

Changes in Physicochemical Characteristics and Volatile Constituents of Yellow- and White-Fleshed Nectarines during Maturation and Artificial Ripening

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Changes in the volatile composition during maturation and artificial ripening of yellow-fleshed nectarines were investigated by means of GC-FID and GC-MS over three years (1999–2001). Unripe and commercially ripe fruits were placed in ripening chambers until complete ripening and compared to tree-ripe nectarines. Firmness, weight, soluble solids (SS), titratable acidity (TA), SS/TA ratio, organic acids, and sugars were also determined. Furthermore, the tree-ripe and artificially ripened yellow-fleshed nectarines were subjected to sensory analysis by a taste panel. In 2001, a similar experiment on white-fleshed nectarines (cv. Vermeil) was performed to compare with the results obtained on yellow-fleshed nectarines. Levels of volatiles compounds, in particular, lactones and C₁₃ norisoprenoids, were found to be the same or higher in the artificially ripened nectarines compared with the tree-ripe nectarines. In addition, no significant difference was observed for sweetness, sourness, and the intensities of “peach odors” or “peach aroma” in the sensory analysis between the tree-ripe samples and the artificially ripened nectarines. However, in the latter the levels of SS and the SS/TA ratio were very close to those observed in the initial unripe samples and significantly lower than those observed in tree-ripe nectarines.

KEYWORDS: Nectarines; flavor; aroma; volatile compounds; artificial ripening; maturation

INTRODUCTION

Because peaches and nectarines are often harvested for commercial reasons before their complete maturity, the consumer often complains about the low quality of these fruits. Aware of this, peach and nectarine growers have tried to modify their practices in order to be able to produce better quality fruits. However, the fruits are often handled just after the harvest and must be picked at a relatively high level of firmness, not acceptable for rapid consumption. Thus, an extra phase of maturation under controlled conditions—or artificial ripening—was added to supply more mature, juicier, and more aromatic peaches and nectarines. Aroma is one of the essential factors for evaluating fruit quality. Peach and nectarine volatiles have been intensively investigated, and ~100 compounds have been identified (1–18). Among them, lactones, particularly γ - and

δ -decalactone, have been reported as character impact compounds in peach aroma, but they act in association with other volatiles, such as C₆ aldehydes, aliphatic alcohols, and terpenes. The formation of aroma compounds in fruit is a dynamic process, during which concentrations of volatiles change both qualitatively and quantitatively. In peaches and nectarines, these changes essentially depend on their degree of maturity (4, 5, 8, 12, 15, 17, 19, 20). In immature fruits, C₆ compounds are the major components, but the levels of these so-called green components decrease drastically and those of lactones, benzaldehyde, and linalool increase significantly during fruit maturation (1, 3, 4, 8). Nevertheless, changes in aroma composition during artificial maturation have been hardly studied, and knowledge is limited to the findings of Do et al. (3) and Bayonove (5) in peaches. In this work changes in physical and chemical characteristics and volatile composition from yellow-fleshed (cv. Springbright) and white-fleshed (cv. Vermeil) nectarines subjected to “on-tree maturation” and artificial ripening were studied to better understand the impact of the latter on the whole quality of fruits.

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Table 1. Effect of Maturity on the Physical Characteristics^a of Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₀		B ₀		C ₀	
	mean	range	mean	range	mean	range
F _p	7.4z	4.5–10.1	4.8y	2.7–7.7		
wt (g)	150z	116–189	180y	137–217	180y	133–225
F _d					54.8	37.1–73.3

^a Values are given as average of a 3-year trial ($n = 90$). F_p = firmness determined using Penefel (kg/0.5 cm²); F_d = firmness determined using Durofel (ID₁₀). Values with different letters are significantly different (based on Newman-Keuls test, $P < 0.05$).

Table 2. Effect of Artificial Ripening on the Physical Characteristics^a of Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₁₈		A ₂₆		B ₁₈		B ₂₆		SSD
	mean	range	mean	range	mean	range	mean	range	
wt (g)	152*	103–210	152*	118–206	174	138–207	179	139–235	6.9
F _d	49.2*	36.2–73.9	48.1*	33.3–67.6	47.0*	38.5–55.7	48.3*	30.7–58.5	2.4

^a Values are given as average of a 3-year trial ($n = 90$). F_p = firmness determined using Penefel (kg/0.5 cm²); F_d = firmness determined using Durofel (ID₁₀). An asterisk (*) indicates significant decrease compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

Table 3. Effect of Maturity on the Chemical Characteristics^a of Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₀		B ₀		C ₀	
	mean	range	mean	range	mean	range
SS	9.6z	8.3–10.9	11.1y	9.1–12.6	11.8y	9.1–13.2
TA	1.02z	0.9–1.1	0.94z	0.8–1.1	0.72y	0.6–0.9
SS/TA	9.42z	8.6–11.1	11.85z	10.2–13.8	16.67y	14–21.9

^a Values are given as average of a 3-year trial ($n = 9$). SS = soluble solid (% Brix); TA = titratable acidity (g/100 g, reported as malic acid). Values with different letters are significantly different (based on Newman-Keuls test, $P < 0.05$).

Table 4. Effect of Maturity on the Sugar and Organic Acid Contents^a of Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₀		B ₀		C ₀	
	mean	range	mean	range	mean	range
sucrose	5.59z	4.5–6.4	6.89y	5.7–7.6	7.42y	6.1–8.2
fructose	0.97	0.9–1	1.00	0.9–1.1	0.98	0.9–1
glucose	0.95	0.9–1	0.98	0.9–1	0.95	0.9–1
citric acid	0.47z	0.4–0.5	0.39y	0.3–0.4	0.20x	0.1–0.2
malic acid	0.67	0.5–0.8	0.74	0.6–1	0.75	0.6–0.9

^a Values expressed in g/100 g are given as average of a 3-year trial ($n = 9$). Values with different letters are significantly different (based on Newman-Keuls test, $P < 0.05$).

MATERIALS AND METHODS

Samples. Nectarines were obtained from a local commercial orchard (R. Montoux-Caillet, Mourières, Bouches-du-Rhône, France). Yellow-fleshed nectarines (cv. Springbright) were hand-picked at three stages of maturity in July 1999, 2000, and 2001, whereas white-fleshed nectarines (cv. Vermeil) were picked at only two stages of maturity in July 2001. The different degrees of maturity were determined according to size, color, and firmness and then classified as “unripe” (A₀), “commercially ripe” (B₀) (only for yellow-fleshed samples), and tree-ripe (C₀). About 500 fruits from A₀ and B₀ stages were placed in trays and stored in ripening chambers maintained at 18 °C (A₁₈ and B₁₈) or 26 °C (A₂₆ and B₂₆) and 85% relative humidity until complete maturity. Firmness of 30 fruits, randomly picked from each ripening sample (A₁₈,

B₁₈, A₂₆, and B₂₆), was daily determined. Complete maturity was determined when the average firmness of the artificially ripened samples was lower than 60 ID₁₀. Firmness lower than 60 ID₁₀ is equivalent to a firmness ranging from 0.5 to 1 kg/0.5 cm².

Fruits were subjected to physical analysis and then sliced; pits were discarded, and fruit slices were frozen with liquid nitrogen and stored at –25 °C until isolation of volatiles and chemical analysis.

Physical Analysis. Firmness of hard fruits (A₀ and B₀) was determined using a hand penetrometer, whereas for soft fruits (A₁₈, B₁₈, A₂₆, B₂₆, and C₀) the firmness was determined using a spring dynamometer. For samples A₀ and B₀, firmness was determined on two opposite sides of 30 nectarines using an electronic Penefel (licensed by Copa-Ctifl) with a 0.50 cm² tip. The exocarp on the area to be pressure-tested was carefully removed from the fruit with a razor blade just before the fruit was punctured with the plunger. The measurements are expressed in kg/0.5 cm². For artificially ripened and C₀ nectarines, firmness was also determined on two opposite sides of 30 nectarines but using an electronic Durofel (licensed by Copa-Ctifl) with a 0.10 cm² tip, as Durofel is more suitable for soft fruits than Penefel. The Durofel, a nondestructive dynamometer, provides a measure of the force used to press a spring-loaded pawl against the fruit surface (21, 22). The measurements are expressed in ID₁₀. The average weight of 30 fruits was also obtained for each maturity stage.

Chemical Analyses. The total soluble solids content (SS) in juice (% Brix) was determined with an Atago PR-101 digital refractometer. Total acidity (TA) was determined by titrating 5 mL of juice to pH 8.1 with 0.1 N NaOH using an automatic titrator Crison Compact II with autosampler. The individual sugars (glucose, fructose, and sucrose) and organic acids (malic and citric) were determined in triplicate from 1 mL of juice using a single injection HPLC technique as described by Doyon et al. (23). These measurements were determined from the juice obtained from 300 g of fruit after homogenization and centrifugation (8500g, 20 min, 4 °C).

Isolation and Concentration of the Volatiles. Two hundred grams of frozen fruits, 100 mL of distilled water, and 133.3 g of (NH₄)₂SO₄ were homogenized in a Waring blender for 3 min. The pulp was then centrifuged (9000g, 30 min, 4 °C), and the clear juice was filtered through glass wool and immediately extracted. Two hundred milliliters of clear juice previously obtained with 32 μg of 4-nonanol (internal standard) added was extracted in batches with 70 mL of analytical grade dichloromethane (further purified by distillation before use) for 30 min under magnetic stirring at 4 °C. Then, the mixture was centrifuged (8500g, 10 min, 4 °C) and then transferred to a 250 mL separatory funnel. The aqueous phase was then re-extracted twice as mentioned above, according to the method of Moio et al. (24). After centrifugation and separation, both organic extracts were dried over anhydrous sodium sulfate, filtered through glass wool, and concentrated to ~1 mL using a small Vigreux column at 45 °C. The extract was then stored at –25 °C until analysis. All analyses were performed in triplicate.

GC-FID Conditions. A Varian 3300 gas chromatograph equipped with an on-column injector was used. The flow of hydrogen carrier gas was 1.5 mL/min. The oven was kept at 40 °C for 3 min, then programmed to 245 °C at 3 °C/min, and kept at 245 °C for 20 min. The injector was kept at 20 °C for 0.1 min, then programmed to 245 °C at 180 °C/min, and kept at 245 °C for 85 min. The FID was kept at 245 °C. One microliter of each sample was injected on a DB-Wax (J&W Scientific, Folsom, CA) capillary column (30 m × 0.32 mm i.d., 0.5 μm film thickness). Levels of volatile compounds were expressed as 4-nonanol equivalent (assuming all response factors were 1). The concentrations are to be considered as relative data because recovery after extraction and calibration factors related to the standard were not determined.

GC-MS Conditions. A Hewlett-Packard 5989 series II gas chromatograph equipped with an on-column injector was used with the same DB-Wax capillary column as above. The flow of helium N60 carrier gas was 1.5 mL/min. The oven and injector temperature programs were as above. A Hewlett-Packard 5889A mass spectrometer with a quadrupole mass filter was used. Mass spectra were recorded in electronic impact (EI) ionization mode at 70 eV. The transfer line, source, and quadrupole temperatures were set, respectively, at 250, 250, and 120 °C. Mass spectra were scanned in the range m/z 29–350 amu

Table 5. Changes in the Concentrations^a of Volatiles during Maturation of Yellow-Fleshed Nectarines (Cv. Springbright)

RI ^b	assignment ^c	compound	A ₀		B ₀		C ₀	
			mean	range	mean	range	mean	range
1163	A	3-penten-2-ol	273	15–830	64	16–167	30	0–89
1220	A	(E)-2-hexenal	590z	375–1004	199y	103–373	70y	8–197
1278	A	acetoin	14z	0–26	40z	9–95	406y	105–951
1406	A	(E)-2-hexen-1-ol	22	12–38	46	3–98	39	12–83
1508	A	benzaldehyde	8	5–12	5	2–13	6	0–14
1539	A	linalool	99	19–196	96	59–169	52	19–107
1589	A	γ-valerolactone	10z	6–12	13z	8–18	22y	14–32
1665	B (25)	1-methyl-2-pyrrolidone	13	4–29	18	5–43	15	3–54
1694	A	γ-hexalactone	308z	269–376	471y	344–821	620x	496–736
1731	A	(E)-pyran linalool oxide	13z	9–20	21y	13–38	13z	5–23
1758	A	(Z)-pyran linalool oxide	4	1–8	6	2–9	2	0–9
1796	A	γ-heptalactone	9	5–14	9	7–11	12	8–19
1912	A	γ-octalactone	14zy	4–35	6y	0–13	27z	6–56
1949	A	3,7-dimethyl-1,5-octadiene-3,7-diol	37z	255–537	388	186–698	243	54–526
1964	A	δ-octalactone	4z	3–6	7z	4–12	14y	4–26
1981	A	linalool hydrate	24	7–50	42	14–76	45	23–74
2029	B (26)	2-pyrrolidinone	10z	0–18	18y	11–29	15zy	10–19
2136	A	γ-decalactone	12z	4–32	22z	16–35	55y	31–70
2164	A	eugenol	5	1–9	5	1–10	8	6–10
2175	B (18)	6-pentyl-α-pyrone	6z	4–13	24y	6–45	39y	17–72
2194	A	δ-decalactone	9z	5–18	39y	7–67	67x	28–101
2320	A	γ-5-hydroxyhexalactone (isomer 1)	93	39–197	64	16–161	100	30–182
2374	A	γ-5-hydroxyhexalactone (isomer 2)	25z	17–36	35z	25–53	100y	26–209
2663	C	3-hydroxy-5,6-epoxy-7,8-dihydro-β-ionone ^d	261	192–347	280	193–455	242	180–340
2681	B (25)	3-hydroxy-7,8-dihydro-β-ionol	7z	6–8	12z	6–32	26y	11–44
2700	B (26)	3-hydroxy-β-ionone	20z	15–29	41y	16–59	52y	40–68
2739	B (25)	3-hydroxy-5,6-epoxy-β-ionone	85	4–166	81	4–186	77	9–172

^a Values expressed in μg/kg are given as average of a 3-year trial ($n = 9$). ^b RI, linear retention index based on a series of n -hydrocarbons. ^c A, identities confirmed by comparing mass spectra and retention time with those of authentic standards; B, identities tentatively assigned by comparing mass spectra with those obtained from the literature (given in parentheses); C, tentatively identified. ^d Major mass spectral fragments [m/e (%): 123 (100), 109 (62), 193 (58), 43 (40), 125 (22), 124 (21), 208 (20), 226 (16, [M]⁺). Values with different letters are significantly different (based on Newman–Keuls test, $P < 0.05$).

Table 6. Effect of Artificial Ripening on the Chemical Characteristics^a of Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₁₈		A ₂₆		B ₁₈		B ₂₆		SSD
	mean	range	mean	range	mean	range	mean	range	
SS	10.8	9.8–11.3	10.4	8.5–11.4	11.0	9.7–12	11.1	9.6–12	
TA	0.84	0.7–1	0.81	0.7–1	0.78	0.7–0.9	0.83	0.6–1.1	
SS/TA	13.01*	11.6–14.7	13.07*	11.3–16.6	14.24	12–16.5	14.04	11.3–19.2	3.01

^a Values are given as average of a 3-year trial ($n = 9$). SS = soluble solid (% Brix); TA = titratable acidity (g/100 g, reported as malic acid). An asterisk (*) indicates significant decrease compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

Table 7. Effect of Artificial Ripening on the Sugar and Organic Acid Contents^a of Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₁₈		A ₂₆		B ₁₈		B ₂₆		SSD
	mean	range	mean	range	mean	range	mean	range	
sucrose	6.06*	5.9–6.2	5.19*	4.7–5.4	6.27*	5.7–7.1	5.84*	5.3–6.2	0.54
fructose	1.08**	1–1.2	1.17**	1–1.3	1.11**	1–1.2	1.03	0.9–1.2	0.1
glucose	0.95	0.9–1.1	1.05	1–1.2	0.98	0.9–1.1	0.92	0.8–1.1	
citric acid	0.39**	0.4–0.4	0.44**	0.3–0.5	0.35**	0.3–0.4	0.36**	0.2–0.5	0.08
malic acid	0.60*	0.6–0.7	0.53*	0.4–0.6	0.62*	0.5–0.7	0.60*	0.5–0.8	0.12

^a Values expressed in g/100 g are given as average of a 3-year trial ($n = 9$). An asterisk (*) indicates significant decrease and two asterisks (**) indicate significant increase compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

at 1 s intervals. Identifications were carried out by comparison of linear retention indices and EI mass spectra with published data or with data from authentic compounds.

HPLC Conditions. A Waters 600 E liquid chromatograph equipped with a Waters 610 fluid unit pump was used. A variable-wavelengths

UV–vis detector (Waters 486) set at 210 nm and a differential refractometer (Waters 410) were connected in series and used as detectors. Twenty microliters of each sample was injected on a 300 mm × 7.8 mm i.d. cation-exchange ICsep ICE-ION-300 column equipped with an ICsep ICE-GC-801/C guard column (Transgenomic, San Jose, CA). The column oven temperature was set at 30 °C, and the flow of mobile phase (0.008 N H₂SO₄) was 0.4 mL/min.

Sensory Evaluation. Sensory evaluation was performed each year on the tree-ripe (C₀) and artificially ripened (A₁₈, A₂₆, B₁₈, and B₂₆) nectarines (cv. Springbright). The fruits were washed and placed into coded cups, using a three-digit random number, for evaluation. The samples were served in a random monadic sequential presentation and were evaluated by a taste panel of 11 members, trained ~12 h per year, for 9 descriptors (firmness, juiciness, mealiness, stringiness, sourness, sweetness, peach aroma, peach external odor, and peach internal odor) by using a structured line scale anchored at the ends by numeric values of 0 and 10.

Statistical Analysis. Analyses of variance and factorial discriminant analyses (FDA) were performed, respectively, using StatboxPro 5.0 (Grimmersoft) and Statlab 3.0 (SLP Infoware). Significant differences were determined at $P < 0.05$.

RESULTS AND DISCUSSION

Yellow-Fleshed Nectarines. The effects of artificial ripening and maturity on the physical and chemical constituents of yellow-fleshed nectarines (cv. Springbright) were investigated over three years. Fruits were picked at three stages of ripening: unripe (A₀), commercially ripe (B₀), and tree-ripe (C₀). Fruits from the two first stages were placed in ripening chambers maintained at 18 °C (A₁₈ and B₁₈) or 26 °C (A₂₆ and B₂₆) and 85% relative humidity until complete ripening. The above temperatures were selected in agreement with current professional practices. Over the three years, the average times required to reach complete maturity of fruits were, respectively, 7.7 days

Table 8. Changes in the Concentrations^a of Volatiles during Artificial Ripening of Yellow-Fleshed Nectarines (Cv. Springbright)

compound ^b	A ₁₈		A ₂₆		B ₁₈		B ₂₆		SSD
	mean	range	mean	range	mean	range	mean	range	
3-penten-2-ol	147	9–556	173**	26–341	26	11–54	92	0–290	143
(<i>E</i>)-2-hexenal	220*	86–429	351*	29–1053	192*	42–547	86	23–226	115
acetoin	47*	5–105	293	19–681	24*	18–30	122*	31–333	225
(<i>E</i>)-2-hexen-1-ol	12	4–21	18	0–32	38	3–95	14	8–28	
benzaldehyde	13	2–24	52**	4–109	10	3–16	8	0–21	24
linalool	21	4–47	123**	5–386	14	5–32	44	19–77	90
γ -valerolactone	15	12–18	38**	15–60	17	11–30	28	13–66	13
1-methyl-2-pyrrolidone	17	5–36	21	0–49	23	3–66	22	8–50	
γ -hexalactone	912	704–1208	1635**	769–2761	822	642–1094	636	489–859	447
(<i>E</i>)-pyran linalool oxide	19	14–29	47**	22–90	18	12–26	19	4–35	16
(<i>Z</i>)-pyran linalool oxide	2	0–6	9	0–28	2	0–7	13**	2–44	10
γ -heptalactone	15	5–24	23	10–33	12	9–18	20	13–33	7
γ -octalactone	35	16–49	59**	19–112	23	9–36	34	15–71	23
3,7-dimethyl-1,5-octadiene-3,7-diol	306	128–444	792**	105–1481	279	132–433	368	130–670	342
δ -octalactone	15	3–25	74**	4–205	15	2–30	22	6–35	44
linalool hydrate	35	19–62	77**	27–113	29	12–47	39	16–63	25
2-pyrrolidinone	12	9–16	108**	8–309	12	7–17	20	10–37	69
γ -decalactone	93	33–166	88	44–140	74	52–108	112**	53–208	46
eugenol	13	4–26	21**	10–44	5	1–8	8	4–19	7
6-pentyl- α -pyrone	121**	39–238	173**	64–332	84	42–152	61	22–113	77
δ -decalactone	61	39–87	183**	64–319	59	32–88	143**	80–233	62
γ -5-hydroxyhexalactone (isomer 1)	61	37–150	168	50–303	48	13–119	65	12–165	
γ -5-hydroxyhexalactone (isomer 2)	43*	13–65	44	7–104	40*	24–65	71	45–102	47
3-hydroxy-5,6-epoxy-7,8-dihydro- β -ionone	205	134–299	279	152–521	202	157–278	276	149–454	
3-hydroxy-7,8-dihydro- β -ionol	30	9–51	183**	28–432	33	15–53	31	13–53	99
3-hydroxy- β -ionone	137**	48–322	155**	88–225	74	49–107	85	55–120	51
3-hydroxy-5,6-epoxy- β -ionone	85	25–121	198**	136–338	67	19–107	84	18–187	68

^a Values expressed in $\mu\text{g}/\text{kg}$ are given as average of a 3-year trial ($n = 9$). ^b For RI and identity assignment see Table 5. An asterisk (*) indicates significant decrease and two asterisks (**) indicate significant increase compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

(range 6–10 days) at 18 °C and 4.5 days (range 2–7 days) at 26 °C for the fruits from stage A and 6 days (range 5–7 days) at 18 °C and 2.3 days (range 1.5–3 days) at 26 °C for the fruits from stage B. Firmness, weight, soluble solids (SS), titratable acidity (TA), SS/TA ratio, organic acids, sugars, and concentrations of main volatile compounds were determined for the three harvests. Furthermore, the fruits were subjected to sensory analysis by a taste panel of 11 members. About 60 volatile compounds were identified and quantified, and 27 were common to all samples over the three years. The effects of maturity and artificial ripening on the physical and chemical characteristics of cv. Springbright nectarines are summarized in Tables 1–8. The data are given as average and minimum–maximum range of the three years of harvest. To follow the evolution of data and to compare differences between unripe, commercially ripe, and tree-ripe samples during the on-tree maturation, data were analyzed using a one-way analysis of variance (ANOVA) with the Newman–Keuls multiple-range test: experimental design, one factor (degree of maturity) with three levels (A₀, B₀, and C₀), complete randomization with nine samples (three replications \times three years). To compare differences between artificially ripened nectarines and tree-ripe nectarines, considered as control, a one-way ANOVA with the Dunnett test was used: experimental design, one factor (ripening mode) with five levels (A₁₈, B₁₈, A₂₆, B₂₆, and C₀), complete randomization with nine samples (three replications \times three years).

As shown in Tables 1 and 2, firmness decreased, whereas weight increased, with natural maturation. No significant change in weight was observed during artificial ripening. Nevertheless, despite the care taken during this study, the average firmness of the tree-ripe samples, although always lower than 60 ID₁₀, was generally significantly higher than that of the artificially ripened nectarines. A factorial discriminant analysis (FDA), using 63 samples (7 ripening modes \times 3 replications \times 3 years)

and 35 variables, was performed to summarize graphically the effects of the natural and artificial maturations on the data displayed in Tables 3–8.

As shown in Figure 1, the results of the FDA show similar effects over the three years, and 82% of the total variance was explained by the two first axes. The first axis (57% of the variance explained) mainly discriminates the artificially ripened nectarines, in particular, samples A₂₆, from the others. These artificially ripened samples are characterized by greater contents of fructose, 3-hydroxy-7,8-dihydro- β -ionol, 3-hydroxy- β -ionone, 3-hydroxy-5,6-epoxy- β -ionone, benzaldehyde, eugenol, 2-pyrrolidinone, *trans*-pyran linalool oxide, 3,7-dimethyl-1,5-octadiene-3,7-diol, γ -valerolactone, γ -hexalactone, γ -heptalactone, γ -octalactone, δ -octalactone, δ -decalactone, 6-pentyl- α -pyrone, and δ -decalactone. Samples A₀, B₀, and C₀ are mainly characterized by high levels of malic acid. The second axis (25% of variance explained) mainly discriminates unripe samples (A₀) from tree-ripe samples (C₀). Samples A₀ are characterized by greater contents of citric acid, TA, 3-penten-2-ol, and (*E*)-2-hexenal, whereas samples C₀ are characterized by greater levels of SS, SS/TA ratio, sucrose, acetoin, and γ -5-hydroxyhexalactone (isomer 2).

Effect of Natural Maturation. The results obtained from the analyses of variance of the chemical and chromatographic data are reported in Tables 3–5. In general, the mean for the three years of harvest of SS, SS/TA ratio, and sucrose content significantly increased with increased maturity. TA and citric acid content significantly decreased with increased maturity. Fructose, glucose, and malic acid contents did not change significantly with maturation. These results are consistent with those previously reported in peaches or nectarines (4, 11, 15, 18, 29, 30).

As shown in Table 5, the levels of (*E*)-2-hexenal, accounting for the “green” note of immature fruits, decreased with increased

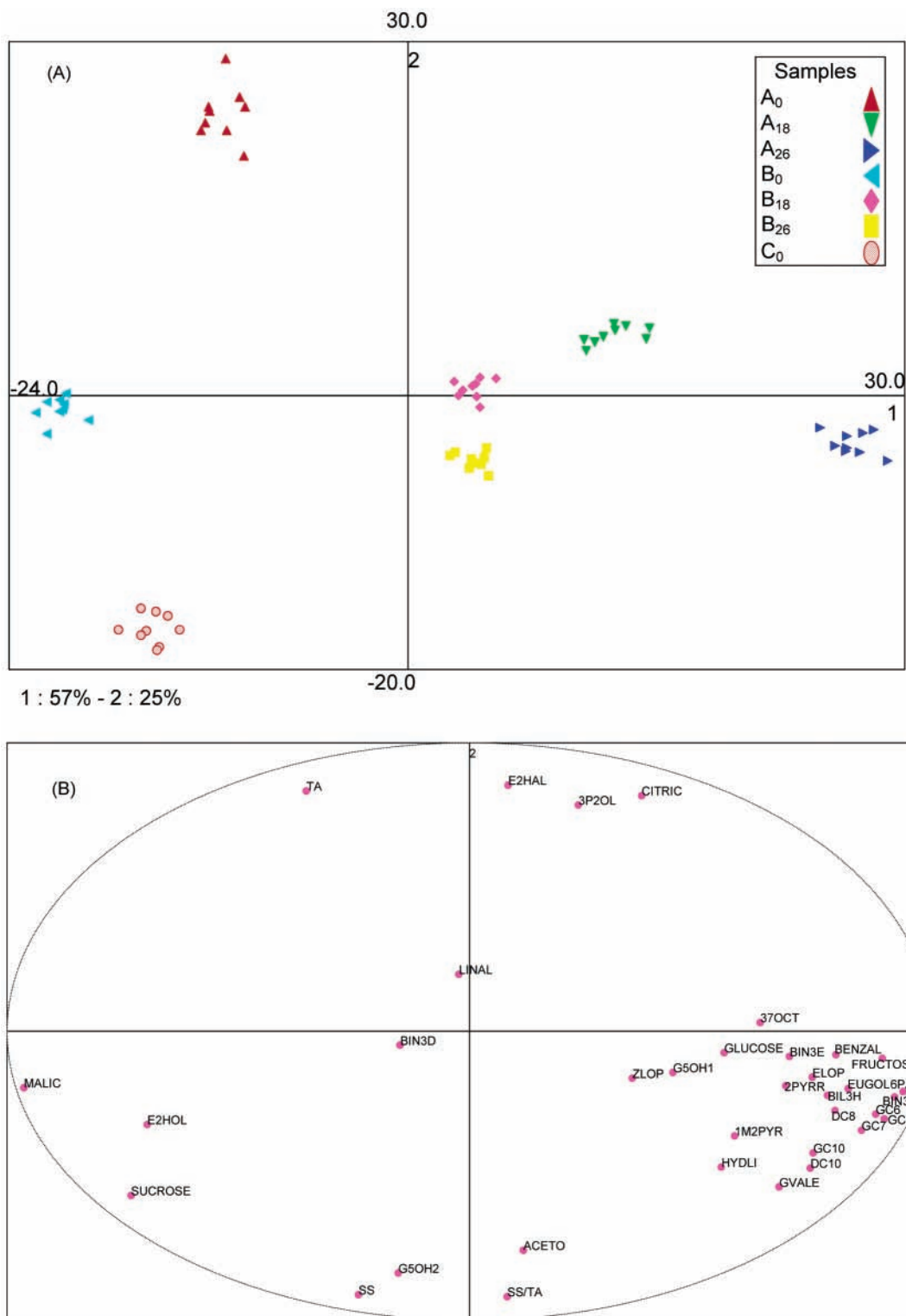


Figure 1. Projection of samples (A) and variables (B) on the first two axes of the FDA carried out using the data given in Tables 3–8. SS, solids soluble; TA, total acidity; 3P2OL, 3-penten-2-ol; E2HAL, (*E*)-2-hexenal; ACETO, acetoin; E2HOL, (*E*)-2-hexen-1-ol; BENZAL, benzaldehyde; LINAL, linalool; GVALE, γ -valerolactone; 1M2PYR, 1-methyl-2-pyrrolidone; GC6, γ -hexalactone; ELOP, (*E*)-pyran linalool oxide; ZLOP, (*Z*)-pyran linalool oxide; GC7, γ -heptalactone; GC8, γ -octalactone; 37OCT, 3,7-dimethyl-1,5-octadiene-3,7-diol; DC8, δ -octalactone; HYDLI, linalool hydrate; 2PYRR, 2-pyrrolidinone; GC10, γ -decalactone; EUGOL, eugenol; 6PAPY, 6-pent- α -pyrone; DC10, δ -decalactone; G5OH1, γ -5-hydroxyhexalalactone (isomer 1); G5OH2, γ -5-hydroxyhexalalactone (isomer 2); BIN3D, 3-hydroxy-5,6-epoxy-7,8-dihydro- β -ionone; BIL3H, 3-hydroxy-7,8-dihydro- β -ionol; BIN3O, 3-hydroxy- β -ionone; BIN3E, 3-hydroxy-5,6-epoxy- β -ionone.

maturity. This is consistent with results previously mentioned (8, 12, 15, 17). On the contrary, the levels of (*E*)-2-hexen-1-ol did not change significantly with maturation. The limited number of C₆ compounds identified in this study and their low

levels are mainly due to the isolation method chosen [enzyme deactivation with saturated (NH₄)₂SO₄ solution during homogenization] inhibiting their formation during the crushing of the fruits from lipids. Thus, their levels can be considered as

Table 9. Means^a of the Sensory Descriptors for the Yellow-Fleshed Nectarines (Cv. Springbright)

	A ₁₈	A ₂₆	B ₁₈	B ₂₆	C ₀	SSD
peach external odor	6.2 ± 2.0	5.6 ± 2.1	5.8 ± 2.4	5.3 ± 2.5	5.7 ± 2.1	
peach internal odor	6.2 ± 2.0	5.8 ± 2.3	5.6 ± 2.3	5.8 ± 2.3	5.7 ± 2.5	
firmness	3.5 ± 2.5*	3.5 ± 2.4*	3.2 ± 2.3*	3.3 ± 2.5*	5.8 ± 2.9	1.5
juicy	6.8 ± 2.1*	6.9 ± 2.2*	7.4 ± 2.0**	7.7 ± 1.9**	5.5 ± 2.6	1.3
mealy	0.9 ± 1.4*	0.7 ± 1.2*	0.8 ± 1.5**	1.3 ± 1.8**	1.1 ± 1.6	
stringy	2.4 ± 2.3*	2.1 ± 1.9*	2.1 ± 2.3**	2.5 ± 2.5**	1.8 ± 2.1	
sourness	4.6 ± 2.1*	3.8 ± 2.7*	3.8 ± 2.5**	4.0 ± 2.7**	3.2 ± 2.6	
sweetness	5.2 ± 2.2*	5.5 ± 2.5*	5.5 ± 2.0**	5.7 ± 2.3**	6.4 ± 2.4	
peach aroma	6.1 ± 1.8*	6.5 ± 2.0*	6.2 ± 2.0**	6.5 ± 1.7**	6.2 ± 2.3	

^a Values are given as average ± standard deviation of a 3-year trial ($n = 3$). An asterisk (*) indicates significant decrease and two asterisks (**) indicate significant increase compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

Table 10. Effect of Maturity and Artificial Ripening on the Physical Characteristics^a of White-Fleshed Nectarines (Cv. Vermeil)

	Newman–Keuls test		Dunnett test	
	A ₀	C ₀	A ₁₈	A ₂₆
F _p	8.9 ± 0.8			
wt (g)	200 ± 46z	207 ± 37z	204 ± 38z	199 ± 41
F _d		50.2 ± 7.8	48.2 ± 4.7	50.2 ± 7.8

^a Values are given as average ± standard deviation ($n = 30$). F_p = firmness determined using Penefel (kg/0.5 cm²); F_d = firmness determined using Durofel (ID₁₀). Values with different letters are significantly different (based on Newman–Keuls test, $P < 0.05$).

Table 11. Effect of Maturity and Artificial Ripening on the Chemical Characteristics^a of White-Fleshed Nectarines (Cv. Vermeil)

	Newman–Keuls test		Dunnett test		SSD
	A ₀	C ₀	A ₁₈	A ₂₆	
SS	12.7 ± 0.1z	15.9 ± 0.1y	12.8 ± 0.1*	12.7 ± 0.1*	0.19
TA	0.4 ± 0.0z	0.2 ± 0.0y	0.4 ± 0.0**	0.4 ± 0.0**	0.01
SS/TA	35.8 ± 0.3z	70.3 ± 1.3y	29.8 ± 0.2*	31.7 ± 0.1*	1.73

^a Values are given as average ± standard deviation ($n = 3$). SS = soluble solid (% Brix); TA = titratable acidity (g/100 g, reported as malic acid). Values with different letters are significantly different (based on Newman–Keuls test, $P < 0.05$). An asterisk (*) indicates significant decrease and two asterisks (**) indicate significant increase compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

representative of the endogenous levels of the fruit. With decreasing concentrations of (*E*)-2-hexenal, lactones become the prominent volatiles in nectarines. This study shows that the levels of lactones, except γ -heptalactone and γ -5-hydroxyhexalactone (isomer 1), increased significantly with increased maturity. This is consistent with results previously observed during maturation of peaches or nectarines (4, 8, 12, 15, 17). Levels of the C₁₃ norisoprenoid compounds, 3-hydroxy-7,8-dihydro- β -ionol and 3-hydroxy- β -ionone, increased with increased maturity, whereas those of 3-hydroxy-5,6-epoxy-7,8-dihydro- β -ionone and 3-hydroxy-5,6-epoxy- β -ionone were found to be very similar for the different degrees of maturity. The occurrence of free and glycosidically bound C₁₃ norisoprenoid compounds has been previously described in white-fleshed nectarines (8, 27, 31, 32), and their importance, in particular, that of β -ionone and β -damascenone, for the specific flavor of these cultivars has been clearly demonstrated by Takeoka et al.

Table 12. Effect of Maturity and Artificial Ripening on the Sugar and Organic Acid Content^a of White-Fleshed Nectarines (Cv. Vermeil)

	Newman–Keuls test				SSD
	A ₀	C ₀	A ₁₈	A ₂₆	
sucrose	8.28 ± 0.04z	11.64 ± 0.01y	8.21 ± 0.07*	7.82 ± 0.09*	0.16
fructose	0.74 ± 0.02z	0.61 ± 0.03y	0.77 ± 0.01**	0.86 ± 0.01**	0.04
glucose	0.69 ± 0.03	0.66 ± 0.03	0.58 ± 0.01	0.69 ± 0.01	
citric acid	0.16 ± 0.01z	0.02 ± 0.00y	0.20 ± 0.01**	0.22 ± 0.01**	0.14
malic acid	0.31 ± 0.00z	0.38 ± 0.00y	0.31 ± 0.00*	0.26 ± 0.00*	0.01

^a Values expressed in g/100 g are given as average ± standard deviation ($n = 3$). Values with different letters are significantly different (based on Newman–Keuls test, $P < 0.05$). An asterisk (*) indicates significant decrease and two asterisks (**) indicate significant increase compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

(10). Nevertheless, the presence of carotenoid-derived volatiles in yellow-fleshed cultivars has not been reported previously. The levels of benzaldehyde were found to be very similar for the different degrees of maturities. Contrary to previous observations in peaches and nectarines (4, 8, 11, 12, 15), the levels of linalool were found to decrease with increased maturity, but this decrease was not statistically significant. However, Visai and Vanoli (17) observed the same decrease of linalool during the last stage of maturity of nectarines (cv. Maria Laura).

Effect of Artificial Maturation. The results obtained from the analyses of variance of the chemical and chromatographic data are summarized in **Tables 6–8**. The leading trend due to artificial ripening was the significant sharp decrease of the SS/TA ratio, particularly for the samples derived from unripe nectarines, as this ratio was used as a maturity index of the fresh fruits (33, 34). In general, SS and TA contents were, respectively, lower and higher in artificially ripened than in tree-ripened nectarines. Sucrose and malic acid contents were significantly lower, whereas fructose and citric acid contents were significantly higher in artificially ripened nectarines. These results are consistent with those of Bayonove (4) and Deshpande and Salunkhe (29).

As shown in **Table 8**, the levels of (*E*)-2-hexenal decreased with artificial ripening but were significantly higher than in the tree-ripened samples, whereas the levels of (*E*)-2-hexen-1-ol were found to be very similar. The highest change in the concentrations of volatile compounds was observed for lactones, the levels of which increased significantly with artificial ripening, exceeding significantly their levels in the tree-ripened control (C₀). The largest amounts of lactones were observed for the samples derived from unripe nectarines, in particular, for the A₂₆ samples ripened at 26 °C. Thus, the levels of γ -hexalactone, γ -octalactone, 6-pentyl- α -pyrone, and γ - or δ -decalactone showed a 2–4-fold increase in the A₂₆ samples compared to C₀, contrary to the findings of Do et al. (3) and Bayonove (4). The same trend was observed for the levels of C₁₃ norisoprenoids, found to be significantly higher in the artificially ripened nectarines A₂₆, as well as the levels of linalool, eugenol, and benzaldehyde.

Sensory Evaluation. The results of the sensory evaluation of artificially ripened and tree-ripened nectarines are summarized in **Table 9**. Data are given as the average of the three years of harvest for each sample expressed in units of a structured line scale anchored at the ends by numeric values of 0 and 10. The only statistically significant differences between the artificially ripened nectarines and the tree-ripened control were observed for the descriptors “firmness” and “juicy”. Tree-ripened samples

Table 13. Changes in the Concentrations^a of Volatiles during Maturation and Artificial Ripening of White-Fleshed Nectarines (Cv. Vermeil)

RI ^b	assignment ^c	compound	Newman-Keuls test				SSD
			A ₀	C ₀	A ₁₈	A ₂₆	
1099	A	hexanal	828 ± 6z	899 ± 18y	1997 ± 53**	1064 ± 31**	87
1124	A	3-pentanol	44 ± 13	70 ± 11	116 ± 31**	170 ± 30**	61
1138	A	2-pentanol	5 ± 1z	7 ± 0y	21 ± 1**	111 ± 2**	3
1166	A	1-penten-3-ol	61 ± 2z	33 ± 1y	109 ± 8**	94 ± 3**	12
1175	A	3-penten-1-ol	158 ± 5	163 ± 4	296 ± 10**	269 ± 28**	41
1220	A	(<i>E</i>)-2-hexenal	4996 ± 20z	2996 ± 26y	8403 ± 152**	2551 ± 29**	213
1313	A	3-hexenyl acetate	110 ± 2z	119 ± 2y	106 ± 29**	139 ± 4**	39
1358	A	hexanol	36 ± 6	27 ± 1	78 ± 2**	288 ± 10**	15
1383	A	(<i>Z</i>)-3-hexen-1-ol	37 ± 0z	29 ± 1y	79 ± 2**	352 ± 4**	6
1406	A	(<i>E</i>)-2-hexen-1-ol	67 ± 0z	34 ± 6y	104 ± 14**	253 ± 1**	21
1444	A	acetic acid	42 ± 4	50 ± 5	92 ± 2**	113 ± 3**	8
1508	A	benzaldehyde	150 ± 0z	556 ± 1y	321 ± 5**	349 ± 5**	10
1539	A	linalool	17 ± 5z	29 ± 3y	18 ± 7	32 ± 12	
1555	A	2-methylpropanoic acid	30 ± 16	36 ± 10	62 ± 17	68 ± 38	
1589	A	γ-valerolactone	25 ± 1z	59 ± 0y	74 ± 2**	105 ± 1**	3
1694	A	γ-hexalactone	414 ± 8z	1591 ± 13y	3709 ± 31**	2865 ± 28**	59
1796	A	γ-heptalactone	12 ± 1z	65 ± 1y	95 ± 2**	82 ± 1**	4
1864	A	benzyl alcohol	28 ± 3z	44 ± 2y	67 ± 6**	94 ± 3**	9
1912	A	γ-octalactone	10 ± 1z	123 ± 2y	368 ± 4**	373 ± 2**	7
1964	A	δ-octalactone	21 ± 2z	187 ± 1y	497 ± 9**	278 ± 7**	15
2136	A	γ-decalactone	58 ± 1z	540 ± 5y	2777 ± 27**	2180 ± 4**	37
2175	B (18)	6-pentyl-α-pyrone	41 ± 9z	103 ± 3y	662 ± 10**	673 ± 49**	68
2194	A	δ-decalactone	19 ± 4z	518 ± 2y	2760 ± 63**	2542 ± 22**	90
2252	B (7)	7-decen-5-olide	27 ± 1z	250 ± 4y	1366 ± 23**	1404 ± 20**	41
2537	A	3-hydroxy-β-damascone	68 ± 6z	159 ± 5y	188 ± 9**	274 ± 7**	17
2567	B (27)	3-hydroxy-7,8-dihydro-β-ionone	166 ± 1z	398 ± 10y	369 ± 31	739 ± 24**	54
2681	B (27)	3-hydroxy-7,8-dihydro-β-ionol	179 ± 4z	131 ± 4y	527 ± 12**	646 ± 4**	18
2702	B (28)	3-hydroxy-β-ionone	94 ± 4z	811 ± 13y	1957 ± 6**	3749 ± 57**	79
2739	B (27)	3-hydroxy-5,6-epoxy-β-ionone	770 ± 33z	1102 ± 8y	1989 ± 21**	1912 ± 27**	48
2980	A	gaiacyl propanol	68 ± 6z	41 ± 15y	139 ± 14**	172 ± 4**	28
3175	A	vomifoliol	951 ± 11z	189 ± 3y	2193 ± 181**	1597 ± 30**	248
	A	coniferyl alcohol	99 ± 26z	119 ± 5y	297 ± 19**	318 ± 13**	32

^a Values expressed in μg/kg are given as average ± standard deviation ($n = 3$). ^b RI = linear retention index based on a series of n -hydrocarbons. ^c A, identities confirmed by comparing mass spectra and retention time with those of authentic standards; B, identities tentatively assigned by comparing mass spectra with those obtained from the literature (given in parentheses); C, tentatively identified. Values with different letters are significantly different (based on Newman-Keuls test, $P < 0.05$). An asterisk (*) indicates significant decrease and two asterisks (**) indicate significant increase compared to control C₀ (based on Dunnett test, $P < 0.05$). SSD = smallest significant difference (Dunnett test).

were found to be significantly “harder” and less “juicy” than the artificially ripened samples. This is consistent with the firmness data shown in **Tables 1** and **2**. On the contrary, the other descriptors were not statistically significantly different. It is noteworthy that the intensities of “peach odors” or “peach aroma” in the tree-ripened and artificially ripened yellow-fleshed nectarines were not significantly different.

White-Fleshed Nectarines. In 2001, an additional experiment on white-fleshed nectarines (cv. Vermeil) was performed to compare with the results obtained on yellow-fleshed nectarines (cv. Springbright). Fruits, obtained from the same local commercial orchard as previously mentioned, were picked in July 2001 at two degrees of maturity only (unripe and tree-ripe). Unripe fruits were placed in trays and stored in ripening chambers until complete ripening under the conditions used for yellow-fleshed nectarines. SS, TA, SS/TA ratio, organic acids, sugar contents and concentrations of volatile compounds were similarly determined.

As shown in **Tables 10–12**, the effects of maturity on the physical and chemical characteristics of white-fleshed nectarines (cv. Vermeil) are consistent with those previously observed for yellow-fleshed nectarines (cv. Springbright). SS, SS/TA ratio, and sucrose contents significantly increased, whereas titratable acidity and citric acid contents significantly decreased with maturation. Glucose and malic acid contents were also found to be very similar in fruits with different degrees of maturity,

but the levels of fructose significantly decreased with maturation. As previously observed for yellow-fleshed nectarines, the leading trend due to artificial ripening was the significant sharp decrease of the SS/TA ratio, and also of SS, sucrose, and malic acid contents. On the contrary, TA, fructose contents, and citric acid were found to be significantly higher than in the control (C₀).

As shown in **Table 13**, the effects of on-tree maturation on the volatile compounds identified in white-fleshed nectarines (cv. Vermeil) are consistent with those previously observed for yellow-fleshed nectarines (cv. Springbright). Although the levels of C₆ compounds decreased, most of the other volatile levels significantly increased with maturation, notably lactones and C₁₃ norisoprenoids compounds, except vomifoliol. However, contrary to what was previously observed in the yellow-fleshed nectarines, the levels of linalool were found to be higher in the tree-ripened samples.

As shown in **Table 13**, the highest changes due to artificial ripening were a significant increase in the levels of most volatiles, notably the levels of lactones, C₆ compounds, aliphatic alcohols, and C₁₃ norisoprenoids compounds. As previously observed for yellow-fleshed nectarines, the levels of C₁₃ norisoprenoids and lactones, in particular, γ-hexalactone, γ- or δ-octalactone, 6-pentyl-α-pyrone, and γ- or δ-decalactone, showed a 2–6-fold increase in artificially ripened samples compared to tree-ripened nectarines C₀. Finally, 3-hydroxy-β-

damascone, 3-hydroxy-7,8-dihydro- β -ionone, 3-hydroxy-7,8-dihydro- β -ionol, 3-hydroxy- β -ionone, 3-hydroxy-5,6-epoxy- β -ionone, and vomifolol have not yet been reported as volatiles in white-fleshed nectarines.

Conclusion. The levels of volatiles could be equal to or higher in the artificially ripened nectarines compared with the tree-ripened samples. Nevertheless, although the levels of volatiles, in particular, lactones and C₁₃ norisoprenoids, were notably higher in artificially ripened samples derived from unripe samples A₀, their levels of soluble solids, titratable acidity, organic acids, and sugars were very close to those observed in the initial unripe samples. In particular, the SS/TA ratio, which has been suggested as an index of the maturity of the fresh fruits (33, 34), was always lower in the artificially ripened samples than in the control. Generally, there is a high consumer acceptance of nectarines with high soluble solids content and SS/TA ratio. Thus, this could suggest that artificially ripened nectarines could be of a lower quality than tree-ripened samples. Nevertheless, no significant difference, excepted for the descriptors "firmness" and "juicy", was observed in the sensory analysis between the artificially ripened and tree-ripened nectarines. Thus, it can be concluded that artificial ripening, under controlled conditions, is an alternative technique adapted with industrial practices to provide a fruit quality similar to tree-ripened.

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