JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Characterization of Odor-Active Volatiles in Apples: Influence of Cultivars and Maturity Stage

EMIRA MEHINAGIC,*,† GAËLLE ROYER,† RONAN SYMONEAUX,† FRÉDÉRIQUE JOURJON,† AND CAROLE PROST§

Laboratoire GRAPPE, Ecole Supérieure d'Agriculture (ESA), 55 rue Rabelais, B.P. 30748, 49007 Angers Cedex 01, France, and Laboratoire de Biochimie Alimentaire, Ecole Nationale des Ingénieurs des Techniques Agro-Alimentaires (ENITIAA), Rue de la Géraudière, B.P. 82225, 44322 Nantes Cedex 3, France

The aroma and texture of three different apple cultivars, harvested at three maturity stages, were analyzed by sensory and instrumental analysis. The emphasis was on the identification of the most potent odorant volatiles, and the challenge was to separate the few most important flavor compounds, which may be trace chemicals, from the vast number of nonodorant compounds present in apple aroma extracts. Thirty-six odorant compounds were detected, 24 of which were common to all extracts. A significant correlation coefficient was found between the aroma intensity scores and overall quantity of the odorant volatiles, which shows that the development of sensory aroma is similar to that of odorant volatiles. This study also showed that the parameters measured by penetrometry and compression were highly correlated with sensory textural attributes. The determination of the optimal maturity stage for different apple cultivars by the usual parameters, such as color, diameter, total soluble solids, and titrable acidity, may not be sufficient to determine the optimal sensory quality for consumers. Moreover, the sensory quality of fruits changes during maturation in a different way from one cultivar to another, and this should be taken into account.

KEYWORDS: Apple; quality; sensory analysis; aroma; olfactometry; texture

INTRODUCTION

Texture and flavor are the main expected quality characteristics of apples that have been mentioned by consumers in different studies (*1*−*3*). Fruit wholesalers are therefore particularly interested in the measurement and control of fruit texture and flavor development. It is particularly important that this development is verified during fruit maturation because the maturity stage will determine the quality of fruits during storage (*4*, *5*). In general, apple fruit maturation is a period defined by physiological and structural changes. These include fruit softening, climacteric cellular respiration, starch hydrolysis, increases in sugars, chlorophyll degradation, membrane changes, specific protein synthesis (*6*), and aroma volatile synthesis (*7*). The chemical changes associated with these physiological and structural changes are well documented and will not be reviewed in this discussion (*8*−*11*). Very few authors have tried to correlate the development of apple eating quality to different instrumental measurements during maturation on the tree.

The aim of this study is to try to relate the development of the overall apple eating quality (texture, aroma, taste) during maturation to different physical and chemical changes that occur in fruits.

We also aimed to identify the most important contributors to apple aroma in different apple cultivars. In fact, the volatile compounds produced by apples have been studied for over 50 years, and over 200 volatile compounds have been found in different apple cultivars (*12*). Most of the recently published papers deal with volatile compound composition due to different rates of maturity or different storage conditions (*13*−*15*). Apple aroma perception is a result of a complex mixture of these volatiles, which include esters, aldehydes, ketones, alcohols, etc. Several studies were realized on the identification of the most potent odorant volatiles by GC-O (*14*−*18*). Thus, butyl acetate, 2-methylbutyl acetate, hexyl acetate, and hexyl hexanoate have been identified as being responsible for the overall apple aroma in several cultivars (*14*, *16*). Compounds known to possess green apple-like odors are hexanal and *trans*-2-hexenal (*17*, *19*), and these compounds are formed after disruption of the cells during processing or chewing. Other compounds, such as butan-1-ol, which possesses a sweet aroma, or ethyl butanoate and ethyl 2-methylbutanoate, which are responsible for a fruity, estery aroma, contribute to apple aroma characteristics as well as to aroma intensity (*18*).The challenge is to separate the few most important flavor compounds, which may be trace chemicals, from the vast number of inactive compounds. To date, no work has been published on the comparison of odorant volatile composition in different cultivars, harvested at different maturity

* Corresponding author [telephone 0033(0)241235555; fax 0033(0)-241235562; e-mail e.mehinagic@groupe-esa.com].
† Ecole Supérieure d'Agriculture.
§ Ecole Nationale des Ingénieurs des Techniques Agro-Alimentaires.

10.1021/jf052288n CCC: $33.50
Published on Web 03/04/2006

Table 1. Principal Characteristics of Three Apple Cultivars Harvested at Three Commercial Maturity Stages

	quality parameter			
	visual aspect (color)	diameter (mm)	soluble solids (°Brix)	titrable acidity (malic acid in g L^{-1})
Golden Delicious harvested				
before maturity stage (Aug 26, 2003)[a]	yellow/green	70/80	12.6	4.76
at maturity (Sept 11, 2003)	yellow/green	70/80	14	4.85
after maturity stage (Oct 7, 2003)	yellow	70/80	15.9	4.55
Fuji harvested				
before maturity stage (Sept 25, 2003)	red/green	70/75	10.8	6.60
at maturity (Oct 15, 2003)	red/yellow	70/75	11.1	5.93
after maturity stage (Nov 5, 2003)	red/yellow	70/75	12.2	5.93
Braeburn harvested				
before maturity stage (Sept 17, 2003)	red/green	75/80	12	3.60
at maturity (Oct 3, 2003)	red/green	75/80	13.2	3.29
after maturity stage (Oct 27, 2003)	red/yellow	75/80	13.6	2.82

[a] Harvest date.

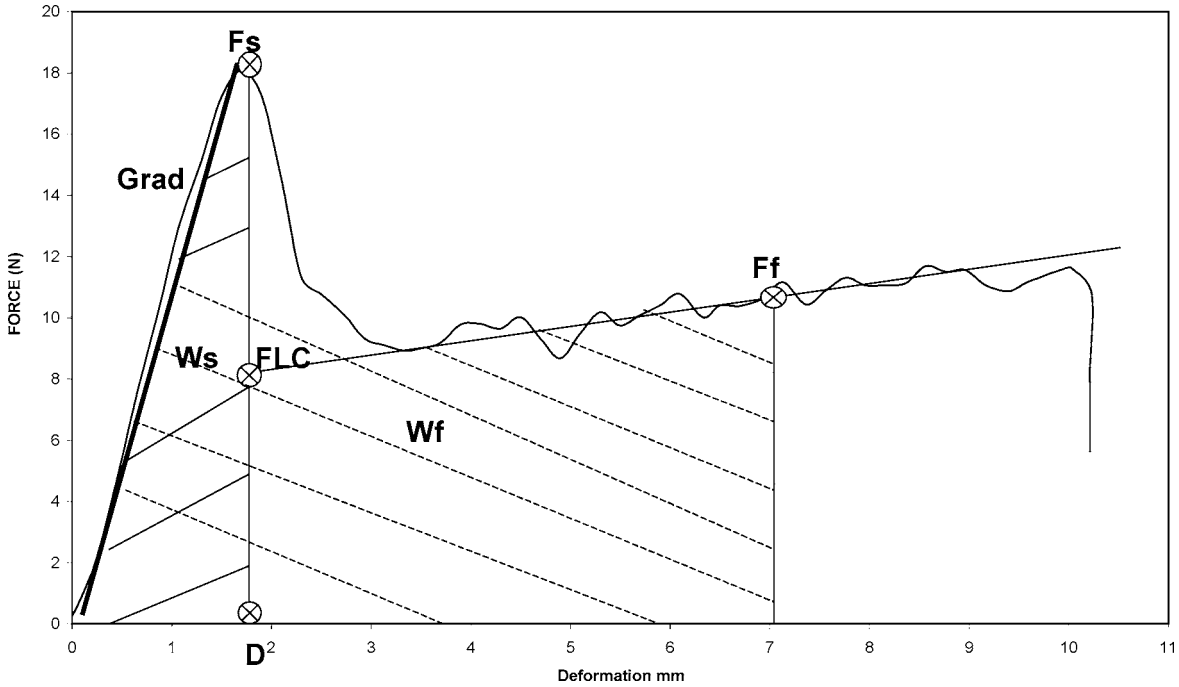

Figure 1. Force/deformation curve obtained during a penetration test on unpeeled apple, using the MTS (Synergie 200H) traction machine (cylindrical probe with a 4-mm-diameter convex tip, penetration speed of 50 mm min^{-1}, and depth of 10 mm).

stages, or established the relationship with the sensory perception of fruit quality.

Three different apple cultivars (Golden Delicious, Fuji, and Braeburn) were harvested at three maturity stages: 3 weeks before commercial maturity, commercial maturity, and 3 weeks after commercial maturity. After aroma analysis, the emphasis was on the identification of the most potent odorant volatiles and the influence of harvest date on these compounds. The sensory profiles were established for different fruits to determine the impact of harvest period on apple sensory quality. The mechanical properties of these fruits were then analyzed by penetrometry and double compression.

MATERIALS AND METHODS

Materials. *Chemicals.* Water was purified by a Milli-Q system (Millipore Corp., Molsheim, France). Dichloromethane, purchased from Sigma-Aldrich Chemical Co. (Saint-Quentin-Fallavier, France), was purified by distillation. Anhydrous sodium sulfate (99% analytical grade) was obtained from Panreac Quimica SA (Barcelona, Spain). Dichloromethane (GC quality), β-ionone, and all of the standard compounds were purchased from Sigma-Aldrich Chemical Co.

Fruits. Two fruit batches were used in this study.

The first batch, composed of commercialized fruits (Golden Delicious, Fuji, and Braeburn) bought in December 2002, was used to characterize by GC-O the odorant volatile compounds from these apple cultivars.

The second batch consisted of Golden Delicious, Fuji, and Braeburn apples harvested in 2003 at the experimental orchard La Morinière (France) at three different maturity stages, as determined by classical tests (diameter, visual aspect, soluble solids, titrable acidity) and mentioned in **Table 1**.

Immediately after picking, fruits were selected for their uniformity (color, diameter, lack of damage) and stored for 4 weeks at 3 °C. They were kept at room temperature for 24 h before analysis. Then the fruits were separated into two groups: 20 fruits were analyzed by sensory analysis, and 40 fruits were characterized by instrumental measurements (penetrometry, double compression, and aroma extraction/analysis).

Methods. *Extraction and Analysis of Aroma Volatile Compounds.* The vacuum hydrodistillation method was used to extract aroma volatile compounds from cored and sliced apples (five to six fruits) according to the method of Mehinagic et al. (*20*). Six hundred grams of cored and sliced apples was placed in a 6 L flask, and 250 mL of ultrapure water and 1 mL of β-ionone (concentration in extract of 0.5 mg mL^{-1}),

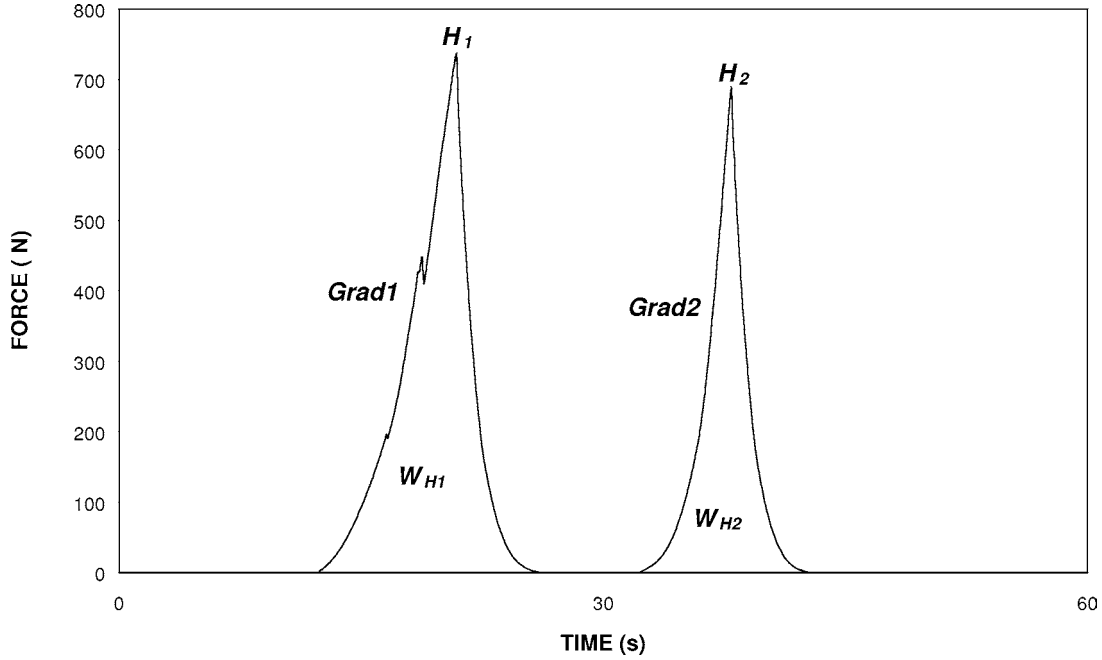

Figure 2. Force/time curve obtained by double compression on unpeeled apples, using an MTS (Synergie 200H) traction machine (two parallel plates, 50 mm min^{-1}, 7 mm).

Table 2. Sensory Descriptors Used for Apples

attribute	definition
flavor	
odor intensity	strength of the external odors in the uncut apple sample
aroma intensity	aroma released during chewing
sour taste	one of the basic tastes (e.g., malic acid)
sweet taste	one of the basic tastes (e.g., sucrose)
astringency	taste in the mouth after swallowing the sample
external touch sensations	
touch resistance	resistance of fruit to thumb pressure
roughness	degree of apple peel roughness as measured by touch
texture	
crunchiness	force required for the first bite plus the noise resulting from this bite
chewiness	time and number of chewing movements needed to grind the sample prior to swallowing
juiciness	amount of liquid released on mastication
mealiness	mealiness
fondant	force required to crush a piece of unpeeled apple between the tongue and palate

as an internal standard, were added. The temperature of the 6 L flask containing the apples was 25 °C, whereas the collector flask was at −1 °C. Three traps were cooled by liquid nitrogen at −196 °C. The residual pressure was maintained at 600 Pa for 4.5 h. The contents of the collector flask and of the three traps were pooled, and the aqueous extract was concentrated using liquid−liquid extraction by dichloromethane: volatile components were extracted with 3 × 40 mL of freshly distilled solvent. The organic extract was dried using 20 g of anhydrous sodium sulfate and then concentrated, using a Kuderna Danish apparatus, to 2 mL and under a nitrogen stream to 0.5 mL. The extract thus obtained was sealed with a Teflon cap and stored at −20 °C prior to use. The whole process was repeated twice for each apple batch (three cultivars × three maturity stages).

For the identification and confirmation of compounds, a Perkin-Elmer mass spectrometer coupled to a gas chromatograph (GC-MS) was used. Samples of 1 μL were injected, and volatile compounds were separated on a capillary column (DB-Wax, 30 m in length × 0.25 mm i.d. × 0.5 μm thickness, J&W Scientific, Folsom, CA). The helium carrier gas linear velocity was 32 cm s^{-1}. The injector (split ratio 1:20) and the detector were at 250 °C. The oven temperature was programmed from 40 °C at 5 °C min^{-1} to 60 °C (held for 30 min at 60 °C), followed by a temperature increase of 5 °C min^{-1} to 240 °C. The electronic impact ionization conditions were as follows: ionization energy, 70 eV; mass range, 33−300 amu; scan rate, 2.0 scan s^{-1}; electron multiplier voltage, 2000 V.

The volatile compounds were identified by matching their spectra to those in the NIST and Wiley NBS mass spectra library. The retention index of each volatile compound, calculated according to the method of van den Dool and Kratz (*21*), was compared with those in the literature. Chemical standards of some volatile compounds were directly injected into the GC-MS.

To quantify the volatile compounds from the extracts, samples of 1 μL were analyzed by gas chromatograph (Star 3400, Varian, Les Ulis, France) equipped with a flame ionization detector. The volatile compounds were separated on a capillary column (DB-Wax, 30 m in length × 0.32 mm × 0.5 μm thickness, J&W Scientific). The helium carrier gas flow was 1 mL min^{-1}. The injector (split ratio 1:20) and the detector were at 250 °C. The oven temperature was programmed from 40 °C at 5 °C min^{-1} to 60 °C (held for 30 min), followed by a temperature increase of 5 °C min^{-1} to 240 °C. Quantitative results are expressed in quantity equivalents of β-ionone per kilogram of fresh fruits (mg equiv kg^{-1}). As three replicates were realized per extract, the quantitative data were averaged.

GC-O. GC-O analysis was performed on apple aroma extracts of Golden Delicious, Fuji, and Braeburn fruits concentrated to 0.5 mL.

GC-O Conditions. The gas chromatography−flame ionization detector−olfactometry (GC-FID-O) system comprised a Varian 3400 GC (Varian, Palo Alto, CA) fitted with a FID at 280 °C and a sniffing port supplied with humidified air at 40 °C. Two microliters from each extract was injected (splitless mode) into a capillary column (DB-Wax, 30 m in length × 0.32 mm i.d. × 0.5 μm thickness, J&W Scientific). Effluent from the end of the GC column was split 1:1 between the FID and the sniffing port. The oven temperature was programmed from 40 °C at 7 °C min^{-1} to 110 °C, followed by a temperature increase of 15 °C min^{-1} to 250 °C (held for 3 min at 250 °C). The injector temperature was maintained at 250 °C. A solution of hydrocarbons (C6−C26) was injected daily under the same conditions to calculate retention indices (RI).

The detection frequency method was applied (*18*). A panel of eight assessors from the ENITIAA, trained in aroma recognition and with experience in GC-O, was selected. The assessors were asked to assign

Table 3. Odorant Volatile Compounds Identified and Quantified in Vacuum Hydrodistillation Extracts Obtained from Golden Delicious, Fuji, and Braeburn Apples (2002)

RI[a]	no.	odor detected by olfactometry	compound	identification[b]	threshold (mL/L)	quantity (mg equiv of β-ionone kg^{-1} of fresh fruit) ($n = 8$)			detection frequency (of eight judges)		
						Golden D	Fuji	Braeburn	Golden D	Fuji	Braeburn
992	**1**	fruity (red fruits, strawberry), floral	*propyl acetate*[c]	a, b	2 (32)[d]	0.06	0.08	0.04	3	3	NS
1010	**2**	pear, apple	*2-methylpropyl acetate*	a, b	0.065 (32)	0.23	0.04	0.34	7	6	7
1034	**3**	fruity (apple, strawberry)	ethyl butanoate	a, b, c, std	0.001 (26)	0.01	0.08	0.09	6	8	7
1051	**4**	fruity, strawberry	ethyl 2-methylbutanoate	a, b, c, std	0.000006 (32)	0	0.01	0	7	8	7
1075	**5**	sweets, fruity	butyl acetate	a, b, c, std	0.066 (26)	2.67	2.61	10.61	8	7	7
1085	**6**	green	1-hexanal	a, b, c, std	0.005 (26)	0.12	0.1	0.14	8	8	6
1103	**7**	plastic	2-methylpropanol	a, b, std	5.3 (33)	0	0	0	7	3	8
1108	**8**	cabbage, garlic, plastic	not identified			0	0	0	6	7	NS[e]
1114	**9**	earthy, animal, sulfur	not identified			0	0	0	7	NS	6
1127	**10**	fruity, apple	2-methylbutyl acetate	a, b, c, std	0.011 (32)	0.48	2.51	16.34	7	6	6
1132	**11**	red fruits, strawberry	butyl propionate	a, b, c	0.025 (32)	0	0	0.01	NS	NS	7
1144	**12**	fresh, apple, green	(*Z*)-hexen-3-ol	a, b, c	0.070 (37)	0.62	1.56	2.05	8	8	7
1176	**13**	fruity, banana	pentyl acetate	a, b, c, std	0.05 (34)	0.07	0.11	0.43	4	3	4
1208	**14**	green	(*E*)-2-hexen-1-al	a, b, c, std	0.017 (32)	0.11	1.34	1.52	NS	3	NS
1212	**15**	grilled	not identified			0	0	0	NS	3	3
1225	**16**	mint, alcohol	3-methylbutanol	a, b, std		0.25	0.31	0.49	NS	3	NS
1231	**17**	rotten fruits	butyl butanoate	a, b, c, std	0.1 (32)	0.04	0.21	0.66	6	5	8
1234	**18**	fruity, green apple	ethyl hexanoate	a, b, c, std	0.001 (32)	0.06	0	0.01	5	NS	NS
1240	**19**	fruity, apple	2-methylbutyl butanoate	a, b, c, std	0.027 (32)	0.06	0.57	0.42	4	5	6
1285	**20**	pear, fruity	hexyl acetate	a, b, c, std	0.002 (26)	0.94	1.41	3.44	7	5	8
1303	**21**	citrus fruits	1-octanal	a, b, c	0.0007 (35)	0.01	0	0	4	3	NS
1314	**22**	mushrooms	not identified			0.02	0.05	0.03	5	5	4
1338	**23**	citrus, strawberry	6-methylhept-5-en-2-one	a, b, c	0.05 (36)	0.03	0.06	0.32	NS	NS	5
1370	**24**	fresh, green	1-hexanol	a, b, c, std	0.150 (33)	1.63	3.57	2.65	7	6	6
1386	**25**	floral, geranium	(*E*)-3-hexen-1-ol	a, b, c, std		0.01	0	0.02	6	4	3
1392	**26**	sulfur, butter, woods	not identified		0.018 (37)	0	0	0	3	3	NS
1407	**27**	green, grass	butyl hexanoate	a, b, c, std	0.7 (32)	0.04	0.13	0.27	3	NS	NS
1440	**28**	green	hexyl butanoate	a, b, c	0.25 (32)	0.07	0.32	0.34	NS	3	3
1452	**29**	butter, biscuits, grilled	not identified			0	0	0.01	3	4	5
1468	**30**	boiled potato, cooked	not identified		0.001 (35)	0.02	0.02	0.03	8	5	6
1491	**31**	boiled potato	not identified			0.01	0.02	0.03	NS	3	NS
1525	**32**	rose, floral, fresh, sweet	pentyl hexanoate	a, b, c		0.01	0.03	0.04	3	3	4
1545	**33**	camphor, pine, spicy	camphor	a, b, c		0.03	0.12	0.06	8	5	6
1565	**34**	grass, pepper	1-octanol	a, b, std	0.110 (35)	0.3	0.78	0.72	6	6	NS
1608	**35**	apple, cucumber	hexyl hexanoate	a, b, c, std		0	0	0	3	3	NS
1659	**36**	butter, mushrooms	not identified			0.01	0.02	0.03	NS	3	NS

[a] Retention index (*17*). [b] "a" means that the identification of volatile compounds was done by the NIST and Wiley spectral databases, "b" means that the identification was done by retention index found in the bibliography, "c" means identification by odor, "std" means that the identification was done by injection of standard molecules. [c] Compounds cited in *italics* were tentatively identified (no standard or odor identification was done for these compounds). [d] Cited references. [e] The compound was not significantly detected (by three or more judges) in the studied extract.

odor properties to each odorant zone. Detection of an odor at the sniffing port by fewer than three assessors was considered to be noise. The final aromagram was obtained by summation of the eight individual sniffings (*22*).

Penetrometry. A cylindrical probe with a 4-mm-diameter convex tip was used to perforate unpeeled apples in a universal testing machine (MTS, Synergie 200H) (*23*). Two perforations were made on opposite paired sides of each apple. Penetration speed was set at 50 mm min^{-1}, and the test was stopped after penetration to 10 mm. Force/deformation curves were analyzed, and seven parameters were studied (**Figure 1**): total puncture force (F_s), flesh rupture breakdown force (F_f), slope of the force−deformation curve (Grad), deformation associated with total puncture force (D), work associated with F_s (W_s), work associated with F_f (W_f), and flesh limit compression force (FLC). For each cultivar and each maturity stage, 10 apples were analyzed.

Compression. Two parallel plates were used to compress unpeeled apples cut into halves in the same universal testing machine (*23*). Apples were compressed twice (double compression) with a 7 mm deformation at 50 mm min^{-1}. Force/time curves were analyzed, and six parameters were studied (**Figure 2**): hardness associated with the first compression (H_1), hardness associated with the second compression (H_2), work associated with H_1 (W_{H_1}), work associated with H_2 (W_{H_2}), slope of the first compression (Grad1), and slope of the second compression (Grad2). For each cultivar and each maturity stage, 10 apples were analyzed.

Sensory Analysis on Fresh Fruits. The panel, composed of 15 permanent expert assessors from the ESA, has been trained to describe apple texture and flavor since 1999 according to the recommendations of Fortin and Desplancke (*24*) and AFNOR (*25*). The sensory attributes studied were *odor intensity*, *aroma intensity*, *touch resistance*, *roughness*, *sour taste*, *sweet taste*, *astringency*, *juiciness*, *crunchiness*, *mealiness*, *chewiness*, and *fondant* (*23*). These sensory attributes are

Table 4. Fifteen Principal Odorant Compounds Detected by ≥75% Assessors in Aroma Extracts of Golden Delicious, Fuji, and Braeburn Apples

compound	odor (olfactometry)	Golden Delicious	Fuji	Braeburn
2-methylpropyl acetate	pear, apple, fruity	7[a]	6	7
ethyl butanoate	fruity (apple, strawberry)	6	8	7
ethyl 2-methylbutanoate	fruity, strawberry	7	8	7
butyl acetate	fruity	8	7	7
1-hexanal	green	8	8	6
2-methylpropanol	plastic	7	3	8
2-methylbutyl acetate	fruity, sweets, apple	7	6	6
butyl propionate	red fruits, strawberry	NS[b]	NS	7
(*Z*)-hexen-3-ol	fresh, green apple, green	8	8	7
butyl butanoate	rotten fruits	6	5	8
2-methylbutyl butanoate	fruity, apple	4	5	6
hexyl acetate	sweets, pear	7	5	8
1-hexanol	fresh, green	7	6	6
camphor	camphor, pine	8	5	6
1-octanol	grass	6	6	NS

[a] Detection frequency (of eight judges). [b] The compound was not significantly detected (by three or more judges) in the studied extract.

defined and described in **Table 2**. The assessors first analyzed the global *odor intensity* as well as the *touch resistance* and the *roughness* of an uncut fruit. Then, they were asked to bite the fruit and to measure other sensory attributes. During a sensory session, each assessor analyzed both sides of one fruit of each cultivar. The washed unpeeled apple halves were randomly presented to the assessors, under red light illumination and at room temperature. A continuous nonstructured scale was used for evaluation. The left end of the scale corresponded to the lowest intensity (value 0) and the right end to the highest intensity (value 10). Each assessor rinsed his/her mouth with mineral water between sample analyses. They all analyzed three apple cultivars harvested at three maturity stages during six sessions.

Statistical Analysis. Data acquisition and statistical treatment were performed with Statgraphics Plus 5.1 software (Sigmaplus, Toulouse, France). Variance analysis was carried out independently for each of the studied variables measured by sensory analysis, penetrometry, and compression. For each analysis, a significance level of 5% was considered.

To compare the effect of maturity stage on the sensory attributes of different cultivars, Fisher's least significant difference (LSD) procedures were applied separately to each cultivar.

The principal component analysis (PCA) was realized on the averaged data (only for apples harvested in 2003) according to the cultivars. The PCA relies on the correlation matrix for all data.

RESULTS AND DISCUSSION

Identification of the Most Important Contributors to Apple Aroma in Different Apple Cultivars. Characterization of volatile compounds contributing to apple aroma extracted by vacuum hydrodistillation was done by GC-O on commercialized fruits bought in 2002: Golden Delicious, Fuji, and Braeburn. Thirty-six odorant compounds were detected, 24 of which were common to all extracts (**Table 3**). Among these 36 compounds, 25 were identified with at least three identification methods and 2 were tentatively identified with two different methods. These compounds corresponded to 16 esters, 6 alcohols, 3 aldehydes, 1 ketone, and 1 terpenoid. All of the identified compounds have already been identified in fresh or processed apples (*26*).

Eighteen of the 27 identified compounds had already been identified as odorants in apple aroma extracts by different extraction methods: dynamic headspace (*14*, *27*), liquid−liquid extraction (*28*), or distillation (*29*). To compare the aromatic profiles of different apple extracts, the detection frequency method was used for each cultivar. The highest detection frequency value was equal to 8, which means that all eight assessors detected the odorant compound at the same retention time.

Fuji. Thirty-one compounds were detected as odorant by three or more assessors in Fuji apple extracts (**Table 3**). Among these compounds, 4 were detected by all assessors [ethyl butanoate (**3**), ethyl 2-methylbutanoate (**4**), hexanal (**6**), and (*Z*)-hexen-3-ol (**12**)], 2 were detected by 7 of eight assessors [butyl acetate (**5**) and compound **8**], and 4 were detected by six assessors [2-methylpropyl acetate (**2**), 2-methylbutyl acetate (**10**), 1-hexanol (**24**), and 1-octanol (**34**)]. These 10 compounds are liable to contribute significantly to Fuji apple aroma. Some of these compounds, such as ethyl butanoate, ethyl 2-methylbutanoate, butyl acetate, 2-methylpropyl acetate, and 2-methylbutyl acetate, were defined by fruity notes (apple, pear, strawberry), whereas others, hexanal, (*Z*)-hexen-3-ol, and 1-octanol, were identified by green notes (grass, green, green apple). The high detection frequency of these compounds can be explained not only by their low odor thresholds but also by their quantity. For example, the odor threshold value for ethyl 2-methylbutanoate (**4**) is very low (6×10^{-6} mgL^{-1}, **Table 3**), and it was detected by all assessors. The odor threshold of 1-hexanol is high, but this compound is present in the greatest quantity (**Table 3**), so this can explain its high detection frequency.

Golden Delicious. In extracts obtained from Golden Delicious apples, 28 compounds were detected as odorant. Among these, 5 were detected by all assessors [butyl acetate (**5**), hexanal (**6**), (*Z*)-hexen-3-ol (**12**), compound **30**, and camphor (**33**)]. Seven compounds were detected by seven of eight assessors: 2-methylpropyl acetate (**2**), ethyl 2-methylbutanoate (**4**), 2-methylpropanol (**7**), compound **9**, 2-methylbutyl acetate (**10**), hexyl acetate (**20**), and hexanol (**24**). Five compounds were identified by six of eight assessors: ethyl butanoate (**3**), butyl butanoate (**17**), (*E*)-3-hexen-1-ol (**25**), and compound **8**. The sensory notes of these compounds were mostly pleasant, fruity, green, or floral, but they can also bring some unpleasant odors such as earthy, plastic, and boiled potatoes. Their high detection frequency is related to their low odor threshold values and their quantity, but there are some exceptions. For example, 2-methylpropanol was detected by seven of eight assessors despite its high odor threshold (5.3 mg L^{-1}, **Table 3**) and low concentration (0.001 mg equiv kg^{-1}). This illustrates the limits of the detection frequency method because the high detection frequency of a compound does not mean that the compound was detected with great intensity. It means only that it was detected by a large number of assessors.

Braeburn. Finally, in Braeburn aroma extracts, 24 compounds were detected as odorant by three or more assessors, among which 3 were detected by all assessors: 2-methylpropanol (**7**), butyl butanoate (**17**), and hexyl acetate (**20**). Six compounds were detected by seven of eight assessors: 2-methylpropyl acetate (**2**), ethyl butanoate (**3**), ethyl 2-methylbutanoate (**4**), butyl acetate (**5**), butyl propionate (**11**), and (*Z*)-hexen-3-ol (**12**). Seven compounds were detected by six of eight assessors: hexanal (**6**), compound **9**, 2-methylbutyl acetate (**10**), 2-methylbutyl butanoate (**19**), hexanol (**24**), compound **30**, and camphor (**33**). All of these compounds are identified by fruity, green, earthy, camphor, or plastic notes. In general, the relationship between detection frequencies and concentration of a compound is hard to determine because the detection of a compound depends on many parameters: the odor threshold, the nature of the compound (e.g., an undesirable odor is often perceived as more intense than a pleasant one), the sensitivity of each assessor, the saturation threshold of each assessor, etc. The method of detection frequency has the advantage of resolving the problem of "scale" used by assessors to evaluate odor intensity, but it cannot distinguish compounds at low concentration detected by all assessors from those at high concentration detected by the same number of assessors: their detection frequency will be the same.

Some odorant compounds were detected by three or more assessors only in Fuji extracts: (*E*)-2-hexen-1-al (green note), 3-methylbutanol (fresh), and compounds **31** and **36** (boiled potatoes, butter, mushrooms). Ethyl hexanoate (fruity and green apple) was detected only in Golden Delicious extracts, whereas Braeburn extracts were characterized by butyl propionate (fruity, red fruits) and 6-methylhept-5-en-2-one (citrus fruits, strawberry).

Fifteen principal odorant compounds, detected by six or more assessors, are listed in **Table 4**. These compounds were principally identified with fruity notes (apple, pear, red fruits), green notes (green, grass, green apple), and some notes of rotten fruit, plastic, and pine. Among these compounds, 1-octanol was

Table 5. Principal Odorant Compounds from Apple Aroma Extracts of Golden Delicious, Fuji, and Braeburn Apples Harvested 3 Weeks before Commercial Maturity (1), at Commercial Maturity (2), and 3 Weeks after Commercial Maturity (3)

			Golden Delicious			Fuji			Braeburn		
volatile compound	RI[a]	identification[b]	1	2	3	1	2	3	1	2	3
2-methylpropyl acetate	1012	a, b	0.07[c]	0.18	0.41	0.19	0.24	0.19	0.45	0.54	0.35
ethyl butanoate	1032	a, b, std	0.02	0.05	0.26	0.12	0.13	0.22	0.28	0.13	0.16
ethyl 2-methylbutanoate	1047	a, b, std	0	0.02	0	0.09	0.2	0.15	0.09	0.09	0.12
butyl acetate	1080	a, b, std	1.1	2.63	28.73	3.06	5.13	3.5	12.95	9.87	12.52
1-hexanal	1093	a, b, std	0.41	0.65	0.82	0.39	0.52	0.43	0.8	0.53	0.38
2-methylpropanol	1097	a, b, std	0	0	0.07	0	0.1	0.11	0.03	0.03	0.05
2-methylbutyl acetate	1126	a, b, std	0.35	0.66	4.14	12.09	16.52	8.45	34.69	25.83	11.23
butyl propionate	1138	a, b, std	0.14	0.21	0.9	0.03	0.23	0.07	0.03	0.03	0.04
(*Z*)-hexen-3-ol	1142	a, b	0.68	1.69	14.74	1.69	3.22	3.01	2.33	2.26	2.67
butyl butanoate	1220	a, b, std	0.01	0.04	0.58	0.29	0.51	0.24	0.69	0.38	0.52
2-methylbutyl butanoate	1268	a, b, std	0.99	1.61	6.75	1.49	2.97	2.58	0.06	0.04	0.03
hexyl acetate	1279	a, b, std	0	0	0.01	0.03	0.08	0.03	7.06	2.98	3.56
1-hexanol	1357	a, b, std	2.81	5.82	34.49	4.5	8.99	6.9	0.02	0	0.08
1-octanol	1566	a, b, std	0.02	0.05	0.22	0.06	0.11	0.17	0.01	0.04	0.01
total			6.60	13.61	92.12	24.03	38.95	26.05	59.49	42.75	31.72

[a] Retention index (*17*). [b] "a" means that the identification of volatile compounds was done by the NIST and Wiley spectral databases, "b" means that the identification was done by retention index found in the bibliography, and "std" means that the identification was done by injection of standard molecules. [c] Quantity (mg equiv kg^{-1}) ($n = 3$).

not detected by three or more judges in Braeburn aroma extract, whereas butyl propionate was detected only in this extract (**Table 4**).

In the next section, we will observe the development of these compounds in different apple extracts related to the fruits harvested at different stages.

Effect of Maturity Stage on Apple Aroma Compounds. Among 15 selected odorant volatile compounds, 14 were identified and quantified at different harvest dates for three apple cultivars harvested from an experimental orchard in 2003.

Table 5 shows that the overall quantity of the selected volatiles increased during maturation for Fuji and Golden Delicious apples, which is related to an increase in quantity of different substrates, such as amino acids, organic acids, alcohols, and aldehydes, used for volatile formation (*29*). The most significant increase in Golden Delicious apple extracts was observed for butyl acetate, 2-methylbutyl acetate, (*Z*)-hexen-3-ol, 2-methylbutyl butanoate, and hexanol (**Table 5**) and concerned the late harvested fruits. In Fuji apples, this increase was optimal for fruits harvested in October (at commercial maturity) and concerned the same volatile compounds (**Table 5**), the predominant compound being 2-methylbutyl acetate.

When the average quantities of different classes of volatiles, extracted from the three cultivars, are observed, it can be seen that the quantities of alcohols and butyric and hexanoic esters increased significantly between early- and late-harvested fruits (*17*), whereas the total quantity of aldehydes decreased for late-harvested fruits (*30*) (**Table 6**). This seems logical as the aldehydes serve as the substrates for the formation of alcohols and esters. Thus, aldehydes are reduced to alcohols that can be esterified then by carboxylic acids present in cells (*30*).

As for Braeburn apples, the results are very different, and a decrease in odorant volatile composition was observed. Contrary to the previous observations, some odorant compounds, such as butyl acetate, (*Z*)-hexen-3-ol, butyl butanoate, or hexyl acetate, attained the lowest concentrations at the stage characterized as commercial maturity for Braeburn apples. These results are surprising as the synthesis of volatile compounds in fruit is rather expected during maturation, in parallel with other observed chemical changes such as the increase of sugar content and the decrease of malic acid content (**Table 1**). This shows the limits of these traditional maturity indexes, which may not be sufficient to determine the optimal date of maturity for the global fruit quality. No explanation or comparable results were found in the literature.

Table 6. Average Quantity of Total Volatile Compounds Extracted by Vacuum Hydrodistillation for Three Harvest Dates

	quantity[a] (mg equiv kg^{-1})		
	before maturity	at maturity	after maturity
acetic esters (8 compounds)	24.37 a	21.95 b	25.14 a
propanoic esters (3 compounds)	0.50	0.46	0.57
butyric esters (5 compounds)	1.86 a	2.23 a	4.44 b
hexanoic esters (4 compounds)	0.83 a	1.09 a	1.93 b
alcohols (12 compounds)	10.35 a	14.44 b	28.64 c
aldehydes (3 compounds)	2.67 a	2.69 a	1.81 b
ketones (2 compounds)	0.09	0.11	0.12
terpenoid (α-farnescene)	2.65 a	3.33 b	12.33 c
total	43.32	46.30	74.98

[a] Letters (a–c) following entries indicate that the observed sensory characteristics are significantly different from one maturity stage to another (LSD test, 5%); no letters indicate there was no difference.

All of these differences had an important impact on sensory characteristics. PCA carried out on averaged data (**Figure 3**) shows that the development of these parameters is different from one cultivar to another, and the most important changes, in aromatic composition as well as in sensory perception of aroma and odor, were observed with Golden Delicious apples. Thus, the aroma intensity increased from the early-harvested to the late-harvested fruits in Golden Delicious (**Figure 4**), whereas it attained its maximum at the commercial maturity stage for Fuji apples (**Figure 5**) and decreased for Braeburn apples (**Figure 6**). A significant correlation coefficient was found between the aroma intensity scores and overall quantity of the odorant volatiles ($R = 0.62$), which shows that the development of sensory aroma is similar to that of odorant volatiles. The aroma intensity scores were the most significantly correlated to the quantity of hexanal ($R = 0.75$), butyl acetate ($R = 0.65$), and 2-methylpropyl acetate ($R = 0.67$), the compounds identified with green and fruity odorant notes. At the same time, the *odor intensity* scores of uncut Golden Delicious and Fuji apples were significantly higher at postmaturity stage. There was no

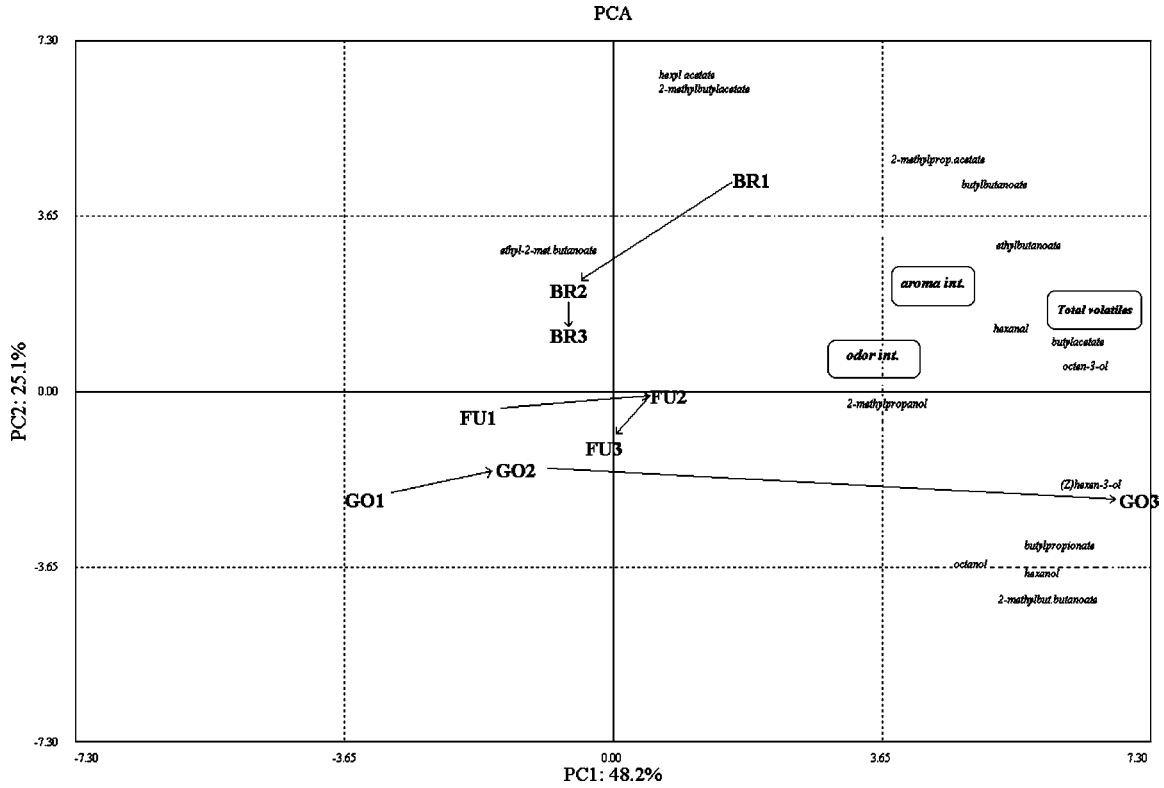

Figure 3. PCA plot of average response values obtained by sensory analysis of fresh fruits and GC analysis of their aroma extracts for three apple cultivars, Golden Delicious (GO), Fuji (FU), and Braeburn (BR), harvested at three maturity stages: 3 weeks before commercial maturity (1), commercial maturity (2), and 3 weeks after commercial maturity (3).

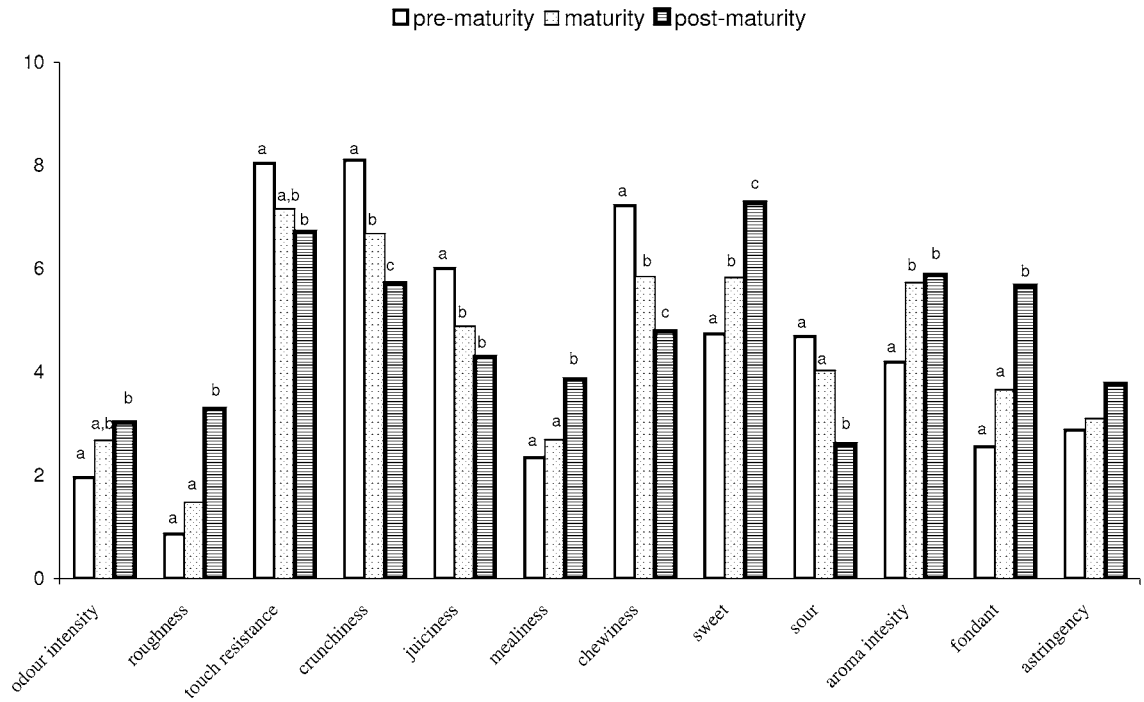

Figure 4. Plot of averaged sensory scores of Golden Delicious apples harvested at three stages: prematurity (3 weeks before commercial maturity), maturity, and postmaturity (3 weeks after commercial maturity). Letters a–c indicate that the observed sensory characteristics are significantly different from one maturity stage to another (LSD test, 5%); no letters were added if there was no difference.

significant correlation between sensory odor intensity and the overall quantity of odorant volatiles found in apple aroma extracts. This can be partially explained by the fact that the aroma extracts were obtained from cut apples, whereas the apple odor was analyzed on uncut apples. However, the odor intensity scores were significantly correlated to the quantity of ethyl butanoate ($R = 0.70$) and 2-methylpropanol ($R = 0.65$), identified successively with fruity and plastic odors.

Changes in Apple Eating Quality in Correlation with Instrumental Measurements. Variance analysis showed that, at the 5% level, the *cultivar* effect was significant for almost all sensory descriptors except *sweet taste* and *fondant* (**Table**

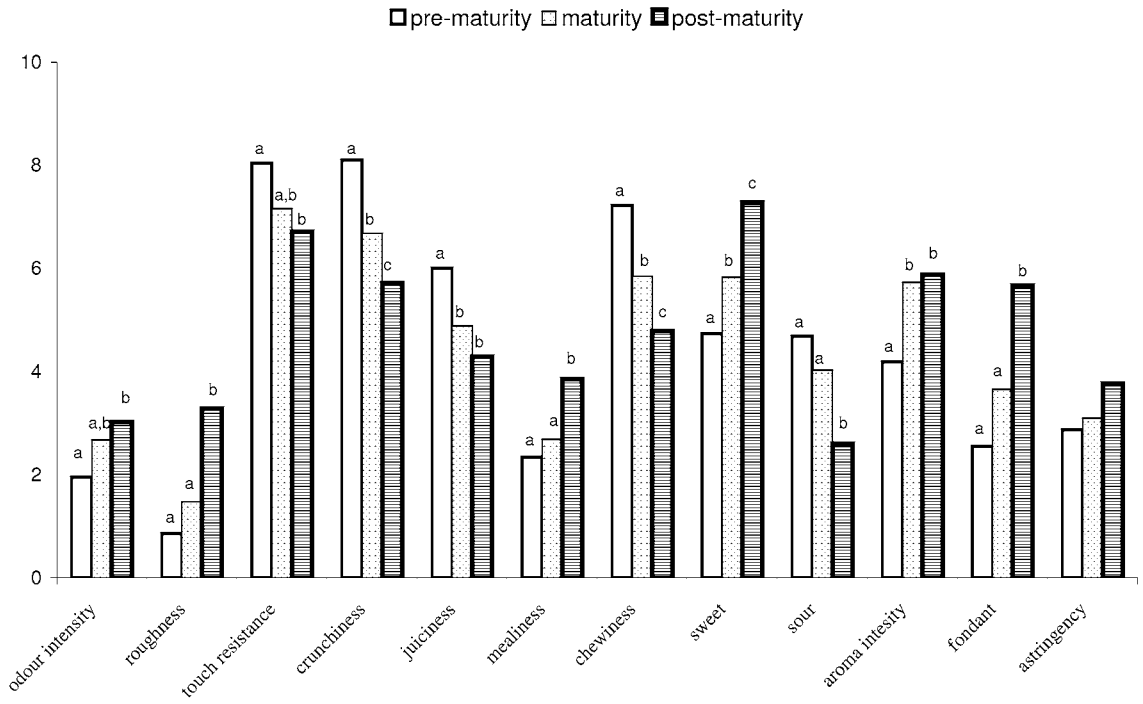

Figure 5. Plot of averaged sensory scores of Fuji apples harvested at three stages: prematurity (3 weeks before commercial maturity), maturity, and postmaturity (3 weeks after commercial maturity). Letters a–c indicate that the observed sensory characteristics are significantly different from one maturity stage to another (LSD test, 5%); no letters were added if there was no difference.

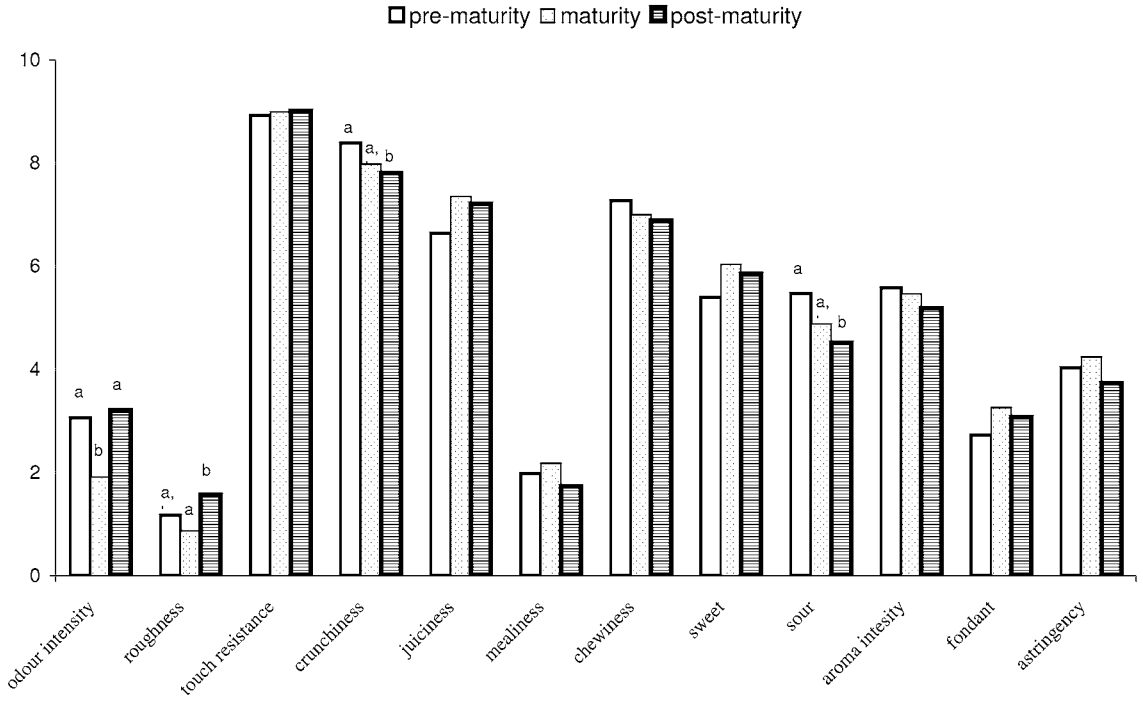

Figure 6. Plot of averaged sensory scores of Braeburn apples harvested at three stages: prematurity (3 weeks before commercial maturity), maturity, and postmaturity (3 weeks after commercial maturity). Letters a–c indicate that the observed sensory characteristics are significantly different from one maturity stage to another (LSD test, 5%); no letters were added if there was no difference.

7), which means that the studied cultivars have very different sensory profiles. In the same way, the *maturity stage* effect was significant for seven descriptors, namely, *sour taste*, *sweet taste*, *touch resistance*, *roughness*, *crunchiness*, *chewiness*, and *fondant*. Moreover, almost all of the parameters measured by penetrometry and double compression discriminated different apple cultivars as well as fruits harvested at different maturity stages (**Table 7**).

To compare the effect of maturity stage on the sensory attributes of different cultivars, Fisher's LSD procedures were applied. **Figures 4**−**6**, produced with averaged sensory scores, show that the development of these parameters is different from one cultivar to another, and the most important changes were observed with Golden Delicious apples. These differences are due not only to the structural differences that exist between the three cultivars but also to the chemical composition of their

Table 7. Two-Way ANOVA Results for Variables Measured by Sensory Analysis, Penetrometry, and Double Compression[a]

variable	cultivar		maturity stage	
	F	*p*	*F*	*p*
Sensory Analysis				
odor intensity	1.76	0.17	3.93	0.02
aroma intensity	3.41	0.034	1.54	0.22
sour taste	26.58	<0.0001	5.13	0.006
sweet taste	0.83	0.44	3.61	0.03
astringency	3.87	0.02	0.71	0.49
touch resistance	17.73	<0.0001	5.29	0.006
roughness	4.90	0.008	11.44	<0.0001
crunchiness	18.99	<0.0001	14.33	<0.0001
chewiness	5.72	0.004	6.24	0.002
juiciness	24.32	<0.0001	0.27	0.76
mealiness	12.52	<0.0001	1.34	0.26
fondant	2.26	0.11	5.25	0.006
Penetrometry				
F_s	31.81	<0.0001	56.38	<0.0001
D	9.86	<0.0001	16.43	<0.0001
Grad	177.14	<0.0001	35.13	<0.0001
W_s	2.96	0.05	28.59	<0.0001
F_f	59.50	<0.0001	57.83	<0.0001
W_f	61.39	<0.0001	74.53	<0.0001
FLC	20.77	<0.0001	22.20	<0.0001
Double Compression				
H_1	56.41	<0.0001	8.39	<0.0001
H_2	60.16	<0.0001	12.53	<0.0001
W_{H_1}	55.65	<0.0001	0.20	0.8153
Grad1	52.72	<0.0001	6.40	0.002
W_{H_2}	46.86	<0.0001	22.64	<0.0001
Grad2	26.20	<0.0001	5.10	0.007

[a] Variance analyses were assessed independently for each variable with a significance level of 5% (or $p = 0.05$).

cells and the enzymatic activity that differs from one cultivar to another (*31*). The mechanical properties, however, changed in the same way: hardness ($H_1$and H_2), as well as total puncture force (F_s), flesh rupture breakdown force (F_f), and energy required for apple flesh rupture (W_f) decreased between prematurity and postmaturity stages for all cultivars. This could explain why the sensory perception of some textural parameters, such as *touch resistance*, *crunchiness*, and *chewiness*, generally decreased during maturation. This "softening" is a normal consequence of maturation and has already been measured instrumentally on apples (*32*, *33*).

The changes in some sensory attributes during maturation are the same for all cultivars (**Figures 4**−**6**). Thus, the intensity of sensory *chewiness* and *crunchiness* decreased during maturation, as did *sourness*. Whereas the *sweet taste*, *fondant*, and *mealiness* scores increased significantly ($p < 0.05$) during the maturation period for Golden Delicious apples, there were no statistically significant differences associated with the maturity stage of Braeburn apples. Similarly, the intensity of *juiciness* decreased significantly only for this cultivar. Barreiro et al. (*34*) observed that sensory panels associate an increase in *mealiness* with a decrease in *hardness* and *juiciness*. These observations were confirmed with Golden Delicious apples. As for the Fuji apples, it seems that their *sweet taste* is highest at commercial maturity and then decreases. Sensory *crunchiness* and *sourness* decreased significantly between maturity and postmaturity stages for Braeburn apples.

To conclude, this study has shown that the sensory quality of apples is highly correlated to different physical and chemical parameters. It was demonstrated that apple aroma composition is very complex, and the 15 most interesting odorant compounds were selected. The changes in these compounds were related to changes in apple aroma intensity, whereas the development of textural parameters was related to the development of the mechanical parameters measured by penetrometry and compression.

Therefore, it seems that the determination of the optimal maturity stage for different apple cultivars by the usual parameters, such as color, calibre, total soluble solids, and titrable acidity, may not be sufficient to determine the optimal sensory quality for the consumers. Moreover, the sensory quality of fruits changes during maturation in a different way from one cultivar to another, and this should be taken into account.

ABBREVIATIONS USED

GC-MS, gas chromatography coupled to mass spectroscopy; GC-O, gas chromatography coupled to olfactometry; GC-FID, gas chromatography with flame ionization detection; ANOVA, analysis of variance; LSD, least significant difference.

ACKNOWLEDGMENT

We thank all of the staff from the Laboratory GRAPPE of ESA (Angers, France) and the Biochemistry Laboratory of ENITIAA (Nantes, France) for their help and advice. We are grateful to the Morinière farm for providing fruits and storage facilities. We epecially thank the members of the trained sensory panel (ESA) and the olfactometric panel (ENITIAA), who worked very conscientiously and never missed a sensory session.

LITERATURE CITED

(1) Stow, J. Quality measurements of apples. *Postharvest News Inf.* **1995**, *6* (*3*), 32−33.
(2) Daillant-Spinnler, D. H. B.; MacFie, H. J. H.; Beyts, P. K. Relationships between perceived sensory properties and major preference directions of 12 varieties of apples from the Southern Hemisphere. *Food Qual. Pref.* **1996**, *7* (2), 113−126.
(3) Jaeger, S. R.; Andani, Z.; Wakeling, I. N.; MacFie, H. J. H. Consumer preferences for fresh and aged apples: a cross-cultural comparison. *Food Qual. Pref.* **1998**, *9* (5), 355−366.
(4) Knee, M.; Hatfield, S. G. S.; Smith, S. M. Evaluation of various indicators of maturity for harvest of apple fruit intended for long-term storage. *J. Hortic. Sci.* **1989**, *64*, 413−419.
(5) Zerbini, E. P.; Pienezzola, A.; Grassi, M. Post-storage sensory profiles of fruit of five apple cultivars harvested at different maturity stages. *J. Food Qual.* **1999**, *22*, 1−17.
(6) Brady, C. J. Fruit ripening. *Annu. Rev. Plant Physiol.* **1987**, *38*, 155−187.
(7) Brown, D. S.; Buchanan, J. R.; Hicks, J. R. Volatiles from apple fruits as related to variety, maturity and ripeness. *Proc. Am. Hortic. Sci.* **1965**, *88*, 98−104.
(8) van Buren, J. P. The chemistry of texture in fruits and vegetables. *J. Text. Stud.* **1979**, *10*, 1−23.
(9) Yoshioka, H.; Aoba, K.; Kashimura, Y. Molecular weight and degree of methoxylation in cell wall polyuronide during softening in pear and apple fruit. *J. Am. Soc. Hortic. Sci.* **1992**, *117* (4), 600−606.
(10) Seymour, G. B.; Gross, K. C. Cell wall disassembly and fruit softening. *Postharvest News Inf.* **1996**, *7*, 45−52.
(11) Harker, F. R.; Redgwell, J. R.; Hallett, I. C.; Murray, S. H.; Carter G. Texture of fresh fruit. *Hortic. Rev.* **1997**, *20*, 121−219.
(12) Dimick, P. S.; Hoskin, J. C. Review of apple flavor—state of the art. *CRC Crit. Rev. Food Sci. Nutr.* **1983**, *18*, 387−409.
(13) Bachmann, U. Volatile compounds in apple dependent on harvesting date and after-ripening conditions. *Acta Hortic.* **1983**, *138*, 77−82.
(14) Plotto, A.; McDaniel, R. M.; Mattheis, J. P. Characterisation of Gala apple aroma and flavor: differences between controlled atmosphere and air storage. *J. Am. Soc. Hortic. Sci.* **1999**, *124*, 416−423.

(15) Lopez, M. L.; Lavilla, M. T.; Recesens, I.; Graell, J.; Vendrell, M. Changes in aroma quality of Golden Delicious apples after storage at different oxygen and carbon dioxide concentrations. *J. Sci. Food Agric*. **2000**, *80*, 311−324.
(16) Young, H.; Gilbert, J. M.; Murray, S. H.; Ball, R. D. Causal effects of aroma compounds on Royal Gala apple flavours. *J. Sci. Food Agric*. **1996**, *71*, 329−336.
(17) Flath, R. A.; Black, D. R.; Guadagni, D. G.; McFadden, W. H.; Schultz, T. H. Identification and organoleptic evaluation of compounds in 'Delicious' apple essence. *J. Agric. Food Chem*. **1967**, *15*, 29−35.
(18) Dixon, J.; Hewett, E. W. Factors affecting apple aroma/flavor volatile concentration: a review. *N. Z. J. Crop Hortic. Sci*. **2000**, *28*, 155−173.
(19) Fuhrmann, E.; Grosch, W. Character impact odorants of the apple cultivars Elstar and Cox orange. *Nahrung* **2002**, *46*, 187−193.
(20) Mehinagic, E.; Prost, C.; Demaimay, M. Representativeness of apple aroma extract obtained by vacuum hydrodistillation: comparison of two concentration techniques. *J. Food Sci*. **2003**, *68*, 2411−2415.
(21) van den Dool, H.; Kratz, P. D. A generalization of the retention index system including linear temperature programmed gas−liquid partition chromatography. *J. Chromatogr*. **1963**, *11*, 463−471.
(22) Le Guen, S.; Prost, C.; Demaimay, M. Evaluation of the representativeness of the odor of cooked mussel extracts and the relationship between sensory descriptors and potent odorants. *J. Agric. Food Chem*. **2001**, *49*, 1321−1327.
(23) Mehinagic, E.; Royer, G.; Bertrand, D.; Symoneaux, R.; Jourjon, F. Prediction of apple sensory quality by physical measurements. *Postharvest Biol. Technol.* **2004**, *34* (3), 257−269.
(24) Fortin, J.; Desplancke, C. *Guide d'Entraînement d'un jury de Dégustation*; Edisen: 1951.
(25) AFNOR V 09-021 (NF ISO 11035). In *Analyse Sensorielle—Recherche et Sélection de Descripteurs pour l'élaboration d'un Profil Sensoriel, par Approche Multidimensionnelle*, 6th ed.; AFNOR: Paris, France, 2002; pp 351−380.
(26) Nijssen, L. M.; Maarse, H.; Visscher, C. A. Fruits. In *Volatile Compounds in Food: Qualitative and Quantitative Data*, 7th ed.; Willemsens, L. C., Boelens, M. H., Eds.; TNO Nutrition and Food Research Institute: Zeist, The Netherlands, 1996.
(27) Rizzolo, A.; Polesello, A.; Teleky-Vamossy, G. Y. GC/sensory analysis of volatile compounds developed from ripening apple fruit. *J. High-Resolut. Chromatogr*. **1989**, *12*, 824−827.
(28) Cunningham, D. G.; Acree, T. E.; Barnard, J.; Butts, R. M.; Braell, P. A. Charm analysis of apple volatiles. *Food Chem*. **1986**, *19*, 137−147.
(29) Song, J.; Bangerth, F. Production and development of volatile aroma compounds of apple fruits at different times of maturity. *Acta Hortic. (ISHS)* **1994**, *368*, 150−159.
(30) DePooter, H. L.; Van Acker, M. R.; Schamp, N. M. Aldehyde metabolism and aroma quality of stored Golden Delicious apples. *Phytochemistry* **1987**, *26*, 89−92.
(31) Huber, J. D. The role of cell wall hydrolases in fruit softening. *Hortic. Rev*. **1983**, *5*, 169−219.
(32) Zerbini, E. P. Compression tests on intact Golden Delicious apples and correlation with other objective measurements. *Lebensm. Wiss. -Technol*. **1981**, *14*, 12−17.
(33) DeEll, J. R.; Saad, F.; Khanizadeh, S. Factors influencing apple fruit firmness. Presented at the 42nd Annual IDFTA Conference, Feb 20−24, 1999, Hamilton, ON, Canada.
(34) Barreiro, P.; Ortiz, C.; Ruiz-Altisent, M.; De Smedt, V.; Schotte, S.; Andani, Z.; Wakeling, I.; Beyts, P. K. Comparison between sensory and instrumental measurements for mealiness assessment in apples. A collaborative test. *J. Texture Stud*. **1998**, *29*, 509−525.
(35) Takeoke, G. R.; Flath, R. A.; Mon, T. R.; Teranishi, R.; Guentert, M. Volatile constituents of apricot (*Prunis armenica*). *J. Agric. Food Chem*. **1990**, *38*, 471−477.
(36) AIHA (American Industrial Hygiene Association). *Odor Thresholds for Chemicals with Established Occupational Health Standards*; AIHA: Washington, DC, 1989; pp 23−61.
(37) Herrmann, K. Die Aromastoffe des Obstes. Teil I: Allgemeine Angaben zu den Aromastoffen, ihren Schwellenwerten und ihrer Zusammensetzung. *Erwerbsobstaub* **1991**, *33*, 4−7.
(38) Buttery, R. G.; Turnbaugh, J. G.; Ling, L. C. Contribution of volatiles to rice aroma. *J. Agric. Food Chem.* **1988**, *36*, 1006−1009.
(39) Buttery, R. G.; Teranishi, R. A.; Flath, R. A.; Ling, L. C. Fresh tomato volatiles. In *Flavor Chemistry: Trends and Sevelopments*; ACS Symposium Series 388; Teranishi, R., Buttery, G., Shahidi, F., Eds.; American Chemical Society: Washington, DC, 1989; pp 211−222.
(40) Richard, H. Connaissance de la nature des arômes alimentaires. In *Les Arômes Alimentaires*; Richard H., Multon, J. L., Eds.; Collection Sciences et Techniques Agro-alimentaires; Tec & Doc Lavoisier: Paris, France, 1992; pp 22−37.

Received for review September 16, 2005. Revised manuscript received January 31, 2006. Accepted February 5, 2006. This research was supported by the Conseil Régional du Pays de la Loire.

JF052288N