

# Controlled-Atmosphere Treatment of Freshly Harvested Oranges at Elevated Temperature To Increase Volatile Flavor Components<sup>†</sup>

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Hamlin and Valencia oranges were treated under several controlled-atmosphere (CA) conditions at 33 or 38 °C for 24 h involving air flow or nitrogen or carbon dioxide gases in closed containers. Laboratory-scale studies showed a maximum 1.5–3-fold increase in acetaldehyde and ethanol and a maximum 3–15-fold increase in 4 volatile esters, while 13 minor volatile components monitored showed no consistent changes. Use of plastic bags under vacuum on individual fruit gave similar results, showing plastic films to be alternatives to inert gases in creating low-oxygen conditions. On a pilot-plant scale Pineapple oranges treated under low-oxygen CA conditions at 33 °C afforded the flavor fractions, aqueous orange essence and essence oil, having 2-fold increases in several volatile components considered to be important to orange flavor. These increases are comparable to those reported earlier from fruit treated under similar CA conditions at room temperature.

## INTRODUCTION

There is an increasing reliance on and demand for natural flavor materials in foods because of consumer demand for healthier and "all-natural" foods. One way to accomplish this end is to treat the raw foodstuff so that flavor components are enhanced prior to processing. Another widely used method in the fruit beverage industry is to isolate flavor fractions during processing for later addition to the processed products. In either case, any postharvest treatment that enhances the levels of volatile flavor components could have great economic benefit for the processor as well as quality improvement for the consumer.

In earlier studies, we used controlled-atmosphere (CA) treatment of oranges immediately after harvest to increase the levels of certain volatile components both in the laboratory (Shaw et al., 1991) and on a pilot-plant scale (Shaw et al., 1990). Those studies were carried out at room temperature. Bruemmer and Roe (1969, 1970) treated grapefruit and oranges at elevated temperatures under CA conditions and found increased levels of ethanol and acetaldehyde in grapefruit, with 33 °C being the optimum temperature of treatment. Shaw et al. (1992) recently reported increases with mandarin and mandarin hybrid fruit at 33 °C.

In the current study, effects of elevated temperatures were studied on levels of 21 volatile components in orange juice when the fruit was treated for 24 h and under CA treatment. A larger, pilot-plant-scale treatment was included as well.

## EXPERIMENTAL PROCEDURES

**Fruit and Juice Samples.** Hamlin, Pineapple, and Valencia orange fruits of uniform size were each harvested evenly from all four quadrants of three trees, and the fruits were washed, dried, and stored at 21 °C until used within 24 h after harvest. Each juice sample was a composite randomly selected sample of

20 fruits from the 100–120 fruits used for each treated or control sample. Fruits were halved and hand-juiced with an electric kitchen juicer and stored at –18 °C until analyzed.

**CA Pretreatments and Storage Conditions.** A 20-kg portion of fruit was used for each control or treated laboratory-scale sample. The air (control), nitrogen, and carbon dioxide treated samples were placed in cylindrical glass jars 28.5 cm in diameter by 61 cm, fitted with glass lids sealed to the top of each jar with stopcock grease. A hole in each lid, 2 cm in diameter, was fitted with a two-holed No. 3 rubber stopper. A 0.25 in. o.d. stainless steel tube inserted into one hole was used to introduce water-saturated air, nitrogen, or carbon dioxide gas into the jar to within 2.5 cm of the bottom. A short piece of glass tubing inserted through the second stopper hole with Tygon tubing attached vented the exit gases from the top of each cylinder to a hood. The oxygen level inside each jar was measured with a YSI Model 5300 oxygen monitor with a Model 5775 oxygen probe and standard membrane inside each chamber (Yellow Springs Instrument Co., Yellow Springs, OH). Treatments were conducted for 24 h in a constant temperature room at 33 ± 1 or 38 ± 1 °C. Gas flow rates into each jar for a given experiment were either 450 mL/min (high flow), as used earlier (Shaw et al., 1991), or 20 mL/min (low flow), as used by Bruemmer and Roe (1969, 1970). When experiments were conducted at the lower gas flow rate, a control sample was placed at the same temperature in a single layer on shelves for the 24-h treatment period. Otherwise, controls were placed inside the vessels with gas purge. After treatments, all samples were placed in single layers on shelves at 21 ± 1 °C and sampled at times indicated in Tables I–III. Oxygen levels were unchanged from that in room air in the sample purged with air at 450 mL/min, 2% or less in the nitrogen atmospheres, and 5% or less in the carbon dioxide atmospheres. The oxygen level in samples purged with air at 20 mL/min dropped to 12% after 4 h and to 4% after 20 h.

Plastic bags used in two tests at 33 °C were Cryovac B540 (ethylene vinyl acetate) shrinkwrap film bags (Cryovac Division, W. R. Grace, Duncan, SC); a vacuum of ca. 25 mmHg was applied to each bag containing a single fruit before the bags were sealed. After the 24-h treatment, these fruits were removed from the bags and stored at 21 °C next to the control and other treated fruit.

**Pilot-Plant CA Treatment and Processing of Fruit.** Forty boxes (1650 kg) of Pineapple oranges were washed, sized, and divided into four 10-box samples containing equal quantities of each size of orange. Two samples in plastic 10-box pallets were placed in a controlled-temperature room at 33 ± 1 °C as a control sample. Two 10-box samples in double 300-gal 3-mil polyethylene bags closed to create airtight chambers were held in similar plastic

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**Table I. Changes in Major Volatile Components in Oranges during Storage after Pretreatment under Low-Oxygen Atmospheres at 33 and 38 °C**

cultivar and conditions <sup>b</sup>	storage time <sup>c</sup>	quantity in juice, <sup>a</sup> ppm														
		acetaldehyde					ethanol					methanol				
		control	air	N <sub>2</sub>	CO <sub>2</sub>	PB <sup>d</sup>	control	air	N <sub>2</sub>	CO <sub>2</sub>	PB	control	air	N <sub>2</sub>	CO <sub>2</sub>	PB
<b>high flow</b>																
Hamlin (38 °C)																
Nov 30	0	— <sup>e</sup>	8.2a	13.1b	15.5b	—	— <sup>e</sup>	650a	1050b	1220b	—	— <sup>e</sup>	29a	29a	25a	—
Dec 1	1	—	8.1a	19.1b	21.6c	—	—	590a	1200b	1770c	—	—	24a	30a	35b	—
Dec 2	2	—	9.2a	21.5b	24.1c	—	—	670a	960a	1120b	—	—	30a	30a	28a	—
Dec 5	5	—	8.0a	19.2b	24.3c	—	—	600a	890a	1230a	—	—	23a	29a	31a	—
Valencia (33 °C)																
April 26	1	9.2a	8.4b	—	—	—	1030a	440b	—	—	—	84a	37b	—	—	—
April 27	2	10.6a	10.3a	—	—	—	770a	520a	—	—	—	59a	40a	—	—	—
April 30	5	10.0a	10.9a	—	—	—	610a	800a	—	—	—	51a	70a	—	—	—
<b>low flow</b>																
Hamlin (33 °C)																
Dec 5	0	7.2a	11.8b	10.1c	17.4d	12.8bc	550a	940ab	1130bc	1710d	1630cd	35a	37a	31a	42a	37a
Dec 6	1	9.5a	19.9b	27.9c	28.8c	11.8d	710a	980a	1640b	1860b	3380c	30a	44a	44a	52b	80c
Dec 7	2	8.6a	16.6b	27.2c	34.2d	8.6a	630a	1100a	1730b	2860c	4770d	45a	59a	56a	59a	101b
Dec 8	3	8.4a	18.6b	30.2c	35.9d	11.9e	660a	1180a	2300b	2330b	6730c	46a	57a	68a	48a	121b
Hamlin (33 °C)																
Nov 15	1	6.4a	14.2b	21.0c	27.8d	25.6d	330a	550b	1390c	1930d	870e	19a	21a	44b	36b	20ac
Nov 16	2	ND <sup>f</sup>	14.9a	21.6b	29.4c	26.1d	ND	660a	1300b	2220c	1220b	ND	25a	40b	36bc	29ac

<sup>a</sup> Values in the same row followed by a common letter are not significantly different ( $P > 0.05$ ). <sup>b</sup> High flow = 450 mL/min and air = control sample; low flow = 20 mL/min and control = control sample. <sup>c</sup> Days stored at 21 °C following 24-h treatment in controlled atmosphere. <sup>d</sup> PB, individual fruit treated in plastic bags. <sup>e</sup> The air-treated sample was the control in this study. <sup>f</sup> ND, not determined because of insufficient sample.

**Table II. Changes in Ethyl Acetate and Ethyl Butyrate in Oranges during Storage after Pretreatment under Low-Oxygen Atmospheres at 33 and 38 °C**

cultivar and conditions <sup>b</sup>	storage time <sup>c</sup>	quantity in juice, <sup>a</sup> ppm									
		ethyl acetate					ethyl butyrate				
		control	air	N <sub>2</sub>	CO <sub>2</sub>	PB <sup>d</sup>	control	air	N <sub>2</sub>	CO <sub>2</sub>	PB
<b>high flow</b>											
Hamlin (38 °C)											
Nov 30	0	— <sup>e</sup>	0.18a	0.30b	0.53c	—	— <sup>e</sup>	1.0a	1.1a	1.4b	—
Dec 1	1	—	0.12a	0.35b	0.67c	—	—	2.9a	9.4b	10.8c	—
Dec 2	2	—	0.11a	0.26b	0.42c	—	—	2.2a	5.8b	6.9b	—
Dec 5	5	—	0.05a	0.16b	0.25c	—	—	1.5a	3.3b	4.0c	—
Valencia (33 °C)											
April 26	1	0.23a	0.18b	—	—	—	0.37a	0.35a	—	—	—
April 27	2	0.29a	0.24a	—	—	—	0.71a	0.65a	—	—	—
April 30	5	0.24a	0.19b	—	—	—	0.69a	0.57a	—	—	—
<b>low flow</b>											
Hamlin (33 °C)											
Dec 5	0	0.12a	0.28b	0.56c	1.3d	0.96e	0.84a	0.67a	0.72a	1.4b	1.2b
Dec 6	1	0.31a	0.35a	0.84b	1.4c	2.1d	1.31a	3.97b	6.23c	10.8d	2.5e
Dec 7	2	0.11a	0.27b	0.69c	0.92d	3.1e	1.5a	4.0b	8.9c	10.2d	2.3a
Dec 8	3	0.11a	0.20b	0.60c	0.72d	4.7e	1.5a	3.4b	6.2c	8.2d	3.4b
Hamlin (33 °C)											
Nov 15	1	0.05a	0.18b	0.34c	0.77d	0.62e	1.0a	2.9b	4.8c	6.8d	6.0d
Nov 16	2	ND <sup>f</sup>	0.11a	0.28b	0.46c	0.42d	ND	2.7a	5.0b	6.6c	5.4b

<sup>a</sup> Values in the same row followed by a common letter are not significantly different ( $P > 0.05$ ). <sup>b</sup> High flow = 450 mL/min and air = control sample; low flow = 20 mL/min and control = control sample. <sup>c</sup> Days stored at 21 °C following 24-h treatment in controlled atmosphere. <sup>d</sup> PB, individual fruit treated in plastic bags. <sup>e</sup> The air-treated sample was the control in this study. <sup>f</sup> ND, not determined because of insufficient sample.

pallets in the same constant-temperature room for CA treatment with nitrogen gas as described earlier (Shaw et al., 1990). The oxygen content dropped to 1–2% after 4 h and remained within the range for the 24-h period of treatment. Immediately after treatment, control and treated fruits were transferred to fresh 10-box pallets (plastic bags removed) and stored at 25 °C for 24 h before they were processed. Control followed by treated samples were both processed on the same day. Fruits were extracted and juice was finished with standard commercial methods, and juice was concentrated in a three-effect four-stage temperature accelerated short time evaporation (TASTE) evaporator as described previously (Shaw et al., 1990). From 100-gal of juice, control fruit produced 919 mL of aqueous essence containing 15.5% ethanol (hydrometer) and 115 mL of essence oil and treated fruit produced 974 mL of aqueous essence containing 28.0% ethanol and 100 mL of essence oil (Johnson and Vora, 1983).

**GC Analyses.** Juices and corresponding standard mixtures were analyzed in duplicate or triplicate with a Perkin-Elmer

Model 8500 gas chromatograph (GC) with a Model HS-6 headspace sampler attached, and a 0.53 mm × 30 m DB-Wax fused silica capillary column with 1.0- $\mu$ m film thickness (J&W Scientific, Folsom, CA). The carrier gas was He at 6 psi column pressure. The FID detector was at 250 °C, the heated injector zone was 180 °C, and the HS-6 temperature was 80 °C with a 15-min juice equilibration time, conditions shown earlier not to cause changes or artifact formation in the components monitored (Moshonas and Shaw, 1992). Oven temperature programming was 40 °C for 6 min with heating at 6 °C/min to 180 °C. The automated injection sequence was as described earlier (Shaw et al., 1991).

Components were identified by comparison of retention times with those of standards added to juice samples (enrichment). Quantification of each component was accomplished using external standards prepared as mixtures of the identified components quantitatively added to a bland juice base. The juice base was prepared by reconstitution to 11.8 °Brix of concentrated orange juice from an evaporator that contained no

Table III. Changes in Ethyl Hexanoate and Methyl Butyrate in Oranges during Storage after Pretreatment under Low-Oxygen Atmospheres at 33 and 38 °C

cultivar and conditions <sup>b</sup>	storage time	quantity in juice, <sup>a</sup> ppm									
		ethyl hexanoate					methyl butyrate				
		control	air	N <sub>2</sub>	CO <sub>2</sub>	PB <sup>d</sup>	control	air	N <sub>2</sub>	CO <sub>2</sub>	PB
high flow											
Hamlin (38 °C)											
Nov 30	0	— <sup>e</sup>	0.07a	0.07a	0.09b	—	— <sup>e</sup>	0.026a	0.036a	0.030a	—
Dec 1	1	—	0.34a	0.63b	0.062b	—	—	0.073a	0.098a	0.082a	—
Dec 2	2	—	0.21a	0.40b	0.39b	—	—	0.068a	0.085a	0.086a	—
Dec 5	5	—	0.07a	0.22b	0.19c	—	—	0.036a	0.055a	0.063b	—
Valencia (33 °C)											
April 26	1	0.07a	0.07a	—	—	—	0.034a	0.039a	—	—	—
April 27	2	0.12a	0.12a	—	—	—	0.050a	0.034a	—	—	—
April 30	5	0.11a	0.10a	—	—	—	0.066a	0.055a	—	—	—
low flow											
Hamlin (33 °C)											
Dec 5	0	0.04a	0.03a	0.05a	0.06ab	0.09b	0.018a	tr <sup>f</sup>	0.007b	0.009b	0.050c
Dec 6	1	0.12a	0.31b	0.30b	0.70c	0.30b	0.03a	0.09b	0.08b	0.11b	0.08b
Dec 7	2	0.15a	0.39b	0.52c	0.53c	0.24a	0.05a	0.09b	0.15c	0.12d	tr
Dec 8	3	0.13a	0.24b	0.29c	0.37d	0.30c	0.05a	0.09b	0.11c	0.10d	—
Hamlin (33 °C)											
Nov 15	1	0.10a	0.33b	0.41b	0.41b	0.43b	0.02a	0.04a	0.06b	0.06b	0.05a
Nov 16	2	ND <sup>g</sup>	0.29a	0.39b	0.35b	0.39b	ND	0.05a	0.07b	0.06c	0.06c

<sup>a</sup> Values in the same row followed by a common letter are not significantly different ( $P > 0.05$ ). <sup>b</sup> High flow = 450 mL/min and air = control sample; low flow = 20 mL/min and control = control sample. <sup>c</sup> Days stored at 21 °C following 24-h treatment in controlled atmosphere. <sup>d</sup> PB, individual fruit treated in plastic bags. <sup>e</sup> The air-treated sample was the control in this study. <sup>f</sup> tr, trace, too small to be accurately quantified. <sup>g</sup> ND, not determined because of insufficient sample.

added flavor fractions. Standard mixtures were prepared in 1:1 ethanol-water as described previously (Nisperos-Carriedo and Shaw, 1990). All standard determinations were carried out in triplicate, and four concentrations of the standard mixture covering the range of values found in the juice samples were measured for determination of a standard curve for each component. With juice samples peak heights were used for all quantitative gas chromatographic measurements. An earlier study showed peak heights to have more precise results than peak areas with this headspace GC method (Moshonas and Shaw, 1992).

Aqueous orange essences (AE) and essence oils (EO) were analyzed on a Hewlett-Packard Model 5880A GC equipped with a 0.32 mm i.d. × 50 m capillary fused silica cross-linked 5% phenylmethyl silicone column (Hewlett-Packard, Avondale, PA) and a capillary inlet system fitted with a 100:1 splitter as described previously (Shaw et al., 1990). Acetaldehyde methanol and ethanol were quantified in AE using a Hewlett-Packard Model 5890 GC equipped with a 0.32 mm × 60 m capillary fused silica DB-Wax column; the oven temperature was 70 °C, and other conditions were as described earlier (Shaw et al., 1990). Peak areas were used for quantitation. Standard mixtures of components quantified in AE were prepared in water (Table IV), and those in EO were prepared in limonene (Table V). Six runs of each standard and AE were averaged to determine amounts shown in Table IV, and four runs of standard and EO were averaged for the values reported in Table V. The coefficient of variation for AE and EO components was generally less than 18% (internal standard method), as described earlier (Shaw et al., 1990).

**Statistical Analyses.** Statistical comparisons were determined by analysis of variance using the general linear model (GLM) procedure, a package program of the Statistical Analysis System (SAS Institute Inc., Cary, NC). Specific differences were determined at least significant difference (LSD). All comparisons were made at a 95% confidence level.

## RESULTS AND DISCUSSION

Hamlin and Valencia oranges were treated under several CA (low oxygen) conditions at 33 and 38 °C to afford the results shown in Tables I–III. The seven volatile juice components in those tables were those most affected quantitatively by these laboratory-scale treatments as monitored by HS-GC. Earlier studies at this laboratory (Bruemmer and Roe, 1969, 1970; Shaw et al., 1992) had

indicated CA treatments involving low-oxygen atmospheres at these elevated temperatures on grapefruit, orange, and mandarin fruits could afford up to 10–20-fold increases in two major volatile components (ethanol and acetaldehyde).

Several sets of conditions were used to create low-oxygen atmospheres for the 24-h treatment periods. When high gas flow in an airtight chamber was used (450 mL/min), the sample with air flowing through the chamber was the control sample. At low gas flow (20 mL/min), air flow was too slow to maintain a normal air oxygen level, and so fruit spread in a single layer at the same temperature as that for treated fruit was the control sample. Gas flows of nitrogen or carbon dioxide into airtight chambers were two treatments used in this study that were also used in an earlier study at room temperature (CA 25–27 °C) (Shaw et al., 1990). Fruits packaged in individual vacuum-packed plastic bags were used for the final conditions for low-oxygen-atmosphere storage with some samples.

Table I shows changes in the three major volatile components for up to 5 days after CA treatment. In general, acetaldehyde and ethanol were increased 1.5–3-fold by both nitrogen and carbon dioxide treatments in Hamlin oranges over the test period, with carbon dioxide often showing the greater increase. Methanol content was little affected by the treatments. These results are not substantially different from those reported in an earlier study under similar CA conditions but at room temperature (Shaw et al., 1990). In the two tests where plastic bags were used, the methanol and ethanol levels were significantly increased over other treatments in the December 6–8 samples but not in the November 15–16 study.

In studies comparing the effects of air in a closed container on content of these three components in juice, high air flow did not lead to any increase, while low air flow (low-oxygen conditions) caused some increases in acetaldehyde and ethanol but generally in levels lower than those where nitrogen or carbon dioxide gas was used.

The four esters that could be monitored by the HS-GC techniques used in this study are shown in Tables II and

**Table IV. Volatile Orange Components Quantified (Parts per Million) during CA Storage Studies**

component	amount present <sup>a</sup>	range
hexanol	0.11	0.07–0.17
<i>trans</i> -2-hexenol	0.027	0.003–0.07
<i>cis</i> -3-hexenol	0.39	0.21–0.60
2-methyl-1-propanol	0.07	0.03–0.14
linalool	0.035	0.005–0.062
$\alpha$ -terpineol	0.39	0.02–1.06
decanal	0.003	
hexanal	0.033	0.001–0.12
octanal	0.007	0.002–0.014
$\alpha$ -pinene	0.05	0.02–0.05
sabinene	0.03	0.02–0.07
$\gamma$ -terpinene	0.01	0.004–0.02
valencene	0.53	0.10–2.30

<sup>a</sup> Mean value in 13 control samples; some components not detected in all control samples.

III. Ethyl acetate and ethyl butyrate were the two volatile components monitored that showed the greatest increases from the treatments (Table II). CA treatment in nitrogen increased ethyl acetate up to 7-fold and ethyl butyrate up to 6-fold at 33 °C and low gas flow. CA treatment in carbon dioxide under the same conditions showed even greater effects on these two esters, increasing ethyl acetate up to 15-fold and ethyl butyrate up to almost 8-fold.

Two less abundant esters monitored were ethyl hexanoate and methyl butyrate, as listed in Table III. The magnitude of increase for these two esters was generally much less than that for the two major esters discussed above. Ethyl hexanoate showed some increase with both high and low flow conditions; the December 6 carbon dioxide treatment afforded the highest increase (about 6-fold). Methyl butyrate increased only under low flow conditions, in which a 3-fold increase was the highest increase observed. The 13 remaining components that were quantified in juice by this HS-GC technique are listed in Table IV. There were no large or sustained changes in these components caused by any of the CA treatments.

An important aspect of this study was what effect elevated-temperature CA treatment had on the composition of the commercially important flavor fractions, aqueous orange essence and essence oil. To produce these fractions on a pilot-plant scale, a minimum of 20 boxes of fruit had to be treated. In an earlier study, conditions were developed for treating this quantity of fruit under CA conditions in a nitrogen atmosphere (Shaw et al., 1990). Using these conditions, 20 boxes each of control and CA-treated fruit at 33 °C were processed, and the aqueous essence and essence oil from control and treated fruits were analyzed.

Quantities of 24 volatile components quantified in aqueous essences for control and treated fruit are listed in Table V. Most of the aqueous essence components that significantly increased were approximately doubled by the CA treatment. These include acetaldehyde, methyl and ethyl butyrate, and ethyl 3-hydroxyhexanoate, compounds believed to be important to fresh orange flavor (Shaw, 1991).

The two components most affected by the treatment were 1-butanol and 1,1-diethoxyethane. 1-Butanol was the aqueous orange essence component most increased in the earlier study at room temperature (Shaw et al., 1990). 1,1-Diethoxyethane is an artifact formed from ethanol and acetaldehyde in the acidic juice medium. Thus, it should be at a higher level in aqueous essence, which contains increased levels of ethanol and acetaldehyde (Nursten, 1970).

Increases in certain volatile components in essence oil

**Table V. Quantities (Parts per Million) of Volatile Components in Aqueous Essences from 33 °C Controlled Atmosphere Treated and Control Pineapple Oranges**

component	control	treated
acetaldehyde <sup>a</sup>	1800	3360 <sup>b</sup>
methanol <sup>a</sup>	6600	7725
ethanol <sup>a</sup>	109000	222000 <sup>b</sup>
acetone	74	290 <sup>b</sup>
1-propanol	80	110
ethyl acetate	210	280
2-methyl-1-propanol	47	61
1-butanol	2	31 <sup>b</sup>
1-penten-3-ol	8	7
1-penten-3-one	34	41
methyl butyrate	5	8 <sup>b</sup>
1,1-diethoxyethane	9	44 <sup>b</sup>
3-methyl-1-butanol	110	140
2-methyl-1-butanol	23	28
ethyl butyrate	120	200 <sup>b</sup>
<i>trans</i> -2-hexenal	75	67
<i>trans</i> -2-hexenol	6	6
octanal	9	12
octanol	10	19 <sup>b</sup>
linalool	53	62
ethyl 3-hydroxyhexanoate	27	57 <sup>b</sup>
terpinen-4-ol	4	5
$\alpha$ -terpineol	9	9
neral	3	3
geranial	2	3

<sup>a</sup> Determined on polar DB-Wax column. <sup>b</sup> Significantly different from control, 95% confidence level.

**Table VI. Quantities (Parts per Million) of Volatile Components in Essence Oils from 33 °C Controlled Atmosphere Treated and Control Pineapple Oranges**

component	control	treated
methanol	170	260 <sup>a</sup>
ethanol	1960	4460 <sup>a</sup>
ethyl acetate	380	590 <sup>a</sup>
1-penten-3-one	119	134 <sup>a</sup>
methyl butyrate	84	114 <sup>a</sup>
ethyl butyrate	2860	6900 <sup>a</sup>
<i>trans</i> -2-hexenal	1740	1470 <sup>a</sup>
<i>trans</i> -2-hexenol	42	41
$\alpha$ -pinene	5980	5520 <sup>a</sup>
sabinene	2670	2320 <sup>a</sup>
myrcene	69900	65700 <sup>a</sup>
ethyl hexanoate	404	458 <sup>a</sup>
octanal	4350	3690 <sup>a</sup>
octanol	68	82 <sup>a</sup>
linalool	3980	3400 <sup>a</sup>
nonanal	794	707 <sup>a</sup>
ethyl 3-hydroxyhexanoate	40	61 <sup>a</sup>
citronellal	450	360 <sup>a</sup>
decanal	3340	2910 <sup>a</sup>
neral	670	520 <sup>a</sup>
carvone	290	290
geranial	850	630 <sup>a</sup>
perillaldehyde	130	100 <sup>a</sup>
valencene	20500	19600 <sup>a</sup>

<sup>a</sup> Significantly different from control, 95% confidence level.

could have the most commercial significance of any of these changes, since essence oil is often fractionated to recover selected flavor fractions, e.g., aldehydes, esters, and alcohols, for "enhancement" of aqueous essence added to citrus beverages (Flora, 1988; Hendrix and Redd, 1991). Table VI lists the essence oil components quantified and those significantly changed by the CA treatment at 33 °C. Of the nine components in Table VI that showed significant increases because of the treatment, two are the major alcohols, methanol and ethanol, and five are the methyl and ethyl esters identified that would be expected to increase as the corresponding alcohols increased. The one ketone that increased was 1-penten-3-one, a potentially important

flavor component first isolated from aqueous orange essence at our laboratory (Moshonas and Shaw, 1973). Octanol was the ninth component showing a significant increase from the CA treatment.

In conclusion, low-oxygen CA treatments of oranges at 33–38 °C caused a maximum 2–3-fold increase in two major volatile components, ethanol and acetaldehyde. These are similar to the increases found earlier at room temperature, rather than the 10–20-fold increases found by Bruemmer and Roe (1969, 1970) under similar conditions. Several esters were increased by much larger amounts in laboratory-scale experiments (ethyl acetate up to 7-fold and ethyl butyrate up to 6-fold). Treatment under low-oxygen CA at 33 °C on a pilot-plant scale resulted in about 2-fold increases in the main volatile components of the commercially important flavor fractions, aqueous orange essence and essence oil. Thus, no greater enhancement in volatile flavor components resulted from low-oxygen CA treatment at 33 °C than was seen at room temperature. Use of plastic bags was effective in causing these changes due to low-oxygen conditions. Plastic films or other oxygen-impermeable coatings may offer practical alternatives to inert atmospheres in altering the composition of volatile components in citrus fruit prior to processing.

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