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Volatile Components of Peel and Leaf Oils of Lemon and Lime Species

MARIE-LAURE LOTA,† DOMINIQUE DE ROCCA SERRA,† FÉLIX TOMI,† CAMILLE JACQUEMOND,‡ AND JOSEPH CASANOVA*,†

Université de Corse, Equipe Chimie et Biomasse, UMR CNRS 6134, Route des Sanguinaires, 20000 Ajaccio, France, and Station de Recherches Agronomiques Institut National de la Recherche Agronomique, Centre International Recherche Agronomique Dévelopement, 20230 San Ghjulianu, France

Peel and leaf oils of 43 taxa of lemons and limes were obtained from fruits and leaves collected from trees submitted to the same pedoclimatic and cultural conditions. Their chemical composition was investigated by capillary GC, GC-MS, and ^{13}C NMR, and the results were submitted to principal component analysis to check for chemical variability. Three major chemotypes were distinguished for lemon peel oils: limonene; limonene/β-pinene/γ-terpinene; and limonene/linalyl acetate/linalool. Two chemotypes were identified for lemon leaf oils: limonene/β-pinene/geranial/neral and linalool/linalyl acetate/α-terpineol. In lime peel oils, four chemotypes were distinguished: limonene; limonene/γ-terpinene; limonene/β-pinene/γ-terpinene; and limonene/γ-terpinene/β-pinene/oxygenated products. Four others were identified for lime leaf oils: β-pinene/limonene; limonene/geranial/neral; limonene/linalool/citronellal; and limonene/sabinene/citronellal/linalool. These results were interpreted using principal component analysis.

KEYWORDS: **Citrus; Rutaceae; lemon; lime; peel oil; leaf oil; GC; GC-MS; ^{13}C NMR; principal component analysis**

INTRODUCTION

Lemons and limes are cultivated in many countries all over the world. Lemons grow in regions with temperate summers and mild winters, particularly in Mediterranean countries, southern California, and Argentina, whereas limes grow in hot subtropical or tropical regions such as southern Florida, India, Mexico, Egypt, and the West Indies. According to the classification by Swingle (*1*), lemons and limes belong to two species: *Citrus limon* (L.) Burm. and *Citrus aurantifolia* (Christm.) Swing (family Rutaceae). Conversely, in the Tanaka system (*2*), they are divided into several species characterized by botanical variability.

Although juice is the main commercial product of lemons and limes, essential oils are exploited and the subject of active trade in food industry. They are widely used as aroma flavor enhancers for soft and alcoholic beverages and food. In pharmaceutical industries they are used as flavoring agents to mask unpleasant tastes of drugs. In perfumery, they form the base of many compositions. They have a higher market value per pound than orange, grapefruit, or tangerine oils. Commercial oils are obtained by cold pressing (peel) or distillation (leaves).

The composition of lemon peel oil has been the subject of numerous studies reviewed by Lawrence (*3*−*5*), although most of these studies concerned commercial oils for which neither the species nor the varieties were specified. A few other studies were carried out on essential oils obtained from *C. limon* species, without specification of the varieties, and others concerned peel oils of *C. limon* of specified varieties (*6*, *7*). Similarly, the composition of lemon leaf oil has been studied many times without specification of the varieties, although some studies concerned specified cultivars.

Lime peel oils have also been widely studied and reviewed by Lawrence (*8*−*13*). In some studies, neither the species nor the variety was specified. In several paper, only acid limes were investigated (*C. aurantifolia* Swing. and *Citrus latifolia* Tan.), although the species and/or varieties were not always specified. Leaf oils have been much less studied. Most of the studies concerned acid limes; only one study concerned sweet limes (*14*).

The main objective of our study was to obtain more information about the chemical variability of lemon and lime oils with respect to the taxonomy. We studied the composition of essential oils of 9 species of lemons and 7 species of limes, represented by 22 cultivars of lemons and 21 cultivars of lime trees, cultivated in the same pedoclimatic and cultural conditions. We compared the chemical composition of both peel oils and leaf oils, analyzed by GC and/or GC-MS and/or ^{13}C NMR. This methodology developed in our laboratory is well-suited for chemical polymorphism studies of *Citrus* essential oils (*15*).

EXPERIMENTAL PROCEDURES

Plant Materials. Clonal propagated trees, grafted on Troyer citrange rootstock, were 12 years old and grown in the same pedoclimatic and cultural conditions in the germplasm collection orchard of the Station

* Corresponding author (telephone + 33-4-95-52-41-21; fax + 33-4-95-52-41-42; e-mail casanova@vignola.univ-corse.fr).
† Université de Corse.
‡ Institut National de la Recherche Agronomique.

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Table 1. Chemical Composition of Lemon Peel Oils[a]

constituent	BP-20	BP-1	pc	rou	mey	voa	spJ	moï	eur	fino	lim	ich	ber	sT	Lis	cor	lap	Men	pan	bar	Bor
α-thujene*	1021	922			0.3	0.1	0.3	**1.7**	0.2	0.2	0.3	0.5	0.3	0.3	0.4	0.3	0.3	0.3	0.4	tr	
α-pinene*	1021	930	0.4	0.2	**1.0**	0.8	**0.8**	**0.5**	**1.6**	**1.6**	1.3	**1.4**	**1.7**	1.7	**1.4**	1.6	2.0	2.2	**2.3**	0.2	0.1
camphene	1066	944			tr	tr	tr	tr	tr		tr	tr	0.1	tr	0.1	tr	0.1	0.1	0.1		
β-pinene	1109	971	0.1	0.1	**0.7**	0.5	**2.9**	**4.9**	**11.7**	**11.2**	12.9	**4.5**	**13.6**	12.3	**14.2**	11.6	15.4	15.8	**15.5**	0.1	0.1
sabinene	1119	964	0.1	**2.0**	0.2	6.3	**0.5**	**0.9**	**2.0**	**1.7**	2.1	**0.9**	**1.9**	1.8	**2.2**	1.7	2.2	2.0	**1.9**	0.1	tr
3-carene	1145	1005					tr						tr			tr		tr	tr		
myrcene	1157	978	1.9	**1.7**	**1.7**	1.8	**1.4**	**1.5**	**1.5**	**1.2**	1.5	**1.6**	**1.2**	1.1	**1.4**	1.1	1.1	1.1	1.1	**1.1**	**0.7**
α-phellandrene	1162	997	tr	tr	tr	tr	tr	tr	tr		tr	**0.6**	tr		tr				tr	tr	tr
α-terpinene	1179	1009			0.2	0.1	0.1	0.3	0.1		0.2	0.4			0.2				0.1	tr	tr
limonene	1199	1021	95.8	**91.4**	**81.8**	81.3	**71.2**	**71.2**	**70.5**	**65.7**	65.6	**63.8**	**63.3**	63.0	**62.6**	61.9	60.9	58.3	**48.6**	**52.5**	**38.1**
β-phellandrene	1208	1021	0.2	0.3	0.4	0.3	0.4	0.3	0.3	0.4	0.4	**4.2**	0.4	0.3	0.4	0.3	0.5	0.5	**0.6**	0.2	0.1
(Z)-β-ocimene	1228	1024	tr	0.1	tr	tr	0.3	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	0.1	0.1
γ-terpinene	1241	1048		tr	**7.3**	3.6	**9.0**	**13.2**	**6.3**	**2.3**	8.7	**18.0**	**3.0**	3.2	**11.1**	2.4	3.4	1.3	**6.8**	**1.0**	**1.4**
(*E*)-β-ocimene	1245	1035	0.4	0.4	0.1	0.1	0.4	tr	0.1	0.1	0.1	0.3	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.4	0.4
p-cymene	1267	1012			**1.4**	0.5	**3.9**	0.1	**0.9**	**5.9**	0.1	0.3	**5.4**	6.0	0.1	7.1	6.8	7.8	**2.2**	0.1	0.1
terpinolene	1278	1078		tr	0.4	0.2	0.4	**0.5**	0.3	0.1	0.4	**0.8**	0.1	0.1	**0.5**	0.1	0.1	tr	0.3	tr	0.1
octanal	1285	978				tr	tr	tr	tr	0.1	tr	tr	0.1	0.1	tr	0.1		0.1	0.1		
6-methylhept-5-en-2-one	1332	942		tr			tr			tr			tr	tr		tr	tr	tr	tr		
nonanal	1388	1082			tr	tr		tr	0.1	0.1	0.1	-	0.2	0.1	0.1	0.1	0.1	0.1	0.3	tr	tr
p-cymenene	1432	1072			0.3								tr							tr	
cis-limonene-1,2-oxide	1440	1116					tr		tr	0.3			0.1	0.5	-	0.4	0.2	0.1	tr		
trans-limonene-1,2-oxide	1451	1120				tr	tr			**0.5**			0.2	0.5	-	0.6	0.4	0.5	tr		
trans-sabinene hydrate	1458	1053		tr	tr	0.1	tr	tr	tr	0.1	0.1		tr	0.1	0.1	0.1	0.1	0.1	0.1		
trans-linalool oxide THF	1464	1072											tr			tr					
octyl acetate	1467	1191		tr								0.1							tr		
citronellal	1473	1130	0.1	0.2	0.2	0.4	tr	tr	tr	0.1	0.1		0.1	0.1	0.1	tr	tr	0.1	0.2		
decanal	1492	1183	tr					tr		tr	tr	tr			tr			tr	tr		
linalool	1539	1082	0.1	0.2	0.4	0.4	0.3	0.2	0.2	0.3	0.2	0.2	0.3	0.3	0.2	0.3	0.2	0.3	0.4	**16.0**	**25.1**
linalyl acetate	1549	1239		0.1		tr						0.1								**23.3**	31.2
cis-*p*-ment-2-en-1-ol	1555	1107																0.1			
bornyl acetate	1573	1270										tr						tr			
trans-α-bergamotene	1580	1432		0.3	0.2	0.2	**1.1**	0.3	0.3	**0.5**	0.3		**0.5**	0.4	0.3	0.6	0.3	0.6	**1.3**	0.3	0.3
β-elemene	1586	1388			0.5							tr									
(*E*)-caryophyllene	1588	1420	0.2	0.2	0.1	0.7	0.3	0.1	0.2	tr	0.2	0.1	0.1	tr	0.2	tr	0.1		0.7	0.1	0.1
terpineol-4	1595	1162			tr	tr	tr	tr	tr	tr	tr	tr	0.1	tr	tr	tr	tr	tr	tr		
trans-*p*-menth-2-en-1-ol	1618	1123												tr		0.1	tr	0.1			
citronellyl acetate*	1654	1332		0.3	tr	0.2	tr	-	tr	tr	tr	0.2	-		tr			tr	0.2		tr
(*E*)-β-farnesene*	1654	1448			0.2		0.1	tr	tr	tr	tr		tr	tr	tr	tr	tr	tr	0.2		
α-humulene	1660	1453										0.2	tr	tr	tr		tr				
neral	1674	1213	tr		tr		**0.8**	**0.4**	**0.6**	**1.0**	1.1	-	**1.1**	1.3	**0.7**	1.3	0.5	0.5	**1.5**	0.1	0.1
terpinyl acetate	1683	1332										**0.5**									
α-terpineol	1688	1172	tr	0.3	0.2	0.2	0.2	0.2	0.2	0.3	0.3		0.3	0.3	0.3	0.3	0.3	0.4	0.3	0.1	
germacrene D	1704	1480	0.1	0.1	0.2	0.1	0.2	**0.8**				0.4	-		tr						
β-bisabolene	1721	1500	-	0.5	0.3	0.3	**1.6**	**0.5**	0.4	**0.8**	0.5	0.2	**0.7**	0.6	**0.5**	0.9	0.5	0.9	**2.0**	0.5	0.4
α-bisabolene	1724	1496																	0.2		
neryl acetate	1725	1340		0.2		0.1	0.1	-	0.4	**0.6**	0.5	0.3	**0.5**	0.5	**0.5**	0.5	0.4	0.6	**3.9**	0.1	0.1
geranial	1742	1242	0.1		0.1		**1.5**	0.8	1.3	1.9	2.1	0.1	**2.0**	2.5	**1.3**	2.6	1.0	1.1	**2.9**	0.2	0.3
geranyl acetate	1748	1358	0.1	0.2	tr	tr	**0.6**		0.4	**0.8**	0.6	0.1	**0.8**	0.6	**0.8**	1.0	0.5	0.7	**3.2**	0.1	tr
citronellol	1756	1207		0.1	tr	0.1	tr						tr		tr				tr		
nerol	1790	1207		tr			0.1				tr		tr		tr				0.1	tr	tr
geraniol	1837	1232			tr		0.2			tr	tr		tr	tr	0.1	tr			0.1		
caryophyllene oxide	1974	1574								0.2			0.1	0.1		0.2	0.3	0.2	tr		
(*E*)-nerolidol	2031	1547					tr													tr	tr
spathulenol	2115	1557					tr											0.1			
thymol	2189	1266			**1.1**	tr								tr							
total			99.6	98.9	99.3	98.4	98.7	98.4	99.6	98.0	99.7	99.8	98.3	97.9	99.9	97.3	97.8	96.1	97.7	96.6	98.8

[a] pc = Poire du Commandeur; rou = Rough; mey = Meyer; voa = Voangiala; spJ = sp Jaffa; eur = Eureka; lim = Limoneira; ich = Ichang; ber = Berna; sT = Santa Teresa; Lis = Lisbon; cor = Corpaci; lap =Lapithou; Men = Menton; pan = Panaché; bar = Barum; Bor = De Borneo. Order of elution and percentages of individual components are given on a BP-20 column except for compounds with an asterisk, for which percentages are given on a BP-1 column). Boldface type indicates components were identified by ^{13}C NMR.

of Agronomic Research of INRA-CIRAD, located at San Ghjulianu, Corsica (latitude 42° 17′ N, longitude 9° 32′ E; Mediterranean climate, average rainfall and temperature, 840 mm and 15.2 °C per annum, respectively; soil derived from alluvial deposits and classified as fersiallitic, pH range 6.0−6.6). Trees were in good vigor, disease free, and without visible insect infestation. The following species and cultivars of lemons and limes, named according to the Tanaka system (2), were investigated:

Lemons, *Citrus limon* (L.) Burm.: Panaché, Lapithou, Menton, Fino, Corpaci, Santa Teresa, Berna, Eureka, Lisbon, Limoneira, Barum. *C. lumia* Risso & Poit: Poire du Commandeur, Citron de Borneo, Citron sp jaffa. *C. assamensis* Dutta & Bhatt.: Ada Jamir. *C. aurata* Risso: Pomme d'Adam. *C. jambhiri* Lush.: Rough lemon. *C. meyeri* Y. Tan.: Meyer. *C. pennivesiculata* (Lush.) Tan.: Moï. *C. pyriformis* Hassk: Poderosa Yuma. *Citrus* sp.: Voangiala, Ichang.

Limes, Citrus aurantifolia (Christm) Swing.: Nouvelle Calédonie, Antillaise, Ambilobe, Kirk, Mexicaine. *C. latifolia* Tan.: Bearss, De Perse, IAC, El Kseur, Tahiti. *C. limettioïdes* Tan.: Bizri, Douce, Brazil, India. *C. limonia* Osb.: Volkameriana, Rangpur Jaune, Rangpur Intermédiaire, Rangpur Rouge. *C. excelsa* Wester: Nestour. *C. karna* Raf.: Khatta. *Microcitrus australasica* (F.Muell.) Swing.: lime Sauvage Digitée d'Australie.

Sampling, Peel and Leaf Essential Oils. Previous results have shown no significant difference in leaf oil composition of different trees

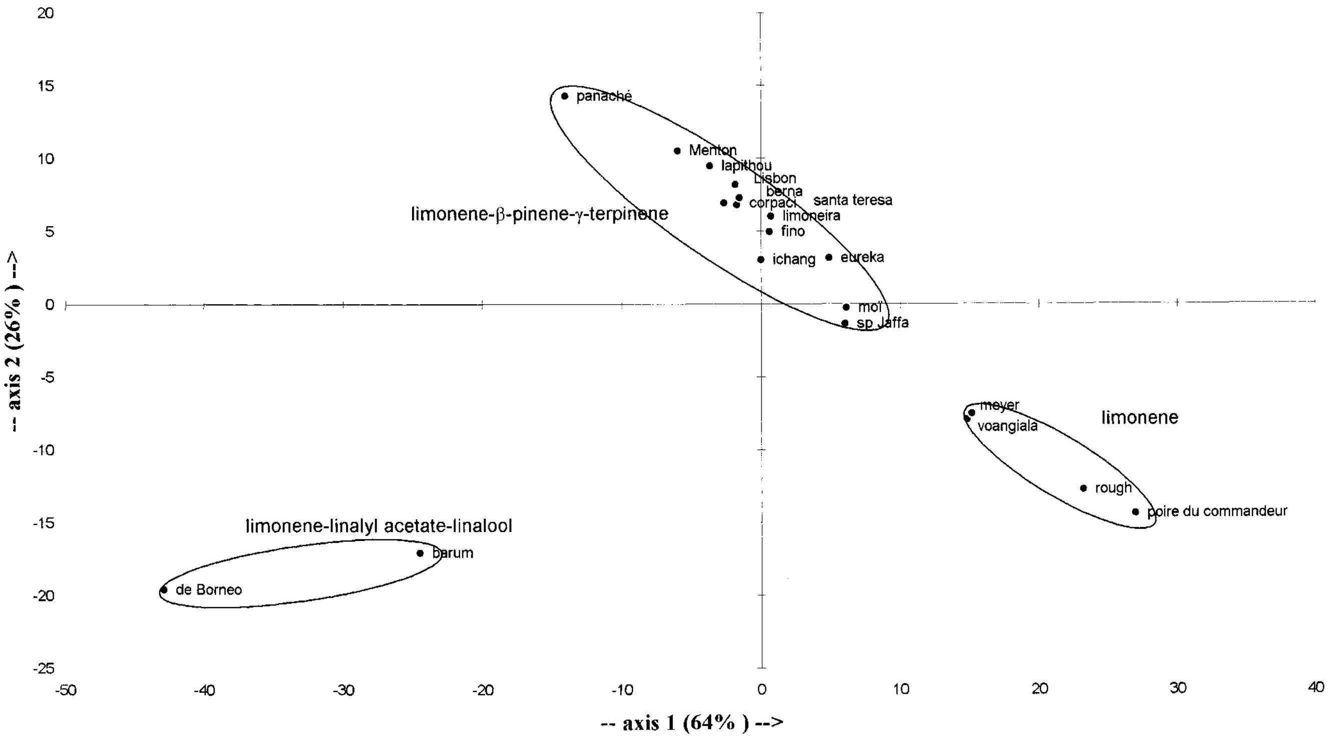

Figure 1. PCA scatterplot of lemon peel oils.

of the same cultivar of clementine. Moreover, the composition of leaf oil of the same cultivar obtained from material picked from the same tree, monthly, during the year, was not significantly influenced by the vegetative stage of the tree. In addition, essential oils from leaves of autumn shoots and from the last spring ones have shown no difference (*16*). Consequently, for each cultivar of lemon and lime, ∼500 g of leaves from the last autumn leaf flush and at least 30 ripe fruits were picked from locations on the same tree, early in the morning and in dry weather, from December 1997 to April 1998.

The peel of fresh fully ripe fruits was cold-pressed, and then the essential oil was separated from the crude extract by centrifugation (10 min at 15000 rpm). Peel oil samples were stored at 6 °C. Fresh leaves were submitted to water distillation, for 3 h, using a Clevenger-type apparatus. To avoid any damage, the storage of leaves was limited to 1 day.

GC, GC-MS, and ^{13}C NMR Analyses. GC analyses were carried out using a Perkin-Elmer Autosystem apparatus equipped with FID and fused-silica capillary columns (50 m × 0.22 mm i.d., film thickness 0.25 μm), BP-1 (dimethyl siloxane), and BP-20 [poly(ethylene glycol)]. Oven temperature was programmed from 60 to 220 °C at 2 °C/min and then held isothermal at 220 °C for 20 min; injector temperature was 250 °C; detector temperature was 250 °C; carrier gas was helium (0.8 mL/min); and split mode 1/60 was used. The relative proportions of the essential oil constituents were expressed as percentage obtained by peak area normalization, without using correcting factors. Retention indices (RI) were determined relative to the retention times of a series of *n*-alkanes with linear interpolation ("Target Compounds" software of Perkin-Elmer).

GC-MS analysis was performed on a Perkin-Elmer quadrupole MS system (model 910) coupled with the above gas chromatograph, equipped with a BP-1 capillary column and operating under the same conditions described above, except for the carrier gas flow rate (1 mL/min.). The MS operating parameters were as follows: ionization voltage, 70 eV; ion source temperature, 230 °C; scan mass range, 35−450 Da.

All NMR spectra were recorded on a Bruker AC 200 Fourier transform spectrometer operating at 50.323 MHz for ^{13}C NMR, equipped with a 10 mm (or 5 mm) probe, in deuterated chloroform, with all shifts referred to internal tetramethylsilane (TMS). ^{13}C NMR spectra were recorded with the following parameters: pulse width, 5 μs (or 3 μs) (flip angle 45°); acquisition time, 1.3 s for 32K data table with a spectral width of 12500 Hz (250 ppm); CPD mode decoupling; digital resolution, 0.763 Hz/point. The number of accumulated scans was 3000 for each sample (200 mg (or 70 mg) of the oil in 2 mL (or 0.5 mL) of $CDCl_3$. Exponential line broadening multiplication (1 Hz) of the free induction decay was applied before Fourier transformation.

Identification of Components. Identification of the individual components was based on (i) comparison of their GC retention indices (RI) on apolar and polar columns, determined relative to the retention time of a series of *n*-alkanes with linear interpolation, with those of authentic compounds, (ii) computer matching with mass spectral libraries (*17*, *18*) and comparison with spectra of authentic samples or literature data (*18*−*21*), and (iii) comparison of the carbon chemical shifts in the ^{13}C NMR spectrum of the mixture with those of the reference spectra compiled in our spectral library with the help of laboratory-produced software (*22*).

All peel and leaf oils were investigated by GC. Thirty-one peel oils and 40 leaf oils were analyzed by ^{13}C NMR, whereas 7 peel oils and 7 leaf oils were analyzed by GC-MS. Samples submitted to GC-MS and ^{13}C NMR analyses were selected on the basis of their chromatographic profile.

Data Analyses. Data analyses were performed using principal component analysis (PCA), a multivariate statistical technique widely applied in essential oil studies (*15*, *23*, *24*). This technique identifies the directions in which the most information is retained in the hyperspace of the variables. This processing was performed with Xlstat-pro software.

RESULTS AND DISCUSSION

The variation of the composition of essential oil from fruits or leaves might be attributed to two major factors: genetics and type of environment (soil, cultural practices, and weather). Here, a real comparative study of the composition of peel and leaf oils of 22 cultivars of lemons and 21 limes could be carried out because all trees were grown in the same conditions of soil, climate, and cultural practices. Extraction conditions were identical for all samples, and the influence of environmental and technical parameters on the chemical composition of essential oils was therefore considered to be negligible.

Lemon Peel Oils. The chemical compositions of the 19

Table 2. Chemical Composition of Lemon Leaf Oils[a]

constituent	BP-20	BP-1	pod	spJ	lim	cor	Lis	pan	lap	eur	ber	fino	Men	sT	pA	aj	voa	rou	moï	pc	ich	bar	Bor	mey
α-thujene*	1021	922		0.1	0.1		0.1	tr	0.1	tr	0.1	0.1	0.1	0.1	tr	0.2	0.2	0.3	tr	0.1	**1.3**	tr	tr	tr
α-pinene*	1021	930	0.6	**1.2**	**1.2**	**1.7**	**1.5**	**0.7**	**1.2**	**0.7**	**1.1**	**1.0**	**1.0**	1.2	**0.5**	**0.9**	1.1	1.2	0.3	1.7	**3.5**	tr	tr	**0.4**
camphene	1066	944	tr	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	tr	tr	tr	tr	tr	0.2	0.1			
β-pinene	1109	971	11.0	**24.8**	**22.2**	**25.1**	**23.8**	**10.5**	**16.8**	**13.9**	**18.0**	**18.2**	**15.9**	18.5	**6.3**	**0.6**	1.5	1.3	**4.5**	41.4	**14.0**	tr	0.1	0.1
sabinene	1119	964	1.7	**3.6**	**4.4**	**4.4**	**5.1**	**1.9**	**3.6**	**2.2**	**2.9**	**3.3**	**3.1**	2.7	**1.9**	**9.0**	30.3	26.0	**1.0**	4.8	**1.8**	tr	0.1	**0.8**
3-carene	1145	1005	tr	tr	0.3	0.4	0.1	**0.6**	0.5	**0.7**	0.3	0.2	0.4	0.4	tr	0.1	tr	tr	0.2	tr	tr			tr
myrcene	1157	978	0.8	**1.0**	**0.8**	**1.0**	**0.9**	**1.0**	**1.1**	**1.1**	**0.9**	**0.8**	**1.0**	0.9	**1.1**	**1.4**		2.5	**0.6**	0.6	**1.2**	**2.6**	**1.9**	**1.5**
α-phellandrene	1162	997		tr	tr	0.1	tr	tr	tr	tr	tr	tr	tr	tr		tr	0.1	tr	tr	tr	**0.7**			tr
α-terpinene	1179	1009	tr	0.1	0.2	0.2	0.2	tr	0.1	0.1	tr	tr	0.1	0.1	tr	0.4	0.7	0.8	tr	0.2	**0.8**	tr	tr	tr
limonene	1199	1021	25.0	**33.5**	**17.8**	**19.2**	**19.1**	**24.2**	**28.9**	**27.9**	**21.8**	**22.0**	21.4	23.8	**30.2**	**17.0**	25.4	31.4	**18.5**	8.6	**3.5**	**3.2**	**3.3**	**75.2**
β-phellandrene	1208	1021	0.2	**0.7**										0.8			0.5	0.4		0.7	**5.1**	tr	tr	
1.8-cineole	1208	1021			**4.1**	**2.6**	**5.2**	**0.8**	**1.6**	**0.9**	**0.8**	**1.0**	**2.1**		**1.6**	**19.2**			**0.4**					**2.6**
(*Z*)-β-ocimene	1228	1024	0.4	**1.1**	0.4	0.3	0.4	0.2	0.4	0.4	0.3	0.3	0.4	0.3	**0.7**	**0.5**	0.9	1.1	**0.4**	0.7	0.2	**1.1**	**0.9**	0.3
γ-terpinene	1241	1048	tr	0.3	0.5	0.4	**0.6**	0.2	0.4	0.3	0.3	0.3	0.4	0.3	0.2	**1.5**	1.2		0.1	0.3	**36.1**	0.4	**0.9**	0.3
(*E*)-β-ocimene	1245	1035	4.3	**1.7**	**2.0**	**1.5**	**2.2**	**1.2**	**2.1**	**2.0**	**1.6**	**1.5**	**2.0**	1.8	**1.1**	**1.9**	3.7	5.0	**1.5**	15.8	**4.0**	**2.4**	**2.5**	**2.6**
p-cymene	1267	1012	0.1	0.1	tr	tr	tr	0.1	tr		0.1	0.1	tr	tr	tr	tr	0.1	-	tr	0.1	**2.1**	tr	tr	0.1
terpinolene	1278	1078	tr	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.2	0.2	tr	0.3	0.3	0.3	0.1	0.1	**1.6**	0.5	**0.5**	tr
octanal	1285	978	tr		tr	tr	tr	0.1	tr	tr	0.1	0.1	tr	tr	0.2	0.1	tr	0.1	0.2				tr	tr
6-methylhept-5-en-2-one	1332	942	1.7	**0.6**	**1.2**	**1.8**	**1.2**	**1.0**	**1.4**	**3.2**	**0.9**	**0.7**	**1.3**	1.7	**1.3**	**1.1**	0.1	0.3	**1.6**	tr	**0.6**	0.1	0.1	0.3
allo-ocimene	1367	1116		tr			tr									tr	tr	tr		tr		tr	tr	
nonanal	1388	1082	0.1		0.2		0.2	0.4	0.2		0.3	0.3	0.1	0.2		**0.6**	tr	tr	**0.4**	tr				0.1
p-cymenene	1432	1072																						tr
cis-linalool oxide THF	1436	1058	0.1	0.1											0.7	0.1			0.1		tr	tr	0.1	
cis-limonene-1,2-oxide	1440	1116	tr	tr				0.1	tr		0.1										tr			
trans-limonene-1,2-oxide	1451	1120					tr															tr	tr	
trans-sabinene hydrate	1458	1053	0.1	tr	0.1	0.1	0.1	0.1	0.1	tr	0.1	0.1	0.1	tr	tr	0.2	0.7	0.5	tr	0.1				
trans-linalool oxide THF	1464	1072													**0.4**				tr	0.1	tr			tr
citronellal	1473	1130	6.4	**2.1**	**0.5**	**0.7**	**0.5**	**1.2**	**1.7**	**1.7**	**1.8**	**1.6**	**1.4**	1.5	**1.8**	1.8	9.5	7.3	**0.6**	2.9	**1.7**	**0.9**	0.1	**4.1**
decanal	1492	1183	0.1					0.1	tr		tr	0.1	tr	tr	**0.3**	0.2	0.1	0.1	0.2			tr	tr	
linalool	1539	1082	1.7	**5.1**	**1.6**	**1.6**	**1.9**	**1.5**	**1.4**	**1.9**	**1.6**	**1.8**	**1.8**	2.1	**8.9**	**7.2**	9.1	7.2	**30.1**	11.2	**4.6**	**38.9**	**47.3**	**3.0**
linalyl acetate	1549	1239															tr		0.1	0.3	tr	**18.5**	**16.5**	
cis-*p*-menth-2-en-1-ol	1555	1107				tr	0.1										0.2	0.2		0.1	tr			tr
bornyl acetate	1573	1270																						0.1
trans-α-bergamotene	1580	1432	tr	tr						tr				tr	tr	tr	tr	tr	tr			tr	tr	tr
β-elemene	1586	1388		tr											0.2		tr			0.8	tr			**0.6**
(*E*)-caryophyllene	1588	1420	0.2	0.2	0.3	0.1	0.3	0.4	0.2	0.2	0.2	0.1	0.1	0.1	0.2	0.1	0.2	0.4	0.1	0.3	0.2	0.1	0.1	0.2
terpineol-4	1595	1162	0.2	**0.6**	**0.7**	**0.7**	**1.0**	0.2	0.4	**0.7**	0.3	0.4	**0.5**	0.5	0.1	**1.5**	2.8	2.6	0.1	1.0	0.4	0.1	0.1	0.2
trans-*p*-menth-2-en-1-ol	1618	1123	tr														0.1	0.1		tr	tr			tr
citronellyl acetate*	1654	1332	0.2	0.2	tr	tr	tr	0.2	0.2	0.1	0.1	0.1	0.1	-	tr	tr	0.1	-	tr	1.1	0.3			0.1
(*E*)-β-farnesene*	1654	1448	tr				tr							0.1				0.1		tr	tr		tr	
α-humulene	1660	1453		tr	tr	tr		tr		tr				tr			tr	tr		0.1	tr	tr		tr
neral	1674	1213	13.6	**5.9**	**13.4**	**12.4**	**11.6**	**16.1**	**10.4**	**10.9**	**13.2**	**12.8**	**14.7**	13.6	**14.8**	**8.6**	1.6	1.9	**12.6**	0.2	**2.3**	0.7	**0.8**	**0.9**
α-terpinyl acetate	1683	1332														**1.6**								
α-terpineol	1688	1172	0.3	0.2	**1.5**	**1.0**	**1.8**	**0.5**	**0.7**	**0.7**	0.4	**0.6**	**0.9**	0.6	**0.5**	**5.3**	0.9	0.4	0.3	0.4	0.3	**11.4**	**8.9**	**0.6**
germacrene D	1704	1480		tr													tr	tr		tr	tr			tr
dodecanal	1708	1389																						tr
β-bisabolene	1721	1500		0.1				tr			tr													
α-bisabolene	1724	1496	0.2					0.1																
neryl acetate	1725	1340	1.1	**0.6**	**1.1**	**1.0**	**0.7**	**4.2**	**3.0**	**3.0**	**5.0**	**5.1**	**2.9**	1.8	0.4	0.2	0.2	0.7	0.2	0.4	**4.4**	**3.4**	**2.5**	
geranial	1742	1242	19.0	**8.6**	**18.4**	**16.8**	**15.9**	**22.6**	**14.3**	**16.4**	**19.1**	**18.1**	**20.1**	18.9	**20.4**	**12.0**	2.3	2.8	**18.0**	0.2	**3.4**	**1.0**	**1.1**	**1.3**
(*E,E*)-α-farnesene	1744	1500				0.1								0.1			tr							tr
geranyl acetate	1748	1358	4.0	**1.3**	**1.3**	**1.2**	**0.8**	**2.5**	**2.4**	**2.0**	**3.2**	**2.3**	**2.5**	2.0	**1.4**	**0.6**	0.2	0.3	**0.6**	0.4	0.4	**5.1**	**4.2**	0.1
citronellol	1756	1207	0.6	**0.6**	0.4	**0.6**	0.4	**0.5**	**0.6**	**1.0**	0.3	**0.5**	**0.6**	0.6	**0.5**	0.3	0.8	0.7	**0.7**	0.5	**1.1**	0.1	0.1	**0.9**
nerol	1790	1207	1.4	**1.4**	**1.9**	**1.8**	**1.2**	**2.7**	**2.4**	**3.4**	**2.2**	**3.3**	**2.1**	2.0	**1.0**	**1.3**	0.3	0.5	**2.6**	0.1	**2.5**	**2.4**	**2.1**	**0.7**
geraniol	1837	1232	1.5	**1.4**	**1.8**	**1.5**	**1.1**	**1.7**	**1.8**	**2.4**	**0.8**	**1.1**	**1.4**	1.6	**1.0**	**1.6**	0.2	0.4	**2.3**	0.1	**0.7**	**6.4**	**5.2**	**0.5**
caryophyllene oxide	1974	1574	0.1					0.1	0.1		tr				0.1				tr	0.2				
(*E*)-nerolidol	2031	1547	tr	0.1	tr		tr	tr	tr			tr	tr	tr		0.5	tr	tr	0.3	0.1	0.2	tr	0.1	tr
β-sinensal	2225	1673																		0.7				
α-sinensal	2323	1726																		0.2				
total			96.7	97.5	98.7	98.6	98.3	98.0	98.4	98.1	98.1	98.1	98.8	98.6	97.8	97.9	95.4	96.9	98.7	96.8	99.1	99.3	99.5	97.6

[a] pod = Poderosa Yuma; spJ = sp Jaffa; lim = Limoneira; cor = Corpaci; Lis = Lisbon; pan = Panache; lap = Lapithou; eur = Eureka; ber = Berna; Men = Menton; sT = Santa Teresa; pA = Pomme d'Adam; aj = Ada Jamir; Voa = Voangiala; rou = Rough; pc = Poire du Commandeur; ich = Ichang; bar = Barum; Bor = De Borneo; mey = Meyer. Order of elution and percentages of individual components are given on a BP-20 colum except for compounds with an asterisk, for which percentages are given on a BP-1column. Boldface type indicates components were identified by ^{13}C NMR.

essential oils from lemons (three lemon trees had no fruits: Ada Jamir, Pomme d'Adam, and Poderosa Yuma) are reported in **Table 1**. The 55 identified components accounted for 96.1−99.9% of the total amount of the oil. For most samples (17 of 19), peel oils consisted almost exclusively of hydrocarbons, the olefin fraction being much higher than the oxygenated one (84.4−99.2 vs 0.4−13.3%). Limonene was always the main constituent (38.1−95.8%) of all oils. β-Pinene (0.1−15.8%), γ-terpinene (trace−18.0%), and linalool/linalyl acetate (up to 23.3 and 31.2%, respectively) were present in appreciable amounts. Among other monoterpenes, α-pinene, sabinene, myrcene, *p*-cymene, β-phellandrene, neral, geranial, and neryl

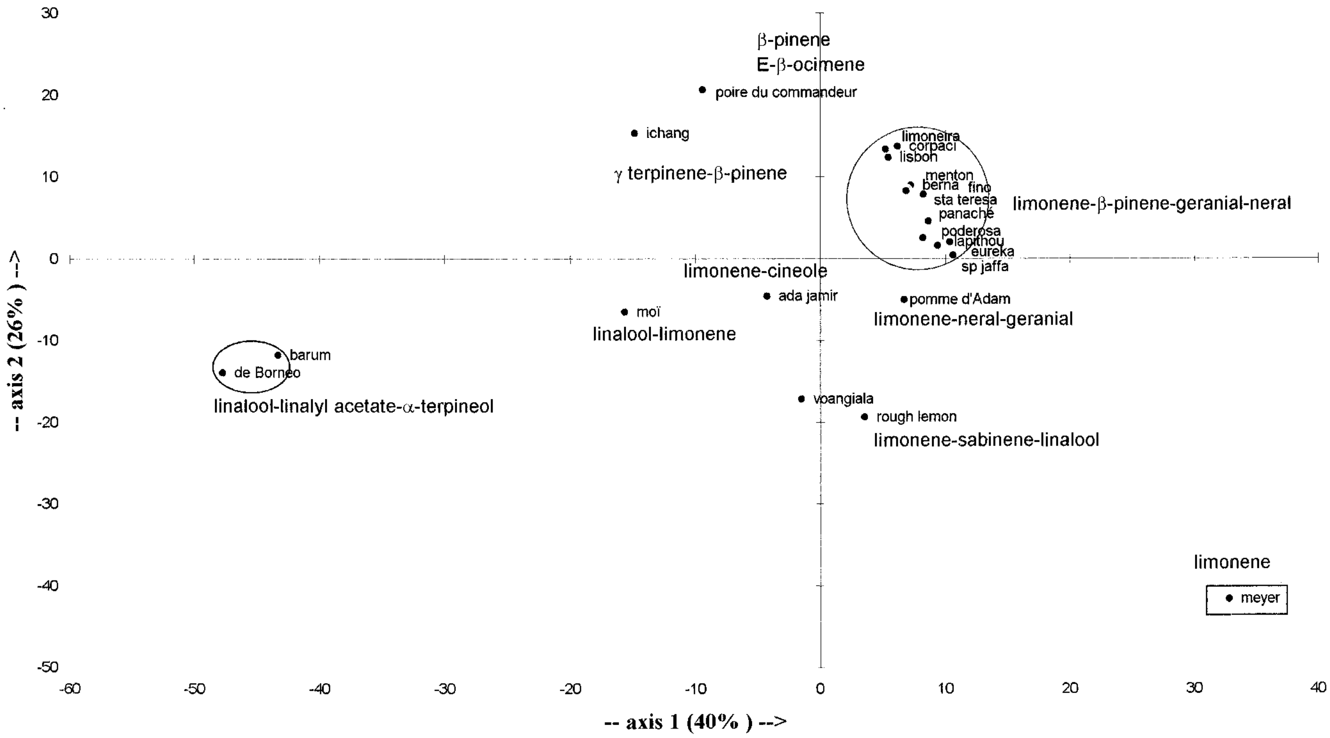

Figure 2. PCA scatterplot of lemon leaf oils.

and geranyl acetates were present in almost all samples at appreciable levels. A few olefinic sesquiterpenes were also present in low amounts, namely, *trans*-α-bergamotene, (*E*)-caryophyllene, germacrene D, and *β*-bisabolene.

PCA (**Figure 1**) suggested the existence of three principal clusters of unequal importance within the essential oils of the lemon taxa with respect to the contents of limonene, *β*-pinene, *γ*-terpinene, linalool, and linalyl acetate as follows:

Cluster 1. Four samples, which belong to four different species, constituted the first group: Poire du Commandeur (*C. lumia*), Rough Lemon (*C. jambhiri*), Meyer (*C. meyeri*), and Voangiala (*C.* sp.). They are characterized by a very high amount (81.3−95.8%) of limonene.

Cluster 2. The 13 samples of the second cluster were dominated by limonene (48.6−71.2%) and *β*-pinene (2.9−15.8%) or *γ*-terpinene (1.3−18.0%). *p*-Cymene, α-pinene, and sabinene were identified in all samples at variable concentrations (0.1−7.8%) as well as geranial, neral, and geranyl and neryl acetates at moderate concentrations. Ten samples belonged to *C. limon* species, the three other samples belonged, respectively, to *C. lumia* and to the monovarietal *C. pennivesiculata* and *C. pyriformis*.

Cluster 3. The oil composition of the two last samples, Barum (*C. limon*) and Citron de Borneo (*C. lumia*), although dominated by limonene (52.5 and 38.1%), exhibited higher contents of linalyl acetate (23.3 and 31.2% vs 0−0.1% in other samples) and linalool (16.0 and 25.1% vs 0.1−0.4%).

It is noticeable that all of the investigated varieties of *C. limon*, Barum excepted, produced oils of cluster 2. Conversely, the oils of the three varieties of *C. lumia* exhibited three different compositions.

The composition of lemon peel oils has been the subject of numerous studies. However, the comparison of our results with those reported is only possible when the species and varieties were specified. Fortunately, several studies concerned peel oils of *C. limon* of specified varieties. The varieties Favazzina, Femminello, Ferres, Giuseppe Larena, Mesaro, Monachello, Verna, Vila (*4*), Interdonato, Massese, Rifiorente, Vaniglia, and Verna, as well as Eureka and Lisbon (*4*, *25*, *26*) and Santa Teresa (*25*), which were also present in our sampling, exhibited the limonene/*β*-pinene/*γ*-terpinene composition that also belonged to our main chemotype (cluster 2). Similarly, we pointed out that all of the samples of commercial oils reported in the literature exhibited the same composition. Incidentally, the variety Meyer of Cameroon (*26*) was reported to contain 84% of limonene comparable to the 82% of our sample (cluster 1). To our knowledge, the limonene/linalool/linalyl acetate composition is reported for the first time for lemon peel oil.

In summary, 10 varieties of lemon produce an essential oil of the common limonene/*β*-pinene/*γ*-terpinene chemotype, 3 varieties have oil dominated by limonene (81−96%), and 2 varieties produce an atypical oil containing limonene, linalool, and its acetate as major components.

Lemon Leaf Oils. The compositions of the 22 oils are reported in **Table 2**. The total of 59 identified components accounted for 95.4−99.5% of the oil. The major components were limonene (3.2−75.2%) or linalool (1.4−47.3%) or *β*-pinene (up to 41.4%). *γ*-Terpinene (up to 36.1%), sabinene (up to 30.3%), geranial (up to 22.6%), 1,8-cineole (up to 19.2%), neral (up to 16.1%), linalyl acetate (up to 18.5%), (*E*)-*β*-ocimene (up to 15.8%), and α-terpineol (up to 11.4%) were present in appreciable levels. Only a few sesquiterpenes were detected in very low amounts.

Due to the diversity of the main components, PCA (**Figure 2**) suggested the existence of (i) two well-defined clusters, (ii) several oils that were related with the major cluster, and (iii) an atypical composition. Leaf oil compositions of the 12 cultivars, which belong to the main cluster, were characterized by the limonene/*β*-pinene/geranial/neral chemotype (percentages of 17.8−33.5, 10.5−25.1, 8.6−22.6, and 5.9−16.1%). All of the cultivars of *C. limon*, Barum excepted, belonged to this group as well as Citron sp Jaffa (*C. lumia*) and Ponderosa Yuma (*C. pyriformis*). Seven oils, most of them belonging to species represented by a single cultivar in our sampling, exhibited

Table 3. Chemical Composition of Lime Peel Oils[a]

constituent	BP-20	BP-1	Bra	dou	biz	Ind	vol	nes	mex	rj	ri	rr	kha	sau	amb	ant	NC	elk	kirk	IAC	bea	Per	Tah
α-thujene*	1021	922				tr	0.4	**0.5**	0.4	0.3	0.4	0.4	0.5	0.2	**0.5**	0.3	0.2	**0.6**	0.3	0.6	**0.7**	0.4	0.4
α-pinene*	1021	930	0.3	0.3	0.2	**0.6**	**1.2**	**1.4**	**1.2**	**1.7**	2.4	2.3	2.7	**1.0**	**1.4**	**2.1**	**1.1**	**2.3**	**1.7**	**1.4**	**2.4**	**1.7**	**1.3**
camphene	1066	944				tr	tr	tr	tr	0.1	0.1	0.1	0.1	tr	0.1	0.1	tr	tr	0.2	tr	0.1	tr	tr
β-pinene	1109	971	tr	tr		0.3	**0.9**	**1.6**	**6.1**	**12.2**	14.4	14.6	15.7	**7.9**	**14.7**	**19.2**	**14.7**	**12.2**	**15.3**	**11.6**	**13.7**	**12.1**	**11.1**
sabinene	1119	964	0.1	0.1	0.1	**7.1**	**3.5**	0.3	**5.4**	**1.8**	2.1	2.1	2.1	**19.6**	**2.0**	**3.1**	**2.2**	**1.8**	**1.0**	**1.8**	**2.1**	**1.7**	**1.6**
3-carene	1145	1005										tr	tr	0.3									
myrcene	1157	978	**1.6**	**1.6**	**1.6**	**2.0**	**1.8**	**1.6**	**1.5**	**1.3**	1.4	1.3	1.3	**1.5**	**1.2**	**1.2**	**1.1**	**1.3**	**0.8**	**1.3**	**1.3**	**1.1**	**1.1**
α-phellandrene	1162	997	tr	tr		tr	tr	tr	tr	tr	0.1	tr	0.1	tr	-	tr	tr	tr	0.1	tr	0.1	0.1	tr
α-terpinene	1179	1009					0.2	0.3	0.3	0.2	0.2	tr	0.2	tr	0.3	0.2	0.1	0.4	0.3	0.4	0.4	0.3	0.2
limonene	1199	1021	**94.4**	**93.4**	**93.0**	**86.1**	**77.4**	**73.5**	**66.8**	**61.7**	62.5	62.0	56.3	**51.1**	**49.9**	**48.8**	**46.4**	**46.5**	**39.9**	**45.8**	**42.7**	**40.3**	**40.4**
β-phellandrene	1208	1021	{ 0.2	{ 0.2	{ 0.2	{ 0.3	{ 0.3	{ 0.2	{ 0.3	**0.6**	{ 0.8	{ 0.5	{ 0.8	**0.6**	**0.4**	**0.5**	{ 0.5			{ 0.5			**0.5**
1.8-cineole	1208	1021																**0.6**	**0.6**		**0.6**	**0.5**	
(*Z*)-*β*-ocimene	1228	1024					0.1	tr	0.1	0.1	0.1		0.1	**0.7**	0.1	0.2	0.3		0.2			0.1	0.1
γ-terpinene	1241	1048	tr	tr		tr	**8.6**	**13.4**	**6.9**	**10.3**	8.6	4.6	9.9	**4.9**	**15.5**	**9.6**	**11.0**	**20.0**	**9.9**	**21.1**	**21.5**	**20.6**	**17.3**
(*E*)-*β*-ocimene	1245	1035	0.1	0.1	0.1	0.1	0.5	tr	0.3	0.2	0.2	0.2	0.3	0.3	0.2	0.3	0.5	0.1	0.4	0.1	0.1	0.1	0.2
p-cymene	1267	1012		tr			**0.7**	0.1	**2.3**	**1.4**	1.2	5.6	1.3	-	**1.3**	**0.6**	0.2	0.4	**5.2**	0.2	0.3	**2.0**	**5.3**
terpinolene	1278	1078				tr	0.4	**0.5**	0.4	0.5	0.4	0.2	0.4	0.2	**0.6**	0.4	0.4	**0.8**	**1.1**	**0.9**	**0.9**	**0.8**	**0.7**
octanal	1285	978				0.1	tr	0.1	tr			0.1	-	tr	-	tr	0.1	tr	tr		tr		
6-methylhept-5-en-2-one	1332	942																					tr
allo-ocimene	1367	1116												tr									
nonanal	1388	1082				0.1	0.1	0.1	tr	tr	tr		tr	tr		tr	0.1	tr	0.1		tr		
p-cymenene	1432	1072																	0.1				
cis-linalool oxide THF	1436	1058																	tr				
cis-limonene-1,2-oxide	1440	1116							tr			0.1		tr					tr			tr	tr
trans-limonene-1,2-oxide	1451	1120	tr			tr			tr	tr		0.2	tr	tr									0.1
trans-sabinene hydrate	1458	1053				0.1	0.1	tr	tr	tr	tr	0.1	tr	0.1	tr	0.1	0.1	0.1		0.1	0.1	0.1	0.1
octyl acetate	1467	1191								tr			tr										
citronellal	1473	1130	**1.1**	**1.1**	**1.6**	0.3	0.4	tr	0.1	0.1	0.1	0.5	0.1	0.2	0.3	0.1	0.1	tr	tr	0.1	0.1	0.2	0.2
decanal	1492	1183				tr		0.1		tr		0.1	0.1			0.1	**0.3**	tr	0.1	tr	0.1	tr	tr
linalool	1539	1082	0.4	**0.7**	**0.5**	0.5	0.2	0.1	0.4	0.1	0.2	0.1	0.2	0.2	0.2	0.3	0.3	0.4	**1.4**	0.4	0.4	**0.5**	0.5
linalyl acetate	1549	1239										tr						tr		tr			
cis-*p*-menth-2-en-1-ol	1555	1107																					
bornyl acetate	1573	1270	tr											tr									
trans-α-bergamotene	1580	1432	0.2	0.4	0.4	0.2	0.3	**0.8**	0.3	**0.8**	0.7	0.7	1.0	0.4	**0.7**	**0.7**	**1.0**	**1.0**	0.5	**1.3**	**1.1**	**1.4**	**1.6**
β-elemene	1586	1388				tr								0.2		0.3	**0.8**	0.2	0.2		0.2	0.2	
(*E*)-caryophyllene	1588	1420				0.1		0.5	-	0.5	0.5	0.2	0.6	0.2	0.2	0.4	0.5	**0.5**	0.4	0.4	0.3	0.4	**0.5**
thymyl methyl oxide	1589	1214					**1.6**		**1.2**														
terpinen-4-ol	1595	1162					tr	tr	**0.9**	tr	tr	tr	0.1	0.5		tr	tr	tr	**1.7**	tr	tr	0.1	0.1
undecanal	1598	1288												tr								tr	
trans-*p*-menth-2-en-1-ol	1618	1123							tr			tr							0.1				
citronellyl acetate*	1654	1332	0.1	0.2	0.1				0.1	0.2	-	0.1	0.2		0.1				0.1			0.1	
(*E*)-*β*-farnesene*	1654	1448						0.1					0.1	-	-	0.1	0.1	0.1	tr	0.1			0.1
α-humulene	1660	1453						0.2	tr			tr		0.2		0.1	0.1	tr		tr	tr		0.2
neral	1674	1213				tr		**1.7**	0.4			tr		**1.2**	**1.5**	**2.1**	**3.1**	**1.6**	**1.7**	**1.7**	**1.6**	**1.9**	**2.3**
α-terpinyl acetate	1683	1332																		tr	tr		
α-terpineol	1688	1172	tr	tr		0.1	0.2	0.2	**0.6**	0.4	0.3	0.1	0.3	0.4	0.3	0.3	0.3	0.4	**4.7**	0.4	0.5	**0.5**	**0.6**
germacrene D	1704	1480	**0.4**	0.5	0.5	0.2	0.1	0.1	0.1	**1.0**	0.6	tr	0.9	0.2	0.2	0.3	0.5	0.1	0.1	0.1	0.2	0.1	tr
dodecanal	1708	1389															tr						
β-bisabolene	1721	1500	0.4	0.5	0.6	0.3		**1.5**	0.4	**1.4**	1.2	1.2	1.6	**1.6**	**1.0**	**1.3**	**2.1**	**1.5**	**0.8**	**1.9**	**1.6**	**2.1**	**2.4**
neryl acetate	1725	1340					tr		0.5					0.1	**0.7**			**1.2**	**0.9**	**1.7**	**1.5**	**1.8**	**2.0**
bicyclogermacrene	1727	1494	tr								0.1		0.1	0.3								**0.4**	**0.4**
piperitenone	1730	1229																					tr
geranial	1742	1242						**0.5**	**0.8**					**2.0**	**2.8**	**4.0**	**6.1**	**2.8**	**2.8**	**3.0**	**2.7**	**3.4**	**4.0**
(*E*,*E*)-α-farnesene	1744	1500									0.6	0.3	1.0		0.4	**1.0**	**1.9**	0.4	**0.6**	0.3	0.3		
geranyl acetate	1748	1358	0.1	0.1	-			0.3	**0.6**	-	-	-	-	0.2	**2.1**	0.5	**0.8**	**0.7**	**1.8**	**0.9**	**0.7**	**2.2**	**2.4**
citronellol	1756	1207	tr	tr			tr		0.1	0.2	tr	tr	0.6	tr		tr			0.1				
nerol	1790	1207							tr										0.1			tr	tr
geraniol	1837	1232					tr		0.1							tr	tr	tr	0.3	tr	tr	tr	tr
caryophyllene oxide	1974	1574										0.1	-						tr			tr	0.1
globulol	2076	1580											-	0.1									
santal-10-en-2-ol	2162	1644						tr					tr			tr		tr		tr	tr		
β-sinensal	2225	1673						tr										0.1		0.1		0.1	
total			99.4	99.2	98.9	98.5	99.0	99.7	98.6	97.1	99.2	97.8	98.7	96.4	98.7	98.3	97.0	98.1	95.5	98.2	98.3	97.3	97.8

[a] Bra = Brazil; dou = Douce; biz = Bizri; Ind = India; vol = Volkameriana; nes = Nestour; mex = Mexicaine; rj = Rangpur Jaune; ri = Rangpur Intermédiaire; rr = Rangpur Rouge; kha = Khatta; sau = Sauvage; amb = Ambilobe; ant = Antillaise; NC = Nlle Caledonie; elk = El Kseur; bea = Bearss; Per = De Perse; Tah = Tahiti. Order of elution and percentages of individual components are given on a BP-20 column except for compounds with an asterisk, for which percentages are given on a BP-1column. Boldface type indicates components were identified by ^{13}C NMR.

compositions close to that of the main cluster. These may be associated with that cluster and consist of Pomme d'Adam (*C. aurata*; limonene, geranial, and neral), Ada Jamir (*C. assamensis*; 1,8-cineole, limonene, neral, and geranial), Voangiala (*C.* sp.; sabinene and limonene), Rough Lemon (*C. jambhiri*; limonene and sabinene), Moï (*C. pennivesiculata*; linalool and limonene), Poire du Commandeur (*C. lumia*; sabinene, (*E*)-*β*-ocimene), and Ichang (*C.* sp.; *γ*-terpinene and *β*-pinene).

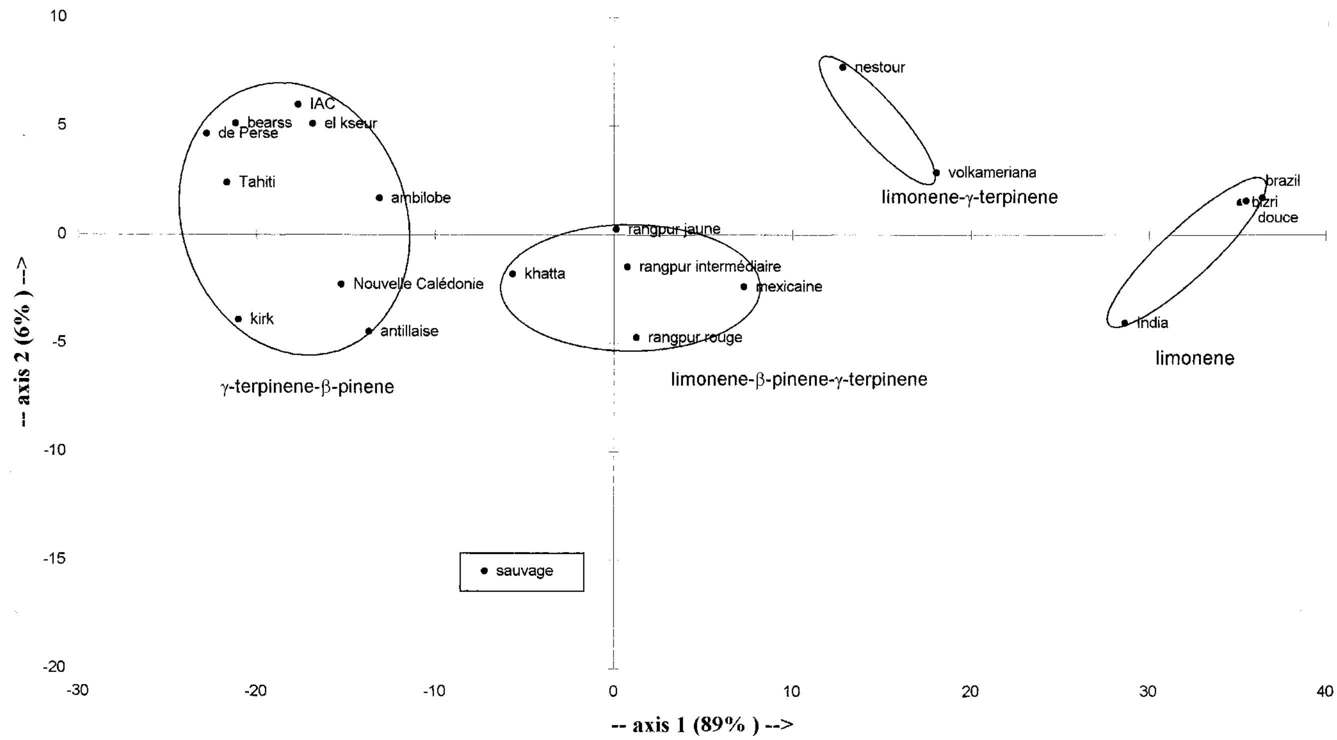

Figure 3. PCA scatterplot of lime peel oils.

In constrast to this large group, two oils obtained from Barum (*C. limon*) and Citron de Borneo (*C. lumia*) cultivars constituted the second cluster, characterized by linalool (38.9 and 47.3%), linalyl acetate (18.5 and 16.5%), and α-terpineol (11.4 and 8.9%) as major components. The percentages of geraniol and geranyl acetate were above that of other samples (4.2−6.4 vs 0.1−4.0%).

Finally, Meyer lemon was alone in the space of the variables and characterized by a high percent of limonene (75.2%) and a low content (below 5%) of all other components.

As we observed for peel oils, all of the investigated varieties of *Citrus limon*, Barum excepted, produced leaf oils belonging to the same cluster. Only oils from Meyer, Citron de Borneo, and Barum cultivars exhibited compositions significantly different from that of the main cluster.

The composition of lemon leaf oil of our main cluster, limonene/β-pinene/geranial/neral, has already been reported in the literature for most studies on unspecified varieties of *C. limon* (*4*, *27*−*29*). Some studies concerned specified cultivars of *C. limon* which belonged to the same chemotype: Eureka (*4*, *14*, *26*, *30*), Lisbon (*4*, *14*, *26*), Monachello (*4*), Verna (*4*, *14*), and Femminello, Favazzina, Giuseppe Larena, San Fernando, Fino, Villafranca, Giorgiana, and plant lemon 1 (*4*). Some *C. limon* oils exhibited compositions with slight differences (*4*, *31*). The composition of our Rough Lemon (*C. jambhiri*) oil is similar to that reported (*14*) as well as Meyer (*C. meyeri*) (*4*, *14*). Conversely, the composition of our sample of Poderosa Yuma (*C. pyriformis*) differed from that reported (sabinene as main component) (*14*).

Sixteen varieties of lemon were investigated for the first time. Fourteen of these produced an essential oil of the common limonene/β-pinene/geranial/neral chemotype or similar in composition, and two varieties produced an oil dominated by oxygenated monoterpenes.

Lime Peel Oils. The 62 identified components accounted for 95.6−99.7% of the total amount of the peel oil (**Table 3**). Limonene was the major component (39.9−94.4%), followed by γ-terpinene (up to 21.5%) and/or β-pinene (up to 19.2%) and/or sabinene (up to 19.6%). *p*-Cymene, α-pinene, myrcene, terpinolene, β-bisabolene, *trans*-α-bergamotene, and germacrene D were present in almost all samples at low levels (0.1−5.6%). Among oxygenated compounds, geranial, neral, geranyl and neryl acetates, α-terpineol, and linalool were found in most samples.

PCA (**Figure 3**) suggested the existence of four clusters and an atypical sample as follows:

Cluster 1. The peel oil composition of the first group was dominated by limonene (86.1−94.4%). All of the samples belonged to the sweet limes: *C. limettioïdes* (Brazil, Douce, Bizri, and India).

Cluster 2. The second group, represented by the two samples Volkameriana and Nestour, showed limonene (73.5 and 77.4%) and γ-terpinene (13.4 and 8.6%) as main products. All other component levels were lower than 3.5%.

Cluster 3. The third group showed limonene in lower quantity (56.3−66.8%), β-pinene (6.1−15.7%), and γ-terpinene (0.1−10.3%). In this cluster we found Mexicaine (*C. aurantifolia*), three Rangpur limes (*C. limonia*), and Khatta (*C. karna*).

Cluster 4. The fourth group, which is the most important with 9 samples of 21, is characterized by the lowest percentage of limonene (39.9−49.9%). Conversely, β-pinene (11.1−19.2%) and γ-terpinene (9.6−21.5%) were present in appreciable amounts. Geranial, neral, and geranyl acetate were present in all of the samples, at moderate contents (2.7−6.1, 1.5−3.1, and 0.5−2.2%, respectively). In this group we found all of the acid limes (except Mexicaine), belonging to *C aurantifolia* and *C. latifolia*: Ambilobe, Antillaise, Nouvelle Calédonie, El Kseur" Kirk, IAC, Bearss, De Pere, and Tahiti.

It is noticeable that the Sauvage lime, which belongs to another botanical genus (*Microcitrus australasica),* close to the *Citrus* genus, showed an original pattern. It differed from all other cultivars in the occurrence of sabinene (19.6%) as the second component, besides limonene (51.1%).

Most of the cold-pressed lime peel oils reported in the

Table 4. Chemical Composition of Lime Leaf Oils[a]

constituent	BP-20	BP-1	ant	amb	mex	kirk	NC	elk	bea	Per	Tah	IAC	nes	Bra	biz	Ind	dou	vol	rj	ri	rr	sau	kha
α-thujene*	1021	922		tr	tr	tr	0.2	tr	tr	tr	tr	tr	tr	tr		0.2	0.2	0.2	0.1	0.2	0.1	0.3	0.2
α-pinene*	1021	930	0.2	0.2	0.2	0.1	**1.1**	0.2	0.2	0.2	0.2	0.3	**0.6**	0.2	0.2	**0.5**	**0.9**	**0.8**	**1.8**	**2.3**	**2.1**	**0.8**	**2.9**
camphene	1066	944											tr			tr	tr	tr	0.2	0.2	0.2	tr	0.2
β-pinene	1109	971	0.3	0.2	0.3	**0.5**	0.2	0.3	0.2	0.2	0.2	0.3	**7.8**	tr		**1.1**	**1.1**	**1.3**	**28.2**	**31.0**	**32.2**	**0.8**	**47.2**
sabinene	1119	964	0.3	**0.6**	0.3	**0.4**	0.1	**1.1**	**0.7**	**0.9**	**0.8**	**0.8**	**2.3**	0.1	tr	**22.7**	**23.7**	**29.3**	**7.0**	**6.6**	**6.1**	**42.5**	**7.8**
3-carene	1145	1005	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr			tr	tr	tr		tr	**1.3**	tr
myrcene	1157	978	**0.9**	**0.9**	**1.0**	**0.8**	**0.6**	**0.9**	**1.0**	**1.0**	**1.1**	**1.5**	**1.1**	**1.3**	**1.1**	**1.3**	**2.0**	**2.1**	**0.9**	**1.0**	**0.9**	**1.4**	**0.7**
α-terpinene	1179	1009						tr	tr	tr	tr	tr	tr				**0.6**	0.6	0.1	0.1	0.1	0.3	0.1
limonene	1199	1021	**31.7**	**32.9**	**34.1**	**22.1**	**22.9**	**38.1**	**37.5**	**37.3**	**46.3**	**53.4**	**32.8**	**63.4**	**50.5**	**39.7**	**35.6**	**26.2**	**27.4**	**27.6**	**27.3**	**18.4**	**11.1**
β-phellandrene	1208	1021	0.3		0.2	0.1	0.1				**1.2**			0.1	0.1	0.2	0.4	0.4			**2.8**	0.3	
1.8-cineole	1208	1021		**1.0**				**1.3**	**2.1**	**2.5**		**1.3**	**1.5**						**5.1**	**3.4**			**3.7**
(*Z*)-β-ocimene	1228	1024	**0.5**	0.4	**0.7**	**0.8**	0.4	0.3	**0.2**	0.2	0.3	0.3	**0.8**	0.2	0.1	0.4	**1.0**	**0.9**	**0.9**	**0.8**	**0.8**	**1.2**	**0.9**
γ-terpinene	1241	1048	tr	0.1	0.1	0.1	tr	0.3	0.1	0.1	0.2	0.2	0.2	0.1	tr	0.2	**1.0**	**0.9**	0.5	**0.6**	**0.6**	**0.8**	**1.0**
(*E*)-β-ocimene	1245	1035	**1.6**	**1.1**	**2.5**	**2.0**	**1.1**	**1.5**	**1.5**	**1.3**	**1.9**	**2.2**	**1.3**	**4.0**	**2.8**	**1.6**	**3.9**	**3.9**	3.7	3.4	3.4	0.5	**4.6**
p-cymene	1267	1012	tr	tr	tr	tr	-	tr	tr	tr	tr	tr	tr	tr	tr	**1.2**	tr	tr	0.1	0.1	0.1	**0.7**	tr
terpinolene	1278	1078					tr	tr	tr			0.1	0.1	tr	tr	0.1	0.2	0.2	0.1	0.1	0.1	0.5	0.1
octanal	1285	978	tr	0.1		tr		tr	tr	tr	0.1	tr	0.1				tr	0.1	0.1	0.1	0.1		0.1
6-methylhept-5-en-2-one	1332	942	**1.5**	**2.5**	**1.7**	**1.6**	**1.1**	**1.1**	**2.2**	**1.4**	**1.9**	**1.3**	**1.2**					tr			tr		tr
allo-ocimene	1367	1116	tr		tr							tr	tr				tr	tr	tr		tr		tr
nonanal	1388	1082	tr	0.1				0.2		0.1	0.2	0.1	0.1		tr	0.3	0.1	0.1	0.2	0.1	0.2	tr	0.3
p-cymenene	1432	1072												0.1		0.2	tr						
cis-linalool oxide THF	1436	1058											0.5			0.3							
cis-limonene-1,2-oxide	1440	1116	0.1	0.1	tr		0.1	-		0.1		tr	tr			0.4			0.2	0.2	0.2		0.4
trans-limonene-1,2- oxide	1451	1120												tr		0.2	tr	tr				tr	
trans-sabinene hydrate	1458	1053						tr	tr	tr	tr	tr	tr			0.1	**0.5**	**0.7**	0.2	0.1	0.1	**0.5**	0.2
octyl acetate	1467	1191							tr								tr					tr	
citronellal	1473	1130	**1.3**	1.2	1.8	3.8	1.7	2.7	1.5	1.0	1.8	0.9	2.0	13.0	17.7	11.3	14.1	14.4	14.7	14.7	14.9	11.5	8.9
decanal	1492	1183	0.2	0.2	**0.4**	**0.4**	**0.6**	**0.4**	0.1	0.1	0.2	0.1	0.2				0.2	0.2	0.2	0.1	0.2		0.1
linalool	1539	1082	**1.2**	**1.5**	**1.2**	**1.2**	**1.1**	**1.4**	**1.5**	**1.4**	**1.4**	**0.9**	**8.1**	**10.6**	**21.3**	**10.1**	**7.8**	**10.5**	**1.7**	**1.3**	**1.7**	**0.6**	**2.1**
linalyl acetate	1549	1239													0.1	0.4						0.2	-
cis-*p*-menth-2-en-1-ol	1555	1107						tr				tr					0.2	0.1	0.1	0.1			0.1
trans-α-bergamotene	1580	1432		tr			0.1	tr	tr				tr				tr						
β-elemene	1586	1388	0.1	0.2	0.1	0.1			tr		0.1	0.2						0.1					
(*E*)-caryophyllene	1588	1420	0.2		0.3	0.2	0.4	0.1	0.1	0.1	0.2	0.3	0.2	0.2	0.1	0.2	0.1	0.1	0.2	0.4	0.2	0.1	0.3
terpinen-4-ol	1595	1162	-	0.1			0.1	0.1	0.2	0.1	0.2	0.2	0.2	tr	tr	**2.5**	**2.1**	**2.0**	**0.7**	0.4	**0.6**	**2.6**	**0.5**
undecanal	1598	1288				0.1		0.1			0.1	0.1											0.1
trans-*p*-menth-2-en-1-ol	1618	1123														0.2	0.1	0.1	tr				tr
citronellyl acetate	1654	1332	0.1		0.1	0.3	0.3	0.2	0.1	0.1	0.2	0.2	0.1	**1.5**	**0.8**	0.2	0.2	0.2	0.4	0.3	0.4	tr	0.1
α-humulene	1660	1453			tr	tr	0.1	tr	tr	tr	tr	tr	tr	tr			tr	tr	tr	tr	tr	0.1	tr
neral	1674	1213	**19.6**	**20.5**	**16.2**	**16.8**	**16.0**	**13.6**	**14.2**	**16.1**	**10.4**	**8.3**	**12.5**	tr	tr		0.1	0.1	0.1	0.1	0.2	0.1	**0.8**
α-terpinyl acetate	1683	1332					0.1				tr												
α-terpineol	1688	1172	0.3	**0.6**	0.2	0.2	0.1	**0.8**	**0.9**	**0.8**	**0.9**	**0.6**	**0.5**	tr	tr	0.4	0.3	0.4	**1.6**	**1.1**	**1.0**	0.1	**1.3**
germacrene D	1704	1480	tr		0.3	tr			tr	tr	tr	tr	-	tr			tr	tr	tr	-	tr		
β-bisabolene	1721	1500																		0.2		0.6	-
neryl acetate	1725	1340	**1.0**	**0.6**	**1.6**	**3.1**	**1.6**	**6.1**	**4.6**	**3.5**	**5.9**	**4.8**	**0.6**		0.1	0.3	0.2	0.4	0.2	0.1	0.1	0.2	0.1
geranial	1742	1242	**26.9**	**26.7**	**23.1**	**23.8**	**23.1**	**19.5**	**20.3**	**22.5**	**14.9**	**11.8**	**18.0**					0.2	0.2	0.2	0.3		**1.1**
(*E*,*E*)-α-farnesene	1744	1500								tr													
geranyl acetate	1748	1358	**3.5**	**2.2**	**4.3**	**8.3**	**4.6**	**4.5**	**3.2**	**2.7**	**3.3**	**4.2**	**1.7**	tr	0.1	-	0.2	0.2	0.2	0.1	0.1	0.2	0.1
citronellol	1756	1207	**0.5**	**0.4**	**0.8**	**1.1**	**0.4**	**0.6**	**0.7**	0.5	**0.5**	0.3	**0.4**	**2.8**	**2.6**	**1.3**	**1.0**	**1.5**	**1.8**	**1.7**	**1.4**	**8.4**	**1.2**
nerol	1790	1207	**2.2**	**2.1**	**2.2**	**3.1**	**1.4**	**1.1**	**2.4**	**1.9**	**1.8**	**1.4**	**1.8**	tr	tr	0.1	tr	0.1	tr	0.1	tr		0.3
geraniol	1837	1232	**2.3**	**1.9**	**2.4**	**3.8**	**1.6**	**1.3**	**2.5**	**1.7**	**1.7**	**1.6**	**1.7**	tr	tr		tr	tr	tr		tr	tr	0.2
caryophyllene oxide	1974	1574	0.2	0.1	0.2	**0.4**	0.3	0.1	tr	tr	tr	tr	0.1						tr		tr		
(*E*)-nerolidol	2031	1547					0.3	0.1	tr	tr	tr	tr	tr					tr	tr		tr		tr
globulol	2076	1580																				**1.0**	
spathulenol	2115	1557	tr														tr					**0.8**	
santal-10-en-2-ol	2162	1644	0.1				**8.5**	tr			tr	tr										tr	
thymol	2189	1266												tr	0.1			tr					
β-sinensal	2225	1673			tr				tr	-		tr	0.1		0.1								
manoyl oxide	2341	1995			**0.7**																		
total			97.1	98.5	97.0	95.2	90.2	98.0	98.0	97.8	98.0	97.7	98.6	97.6	97.8	97.7	97.8	98.3	98.9	98.8	98.5	96.8	98.8

[a] ant = Antillaise; amb = Ambilobe; mex = Mexicaine; NC = N. Calédonie; elk = El Kseur; bea = Bearss; Per = De Perse; Tah = Tahiti; nes = Nestour; Bra = Brazil; biz = Bizri; Ind = India; dou = Douce; vol = Volkameriana; rj = Rangpur Jaune; ri = Rangpur Intermédiaire; rr = Rangpur Rouge; sau = Sauvage; kha = Khatta. Order of elution and percentages of individual components are given on a BP-20 column except for compounds with an asterisk, for which percentages are given on a BP-1column. Boldface type indicates components were identified by ^{13}C NMR.

literature exhibited limonene, β-pinene, and γ-terpinene as major components whatever the species and whether the varieties were unspecified (*9*, *10*, *12*) or specified: Mexicaine (*C. aurantifolia*) (*9*, *32*), Key (*C. aurantifolia*) (*9*, *13*), De Perse (*C. latifolia*) (*9*, *13*), Tahiti (*C. latifolia*) (*13*, *32*). *Microcitrus* peel oil has been reported by Ruberto and Rocco (*33*). The composition of 17 lime peel oils is reported for the first time. Although the oils exhibited limonene, β-pinene, and γ-terpinene as major components and so are qualitatively similar, quantitative differences of these three compounds allowed a classification into four clusters of unequal importance.

Lime Leaf Oils. The compositions of the 21 oils are reported in **Table 4**. The total of 59 identified components accounted for 90.3−99.3% of the oil. We observed a higher variability

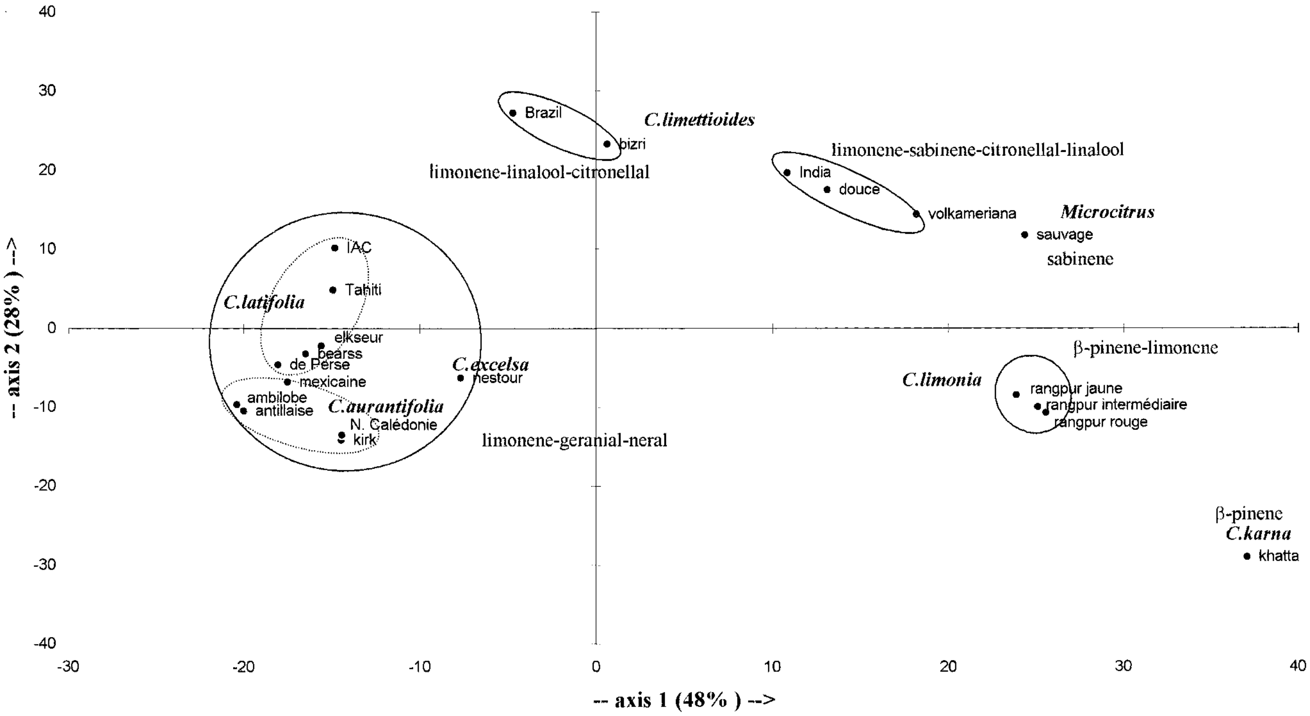

Figure 4. PCA scatterplot of lime leaf oils.

than in peel oils, and indeed the major components were limonene (1.5−63.4%), β-pinene (up to 47.2%), and sabinene (up to 42.5%). (E)-β-Ocimene (0.5−4.6%), α-pinene (0.1−2.9%), myrcene (0.6−2.3%), and (Z)-β-ocimene (0.1−1.2%) were present in all samples. The oxygenated fraction (11.4−77.1%) mainly consisted of acyclic monoterpenes: linalool, geranial, linalyl acetate, citronellal, neral, citronellol, geranyl acetate, neryl acetate, geraniol, and nerol. α-Terpineol was also found in all samples.

PCA suggested the existence of four principal clusters and two atypicals (**Figure 4**):

Cluster 1. Eleven samples belonged to the limonene/geranial/neral chemotype (22.1−53.4%/11.8−26.9%/8.3−20.5% respectively). This group was also characterized by variable concentrations of geranyl acetate (1.7−8.3%), neryl acetate (0.6−6.1%), geraniol (1.3−3.8%), and nerol (1.1−3.1%). The cultivar Nouvelle Calédonie was characterized by an original compound: santal-10-en-2-ol (8.5%), previously found in *Dysoxylum spectabile* (*34*). All of the cultivars of *C. aurantifolia* and *C. latifolia* belonged to this group: Antillaise, Ambilobe, Mexicaine, Kirk, Nouvelle Calédonie, El Kseur, Bearss, De Perse, Tahiti, and IAC, as well as the cultivar Nestour from *C. excelsa* species.

Cluster 2. Two samples, Brazil and Bizri (*C. limettioïdes*), exhibited contents of limonene (63.4 and 50.5%) comparable to that of the previous group. However, citronellal (13 and 17.7%) and linalool (10.6 and 21.3%) were important components.

Cluster 3. The two other sweet limes of our sampling, India and Douce, contained less limonene (39.5 and 35.6%) and comparable quantities of citronellal and linalool, and particularly sabinene (22.7 and 23.7%). They constitute a cluster with Volkameriana (*C. limonia*) that exhibited a comparable composition: limonene/sabinene/citronellal/linalool.

Cluster 4. The fourth group was distinguished from others by high contents of β-pinene (28.2−32.2%) and limonene (27.3−27.6%). Citronellal (14.7−14.9%) was found in all samples at an appreciable level. This cluster corresponds to Rangpur limes (*C. limonia*).

The cultivar Sauvage was close to Volkameriana and had some characters of sweet limes *C. limettioïdes*, but differed by a higher content of sabinene and a lower amount of linalool.

The cultivar Khatta (*C. karna*) was found to be atypical with β-pinene as the main component (47.2%) and a small amount of limonene (11.1%).

Leaf oils have been less studied, although the first studies date back to 1957 (*11*). Most of the studies concerned acid limes. The compositions were dominated by limonene, geranial, or neral in unspecified varieties (*11*, *35*), Tahiti and Mexicaine (*14*, *26*), Key (*11*), and an unspecified variety of *C. limonia* (*14*). Only one study concerned an unspecified variety of sweet lime, *C. limettioïdes* (*14*).

The compositions of 19 lime leaf oils are reported for the first time. Most of the oils exhibited limonene, geranial, and neral as major components and so are qualitatively similar to the 3 oils previously reported (Tahiti, Mexicaine, and Key). Three other chemotypes and two atypicals are reported. Acid limes (*C. aurantifolia* and *C. latifolia*; cluster 1) can be distinguished from sweet limes (*C. limettioïdes*; clusters 2 and 3) and Rangpur limes (*C. limonia*; cluster 4) by the presence of oxygenated monoterpenes (neral and geranial) in leaf oils.

In summary, our results in association with literature data provide a new insight into the chemical variability of lemon and lime peel and leaf oils. Peel oils of most varieties of lemons exhibit the common limonene/β-pinene/γ-terpinene chemotype, whereas the peel oil of a few varieties is dominated by limonene. An unusual peel oil composition, containing limonene, linalool, and its acetate as major components, was found for two samples. Most varieties of lemon produced leaf oils exhibiting the limonene/β-pinene/geranial/neral chemotype or similar composition. Linalool and linalyl acetate were the major components of two varieties that produced an oxygenated monoterpene-rich peel oil. In the species *C. limon*, peel oil and leaf oil of the different varieties showed a great homogeneity in their composition, except for one cultivar. Conversely, the species *C. lumia* presented a great variability because each cultivar analyzed belonged to one of the three chemotypes observed. The specificity of Meyer lemon (*C. meyeri*) leaf oil can be explained by the fact that this cultivar is a supposed hybrid between *C. limon* and *C. sinensis* (orange tree). Obviously, acid limes (*C. aurantifolia* and *C. latifolia*) can be easily distinguished from sweet limes (*C. limettioïdes*) and Rangpur limes (*C. limonia*) by the presence of oxygenated monoterpenes (neral and geranial) in leaf oils and by a lower amount of limonene in leaf and peel oils. These results may permit classification of *Citrus* based not only on DNA fingerprints but also on essential oil profiles.

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