JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY



# Quantitative Determination of Geosmin in Red Beets (*Beta vulgaris* L.) Using Headspace Solid-Phase Microextraction

GUIPING LU,*,† JOHN K. FELLMAN,‡ CHARLES G. EDWARDS,† D. SCOTT MATTINSON,‡ AND JOHN NAVAZIO§

Department of Food Science and Human Nutrition, Washington State University, Pullman, Washington 99164-6376; Department of Horticulture and Landscape Architecture, Washington State University, Pullman, Washington 99164-6414; and Alf Christianson Seed Company, Mt. Vernon, Washington 98273

An improved analytical method for the determination of geosmin in red beets was developed using headspace solid-phase microextraction (HSPME). Volatiles of beet juice were extracted in headspace for 2 h using a polydimethylsiloxane/divinylbenzene fiber, thermally desorbed from the fiber, and analyzed by gas chromatography. The HSPME method was determined to be suitable for geosmin analysis as evidenced by high relative recovery (99.2%), low relative standard deviation (7.48%), and reasonable detection limit (1 $\mu$g/kg of beet root tissue). The concentrations of geosmin in four beet cultivars ranged from 9.69 $\pm$ 0.22 to 26.7 $\pm$ 0.27 $\mu$g/kg, depending on cultivar.

**KEYWORDS: Geosmin; headspace solid-phase microextraction (HSPME); purge-and-trap; red beets (*Beta vulgaris* L.)**

## INTRODUCTION

Red beets (*Beta vulgaris* L.) are nutritious vegetables, the roots being rich in folic acid and iron and the leaves high in vitamins A and C (*1*). However, production of red beets destined for processing in the United States amounts to only 8000 acres, a small amount compared to other vegetables (*2*, *3*). One potential reason for low production is the characteristic "earthy" flavor some people find objectionable (*4*), a flavor due to the presence of geosmin (*5*, *6*).

Geosmin, *trans*-1,10-dimethyl-*trans*-(9)-decalol (**Figure 1**), has been extracted and analyzed from water, soil, or fish samples by various methods including closed loop stripping, purge-and-trap, and microwave distillation (*7*−*9*). Most of these methods include a preconcentration step before analysis by gas chromatography−flame ionization detection (GC−FID) or gas chromatography−mass spectrometry (GC−MS) (*7*, *8*, *10*, *11*). For instance, Tyler et al. (*12*) developed a liquid−liquid extraction method for red beets using Freon 113, followed by purification using a Florisil column and GC analysis. Unfortunately, many of these methods require special or expensive equipment and/or relatively large samples. As an example, Buttery et al. (*13*−*15*) developed a closed loop purge-and-trap method that required a sample size of 100 g and utilized an all Teflon diaphragm pump to sweep volatiles from food and plant materials onto a 10 g Tenax trap.

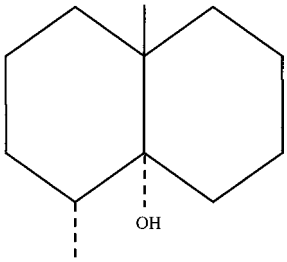


**Figure 1.** Chemical structure of geosmin.

Recently, headspace solid-phase microextraction (HSPME) was investigated for the quantitative analysis of geosmin in water (*11*, *16*). Using a dual coated fiber of polydimethylsiloxane/carboxen/divinylbenzene (PDMS/CAR/DVB), HSPME exhibited a highly linear detector response ($R^2 = 0.999$) for 30 mL aqueous solutions of geosmin at concentrations ranging from 1 to 20 ng/L (*17*). In fact, Watson et al. (*11*) reported a detection limit of 3.3 ng/L with a relative standard deviation (RSD) of only 1.9% using PDMS/DVB fibers. In general, HSPME is very simple to perform because the coating of polymer on a fused silica fiber combines extraction, concentration, and injection into a single process (*18*). Furthermore, this method does not utilize organic solvents and requires a smaller sample. Because of these advantages, the objective of this study was to develop a sensitive quantitative HSPME method for the analysis of geosmin in red beets.

* Address correspondence to this author at 416 W. P. Garrigus Building, Department of Animal Sciences, University of Kentucky, Lexington, KY 40546-0215 [e-mail gpl@uky.edu; telephone (859) 257-5985; fax (859) 257-7537].

† Department of Food Science and Human Nutrition, Washington State University.

‡ Department of Horticulture and Landscape Architecture, Washington State University.

§ Alf Christianson Seed Co.

10.1021/jf020806d CCC: $25.00 
Published on Web 01/10/2003

## MATERIALS AND METHODS

**Chemical and Plant Materials.** Standard geosmin (98% purity) was obtained from Sigma (St. Louis, MO), whereas the internal standard, (−)-menthone (95% purity), was purchased from Aldrich (Milwaukee, WI). NaCl (crystals, reagent, 99.9% purity), methanol (HPLC grade), and anhydrous $Na_2SO_4$ were obtained from J. T. Baker (Phillipsburg, NJ), and anhydrous diethyl ether was purchased from Fisher Scientific (Pittsburgh, PA). Tenax TA (80/100 mesh) was acquired from Supelco (Bellefonte, PA). Different cultivars of red beet roots, Chioggia, Crosby Green Top, Crosby Egyptian, Cylindra, Detroit Dark Red, Lutz Green Leaf, and Round Red, were obtained from Alf Christianson Seed Co. (Mt. Vernon, WA); additional beet roots (unknown cultivar) were purchased from a local grocery store.

**Purge-and-Trap.** Geosmin was extracted using a closed loop purge-and-trap system based on that of Buttery et al. (*14*) and later modified. Beet roots (200 g of Round Red cultivar) were blended in a two-speed blender (Waring) with 120 mL of distilled water and 135 g of NaCl at high speed for 1 min before transfer to a 2 L flask. Another 80 mL of distilled water was used to rinse the beet residue from the blender into the flask. Samples were purged in the closed loop purge-and-trap system at room temperature (*14*) while stirred using a spin bar at 800 rpm (Mag-Mix, Precision Scientific Co., Chicago, IL). After 2.5 h of purging, the traps were removed and eluted with 50 mL of diethyl ether. The ether eluate was filtered through 5 g of anhydrous $Na_2SO_4$ to remove water and concentrated to ~30 $\mu$L in a 45 °C water bath.

Gas chromatography was accomplished using a Hewlett-Packard 5890 series II GC equipped with an autosampler (HP 7673) and a flame ionization detector. The temperatures of the injector and detector were maintained at 200 and 275 °C, respectively. The ether concentrate was injected (1 $\mu$L) into the GC under splitless mode. Separation was achieved using a 30 m × 0.32 mm i.d., 0.25 $\mu$m film thickness, SE-54 fused silica capillary column (Alltech, Deerfield, IL). The temperature program was as follows: initial temperature, 50 °C; increased at 2.5 °C/min to 135 °C; increased at 10 °C/min to 280 °C; and held for 20 min. Chromatographic data were analyzed using Chrom Perfect program (Mt. View, CA). All extractions were performed in triplicate. Tenax traps were conditioned at 210 °C for 2 h by passing prepurified $N_2$ through the trap at ~200 mL/min prior to use.

**HSPME.** Beet roots were blended with distilled water (1:1 w/w) in a Waring blender for 1 min at high speed. Aliquots (10 g) of the beet juice were weighed into 81 × 29 mm vials, and headspace volumes were altered by addition of 0, 5, 10, or 15 g of distilled water and 3.49, 5.36, 7.20, or 9.00 g of NaCl, respectively. Thus, the headspace/total volume ratios for the four treatments were 75, 62.5, 50, and 37.5%, respectively. Each vial was then sealed by a cap containing a PTFE/silicone septum and frozen. Once the beet juice was thawed in a 60 °C water bath, a spin bar was added for mixing of the beet juice (750 rpm), and headspace was extracted for 1 h using 65 $\mu$m thick PDMS/DVB fibers (Supelco). A 1.4 cm thick rubber spacer was placed between the vial cap and the fiber holder to position the fiber and prevent the fiber from contamination by beet juice. Replicate samples containing 10 g of beet juice, 5 g of distilled water, and 5.36 g of NaCl were extracted for different times (0.5, 1, 1.5, 2, and 2.5 h).

PDMS/DVB fibers were thermally desorbed of geosmin at 260 °C for 10 min into the injector block of the same GC system, equipped with a 0.75 mm i.d. injection sleeve for SPME (Supelco). The injector and detector temperatures were maintained at 260 and 275 °C, respectively. The temperature program was as follows: 35 °C for 1 min; increased at 2 °C/min to 140 °C; increased at 10 °C/min to 280 °C; and held for 5 min. The total run time was 72.5 min. New PDMS/DVB fibers were conditioned for 30 min at 260 °C prior to use.

A standard curve to check the linear response of HSPME was prepared by adding standard geosmin using a microsyringe (Precision Sampling Corp., Baton Rouge, LA) to vials containing 15 g of distilled water and 5.60 g of NaCl to yield concentrations from 46.7 to 2770 ng/L of water. Vial headspace was sampled using HSPME for 2 h at 60 °C, and a calibration curve was prepared by plotting peak area against concentration of geosmin.

To determine the relative recovery of geosmin from beet roots, vials containing 10 g of beet juice, 5 g of distilled water, and 5.36 g of NaCl were spiked with standard geosmin to yield concentrations of 0.505−21.6 $\mu$g/kg of beet root tissue and the internal standard, (−)-menthone, at a constant concentration of 5.64 $\mu$g/kg beet root tissue. Vial headspace was then sampled for 2 h at 60 °C. A calibration curve for calculation of the relative recovery of geosmin from beet roots was prepared by plotting the area ratio of geosmin to (−)-menthone versus the concentration ratio of geosmin to (−)-menthone. The calculation of the relative recovery of geosmin from beet roots was based on the following equation:

$$\text{relative recovery} = \frac{(\text{geosmin total} - \text{geosmin originally in beets})}{\text{geosmin spiked}} \times 100\%$$

**Statistical Analysis.** Data were analyzed by Tukey's pairwise comparison using SAS (SAS Institute Inc., Cary, NC) at $p \leq 0.05$ defined as a significant difference.

**Table 1.** Concentration of Geosmin in Several Beet Cultivars Determined Using the Purge-and-Trap Method

| cultivar | geosmin[a] ($\mu$g/kg) | RSD[b] (%) |
|---|---|---|
| Detroit Dark Red | 2.02 ± 0.43[c] | 21.3 |
| Crosby Green Top | 3.19 ± 1.1[cd] | 34.5 |
| Lutz Green Leaf | 3.88 ± 1.6[cd] | 41.2 |
| Crosby Egyptian | 4.15 ± 0.57[cd] | 13.7 |
| Cylindra | 4.28 ± 0.86[cd] | 20.1 |
| Chioggia | 6.09 ± 2.5[d] | 41.1 |

[a] Based on wet weight of beets. Means with different letters are significantly different ($p \leq 0.05$). [b] Relative standard deviation (RSD) = standard deviation/mean.

## RESULTS AND DISCUSSION

**Purge-and-Trap.** The concentrations of geosmin in the roots of six beet cultivars were determined using the previously described purge-and-trap method. As shown in **Table 1**, the concentration of geosmin ranged from 2.02 $\mu$g/kg for Detroit Dark Red to 6.09 $\mu$g/kg for Chioggia. Chioggia and Detroit Dark Red were the only beet cultivars significantly different in geosmin concentration. These values were higher than those published by Tyler et al. (*19*), who noted geosmin concentrations of 0.8, 0.8, and 1.0 $\mu$g/kg for cultivars Detroit Dark Red, Cylindra, and Crosby Egyptian, respectively. However, relative standard deviations (RSD = standard deviation/mean) for the means obtained by the purge-and-trap method were quite high, ranging from 13.7 to 41.2% depending on beet cultivar. Although the same purge-and-trap method was used by Buttery et al. (*13*, *14*) to quantify other volatiles present in vegetables, variability was not reported. Because the purge-and-trap method yielded such high variability, this method was discarded in favor of developing an SPME technique for the analysis of geosmin in red beets.

**Optimization of HSPME.** Although HSPME is a simple equilibrium sampling technique, the method requires careful control of sampling conditions for efficient recovery and quantitative analysis of compounds. Such sampling conditions include extraction mode (immersion or headspace), addition of salt, fiber type, temperature, sample agitation, fiber position, sample size, headspace volume, and extraction time (*18*, *20*). To simplify the development of a quantitative method, headspace sampling at 60 °C with the addition of 37% NaCl (saturated concentration at 60 °C) and PDMS/DVB fibers were used on the basis of the reports of others (*11*, *16*, *17*, *21*, *22*). Fiber position in the headspace and stirring rate were also fixed. A sample size of 5 g was chosen because preliminary experiments showed that extraction of a 5 g beet root sample yielded

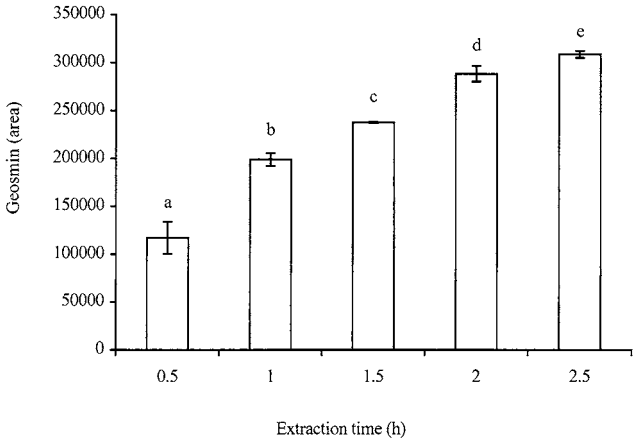


**Figure 2.** Area of geosmin peak extracted from beet roots for different times using HSPME. Means with different letters are significantly different ($p \leq 0.05$); error bars indicate one standard deviation.

an adequate GC−FID signal (**Figure 2**). The variables optimized were headspace volume and extraction time.

To change headspace volumes, different amounts of distilled water were added to vials containing 10 g of beet juice. For the HSPME technique, the equilibria are established among the concentrations of geosmin in the sample, in the headspace above the sample, and in the polymer coating on the fused silica fiber (*18*). Thus, changing the headspace volume may change the recovery efficiency of geosmin. However, varying the headspace volume through the addition of different amounts of distilled water produced no significant differences in the recovery of geosmin from beets (data not shown). In contrast, Lloyd et al. (*23*) reported that the recovery of geosmin from water increased dramatically when the ratio of headspace volume to liquid volume (percent headspace) decreased from 65 to 30%. However, it is difficult to compare the recovery of geosmin reported by Lloyd et al. (*23*) with that from the present study because these authors used a different fiber (PDMS) and varied both the vial size and sample size. Bao et al. (*21*) found a decrease in headspace from 81.9 to 45.6% only slightly increased the recovery of geosmin from water using PDMS/DVB fibers. Because beets and water are different matrices, disagreement between the result of this study and the literature is expected. In general, the optimum headspace/total volume should be 25% or less (*23*). Unfortunately, none of the treatments in this study reached this value because lower headspace volumes would have resulted in immersion of the fiber into the sample in the 40 mL vial. Although there was no difference in recovery of geosmin by changing headspace volume, the signals of geosmin peak for the four treatments were adequate (above 170000 peak area). Therefore, 62.5% of headpsace to total volume (i.e., addition of 5 g of water) was chosen because the water could be used to wash down beet residues on the vial wall, and less NaCl would be used to saturate beet samples compared to choosing lower headspace volume.

The amount of geosmin extracted from beet samples increased with an increase in extraction time (**Figure 2**). In fact, extraction equilibrium had apparently not been achieved after an extraction time of 2.5 h. In agreement, Bao et al. (*21*) noted that equilibration was not reached by 2 h when PDMS/DVB fibers were used for geosmin analysis of water samples at room temperature, and equilibration time for an analyte depended on the polarity and the relative molecular mass of the analytes in water. In the present experiment, extraction equilibrium was not reached at 2.5 h, possibly because geosmin is a relatively large polar molecule (molecular weight of 182) and beet juice is a complex matrix. Because the increase in recovery of geosmin between 2 and 2.5 h was only 7.08%, 2 h was chosen as the extraction time.

**Table 2.** Relative Recovery of Geosmin Extracted from Beet Roots using HSPME

| spiked geosmin ($\mu$g/kg) | relative recovery (%) | RSD[a] (%) |
|---|---|---|
| 21.6 | 97.5 ± 8.7 | 8.92 |
| 11.4 | 108 ± 0.63 | 0.581 |
| 5.50 | 100 ± 2.4 | 2.34 |
| 2.05 | 90.5 ± 13 | 14.4 |
| 1.03 | 75.5 ± 16 | 21.8 |
| 0.505 | nd[b] | |
| av[c] | 99.2 ± 7.4 | 7.48 |

[a] Relative standard deviation (RSD) = standard deviation/mean. [b] No difference in area between unspiked and spiked samples. [c] Average excluding samples spiked with ≤1.03 $\mu$g/kg geosmin.

**Linearity of Response.** In this study, a calibration curve was prepared by adding standard geosmin to distilled water to yield concentrations from 46.7 to 2770 ng/L prior to extraction. HSPME exhibited a high degree of linearity ($R^2 = 0.998$) for a correlation equation of area = 242006 × concentration (ng/L) + 66189 and reasonable precision at these concentrations (RSD from 2.45 to 9.82%). Similarly, a high linearity in FID response for geosmin extracted from water ($R^2 = 0.999$) at concentrations from 1 to 20 ng/L using HSPME has been reported (*17*). Watson et al. (*11*) also found a highly significant linear detector response over a range of 1−80 ng/L for the analysis of geosmin in water ($R^2 = 0.993$), concentrations lower than those found in beets (*19*).

**Relative Recovery and Detection Limit.** To minimize run-to-run variation and matrix effects, (−)-menthone was added to beet samples as an internal standard. After an extensive search, (−)-menthone was chosen as the internal standard because preliminary study showed that it separated well from other compounds and achieved a relative recovery close to 100%. A GC chromatogram of volatiles extracted from Chioggia beet roots indicating the elution of geosmin and (−)-menthone is shown in **Figure 3**.

The calibration curve for calculation of the relative recovery of geosmin from beet roots was prepared. The HSPME using PDMS/DVB fibers also produced a highly linear detector response over the concentrations spiked in beet samples ($R^2 = 0.995$). The correlation equation was weight ratio of geosmin/menthone = 1.3711 × area ratio of geosmin/menthone − 0.0106. The recovery of geosmin ranged from 90.5 to 108% with a mean value of 99.2% for concentrations of spiked geosmin from 2.05 to 21.6 $\mu$g/kg (**Table 2**). The value of 99.2% indicates that (−)-menthone and geosmin had similar volatilities in beet juices. Furthermore, the low RSD (0.581−14.4%) implies that the method also exhibited high precision and reproducibility.

Detection limits are traditionally described as the concentration of a compound that gives a detector signal equal to 3 times the peak-to-peak noise level of the baseline (*24*). Because geosmin is a characteristic compound of red beets, it is difficult to determine the detection limit of geosmin in red beets for the HSPME method on the basis of this definition. Hence, the minimum concentration of geosmin that can be reported with 99% confidence to be greater than the blank is defined as the detection limit of the HSPME method (*24*). Spiking geosmin at 0.505 $\mu$g/kg did not produce a significantly higher detector

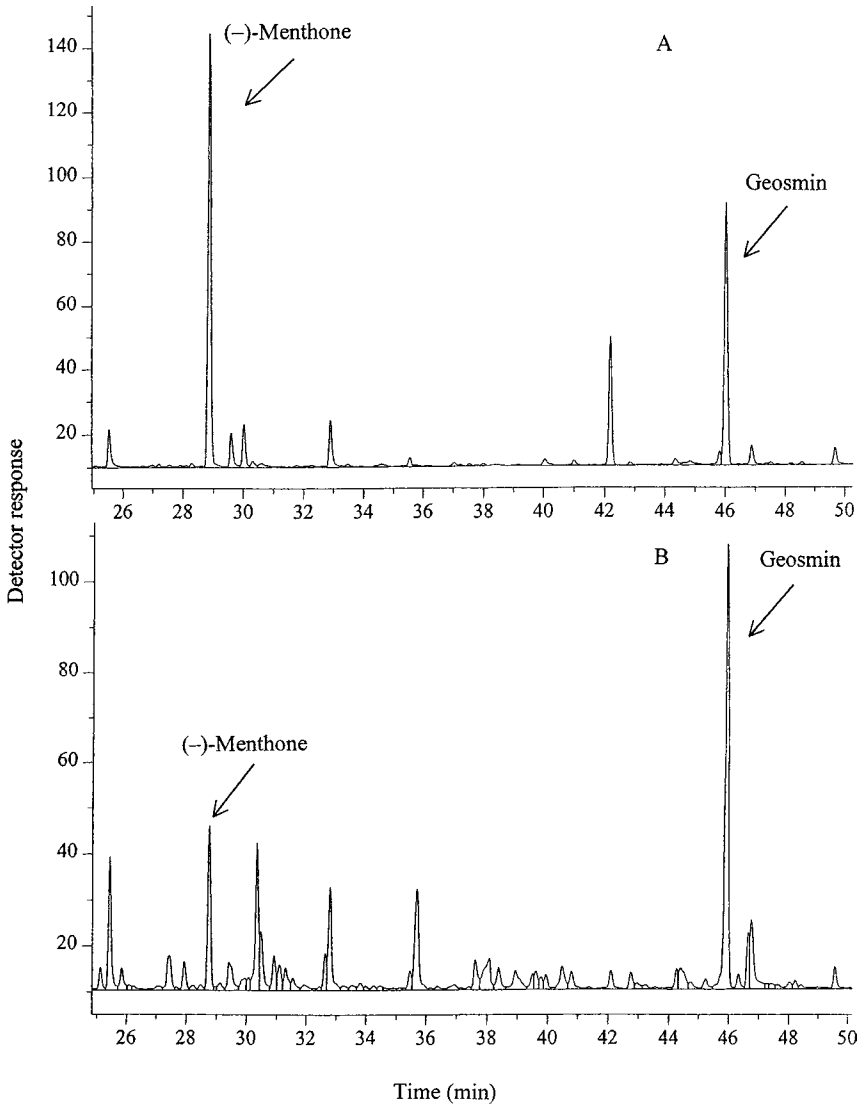


**Figure 3.** Chromatograms of standard geosmin and (–)-menthone solutions (A) and an extract of cv. Chioggia red beet roots using HSPME (B).

**Table 3.** Concentration of Geosmin in Several Red Beet Cultivars Determined Using the HSPME Method

| cultivar[a] | geosmin[b] ($\mu$g/kg) | RSD[c] (%) |
|---|---|---|
| Detroit Dark Red | $9.69 \pm 0.22^{d}$ | 2.27 |
| Crosby Green Top | $15.0 \pm 0.07^{e}$ | 0.467 |
| Lutz Green Leaf | $19.6 \pm 0.94^{f}$ | 4.80 |
| Chioggia | $26.7 \pm 0.27^{g}$ | 1.01 |

[a] Obtained in different seasons from those in **Table 1**. [b] Based on wet weight of beets. Means with different letters are significantly different ($p \leq 0.05$). [c] Relative standard deviation (RSD) = standard deviation/mean.

signal for geosmin than the corresponding unspiked samples. Thus, 1.03 $\mu$g/kg was considered to be the detection limit, which was higher than the detection limit of 0.31 $\mu$g/kg reported by Tyler et al. (*12*). The higher detection limit of the HSPME method is probably due to the small sample size (5 g of beets) used for extraction as opposed to 1 kg used by Tyler et al. (*12*).

**Analysis of Red Beet Cultivars.** The geosmin concentrations of four red beet cultivars determined using the HSPME method were significantly different (**Table 3**). Here, Detroit Dark Red was lowest ($9.69 \pm 0.22$ $\mu$g/kg) and Chioggia the highest ($26.7 \pm 0.27$ $\mu$g/kg). These values were much higher than those determined by the purge-and-trap method, probably because HSPME obtained higher recovery efficiency for geosmin and beets grown in different conditions were used for the two methods. These values were also much higher than those previously reported by Tyler et al. (*19*), ranging from 0.6 to 3.7 $\mu$g/kg, possibly due to the analysis of different cultivars and/or different growth conditions. Lower values reported by Tyler et al. (*19*) were probably also because the authors did not add the internal standard in the initial step for the extraction of geosmin (*12*). Consequently, a lower ratio of geosmin to the internal standard was obtained and a lower geosmin concentration was calculated on the basis of the calibration curve for relative recovery of geosmin.

## ABBREVIATIONS USED

GC−FID, gas chromatography−flame ionization detection; GC−MS, gas chromatography−mass spectrometry; HSPME, headspace solid-phase microextraction; PDMS/CAR/DVB, polydimethylsiloxane/carboxen/divinylbenzene; RSD, relative standard deviation.

## LITERATURE CITED

(1) Mattern, V. You can't beat beets! *Org. Gardening* **1995**, March, 40−44.

(2) Swiader, J. M.; Ware, G. W. The vegetable industry. In *Producing Vegetable Crops*, 5th ed.; Interstate Publishers: Danville, IL, 2002; pp 1−31.

(3) USDA. http://usda.mannlib.cornell.edu/reports/nassr/fruit/pvgbban/vgano1o1.txt (accessed Oct 2001).
(4) Keough, B. Better beets. *Org. Gardening* **1988**, Nov, 28−33.
(5) Acree, T. E.; Lee, C. Y.; Butts, R. M.; Barnard, J. Geosmin, the earthy component of table beet odor. *J. Agric. Food Chem.* **1976**, *24*, 430−431.
(6) Murray, K. E.; Bannister, P. A.; Buttery, R. G. Geosmin: an important volatile constituent of beetroot (*Beta vulgaris*). *Chem. Ind. (London)* **1975**, *15*, 973−974.
(7) Hwang, C. J.; Krasner, S. W.; McGuire, M. J.; Moylan, M. S.; Dale, M. S. Determination of subnanogram per liter levels of earthy-musty odorants in water by the salted closed-loop stripping method. *Environ. Sci. Technol.* **1984**, *18*, 535−539.
(8) Lloyd, S. W.; Grimm, C. C. Analysis of 2-methylisoborneol and geosmin in catfish by microwave distillation-solid-phase microextraction. *J. Agric. Food Chem*. **1999**, *47*, 164−169.
(9) Stahl, P. D.; Parkin, T. B. Purge-and-trap extraction of geosmin and 2-methylisoborneol from soil. *Soil Sci. Soc. Am. J.* **1994**, *58*, 1163−1167.
(10) Palmentier, J.-P. F. P.; Taguchi, V. Y. The determination of six taste and odour compounds in water using Ambersorb 572 and high-resolution mass spectrometry. *Analyst* **2001**, *126*, 840−845.
(11) Watson, S. B.; Brownlee, B.; Satchwill, T.; Hargesheimer, E. E. Quantitative analysis of trace levels of geosmin and MIB in source and drinking water using headspace SPME. *Water Res.* **2000**, *34*, 2818−2828.
(12) Tyler, L. D.; Acree, T. E.; Nelson, R. R.; Butts, R. M. Determination of geosmin in beet juice by gas chromatography. *J. Agric. Food Chem.* **1978**, *26*, 774−775.
(13) Buttery, R. G.; Teranishi, R.; Ling, L. C.; Flath, R. A.; Stern, D. J. Quantitative studies on origins of fresh tomato aroma volatiles. *J. Agric. Food Chem*. **1988**, *36*, 1247−1250.
(14) Buttery, R. G.; Stern, D. J.; Ling, L. C. Studies on flavor volatiles of some sweet corn products. *J. Agric. Food Chem*. **1994**, *42*, 791−795.
(15) Buttery, R. G.; Ling, L. C. Methods for isolating food and plant volatiles. In *Biotechnology for Improved Foods and Flavors*; Takeoka, G. R., Teranishi, R., Williams, P. J., Kobayashi, A., Eds.; ACS Symposium Series 637; American Chemical Society: Washington, DC, 1996; pp 240−248.
(16) McCallum, R.; Pendleton, P.; Schumann, R.; Trinh, M.-U. Determination of geosmin and 2-methylisoborneol in water using solid-phase microextraction and gas chromatography chemical ionisation/electron impact ionisation−ion-trap mass spectrometry. *Analyst* **1998**, *123*, 2155−2160.
(17) Supelco. *Application: Solid-Phase Microextraction of Odors in Drinking Water, for Analysis by GC/MS*; Note 147; Bellefonte, PA, 1998; p 2.
(18) Pawliszyn, J. *Solid-Phase Microextraction: Theory and Practice*; Wiley-VCH: New York, 1997.
(19) Tyler, L. D.; Acree, T. E.; Becker, R. F.; Nelson, R. R.; Butts, R. M. Effect of maturity, cultivar, field history, and the operations of peeling and coring on the geosmin content of *Beta vulgaris*. *J. Agric. Food Chem*. **1978**, *26*, 1466−1469.
(20) Supelco. *Solid-Phase Microextraction: Theories and Optimization of Conditions*; Bulletin 923; Bellefonte, PA, 1998; p 5.
(21) Bao, M.; Griffini, O.; Burrini, D.; Santianni, D.; Barbieri, K.; Mascini, M. Headspace solid-phase microextraction for the determination of trace levels of taste and odor compounds in water samples. *Analyst* **1999**, *124*, 459−466.
(22) Jia, M.; Zhang, Q. H.; Min, D. Optimization of solid-phase microextraction analysis for headspace flavor compounds of orange juice. *J. Agric. Food Chem*. **1998**, *46*, 2744−2747.
(23) Lloyd, S. W.; Lea, J. M.; Zimba, P. V.; Grimm, C. C. Rapid analysis of geosmin and 2-methylisoborneol in water using solid phase microextraction procedures. *Water Res.* **1998**, *32*, 2140−2146.
(24) Harris, D. C. Atomic spectrometry. In *Quantitative Chemical Analysis*, 5th ed.; Freeman: New York, 1998.

**Received for review July 22, 2002. Revised manuscript received November 13, 2002. Accepted November 15, 2002. We sincerely thank Alf Christianson Seed Co. (Mt. Vernon, WA) and the Robert MacDonald Vegetable Seed Memorial Fund for financial support of this research.**

JF020806D