Carotenoids in Finnish Foods: Vegetables, Fruits, and Berries

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In this study 69 items of vegetables, fruits, berries, mushrooms, and their respective products were analyzed for carotenoids by a high-performance liquid chromatographic (HPLC) method. Lutein and β-carotene were the predominant carotenoids in all samples except rutabaga, celeriac, mushrooms, and raisins. The amount of lutein (possibly containing some zeaxanthin) was at its highest in the green vegetables parsley, celery, dill, and spinach (≥4400 μg/100 g). Carrot, parsley, dill, spinach, broccoli, leek, celery, sweet red pepper, tomato ketchup, and chantarelle were very rich in β-carotene (1000–7600 μg/100 g), and thus these items also contribute significantly to the vitamin A activity expressed as retinol equivalents (RE). α-Carotene (range 12–530 μg/100 g) was present in carrot, bean, sweet yellow pepper, orange, mandarin, banana, avocado, cloudberry, raspberry, frozen corn, and prune, with traces found in many other samples. γ-Carotene was found only in tomato, where lycopene was the predominant carotene as it was in tomato ketchup. Small amounts of cryptoxanthin were present in some vegetables, fruits, berries, and their products. In sweet red pepper capsanthin was also quantified. The variation in β- and α-carotene, lutein, and lycopene was studied by analyzing carrot, lettuce, and tomato bought from retail food stores at different seasons of the year.

The present study is part of an effort to determine the retinoid and carotenoid contents of the main food items in all categories of food common in typical Finnish diets. The purpose of the study was to produce analytically homogeneous and up-to-date data to be included in the national food composition data bank and composition tables. The results will also be used in the reestimation of the vitamin A intake of the Finnish population. The previous estimations are based on composition data produced with miscellaneous methods in a number of laboratories or even on data of unknown origin, thus causing a considerable uncertainty factor in the results.

Vegetables and fruits is a category of foods where a great variety of carotenoids is found (Goodwin, 1976; Kläui and Bauernfeind, 1981; Weedon, 1971). In this study the identification of unknowns had to be compromised very strongly for capacity reasons, and only the most abundant carotenoids could be included in the analytical scheme. The main provitamin A active species were determined, as were the most prominent oxidized carotenoids.

Samples were taken of 69 items of vegetables, fruits, berries, and their products. They represent the most common items distributed by the retail food stores in Finland. Because of the extensive coverage of commodities, the number of analyses had to be minimized by pooling the subsamples of each items. This technique gives average compositions, but the information of variation is lost. However, the average values are useful, since the present food distribution systems also efficiently average out food bought by the consumer.

EXPERIMENTAL PART

Sampling. Vegetables, fruits, berries, and mushrooms were sampled during the summer and fall of both 1985 and 1986. Samples of imported apples, pears, and citrus fruits were taken in December 1985. Most of the fresh vegetables and fruits and all of the berries and mushrooms were bought from marketplaces or market halls. The sampling of the other fresh vegetables and fruits, and of all of the vegetable and fruit products, was designed to produce composite samples of food items sold by the four major wholesale food chains (about 90% of total sales) in the Helsinki area. Eight to ten subsamples of 0.25–1 kg were obtained of each food item. The food products for the same item were purchased on the same day.

For each item, one pooled sample was made up from the subsamples. Equal weight of food from each store was composited. Only the edible part of the fresh vegetables, fruits, berries, and mushrooms was analyzed. Green leafy vegetables (except celery and leek) and potatoes, carrot, rutabaga, red cabbage, and tomato, as well as cucumber, were analyzed within 1–3 days of purchase. Peach, banana, avocado, kiwi fruit, watermelon, strawberry, orange juice, and pickled outdoor cucumber were analyzed without a storage phase. The other pooled samples of vegetables, fruits, berries, and mushrooms were vacuum-packed into portions, weighing approximately 100 g each, in polyethylene–nylon laminate bags and stored in the dark at −20 °C generally for no more than 2 weeks until analysis. Any liquid or syrup of the canned vegetables and fruits was discarded before pretreatment.

The seasonal effect on the carotenoid content was studied in carrot, leaf lettuce, and tomato. Ten subsamples of these vegetables were obtained in October, December, March, June, and August from marketplaces and market halls (June and August) or from 10 different stores of the four wholesale food chains. The samples were not frozen before analysis.

Extraction. Fresh or frozen samples were homogenized in a blender. Aliquots of sample homogenates (5 or 10 g) were extracted with acetone in an Omni-Mixer with use of Na_2SO_4 and $MgCO_3$ as desiccants, vacuum-filtered, and concentrated prior to saponification (Bushway and Wilson, 1982). The samples were extracted at least three times to remove all the carotenoids. BHT (0.1% in acetone) was used as antioxidant. The concentrate was saponified overnight at room temperature with 1 or 5 mL (green vegetables) of potassium hydroxide (100 g of KOH + 100 mL H_2O) in a mixture of ethanol (50 mL) and water (20 mL) by a modified method described by Piironen et al. (1984). Ascorbic acid was used as antioxidant (1 g/20 mL of H_2O). Before solvent extraction, the saponification extract was diluted with a solution of sodium chloride (10% in H_2O). Carotenoids were extracted with *n*-hexane and diethyl ether (70:30) with BHT (0.1% in hexane) as antioxidant. The extraction procedure has been decribed in greater detail elsewhere (Heinonen et al., 1988).

High-Performance Liquid Chromatography. All samples were analyzed with the nonaqueous reversed-phase (NARP) system. The two Varian Vista 5500 liquid chromatographs (Varian) were equipped with Varian UV-200 detectors and Varian 4270 integrators. In the NARP chromatography of carotenoids (Nelis and De Leenheer, 1983) the Zorbax ODS (5–6 μm, 25 × 0.46 cm (i.d.)) column (DuPont) was preceded by a guard column (5 × 0.46 cm (i.d.)) packed with Bondapak AX/Corasil (37–50 μm) (Waters). The elution mixture was acetonitrile, dichloromethane, and methanol (70:20:10); the flow rate was 2 mL/min.

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Table I. Carotenoids and Vitamin A Activity in Vegetables and Mushrooms

item	carotenoid, μg/100 g (fresh product)						RE[a]
	α-carotene	β-carotene	γ-carotene	cryptoxanthin	lutein[b]	lycopene	
potato							
new (Aug)	–[f]	3.2	–	–	13	–	0.5
old (March)	tr	7.7	–	nd	60	–	1.3
carrot	530	7600	nd	–	300	–	1300
rutabaga	–	tr	nd	nd	–	–	0
turnip	tr	72	–	–	tr	–	12
white cabbage	tr	66	nd	–	150	–	11
red cabbage	tr	15	–	nd	26	–	2.5
Chinese cabbage	tr	15	–	tr	40	–	2.5
cauliflower	–	11	–	–	33	–	1.9
broccoli	tr	1000	nd	24	1800	–	170
Brussels sprouts	–	430	–	nd	920	–	71
spinach	–	3300	–	–	4400	–	550
lettuce							
leaf	tr	980	–	–	1800	–	160
American	–	73	–	nd	250	–	12
celery	–	2900	nd	nd	7200	–	490
celeriac	–	–	nd	nd	tr	–	0
onion, yellow	–	6.9	–	–	16	–	1.2
leek	–	1000	nd	–	1900	–	170
radish	–	9.4	–	–	12	–	1.6
bean	39	180	nd	–	440	–	33
dill	–	4500	–	–	6700	–	750
parsley	–	5600	–	–	10200	–	940
cucumber	tr	130	–	–	470	–	22
tomato	–	660	170	nd	100	3100	120
pepper							
red[c]	nd	2900	nd	nd	nd	nd	480
yellow	92	150	–	–	770	–	33
green	tr	240	nd	–	700	–	40
chantarelle[d]	tr	1300	–	–	–	–	220
false morel[e]	–	–	–	–	–	–	0

[a] Micrograms of RE/100 g = 0.167 (μg β-carotene) + 0.083 (μg α-carotene + μg γ-carotene + μg cryptoxanthin). [b] Lutein + zeaxanthin. [c] Contains capsanthin, 3200 μg/100 g. [d] *Cantharellus cibarius*. [e] *Gyromitra esculenta*. [f] Abbreviations: – = not detected at a detection limit of 0.5 μg/100 g; tr = trace; nd = not determined.

This system gave a good resolution of α-, β-, and γ-carotene, cryptoxanthin, and lycopene. Lutein and zeaxanthin were not separated from each other.

For most extracts, a combination of isocratic and gradient chromatography (Khachik et al., 1986) was also used to separate the oxygenated carotenoids (xanthophylls) from the hydrocarbon carotenoids (carotenes). The column was Spherisorb ODS2 (5 μm, 25 × 0.46 cm (i.d.) (Phase Sep)) and the Uptight (5 × 0.2 cm (i.d.) (Upchurch Scientific)) guard column was packed with Spherisorb ODS2 (5 μm, (Phase Sep)). An isocratic mixture of acetonitrile (75%), methanol (15%), dichloromethane (5%), and hexane (5%) at a time 0 was followed by a gradient beginning at time 6 and completed at time 20 (minutes). The final composition of the gradient mixture was acetonitrile (40%), methanol (15%), dichloromethane (22.5%), and hexane (22.5%). The flow rate was 1 mL/min. The UV detection of both chromatographic systems was set at 450 nm, and the columns were run at 30 °C. Both standards and samples were injected via full loop, approximately 55 μL.

Identification and Quantitation. The carotenoids were identified by comparing their retention times with those of authentic standards. The quantitation was based on an external standard method where the calibration curves (range 50–3600 ng/mL) were determined daily. A single peak of lutein possibly containing zeaxanthin was quantified as lutein. *All-trans*-β-, 15-*cis*-β-, α- and β-carotene, (3*R*)-cryptoxanthin, lutein, zeaxanthin, lycopene, and capsanthin (inpure) were obtained from F. Hoffman La Roche. Lycopene (90–95%), lutein, and α-carotene were also purchased from Sigma Chemical Co. The concentrations of stock standard solutions were determined spectrophotometrically (Davies, 1976). The extinction coefficient ($E^{1\%}_{1cm}$) used for an ethanolic solution of lutein was 2550 (445 nm), for zeaxanthin 2480 (452 nm), and for capsanthin in benzene 2072 (483 nm). The extinction coefficients ($E^{1\%}_{1cm}$) used for *all-trans*-β- (and 15-*cis*-β-), α- and γ-carotene, (3*R*)-cryptoxanthin, and lycopene in hexane were 2592 (453 nm), 2725 (446 nm), 2720 (461 nm), 2470 (452 nm), and 3450 (472 nm), respectively. All standard solutions were stored in the dark at −70 °C under nitrogen.

More information about the carotenoids present in some vegetables, fruits, and berries was obtained by the diode array technique (Hewlett-Packard 1090 liquid chromatograph, UV–vis diode array detector and 85B data system) available at the National Public Health Institute (NPHI). The purity of the carotenoids quantified in leaf lettuce, carrot, tomato, frozen pea, frozen corn, sweet red pepper, canned peach, orange, cloudberry, and strawberry was checked. The recovery of β-carotene was 101% (n = 8, checked with tomato, carrot, and broccoli). The recoveries of lycopene in tomato and of lutein in broccoli were 103% (n = 4) and 103% (n = 2), respectively.

The mean, standard deviation, and coefficient of variation were calculated from the data of triplicate analysis. A q-test was used to reject the error results (Fritz and Schenk, 1979). The mean coefficient of variation was 10% (range 0.8–44%), depending on the amount and complexity of the carotenoids in the food item. When "not determined", the compound was not quantified. The individual carotenoid values were converted into retinol equivalents, RE (micrograms/100 g), according to the guideline of the Food and Nutrition Board (1980).

RESULTS AND DISCUSSION

The carotenoid composition of fresh vegetables, fruits, berries, and mushrooms and their products is shown in Table I–III. The results presented here are generally in accordance with the vitamin A and carotenoid data presented in current food composition tables. However, there are discrepancies in the carotenoid concentration of some items, especially of fruits and their products. As the current food composition tables (Levnedsmitteltabeller, 1985; Paul and Southgate, 1985; Livsmedelstabeller, 1986; Souci et al., 1986) list only total carotenoids (carotenes) or vitamin A active carotenes, the information of the distribution of the individual carotenoids is not always available.

Table II. Carotenoids and Vitamin A Activity in Fruits and Berries

item	carotenoids, μg/100 g (fresh product)						RE[a]
	α-carotene	β-carotene	γ-carotene	cryptoxanthin	lutein[b]	lycopene	
apple							
domestic	–[c]	12	–	–	48	–	1.9
imported	–	39	–	nd	42	–	6.5
pear	–	17	–	nd	110	–	2.8
orange	19	38	nd	nd	27	nd	7.9
mandarin	20	38	nd	nd	20	nd	7.9
grapefruit	tr	2.3	nd	3.3	9.5	–	0.7
lemon	–	3.4	–	nd	12	–	0.6
peach	tr	86	nd	51	14	nd	19
banana	12	14			3.3		3.4
avocado	19	34	nd	38	320	nd	10
kiwi fruit	–	43	–	3.7	180	–	7.4
grapes	tr	33	nd	nd	72	–	5.4
watermelon	tr	230	nd	nd	14	4500	38
plum	nd	430	nd	nd	240	–	71
rhubarb	–	61	–	–	170	–	10
black currant	tr	99	nd	–	440	–	16
red currant	–	25	–	–	47	–	4.1
gooseberry							
green	tr	110	–	–	170	–	19
red	tr	62	nd	–	180	–	10
bilberry	tr	47	nd	nd	260	–	7.9
strawberry	–	8.9	–	–	31	–	1.5
lingonberry	tr	9.1	–	–	22	–	1.5
cranberry	tr	22	–	nd	28	–	3.6
cloudberry	59	140	nd	nd	37	–	29
raspberry	13	6.4	nd	nd	76	–	2.2

[a] Micrograms of RE/100 g = 0.167 (μg β-carotene) + 0.083 (μg α-carotene + μg γ-carotene + μg cryptoxanthin). [b] Lutein + zeaxanthin. [c] Abbreviations: – = not detected at a detection limit of 0.5 μg/100 g; tr = trace; nd = not determined.

Table III. Carotenoids and Vitamin A Activity in Vegetable and Fruit Products

item	carotenoid, μg/100 g (fresh product)						RE[a]
	α-carotene	β-carotene	γ-carotene	cryptoxanthin	lutein[b]	lycopene	
pea, frozen	tr	360	–	9.2	1700	–	61
corn, frozen	50	51	nd	nd	730	–	13
red beet, pickled	–[c]	tr	–	–	4.4	–	0
pumpkin, pickled	tr	490	nd	nd	630	–	81
outdoor cucumber, pickled	–	180	–	nd	510	–	29
champignon, canned	–	–	–	–	–	–	0
olive, green	–	280	–	19	510	–	47
tomato ketchup	–	5000	nd	nd	210	9900	830
raisin	–	–	–	–	tr	–	0
prune	31	140	nd	nd	120	–	27
peach, canned	tr	100	nd	82	28	nd	23
apricot, canned	–	560	nd	tr	3.5	nd	94
pineapple, canned	tr	18	nd	1.8	1.6	nd	3.2
orange juice	–	2.8	–	10	27	–	1.3
orange marmelade	tr	16	nd	6.2	5.6	–	3.2

[a] Micrograms of RE/100 g = 0.167 (μg β-carotene) + 0.083 (μg α-carotene + μg γ-carotene + μg cryptoxanthin). [b] Lutein + zeaxanthin. [c] Abbreviations: – = not detected at a detection limit of 0.5 μg/100 g; tr = trace; nd = not determined.

Khachik et al. (1986) reported a loss of over 80% for some xanthophylls, 37% for *all-trans*-lutein, and 6% for total β-carotene when methanolic potassium hydroxide (30%) was used in saponification of the carotenoid extracts from broccoli. The lutein concentration of broccoli reported in the present paper does not show marked losses, which may be explained by the mild conditions of saponification. However, no information was obtained about the possible losses of xanthophylls other than lutein.

Lutein (possibly containing some zeaxanthin) and β-carotene were the predominant carotenoids in vegetables. Both were found in all samples except rutabaga, celery, mushrooms, and raisins. The highest lutein values (≥4400 μg/100 g) were obtained in green vegetables, such as parsley, celery, dill, and spinach. The amount of lutein was moderately high (700–1800 μg/100 g) in broccoli, Brussels sprouts, leaf lettuce, leek, and both yellow and green pepper. Normally, the amounts of lutein are considerably greater than those of zeaxanthin, although there are many exceptions. Thus, zeaxanthin is a major pigment in yellow corn (Weedon, 1971). In the present study, the ratio of lutein to zeaxanthin in frozen corn was approximately 1:1, although both xanthophylls were quantified as lutein (730 μg/100 g).

In their study, Khachik et al. (1986) reported xanthophylls and carotenes in broccoli, cabbage, spinach, Brussels sprouts, and kale. The xanthophylls identified were neoxanthin, violaxanthin, lutein epoxide, and lutein with its several mono-cis isomers. The only carotenes found were *all-trans*-β-carotene and its 15-cis isomer, which was also the case in the present study. Cis isomers occur in a number of plants, but they are also formed in isolation procedures, under storage conditions, and during processing (Gortner and Singleton, 1961; Sweeney and Marsh, 1971; Weedon, 1971). On the other hand 13-*cis*- and 9-*cis*-β-carotenes have been reported in both raw and pro-

Table IV. Carotenoid Content of Carrot, Lettuce, and Tomato Purchased from Retail Food Stores Five Times during 1 Year

item	carotenoid, μg/100 g (fresh product)			
	α-carotene	β-carotene	lutein[a]	lycopene
		June to August		
carrot	2400–2900	2600–5500	260	–
lettuce	tr	730–790	1200–1400	–
tomato	–[b]	390–580	50–110	3800–6600
		October to March		
carrot	530–2200	4600–7700	220–300	–
lettuce	tr	860–1100	1800–2200	–
tomato	–	610–850	100	2600–3100

[a] Lutein + zeaxanthin. [b] Abbreviations: – = not detected at a detection limit of 0.5 μg/100 g; tr = trace.

cessed fruit and vegetable samples (Bushway, 1985; Chandler and Schwartz, 1987; Quackenbush, 1987). 15-*cis*-β-Carotene was only a minor component. In the present study, the 15-*cis*-β-carotene was tentatively identified according to the characteristics of its absorption spectrum, especially that of the subsidiary peak in the near-ultraviolet region.

Carrot, parsley, dill, spinach, broccoli, leek, sweet red pepper, tomato ketchup, and chantarelle were very rich in β-carotene (1000–7600 μg/100 g), and thus these vegetables also contributed significantly to the vitamin A activity expressed as retinol equivalents (RE). The amount of the tentative 15-*cis*-β-carotene in broccoli, Brussels sprouts, spinach, lettuce (American), red cabbage, dill, parsley, and cucumber varied from 5% to 10% of the total amount of β-carotene.

In higher plants, β-carotene is often associated with smaller amounts of α-carotene and traces of γ-carotene (Weedon, 1971). α-Carotene (range 12–530 μg/100 g) was present in carrot, bean, yellow pepper, orange, mandarin, banana, avocado, cloudberry, raspberry, frozen corn, and prune, with traces found in many other samples. γ-Carotene could be determined in only tomato and tomato ketchup. According to Goodwin (1976) lycopene occurs in red tomatoes and in a number of other fruits and in many berries. In the present study, lycopene was present in watermelon (4500 μg/100 g).

β-Cryptoxanthin is one of the major pigments in ripe peaches (Katayama et al., 1971) and oranges (Stewart, 1977). The amount of both β-cryptoxanthin, and β-carotene was higher in canned peaches, orange juice and orange marmelade than in the respective fresh fruit. Thus, many processed fruit products may be good sources of vitamin A. Also broccoli, avocado, frozen peas, green olives, grapefruit, kiwi fruit, and canned pineapple contained small amounts of β-cryptoxanthin.

A complex carotenoid pattern has been found in red pepper, including capsanthin, capsorubin, cryptocapsin, β-carotene, and cryptoxanthin (Cholnoky et al., 1955). In the present study the amount of capsanthin found in red pepper was 3200 μg/100 g; this carotenoid could not be quantified in yellow and green peppers.

Concentrations of carotenoids in carrot, leaf lettuce, and tomato purchased in different seasons of the year are shown in Table IV. The β-carotene and lutein levels were to their lowest in summer (June and August). The lycopene concentration in tomato was high in summer and low in winter (October to March). The carrot samples analyzed were all of domestic origin, whereas leaf lettuce and tomato samples were imported in winter.

The average per capita consumption of potatoes, other fresh vegetables, fresh fruits, fruit juices, and berries was 187, 101, 126, 28, and 30 g, respectively (Agricultural Economics Research Institute, 1986). The daily consumption of mushrooms is 3 g per capita. Tomatoes, carrots, cucumbers, and cabbages account for more than 70% of the total consumption of fresh vegetables other than potatoes (Central Statistical Office of Finland, 1986). The consumption of fresh vegetables in summer is about twice that in winter (Ylätalo, 1985).

As a conclusion, all green tissue of higher plants generally contain the same major carotenoids, which include β-carotene and lutein (with some zeaxanthin). The green leafy vegetables, carrot, and red pepper are also good sources of vitamin A. Pigments that frequently appear in smaller amounts in, for example, many fruits and berries are α-carotene and cryptoxanthin. Larger amounts of individual carotenoids are encountered in some specific samples, such as lycopene in tomato and capsanthin in red pepper. Finally, the carotenoid level in vegetables, fruits, berries, and mushrooms is determined by the maturity of the tissue and variety.

Registry No. α-Carotene, 7488-99-5; β-carotene, 7235-40-7; γ-carotene, 472-93-5; cryptoxanthin, 472-70-8; lutein, 127-40-2; lycopene, 502-65-8; zeaxanthin, 144-68-3; capsanthin, 465-42-9; vitamin A, 11103-57-4.

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Volatile Components of Chickpea (*Cicer arietinum* L.) Seed

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Headspace material was collected from floured *Cicer arietinum* seed and analyzed by capillary GC–mass spectrometry. Structural assignment was achieved for 132 components of the chickpea volatiles by mass spectra, by cochromatography, and through their Kovats indices. All the substances, with two exceptions, present in chickpea headspace in an amount above 0.5% of the total volatiles, could be identified. Besides aliphatic hydrocarbons, the dominant chemical classes in the chickpea volatiles were terpenoids (35%) and alcohols (18%). With the sampling method applied for this analysis, a total of 2.4 μg of volatiles was collected from 1.5 L of headspace volume. The main individual component was α-pinene (≈0.3 μg).

Chickpea (*Cicer arietinum* L., Fabaceae) is a legume of economic importance, which is mainly grown in the hot climates of India, Pakistan, Iran, Ethiopia, Mexico, and the Mediterranean area. Its most important insect pest is *Heliothis armigera* Hübner (Lepidoptera: Noctuidae), a polyphagous night-active moth identified in India on 181 host plant species (Manjunath et al., 1985). On chickpea, the larvae fed on leaves, flowers, buds, pods, and seeds of different maturation stages. Preliminary studies have demonstrated an attraction of *H. armigera* larvae by chickpea seed volatiles (Saxena and Rembold, 1984). The present study was undertaken to characterize such headspace material, which is volatile at 43 °C. Its volatiles profile could also be of interest for food chemists if used as chemical fingerprint for identification of chickpea seed samples. Such an analytical characterization is still missing, according to corresponding literature (van Straten and Maarse, 1983).

EXPERIMENTAL SECTION

Materials. For this stock-taking study, one batch of commercial standard chickpea seed of kabuli type (Scandimport, Maisach, FRG) was used. The material was dried at 40 °C overnight and, if required, ground in 25-g portions in an IKA-M-20 Universal mill (Janke & Kunkel, Staufen, FRG) under intensive water cooling. Headspace was collected immediately afterward.

Isolation of Volatiles Using Tenax Traps. The seed flour was placed in a 110-mL graduated flask fitted with three ground glass stoppers and maintained at 43 °C in a water bath. Two traps filled with Tenax TA (150 mg, 80–100 mesh, package of 56-mm length fixed with silanized glass wool in the middle of a glass tube with 20-cm length and 4-mm i.d.) were directly connected through ground-glass connections with the flask. The third inlet was for sample introduction and was closed with a ground-in stopper. After 10 min, purified nitrogen was flown (100 mL/min) via a Teflon tube connection through one of the Tenax tubes into the flask. Headspace was collected in the second Tenax tube for 15 min. The commercially available authentic chemical samples used for identification purposes were trapped in a similar way as the headspace material.

Capillary Gas–Liquid Chromatography–Mass Spectral (GC–MS) Analysis. The method of thermal desorption of the Tenax trap and transfer of the volatiles via an intermediate trap onto the capillary column has been described already (Nitz et al., 1984; Wächter et al., 1986). For the present study, a desorption temperature of 150 °C for the Tenax traps, helium flux of 20 mL/min, and time period of 10 min were applied. The chickpea volatiles are completely desorbed under these conditions. A Finnigan 1020 quadrupole automated GC–MS system, directly coupled to a Sigma III gas chromatograph (Perkin-Elmer) equipped with a modified PTV injector from Dani as described elsewhere (Nitz et al., 1984), was used. Separation was performed with a J&W fused silica capillary column (30 m × 0.25 mm (i.d.)) coated with SE54 (film thickness 0.25 μm). Carrier gas was helium (29 cm/s), and the oven, after having been kept at 0 °C for 12 min, was programmed to 250 °C at a rate of 2 °C/min. The mass spectra were measured by electron impact at 70 eV.

RESULTS AND DISCUSSION

With the technique described, a solvent-free sample collection is achieved. Control experiments using the empty manifold under our standard sampling conditions showed that only insignificant impurities were present. Practically no serious breakthrough effect of the volatiles was discernible in the second trap if two Tenax traps were used in line for sample collection. Only some part of the

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