

# Gas chromatography/sniffing port analysis and sensory evaluation of commercially dried bell peppers (*Capsicum annuum*) after rehydration

Saskia M. van Ruth & Jacques P. Roozen

Wageningen Agricultural University, Department of Food Science, PO Box 8129, NL-6700 EV Wageningen, The Netherlands

(Received 13 December 1993; revised version received and accepted 14 February 1994)

Cultivated, cut and commercially dried red bell peppers (origins Chile, Hungary, and Turkey) were rehydrated and then evaluated by descriptive and hedonic panels. The types of pepper did not differ significantly in sensory values for the attributes cucumber, cooked vegetables, burned, mushrooms, bell pepper, fruity, grassy/green vegetables, fresh, spicy and sweet. The bell peppers of Chilean origin were rated higher in sour, bitter, sharp and pungent attributes than the Turkish, and higher in bitter and pungent attributes than the Hungarian. The Hungarian rated higher in 'sour' than the Turkish. Volatile compounds were analysed by gas chromatography, using flame ionisation detection, mass spectrometry and sniffing port detection. Forty-six compounds were identified, 12 of which possessed odours: 2-methylpropanal (chocolate); 2- and 3-methylbutanal (chocolate); 2,3butadione (caramel/butter); 1-penten-3-one (plastic/chemical); hexanal (grassy/ green); heptanal (lemon/orange); β-ocimene (fish/rotten/sickly); trans-3-hepten-2one (mushrooms); dimethyltrisulphide (rotten/onion/leek); 2-methoxy-3-isobutylpyrazine (bell pepper); and  $\beta$ -cyclocitral (fruity). The compositions of the volatiles were similar for the three origins, although the Hungarian one generally exhibited the largest peak areas. The bell peppers from Chile were appreciated more by the panel than the ones from Turkey. This was probably due to the higher intensity of taste attributes (sour, bitter, sharp and pungent) because composition of the volatile compounds and intensity of odour attributes were similar for both origins.

## INTRODUCTION

Fruits of the genus *Capsicum*, both fresh and dried, are widely accepted for use as spices and as staples in the diet. Bell peppers differ from most other members of the genus in that they generally do not have the 'hot' taste associated with the Chili and Tabasco types. Bell peppers possess a characteristic pleasant aroma, although various varieties have distinctive aromas (Heath, 1981).

Some aroma compounds are present in the intact bell peppers; many others are produced only when the tissue of the raw vegetable is broken, such as by cutting, chewing or blending (Buttery, 1981). In many plants, disruption of tissues gives rise to rapid hydrolytic and oxidative degradation of endogeneous lipids into various products, among which are volatile compounds found to be responsible for desirable and undesirable flavours (Tressl *et al.*, 1981; Hatanaka *et al.*, 1983). Wu and Liou (1986) showed that tissue disruption stimulated enzymic formation of hexanal, *trans*-2-hexenal, hexanol, *cis*-3-hexen-1-ol and *trans*-2-hexen-1-ol, while the contents of 2-methoxy-3-isobutylpyrazine, linalool, *trans*- $\beta$ -ocimene and benzaldehyde were similar before

and after tissue disruption. Cooking or processing of the vegetable gives rise to other volatile flavour compounds, usually breakdown products of its major components, e.g. carbohydrates, proteins and lipids (Buttery, 1981). The aroma of fresh bell peppers, mainly due to 2-methoxy-3-isobutylpyrazine, is modified to a cooked bell pepper aroma by the increased amounts of  $C_{9}$ ketones in particular (Govindarajan, 1986). As a result the flavour of rehydrated bell pepper cuttings is due to compounds present in the intact vegetable and to compounds formed during processing and rehydration.

Some investigations have examined hedonic (likedislike) responses to peppers (Weisenfelder *et al.*, 1978; Saldana & Meyer, 1981). Studies of Rozin and Schiller (1980) and Rozin *et al.* (1981) noted that people typically like the widely varying flavours of the different varieties of the fruit, in addition to acquiring a liking for the intensity of the pungency of *Capsicum*. According to Chitwood *et al.* (1983), the volatile fraction of fresh *C. annuum* yielded 11 components, nine of which were described by a gas chromatograph effluent sniffing panel. Descriptors used were: green; grassy; bitter almond; floral; green bell pepper; wintergreen-like; apple-like; fruity; violet-like and woody.

The present study deals with the relationship between the volatile constituents and flavour characteristics of bell peppers cultivated and dried in Chile, Hungary and Turkey.

### MATERIALS AND METHODS

Commercially dried red bell pepper cuttings from Chile, Hungary and Turkey were supplied by Top Foods b.v. (Elburg, The Netherlands). The bell peppers were stored for a maximum of 2 months in glass jars at 4°C and in the absence of light.

#### **Instrumental analysis**

Bell pepper cuttings (3 g) were rehydrated by adding 35 ml water and 0.1 ml 0.3% anti-foam-agent Polysiloxan (A. Smit & zn b.v., Weesp, The Netherlands), then heating in a waterbath at 100°C for 10 min, and cooling in a waterbath at 25°C for 4 min. The cuttings were transferred into the sample flask of a purge-andtrap mouth model system and 25 ml artificial saliva was added (van Ruth et al., 1993). This saliva consisted of 5.208 g NaHCO<sub>3</sub>, 2.160 g mucin, 1.369 g K<sub>2</sub>HPO<sub>4</sub>, 0.877 g NaCl, 0.500 g NaN<sub>3</sub>, 0.447 g KCl, 0.441 g CaCl<sub>2</sub>. 2aq and 200 000 units  $\alpha$ -amylase (Sigma Chemical Co., St Louis, MO) in 1 litre of distilled water (adjusted to pH 7). A flow of purified nitrogen (20 ml/min) passed through the bell pepper cuttings/saliva mixture at 37°C for 2 h to trap the volatiles in 0.10 g Tenax TA, 35/60 mesh (Alltech Nederland b.v., Zwijndrecht, The Netherlands), positioned in a glass tube, 3 mm i.d. and 10 cm long.

Thermal desorption of the volatiles from Tenax was performed by a thermal desorption (200°C, 10 min)/ cold trap (-100°C) device (Carlo Erba TDAS 5000; Interscience b.v., Breda, The Netherlands). Gas chromatography (GC) was carried out on a Carlo Erba MEGA 5300 (Interscience b.v., Breda, The Netherlands) equipped with a Supelcowax 10 capillary column, 0.25 mm i.d. and 60 m long and a flame ionisation detector (FID) at 275°C. An initial oven temperature of 40°C for 4 min was used followed by a rate of  $2^{\circ}$ C/min to  $92^{\circ}$ C and then of 6°C/min to  $272^{\circ}$ C.

GC-sniffing analyses were performed with a Carlo Erba gas chromatograph, type 6000 VEGA series (Interscience b.v., Breda, The Netherlands). A thermal desorption/ cold trap device (Chrompack TCT injector 16200; Chrompack, Middelburg, The Netherlands) was used as well as the column and temperature conditions described before. At the end of the capillary column the effluent was split 30:35:35 for FID, sniffing port 1 and sniffing port 2, respectively (Linssen *et al.*, 1993). Twelve assessors were selected on their availability, sensitivity, memory and ability to recognise odours. Prior to sniffing bell pepper samples, the assessors were trained on the technique of sniffing. Assessors used portable

Descriptors	Attributes		
(GC-sniffing panel)	(analytical sensory panel)		
Bell pepper	Bell pepper		
Burned/rubber	Burned		
Cooked vegetables	Cooked vegetables		
Fresh vegetables	Fresh		
Fruity	Fruity		
Grassy/green	Grassy/green		
Mushrooms	Mushrooms		
Sour	Sour		
Spicy	Spicy		
Sweet	Sweet		
Butter	Bitter		
Caramel	Cucumber		
Chocolate	Pungent		
Coffee	Sharp		
Fish			
Lemon/orange-like			
Onion/leek			
Plastic/chemical			
Rotten			
Sickly			

Table 1. Comparison of the descriptors of the GC-sniffing panel and the attributes of the analytical sensory panel

computers with a program in Pascal for data collection. The data were converted from the field disks into Lotus 123 software in order to process the raw data. Flavour descriptors were generated during preliminary GC-sniffing experiments and clustered after group sessions of the panel had been undertaken, resulting in a list of 20 descriptors (Table 1). These descriptors and 'other/I do not know' had to be used for each component detected by the assessors at the sniffing port. Tenax tubes without adsorbed volatile compounds were used as dummy samples for determining the signal-to-noise level of the group of assessors.

The volatile components trapped on Tenax TA were identified by Dr M. A. Posthumus, Wageningen Agricultural University, Department of Organic Chemistry, using combined GC (Pye 204, Unicam Ltd, Cambridge, UK) and mass spectrometry (MS; VG MM 7070 F, Fisons Instruments, Weesp, The Netherlands). The thermal desorption device, capillary column, and oven temperature program were the same as described for the GC-sniffing analyses. Mass spectra were recorded in the electron impact mode at an ionisation voltage of 70 eV and scanned from m/z = 300 to 25 with a cycle time of 1.8 s.

#### Sensory evaluation

A panel of 24 judges (aged 20–65) was selected and trained for analytical sensory analysis. Part of the training was utilisation of a 70 mm visual analogue scale on a portable computer screen for scoring perceived flavour intensities. A computer interactive interviewing system for composing questionnaires was used to gather survey information (Ci2 system; Sawtooth Software Inc., Ketchum, ID, USA). The SPSS program (MANOVA) was used tor statistical evaluations (Linssen

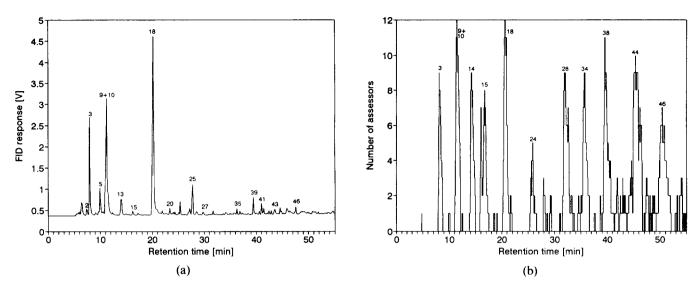


Fig. 1. Gas chromatograms of volatile components of rehydrated Hungarian bell pepper cuttings. (a) FID chromatogram and (b) chromatogram of the components detected at the sniffing port.

et al., 1991). Samples of 3 g dried red bell pepper cuttings were rehydrated by adding 20 ml water and heating in a waterbath at  $100^{\circ}$ C for 10 min. They were subsequently served to the panel as 20 g portions at room temperature and assessed for flavour evaluation. Flavour attributes were generated during training sessions and clustered after group sessions, which resulted in a list of 14 attributes (Table 1).

In a paired comparison test, a hedonic panel of 203 assessors (students and their parents) provided flavour preference responses to bell peppers from Chile and Turkey. Red bell pepper cuttings were rehydrated and served as described before.

#### **RESULTS AND DISCUSSION**

Dynamic headspace gas chromatography of rehydrated bell pepper samples was used to analyse the release of

volatiles at mouth conditions (artificial saliva, 37°C). Figure 1 represents the chromatograms of rehydrated Hungarian bell pepper cuttings, obtained by FID and sniffing port detection. The numbers above the peaks in Fig. 1 refer to the compounds listed in Table 2. GC-sniffing of dummy samples revealed that detection of an odour at the sniffing port by three or less out of 12 assessors can be considered as 'noise'. FID and sniffing chromatograms of Chilean and Turkish bell peppers were similar to the Hungarian bell pepper chromatograms as presented in Fig. 1. The volatile compounds of dynamic headspace samples of rehydrated red bell pepper cuttings from Chile, Hungary and Turkey were identified by GC/MS (Table 2). The compounds were further characterised by their retention times, their peak areas and the odours described by the assessors at the sniffing port. Overall there is no difference in composition of volatiles between the bell peppers. However, the average peak areas (n = 6) of

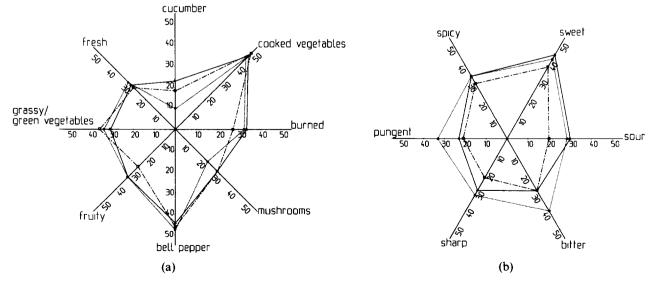


Fig. 2. Spider-web diagrams of rates for sensory attributes of rehydrated red bell pepper cuttings from Chilean (.....), Hungarian (\_\_\_\_) and Turkish origin (\_\_\_\_). (a) odour attributes, (b) taste attributes.

the volatile compounds differ substantially between the different origins. In general, Hungarian bell peppers exhibit the largest peak areas. Smaller, highly volatile compounds were identified in comparison with other studies (Buttery *et al.*, 1969; Keller *et al.*, 1981; Chitwood *et al.*, 1983; Wu *et al.*, 1986), i.e. dimethyl-sulphide, propanal, 2-methylpropanal, methyl acetate, 2-methylfuran, butanal, diethoxyethane, 3-buten-2-one, 2-pentanone and 2,3-butadione. Most of the odour active volatile compounds, as hexanal, heptanal,  $\beta$ -

ocimene, *trans*-3-hepten-2-one and 2-methoxy-3-isobutylpyrazine, were also reported by Wu *et al.* (1986). In the study of Buttery (1981) 2-methoxy-3-isobutylpyrazine, *trans*- $\beta$ -ocimene and limonene contributed to the vacuum steam volatile oil of green bell peppers for 37%. The only similar odour active compound of bell peppers described by a GC-effluent sniffing panel is 2-methoxy-3-isobutylpyrazine, which was reported by Chitwood *et al.* (1983). This characteristic 2-methoxy-3-isobutylpyrazine was identified in

Table 2. Volatile components of rehydrated Capsicum cuttings of three different origins: their retention times, odour descriptions and
average peak areas $(n = 6)$

Peak no.	Retention time (min)	Component	Odour description	Peak area (mV.s)		
				Chile	Hungary	Turkey
1	6.27	Dimethylsulphide		1 840 878	935 803	5612917
2	7.30	Propanal		1 0 2 6 6 1 8	3 348 740	1 1 7 2 6 1 4
3	7.83	2-Methylpropanal	Chocolate	12936741	26973318	12024000
4	8.11	Methyl acetate		88 4 57	77 390	272 909
5	9.50	2-Methylfuran		756175	3 096 111	1 249 419
6	9.62	Butanal		783 889	1 279 480	1 035 635
7	9.99	Diethoxyethane		163 819	1 171 945	947 330
8	10.33	2-Butanone		1 772 454	1 688 028	4 393 976
9	10.79	2-Methylbutanal	Chocolate	21 058 713	54 528 985	29957213
10	11-24	3-Methylbutanal	Chocolate	44 997 203	75734382	61 066 495
11	12.69	3-Buten-2-one		1 612 069	525114	848 469
12	13.95	2-Pentanone		107 232	<10000	674 363
13	14.05	Pentanal		5005615	25 434 392	4985086
14 15	14·26 16·18	2,3-Butadione 1-Penten-3-one	Caramel; butter Plastic/chemical	597 711	2 958 329	332 518
			Plastic/chemical	688 652	3 9 3 9 2 9 0	288 165
16	19.53	2,3-Pentadione		123 015	1 779 999	287 485
17	20.10	Dimethyldisulphide		1 242 240	97 820	3 317 768
18	20.55	Hexanal	Grassy/green	16 992 232	212 369 451	15687703
19 20	20.95 23.42	3-Penten-2-one 1-Methyl-1H-pyrrole		62 132 3 820 132	41 490 7882 184	45 331 5 012 893
21	23.72	1-Butanol		25171	1 389 427	<10000
22 23	25·42 27·06	1-Penten-3-ol		644 311	3 096 888	262 994
23 24	27.86	2-Heptanone	Lamon/orango	419 162 2 306 133	7850928	775712
24 25	28.62	Heptanal Limonene	Lemon/orange	2 306 133 96 643	81 323 827 14 213 807	1 471 963 282 703
26	29.76	3-Methyl-1-butanol		1 278 667	2678512	987 219
27	31.02	trans-2-Hexenal		81 841	880410	52 383
28	31.12	β-Ocimene	Fish; rotten; sickly	958 810	406 779	311 249
29	31.33	4-Methyl-2-hexanone	r ion, rotten, slekty	186 340	1 489 611	151 439
30	31.74	l-Pentanol		270 199	2 330 559	203 288
31	31-93	2,3-Hexadione		179 561	81 517	84 299
32	34.01	5-Methyl-2-hexanone		144 178	659495	113 354
33	34.26	Octanal		583 280	1688011	474113
34	34.97	trans-3-Hepten-2-one	Mushrooms	689 214	1 104 1 59	1 188 447
35	36-28	cis-2-Heptenal		1 258 834	3 4 50 004	524 969
36	36.83	6-Methyl-5-hepten-2-one		1 397 735	3 299 050	1 444 512
37	37.37	1-Hexanol		542 270	666 91 5	360 496
38	38.40	Dimethyltrisulphide	Rotten: onion/leek	478 317	128618	525 891
39	39.43	Nonanal		198 179	2 372 835	198 948
40	40.53	tert-Dodecanethiol		106 942	998 882	73 005
41	41.04	1-Octen-3-ol		1 069 494	5 370 009	521912
42	41.77	Acetic acid		841 632	145 456	199007
43	43.46	Decanal		1 451 095	4 608 510	159129
44	43.94	2-Methoxy-3-isobutylpyrazine	Bell pepper	193 529	375 886	85183
45	44.57	Benzaldehyde	Facility	617 389	6990902	608 350
46	47.81	$\beta$ -Cyclocitral	Fruity	21 081	440 575	47216

Table 3. Number of assessors preferring rehydrated red bell pepper cuttings from Chilean or Turkish origin in a hedonic paired comparison test (n = 203)

Country of origin	No. of assessors		
Chile	127*		
Turkey	76		

\*Binomially significant (p < 0.05).

sweet bell peppers, as well as in Chili peppers at a lower level (Murray & Whitfield, 1975).

An analytical sensory panel of 24 judges evaluated the bell peppers from Chile, Hungary and Turkey. Averages tor the intensities of sensory attributes of three origins of Capsicum are presented in spider-web diagrams in Fig. 2. Although individual judges responded differently to the three origins, the panel was consistent across replications. The origins of bell peppers did not differ significantly for the attributes cucumber, cooked vegetables, burned, mushrooms, bell pepper, fruity, grassy/green vegetables, fresh, spicy and sweet (Student's *t*-test, p < 0.05). The Chilean origin was rated significantly higher than the Turkish in sour, bitter, sharp and pungent, and also higher than the Hungarian in bitter and pungent. The attribute sour rated higher in the Hungarian than in the Turkish origin. Therefore, the bell peppers were more different in 'taste' attributes like sour, bitter, sharp and pungent than in 'odour' attributes. This could be due to differences in concentration of capsaicinoids, which contribute to the pungency of red pepper (Bennett & Kirby, 1968; Huffman et al., 1978; Rowland et al., 1983).

Most of the attributes generated by the sensory panel were similar to the descriptors of the sniffing panel (Table 1). Apparently, volatile compounds contribute considerably to the flavour of rehydrated bell peppers. This is in agreement with studies of Chitwood et al. (1983) in which a similarity between sniffing descriptors and sensory attributes of bell peppers was observed. Although FID responses of the odour active compounds 1-penten-3-one, hexanal and heptanal showed a considerable difference between the Hungarian and the other bell pepper origins (Table 2), this difference was apparently not sufficient to cause significant changes in the values of sensory attributes. This is probably due to the fact that sensory intensity is related to the log physical concentration as in the Fechner equation (Meilgaard et al., 1991). In preliminary sensory experiments the intensity rate of the attribute 'grassy/green flavour' increased 1.3 times per doubled concentration of hexanal. Hexanal concentration detected at the FID was about 13 times  $(2^{3.7})$  higher for the Hungarian peppers compared with the other two origins. Therefore, a difference in intensity of 4.8 could be expected. For a significant difference (Student's *t*-test, p < 0.05) the increase in intensity rate of the attribute 'grassy/ green flavour' should have been about 10 in the bell pepper experiments. Thus, the difference is not likely to cause a significant difference in the intensity rate for the attribute 'green/grassy flavour'.

In the paired comparison test the hedonic panel provided preference responses to bell peppers from Chile and Turkey (Table 3). The bell peppers from Chile were significantly more appreciated (binomial probability < 0.05) than the Turkish bell peppers. The two origins differed in 'taste' attributes like sour, bitter, sharp and pungent only, according to the sensory analysis results. This is in agreement with the GC-FID and GC-sniffing results, which indicated only small differences in composition of the volatile compounds between the two origins.

## CONCLUSIONS

Compositions of the volatile compounds were similar for Chilean and Turkish bell peppers, as well as the rates of 'odour' attributes in sensory analysis. Therefore, 'taste' attributes are expected to be responsible for the difference in appreciation of Chilean and Turkish bell peppers.

## REFERENCES

- Bennett, D. J. & Kirby, G. W. (1968). Constitution and biosynthesis of capsaicin. J. Chem. Soc., C, 442-6.
- Buttery, R. G. (1981). Vegetable and fruit flavours. In Flavour Research—Recent Advances, ed. R. Teranishi, R. A. Flath & H. Sugisawa. Marcel Dekker, New York, pp. 175-216.
- Buttery, R. G., Seifert, R. M., Guadagni, D. G. & Ling, L. C. (1969). Characterization of some volatile constituents of bell peppers. J. Agric. Food Chem., 17, 1322–7.
- Chitwood, R. L., Pangborn, R. M. & Jennings, W. (1983). GC/MS and sensory analysis of volatiles from three cultivars of *Capsicum. Food Chem.*, 11, 201–16.
- Govindarajan, V. S. (1986). Capsicum—Production, technology, chemistry and quality. Part III. Chemistry of the colour, aroma, and pungency stimuli. CRC Crit. Rev. Food Sci. Nutr., 24, 245–355.
- Hatanaka, A., Sekiya, J. & Kajiwara, T. (1983). Linolenic acid and its 13-hydroperoxide inhibit hexanal formation from linolenic acid in plant tissues. J. Agric. Food Chem., 31, 176-8.
- Heath, H. C. (1981). Source Book of Flavours. AVI, Westport, CT.
- Huffman, V. L., Schadle E. R., Villalon, B. & Burns, E. E. (1978). Volatile components and pungency in fresh and processed Jalapeño peppers. J. Food Sci., 43, 1809–11.
- Keller, U., Flath, R. A., Mon, T. R. & Teranishi, R. (1981). Volatiles from red pepper (*Capsicum spp.*). In *Quality* Selected Fruits and Vegetables of North America, ed. R. Teranishi & H. Barrera-Benitez. ACS Symposium Series 31, ACS, Washington, DC, pp. 137–46.
- Linssen, J. P. H., Janssens, J. L. G. M., Reitsma, J. C. E. & Roozen, J. P. (1991). Sensory analysis of polystyrene packaging material taint in cocoa powder for drinks and chocolate flakes. *Food Add. Contamin.*, 8, 1–7.
- Linssen, J. P. H., Janssens, J. L. G. M., Roozen, J. P. & Posthumus, M. A. (1993). Combined gas chromatography and sniffing port analysis of volatile compounds of mineral water packed in polyethylene laminated packages. *Food Chem.*, 46, 367–71.

- Meilgaard, M., Civille, G. V. & Carr, B. T. (1991). Sensory evaluation techniques. CRC Press, Boca Raton, FL.
- Murray, K. E. & Whitfield, F. B. (1975). The occurrence of 3-alkyl-2-methoxypyrazines in raw vegetables. J. Sci. Food Agr., 26, 973-86.
- Rowland, B. J., Villalon, B. & Burns, E. E. (1983). Capsaicin production in sweet bell and pungent Jalapeño peppers. J. Agric. Food Chem., 31, 484–7.
- Rozin, P. & Schiller, D. (1980). The nature and acquisition of a preference for chili pepper by humans. *Motivation & Emotion*, 4, 77–101.
- Rozin, P., Mark, M. & Schiller, D. (1981). The role of desensitization to capsaicin in chili pepper ingestion and preference. *Chem. Sens.*, 6, 23–31.
- Saldana, G. & Meyer, R. (1981). Effects of added calcium on texture and quality of canned Jalapeño peppers. J. Food Sci., 46, 1518-22.
- Tressl, R., Bahri, D. & Engel, K.-H. (1981). Lipid oxidation in fruits and vegetables. In *Quality of Selected Fruits and*

Vegetables of North America, ed. R. Teranishi & H. Barrera-Benitez. ACS Symposium Series 31, ACS, Washington, DC, pp. 213-31.

- van Ruth, S. M., Roozen, J. P. & Cozijnsen, J. L. (1994). Comparison of dynamic headspace mouth model systems for flavour release from rehydrated bell pepper cuttings. In *Trends in Flavour Research*, eds H. Moarse & D. G. van der Hey. Elsevier, Amsterdam, pp. 59–64. Proc. 7th Weurman Symp., Noordwijkerhout, The Netherlands.
- Weisenfelder, A. E., Huffman, V. L., Villalon, B. & Burns, E. E. (1978). Quality and processing attributes of selected Jalapeño pepper cultivars. J. Food Sci., 43, 885–8.
- Wu, C.-M. & Liou, S.-E. (1986). Effect of tissue disruption on volatile constituents of bell peppers. J. Agric. Food Chem., 34, 770-2.
- Wu, C.-M., Liou, S.-E. & Wang M.-C. (1986). Changes in volatile constituents of bell peppers immediately and 30 minutes after stir frying. J. Am. Oil Chem. Soc., 63, 1172-5.