuts with no or minimal extraction of the triglyceride fraction. A substantial amount of nonvolatile lipid material in the extract was undesirable since it would degrade the front end of the capillary GC column. Since lipid solubility increases with increasing density and temperature of

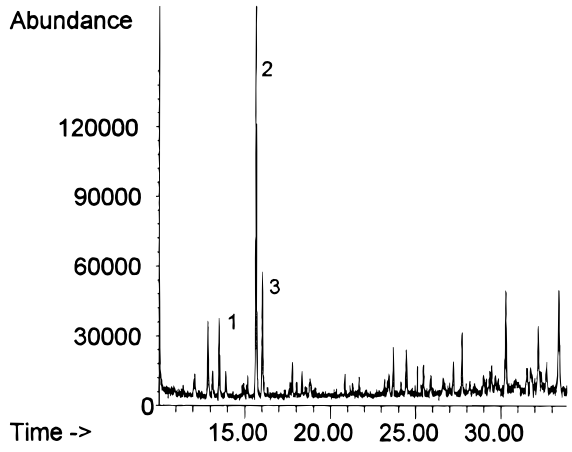

Figure 1. Peak profile for extract from mildly roasted peanuts (145 °C, 3 min): 1, methylpyrrole; 2, hexanol; 3, hexanal.

Table 1. Range of Supercritical Fluid Extraction Conditions Examined and Conditions Chosen for Recovery of Roasted Flavor Components

	range examined	conditions chosen for recovery of flavor components
chamber temp (°C)	50−55	50
CO_2 density (g/mL)	0.35−0.40	0.35
CO_2 flow rate (mL/min)	1.5−2.0	2.0
extraction times (min)		
static	5−10	10
dynamic	5−10	10
trap temperatures	−5 and 5	−5

the supercritical CO_2 phase (Friedrich and Pryde, 1984; Gere et al., 1993; Majors, 1994; Santerre et al., 1994), low-density (<0.40 g/mL) and low-temperature (< 55 °C) extraction conditions were explored. Table 1 summarizes the range of extraction conditions that were studied as well as the conditions within this range that resulted in maximum recovery of roasted flavor compounds. These extraction conditions were used for all of the reported analysis. Samples were extracted a second time, at the same SFE conditions, to confirm that a single extraction step was sufficient for complete extraction of roasted flavor compounds. Complete removal of the volatiles from the analyte trap was confirmed by GC−MS analysis of a second rinse sample from the trap.

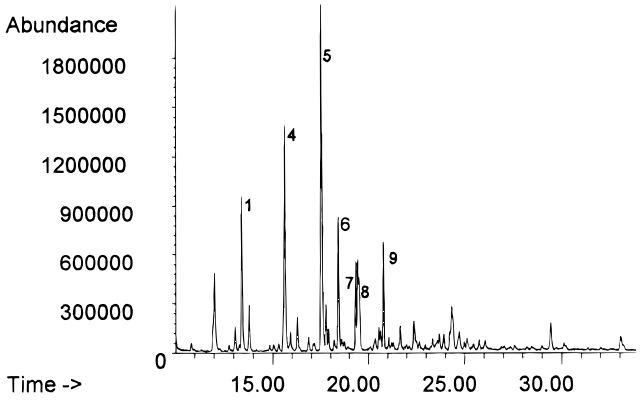

Figure 2. Peak profile for extract from peanuts roasted at severe conditions (170 °C, 17 min): 1, methylpyrrole; 4, methylpyrazine; 5, 2,6-dimethylpyrazine; 6, 2-furancarboxaldehyde; 7, 2,3,5-trimethylpyrazine; 8, 2-ethyl-5-methylpyrazine and 2-ethyl-6-methylpyrazine; 9, 3-ethyl-2,5-dimethylpyrazine.

Trials were also conducted to ensure that the flavor components were not lost during sample preparation for SFE. Cryogenic grinding with dry ice was tested. However the GC−MS results for cryogenic grinding were identical to the results when samples were ground directly from the freezer (−10 °C).

Diatomaceous earth was added to some of the ground samples to determine if this improved CO_2 permeation. The dispersant was not effective in enhancing extraction so it was not used.

GC−MS Analysis. Total ion chromatograms for two peanut extracts are shown in Figures 1 (mild roasting conditions) and 2 (severe roasting conditions). Selected peaks associated with roasted flavor are identified on the total ion chromatograms. Table 2 is a summary of flavor components identified in extracts of raw and roasted peanuts with quantitative estimates of the amounts produced at different roasting conditions. For the flavor compounds such as 2-furancarboxaldehyde, 2-ethyl-, 5-methylpyrazine; 3-ethyl-2,5-dimethylpyrazine, and benzene acetaldehyde, only qualitative estimates (i.e., present or absent) were possible, and these are presented in Table 3.

Table 2. Components in Supercritical CO_2 Extracts of Raw and Roasted Peanuts (Quantitative Identification)[a]

roasting conditions		methyl-pyrrole	hexanol	methyl-pyrazine	hexanal	2-furan-methanol	2,6-dimethyl-pyrazine	ethyl-pyrazine	2,3-dimethyl-pyrazine	2,3,5-trimethyl-pyrazine
temp (°C)	time (min)	(μg/g)	(μg/g)	(μg/g)	(μg/g)	(μg/g)	(μg/g)	(μg/g)	(μg/g)	(μg/g)
raw peanuts		*	*	1.24E−03	*	*	*	*	*	*
raw peanuts		*	*	9.99E−04	*	*	*	*	*	*
raw peanuts		*	*	1.16E−03	*	*	*	*	*	*
145	5	8.95E−02	2.97E−03	*	1.99E−02	*	5.13E−04	*	5.82E−04	2.07E−04
145	5	1.11E−01	2.87E−03	*	1.52E−02	*	7.62E−04	*	*	2.59E−03
145	10	1.12E−01	1.95E−03	*	1.64E−02	*	2.61E−03	*	*	6.12E−04
145	10	9.78E−02	2.41E−03	*	1.29E−02	*	2.23E−03	*	*	6.55E−04
150	10	1.07E−01	9.30E−04	8.84E−04	1.09E−02	2.43E−03	3.51E−03	*	*	8.19E−04
150	10	1.36E−01	1.30E−03	1.25E−03	1.29E−02	*	3.97E−03	2.24E−04	4.32E−04	9.48E−04
150	20	4.22E−01	3.06E−03	4.78E−03	1.17E−02	9.54E−04	1.15E−02	6.97E−04	*	2.41E−03
150	20	3.62E−01	2.97E−03	4.26E−03	1.40E−02	1.12E−03	1.07E−02	6.89E−04	*	2.33E−03
165	20	112E+00	*	1.66E−02	*	1.95E−03	2.05E−02	2.00E−03	1.16E−03	4.48E−03
165	20	9.33E−01	*	1.66E−02	*	2.91E−03	2.13E−02	2.08E−03	1.41E−03	4.14E−03
170	17	1.66E+00	*	2.29E−02	2.46E−02	3.62E−03	2.73E−02	*	*	5.26E−03
170	17	1.90E+00	*	2.81E−02	2.57E−02	4.93E−03	3.11E−02	3.52E−03	1.91E−03	6.81E−03

[a] An asterisk (*) indicates below minimum limit for quantification. Estimates of minimum limits for quantification (based on extraction of 2.0−2.5 g of peanuts): methylpyrrole, 8E−02 μg/g; hexanol, 4E−04 μg/g; methylpyrazine, 8E−04 μg/g; hexanal, 1E−02 μg/g; 2-furanmethanol, 1E−03 μg/g; 2,6-dimethylpyrazine, 5E−04 μg/g; ethylpyrazine, 2E−04 μg/g; 2,3-dimethylpyrazine, 1E−03 μg/g; 2,3,5-trimethylpyrazine 2E−04 μg/g.

Table 3. Components in Supercritical CO_2 Extracts of Raw and Roasted Peanuts (Qualitative Identification)[a]

temp (°C)	time (min)	2-furancarbox-aldehyde	2-ethyl-5-methylpyrazine and 2-ethyl-6-methylpyrazine	3-ethyl-2,5-dimethylpyrazine	benzene acetaldehyde
raw peanuts	–	–	–	–	
145	5	–	–	–	–
145	10	–	+	+	+
150	10	+	+	+	+
150	20	–	+	+	–
165	20	+	+	+	–
170	17	+	+	+	–

[a] Analyses for duplicate samples were identical. (+) compound present; (−) compound not present.

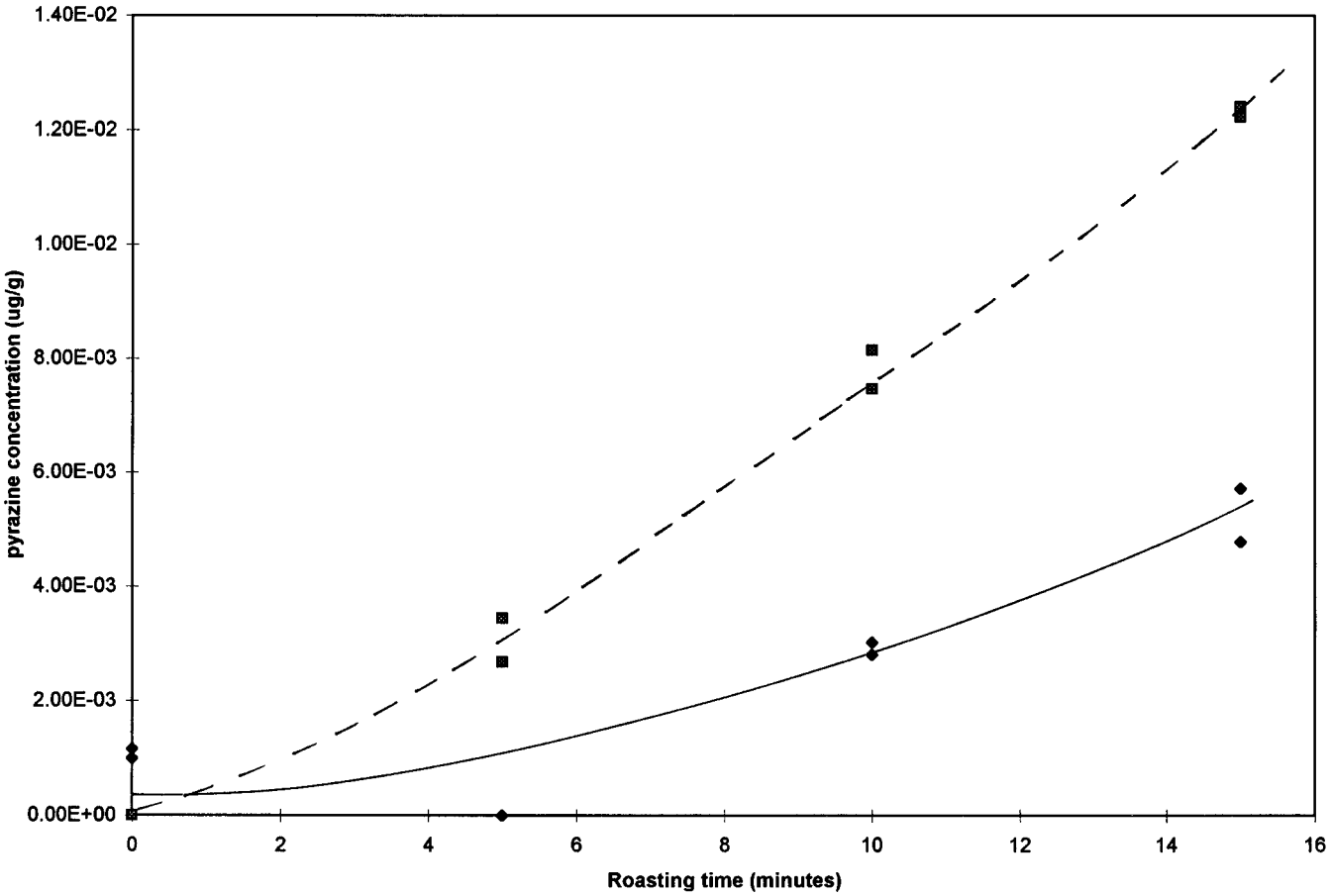

Figure 3. Pyrazine components identified in supercritical extracts of peanuts roasted at 160 °C from 5 to 15 min (◆, methylpyrazine; ■, sum of ethyl-, dimethyl-, and trimethylpyrazines).

As roasting conditions changed from mild (i.e., low temperatures, short times) to severe (i.e., high temperatures, long times), a range of pyrazine compounds was formed as well as other flavor compounds such as methylpyrrole. Under the GC conditions reported here, the hexanol and methylpyrazine peaks were not well resolved for certain roasting conditions. In lightly roasted samples, the hexanol peak was dominant, but there was always a trailing shoulder present. Inspection of the mass spectra of these shoulders revealed that they were likely methylpyrazine. However, it was not possible to integrate two separate peaks, and hexanol estimates in Table 2 were based on total areas (i.e., including shoulders). In peanut samples roasted under severe conditions, the methylpyrazine peak was identified but also included a distinct leading shoulder. An examination of this region showed an ion profile representative of hexanol. While complete GC separation of hexanol and methylpyrazine was not possible for samples representing extreme roasting conditions, these components could be resolved for moderate roasting conditions (e.g., 150 °C and 10−20 min).

Sensory Analysis. Roasting at high temperatures (>120 °C) produces a wide range of chemicals in peanuts due to Maillard reactions. Methylpyrazine is associated with "roasted" flavor and is desirable at low concentrations. However, as the concentration of methyl and other pyrazine compounds increases, the flavor becomes increasingly bitter. Air temperature and roasting time directly affected the levels of pyrazine derivatives (Table 2 and Figure 3).

Sensory analysis of these roasted peanuts confirmed a close relationship between the level of pyrazine compounds and perception of roasted flavor. Panelists evaluated four levels of roasted flavor (weak, moderate, strong, and burnt) and four levels of overall quality (excellent, acceptable, marginal, and unacceptable) for each sample. The group's overall opinion was summarized as a truth value between 0.0 (false) and 1.0 (true) for each level of roasted flavor and overall quality. Figure 4 shows truth values for the linguistic descriptors as the roasting time changed from 5 to 15 min (160 °C). When Figure 4 is compared to Figure 3, it is clear that the increase in pyrazine compounds corresponded to a

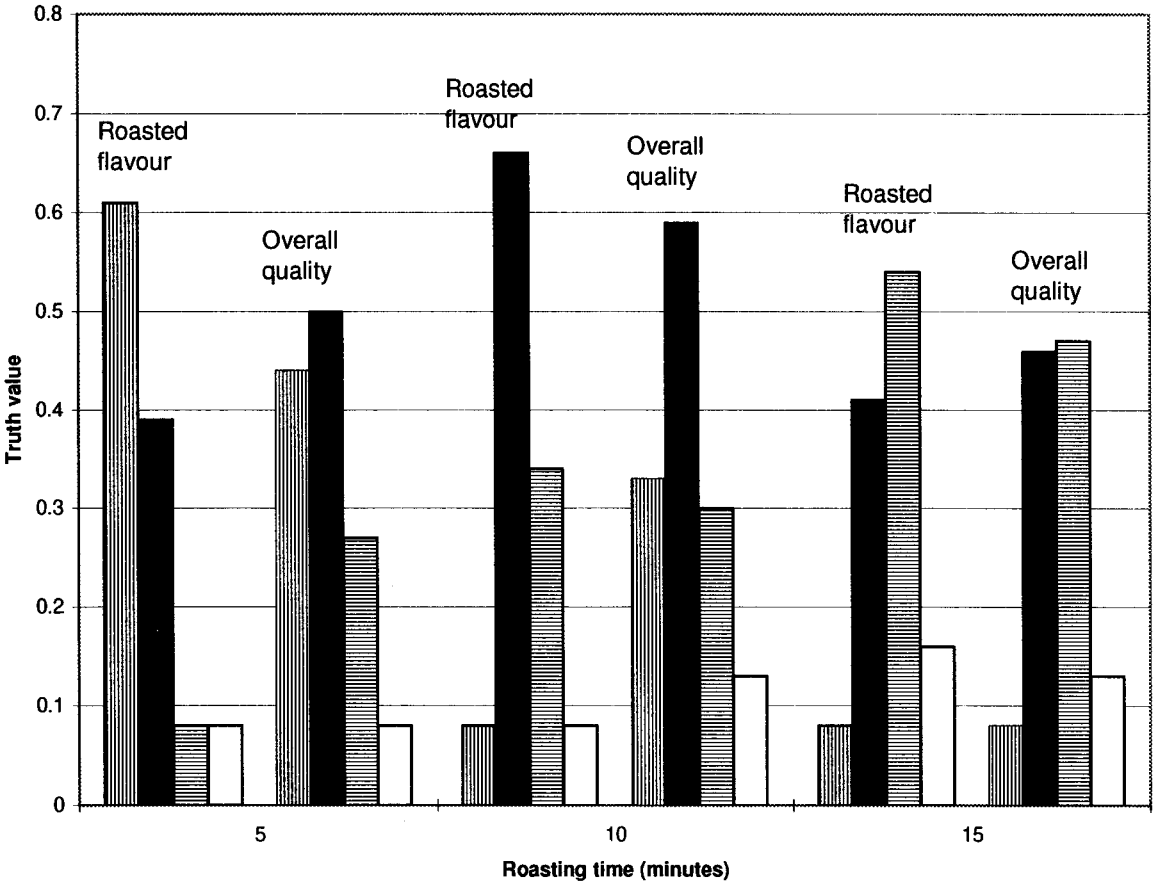

Figure 4. Sensory ratings for peanuts roasted at 160 °C from 5 to 15 min. Roasted flavor: (vertically striped bar) weak; (solid bar) moderate; (horizontally striped bar) strong; (open bar) burnt. Overall quality: (vertically striped bar) excellent; (solid bar) acceptable; (horizontally striped bar) marginal; (open bar) unacceptable.

transition between weak and strong roasted flavor. As the concentration of pyrazine compounds increased, the judgment on overall quality of the peanuts changed from clearly excellent to a split decision between acceptable and marginal.

It is recognized that many other compounds can affect the perception of roasted flavor and overall quality. However the objective of this study was to determine changes in specific compounds associated with flavor impact as roasting conditions changed and to relate these changes to the consumer perception of roast flavor and overall quality. The authors recognize that correlation between observations is not always equivalent to a causal relationship. However it often gives a useful starting point for making process control decisions. The work will continue to develop roasting control strategies that can make reasonable compromises between conflicting objectives such as maximum throughput and consistent acceptable quality.

Conclusions. An optimum set of SFE conditions was developed to extract some of the important flavor compounds from roasted peanuts. The use of a 10-min static equilibration time, followed by a 10-min dynamic extraction time, with a chamber temperature of 50 °C, a CO_2 density of 0.35 g/mL, and a pressure of 96 bar, resulted in a rapid and efficient extraction procedure. This SFE method proved advantageous over ECID and purge-and-trap methods since the peanut extract could be taken up directly into a solvent (methylene chloride) and prepared for injection in a manner similar to the external standard. In addition, the extraction could be performed at lower temperatures than ECID stripping, thereby avoiding any further roasting of the peanuts. Subsequent GC−MS analysis confirmed the presence of a range of pyrazine compounds as well as hexanol, hexanal, and methylpyrrole that were related to the severity of roasting conditions and sensory perceptions of a taste panel. The concentration of methylpyrazine and other pyrazines was directly related to the human perception of "roasted" flavor and overall quality.

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