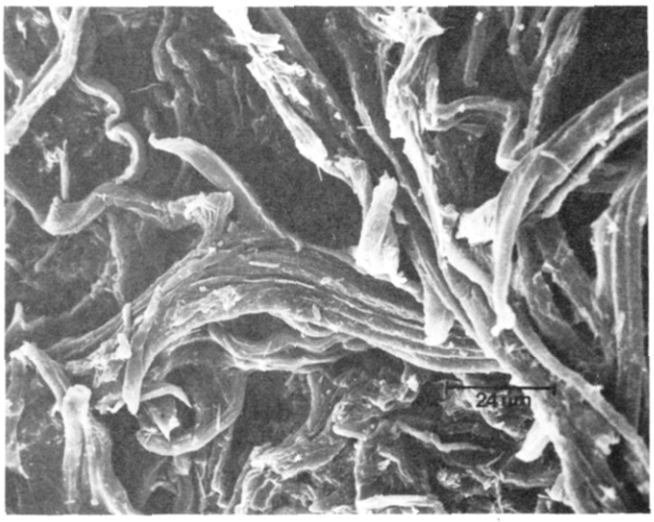

Figure 8. Dioxane–water extraction of material wet oxidized at 200 °C for 30 min.

walls in material pretreated by autohydrolysis and wet oxidation appeared to depend more on the removal of hemicellulose than cellulose (Biermann, 1984).

Registry No. Lignin, 9005-53-2.

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Epicuticular Leaf Waxes of *Citrus halimii* Stone

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Epicuticular leaf wax from *Citrus halimii* Stone was analyzed and found to contain *n*-alkanes, wax esters, primary alcohols, fatty acids, and large amounts of triterpenols and their derivatives. Benzoic acid esters and sterols were also isolated. The main components of this wax were the free triterpenols β-amyrin, α-amyrin, and lupenol. β-Amyrin and α-amyrin were also found esterified with very long chain fatty acids. Triterpenol ketones lupen-3-one and friedelan-3-one could be identified. The sterols isolated were identified as cholesterol, campesterol, stigmasterol, and β-sitosterol.

Citrus halimii is a wild species that has been reported from only a few places of the primary hill forests of southern Thailand and peninsular Malaysia. It has been described and placed into the subgenus *Citrus* in the orange subfamily Aurantioideae (Stone et al., 1973). Its potential for rootstock use, breeding, and conservation purposes has been explored neither in its native areas nor in research stations of other countries (Jones and Ibrahim, 1984). Phytochemical investigations of *C. halimii* so far have been carried out on the fruit flavanones, cations and major rind oil components (Stone et al., 1973), on essential leaf oil composition, oxidase browning of young leaf shoots, and on amylase, catalase and peroxidase isozymes of leaves (Scora et al., 1976). The *n*-alkanes from leaf tissues of various *Citrus* and *Fortunella* fruits were investigated by Nordby et al. (1979); those for 71 citrus biotypes were also reported by Scora et al. (1982). The potential usefulness of *Citrus* long-chain leaf hydrocarbons for taxonomic purposes was reported by Nordby and Nagy (1974), as well as the possibility to separate zygotic from nucellar seedlings so important in *Citrus* breeding (Norby et al., 1975). None of these reports, however, included *C. halimii*, which is a new and taxonomically still imperfectly understood taxon. We therefore report on the soluble cuticular lipids of *C. halimii*, which influence the adherence of agricultural sprays, act as a barrier to pathogen infection, and also play a role in the water economy of the plants.

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EXPERIMENTAL PROCEDURES

Healthy, mature leaves taken from the 1985 spring flush of 14-year-old seedlings of *C. halimii* (Voucher: Scora 3174

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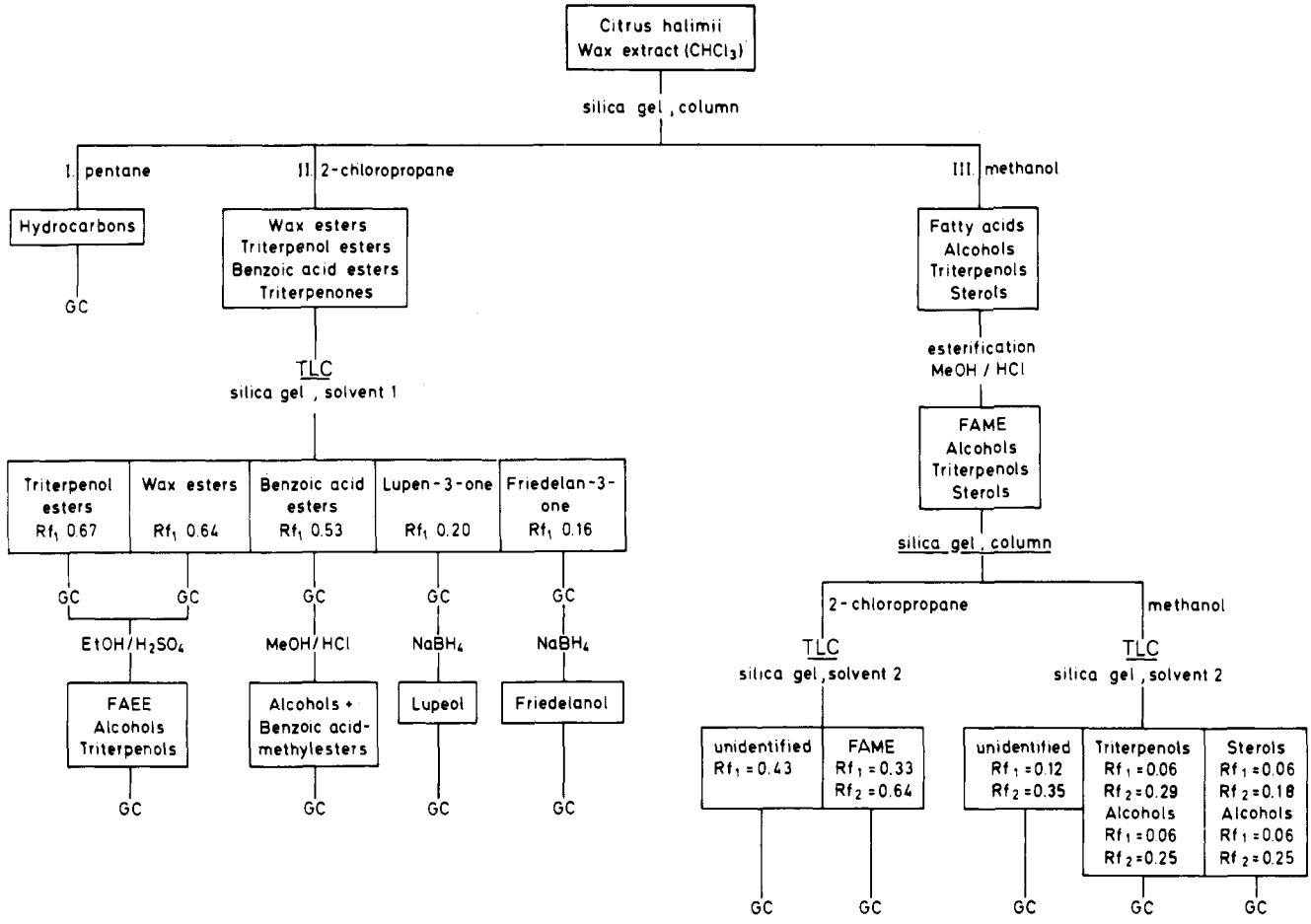

Figure 1. Flow diagram for fractionation, separation, and identification of epicuticular waxes from *C. halimii*.

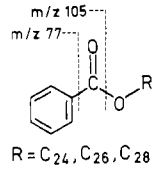

Figure 2. Benzoic acid alkyl esters.

UCR) grown on their own rootstock at the Citrus Variety Collection, University of California at Riverside, were harvested. These were dipped consecutively into three 800-mL beakers of chloroform for a total of 6 min to extract the epicuticular waxes. The combined extracts were taken to dryness. Three hundred and forty grams of fresh leaf material yielded 1.035 g of crude wax (0.3%). The crude leaf wax was redissolved in 50 mL of pentane and fractionated on a silica gel column (Merck 60, Darmstadt) with solvents of increasing polarity (Gülz, 1985, 1986; Scora et al., 1986) into three fractions (I–III). Pentane (600 mL) was used for the elution of hydrocarbons (I), 2-chloropropane (800 mL) for the elution of esters and ketones (II), and methanol (800 mL) for the elution of alcohols and acids (III). The yield and composition of the individual fractions are reported in Table I.

Fraction I was analyzed directly by GC, and fraction II had to be separated again by TLC (C_6H_6), as did fraction III after esterification of the fatty acids (TLC: CH_2Cl_2/EtOAc, 24:1). A system for fractionation, separation, and identification of the individual components is shown in Figure 1.

For TLC, precoated silica gel plates (Merck 60, Darmstadt) were used. The solvent system was C_6H_6 for R_{f_1}; CH_2Cl_2/EtOAc (24:1) for R_{f_2}. Plates impregnated with $AgNO_3$ in acetonitrile were run in CH_2Cl_2/EtOAc for R_{f_3}.

Table I. Quantitative Composition of *C. halimii* Epicuticular Leaf Wax

fraction	mg	% wax	mg	% fraction	R_{f_1}
I. pentane	66	6.0			
n-alkanes		5.9	65.2	98.8	0.7
branched alkanes		0.1	0.8	1.2	
II. 2-chloropropane	162	16.0			
wax ester / triterpenol ester		0.8	8.3	5.1	0.64
benzoic acid ester		0.4	4.0	2.5	0.53
lupen-3-one		2.9	29.0	17.9	0.20
friedelan-3-one		11.9	120.7	74.5	0.16
III. methanol	716	69.4			
primary alcohols		3.7	38.0	5.3	0.06
β-amyrin		15.5	160.4	22.4	0.06
α-amyrin		29.9	308.7	43.1	0.06
lupeol		16.8	173.2	24.2	0.06
sterols		0.9	9.5	1.3	0.06
fatty acids		2.6	26.4	3.7	0.02
unidentified	37	3.6			
lost on column	54	5.0			
total crude wax	1035	100			

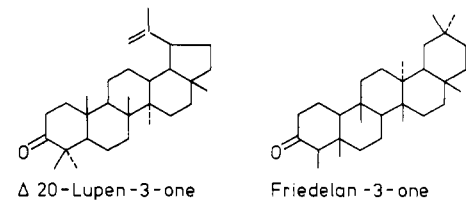

Figure 3. Structures of triterpene ketones.

The spray reagent used was bromothymol blue, and carbazole was used for triterpenols and sterols (Ghosh and Thakur, 1982). For gas chromatography, a Hewlett-

Table II. Quantitative Composition of Epicuticular Leaf Wax Components of *C. halimii* (Peak Area %)

no. C atoms	alkanes	primary alcohols		fatty acids		wax esters	
		free	esterified	free	esterified	no. C atoms	%
16				34.7	40.0		
17				+	+		
18:2				12.1			
18:1				12.1			
18:0				13.2	7.8	42	+
19	+			+	0.6	43	+
20	0.2		0.5	12.9	32.6	44	1.9
21	0.2		+	+	+	45	2.5
22	0.7		0.2	7.7	10.1	46	13.0
23	0.5		+	+	+	47	4.4
24	0.9	+	7.6	5.5	4.7	48	24.3
25	1.6	+	0.4	+	+	49	7.2
26	1.7	14.2	4.4	2.0	2.6	50	21.9
27	3.2	+	0.9	+	+	51	5.0
28	2.9	35.6	29.1	+	1.5	52	13.2
29	10.6	+	8.2	+	+	53	2.9
30	4.7	21.6	34.1	+	+	54	3.4
31	38.7	+	4.7				
32	12.7	21.0	9.9				
33	20.1	+	+				
34	0.7	7.6	+				
35	0.3						
36	0.2						
37	+						

Packard Model 5710A with fid and integrator 3380S was used. A 25-m glass capillary column (DuHT-OV 101) was programmed from 160 to 340 °C with a 4 °C/min advance. For all fractions, GC–MS detection was by Finnigan-Mat 4510, 70 eV, EI.

MS data [m/z (rel intens)] of triterpenols and triterpenone derivatives follow.

Friedelan-3-one: 69 (100), 257 (0.3), 273 (1.6), 287 (0.2), 302 (0.5), 411 (M − 15, 0.1), 426 (M^+, 0.3).

Lupen-3-one: 55 (100), 205 (20), 218 (10), 232 (1.4), 245 (1.8), 313 (1.0), 381 (M − 43, 2.0), 409 (M − 15,0.4), 424 (M^+, 0.8).

β-Amyrin: 43 (100), 55 (100), 203 (45), 218 (100), 257 (1), 411 (M − 15, 0.2), 426 (M^+, 0.6).

α-Amyrin: 43 (100), 55 (100), 203 (15), 218 (50), 257 (1), 411 (M − 15, 0.2), 426 (M^+, 0.8).

Lupeol: 43 (100), 55 (95), 189 (20), 207 (15), 218 (15), 257 (0.4), 315 (0.2), 383 (M − 43, 0.1), 426 (M^+, 0.4).

MS data of the isolated triterpenes are identical with those from authentic samples (Carl Roth, D-7500 Karlsruhe 21, FRG).

MS data [m/z (rel intens)] of sterols follow.

Cholesterol: 43 (100), 55 (100), 231 (2), 247 (1), 255 (15), 275 (3), 301 (1.5), 353 (M − 33, 0.5), 368 (M − 18, 0.5), 386 (M^+, 1).

Campesterol: 43 (100), 55 (60), 231 (2), 255 (2), 273 (10), 289 (20), 315 (20), 368 (M − 33, 0.5), 382 (M − 18, 0.5), 400 (M^+, 1.0).

Stigmasterol: 43 (45), 55 (100), 83 (55), 255 (2.5), 271 (1.5) 300 (0.5), 379 (M^+ − 33, 0.1), 394 (M − 18, 0.1), 412 (M^+, 0.5).

β-Sitosterol: 43 (100), 55 (60), 81 (40), 231 (2), 255 (1.5), 273 (1.0), 303 (1.2), 329 (1.0), 381 (M − 33, 0.2), 396 (M − 18, 0.5), 414 (M^+, 1.0).

MS data of the isolated sterols are identical with those from authentic samples (Roth, Karlsruhe).

MS data [m/z (rel intens)] of benzoic acid esters follow.

Benzoic acid tetracosanyl ester: 77 (20), 105 (30), 123 (100), 458 (M^+, 0.2).

Benzoic acid hexacosanyl ester: 77 (20), 105 (45), 123 (100), 486 (M^+, 0.1).

Benzoic acid octacosanyl ester: similar fragment ions as the above esters.

MS data of the isolated benzoic acid esters are identified with those from the synthesized esters.

For methanolysis, substances were refluxed with 2 N HCl/MeOH for 2 h. After evaporation of the solvent, the residue was dissolved in CH_2Cl_2 and reduced to dryness once more to completely remove the HCl. Finally, the product was dissolved in hexane and analyzed.

For ethanolysis, wax esters were refluxed with 2 N HCl/EtOH for 14 h. The reaction products were extracted three times with ether/petroleum ether, washed until neutral, dried over $MgSO_4$, filtered, and evaporated.

For reduction, the solution of the triterpene ketones in benzene was stirred for 2 h with $NaBH_4$, deposited on aluminia (McKillop and Young, 1979), filtered, and analyzed.

For hydrogenation, the substance was dissolved in 10 mL of ethyl acetate, and Pd-catalysator (100 mg) was added. The mixture was stirred under an H_2 atmosphere for 2 h. The catalysator then was filtered and the solvent evaporated.

For the synthesis of benzoic acid esters, a mixture of hexacosanol (100 mg) with an excess of benzoic acid and a few drops of H_2SO_4 was heated to 80 °C for 1 h. The reaction products were extracted with ether/petroleum ether (1:1), washed for neutrality, dried over $MgSO_4$, filtered, and evaporated. The esterification was checked by TLC, GC, and GC–MS.

RESULTS AND DISCUSSION

Epicuticular waxes from *C. halimii* leaves, extracted with $CHCl_3$, show a very complex and unusual composition. In this investigation, it was not those substances resulting from lipid metabolism (alkanes, wax esters, primary alcohols, fatty acids) that were the dominant ones, but the triterpenols and their derivatives that are products from the isoprene metabolism. The latter comprised more than 77% of the total raw wax. In addition, there were also unusual benzoic acid esters present.

Alkanes. Hydrocarbons were found to be only saturated *n*-alkanes (R_{f_1} 0.70) in a series of 19 homologues ranging from C_{19} to C_{37}. The dominating component was

Table III. Composition of Benzoic Acid Esters in *C. halimii* Leaf Wax

no. C atoms	TLC R_{f_1}	GC ret[a]	GC–MS M^+	ester alcohols no. C atoms	ester alcohols %
31	0.53	1.000	458	24	4.4
33	0.53	1.118	486	26	75.5
35	0.53	1.218	514	28	20.1

[a] Relative emergence times.

hentriacontane ($C_{31}H_{64}$) with 38.7%. The odd-numbered homologues comprised about 75%; the even-numbered, about 25% (Table II).

Wax Esters. Wax esters (R_{f_1} 0.64) had to be separated from the 2-chloropropane fraction (II) by TLC (C_6H_6). They were accompanied by triterpenol esters. A separation of the latter succeeded only incompletely in this case. Wax esters were found in homologue series ranging from C_{42} to C_{54}. C_{48} and C_{50} esters yielded the highest percentage with more than 20%, followed by C_{46} and C_{52} esters, with about 13% (Table II).

Ethanolysis of the esters resulted in alcohols and saturated fatty acids in the form of their ethyl esters (FAEE). FAEE range from C_{16} to C_{30}. The largest component was C_{16} with 40%, followed by C_{20} with 32.6%. Primary alcohols were found in series from C_{20} to C_{34}, with C_{28} comprising about 29% and C_{30} about 34% (Table II). Triterpenols were found to accompany the alcohols. They could be identified as α- and β-amyrin and were also detected in a concentrated fraction of triterpenol esters.

Benzoic Acid Esters. In addition, esters with R_{f_1} 0.53 could be separated by TLC (C_6H_6) (see Figure 1). These esters were transesterified by HCl/MeOH and by heating for 2 h. This method yielded even-numbered primary alcohols ranging from C_{24} to C_{28} with hexacosanol ($C_{26}H_{54}O$) as the main component (75.5%). The structure of the acid component of these esters was elucidated by GC–MS. The MS spectra showed significant fragment ions m/z 77 and 105, characteristic for benzoic acid derivatives. These M^+ fragment ions could also be seen in all benzoic acid esters. The coordination of the benzoic acid to the corresponding alcohols is listed in Table III. The structure of these esters was verified by synthesis. These synthetic benzoic acid tetracosanyl and hexacosanyl esters showed the same chromatographic behavior and MS data as the isolates from *C. halimii* leaf wax. A benzoic acid ester in the form of benzyl benzoate was reported in the essential oils of Cistus leaves (Proksch et al., 1980). Analogue benzoic acid esters of long-chain alcohols were also found recently in *Euphorbia dendroides* leaf wax (Gülz et al., 1987). Furthermore, two bands with R_{f_1} 0.20 and 0.16 (Figure 1) could be separated by TLC in fraction II. These were identified as triterpene ketones and will be discussed later in detail.

Fatty Acids. Fraction III contained fatty acids, primary alcohols, triterpenols, and sterols. After esterification of the fatty acids to fatty acid methyl ester (FAME), these substances were separated by TLC with the solvent system CH_2Cl_2/EtOAc (24:1). The fatty acid composition analyzed as FAME (R_{f_2} 0.41) ranged from C_{16} to C_{30}. Dominant was palmitic acid ($C_{16}H_{32}O_2$) with 34.7%, followed by stearic acid ($C_{18}H_{36}O_2$) with 13.2%, and the unsaturated oleic (C 18:1) and linoleic acid (C 18:2). These were found in amounts of 12% each (Table II). The presence of these unsaturated fatty acids was proved by hydrogenation.

Alcohols. The primary alcohols (R_{f_2} 0.25) ranged from C_{24} to C_{34}, with octacosanol ($C_{28}H_{58}O$) as the main component with 35.6%, followed by triacontanol ($C_{30}H_{62}O$) and dotriacontanol ($C_{32}H_{66}O$), each with about 21% (Table II).

Table IV. Characterization of Triterpenols and Derivatives in *C. halimii* Leaf Wax

	TLC R_{f_1}	TLC R_{f_3}	TLC carbazol	GC ret[a]	GC/MS M^+
I. triterpenol ester					
triterpenol esters	0.64		+		
β-amyrin (ester)	0.06	0.30	+	1.000	426
α-amyrin (ester)	0.06	0.30	+	1.027	426
II. triterpen-3-one					
lupen-3-one	0.20	0.49	+	1.018	424
$NaBH_4 \rightarrow$ lupeol	0.06	0.20	+	1.033	426
friedelan-3-one	0.16	0.53	–	1.095	426
$NaBH_4 \rightarrow$ friedelanol	0.12	0.40	–	1.086	428
III. triterpenols					
β-amyrin	0.06	0.30	+	1.000	426
α-amyrin	0.06	0.30	+	1.027	426
lupeol	0.06	0.20	+	1.033	426

[a] Relative emergence times; β-amyrin = 1.000.

Triterpenols and Derivatives. The main components in *C. halimii* leaf wax were found to be triterpenols and their derivatives, comprising more than 77% of the raw wax. About 62% of this wax was free triterpenols. They were isolated from fraction III by TLC with the solvent CH_2Cl_2/EtOAc (24:1), showing a R_{f_2} value of 0.29 and a positive color reaction with carbazol (Ghosh and Thakur, 1982). Three peaks were separated by GC, which could be identified with authentic samples to be β-amyrin, α-amyrin, and lupeol. β-Amyrin and α-amyrin have quite different retention times for a positive detection. But α-amyrin and lupeol show only very small differences. According to the different positions of the double bond in these two triterpenols, they could be separated on silica gel plates that were impregnated with $AgNO_3$. β-Amyrin and α-amyrin have an R_{f_3} value of 0.30; lupeol has one of 0.20 (Table IV). Additionally, these triterpenols were elucidated by their GC–MS data. They were identical with those from authentic samples and in agreement with data reported by Tulloch (1982).

Not all of these free triterpenols were found to be esterified with long-chain fatty acids. In the saponification products of the triterpenol esters, only β-amyrin and α-amyrin were found, but not Lupeol. β- and α-amyrin were esterified with fatty acids of chain lengths ranging from C_{18} to C_{28}.

Triterpene derivatives were also found in the form of ketones, in fraction II, with different R_{f_1} values, 0.20 and 0.16. They could be separated by TLC (C_6H_6) and identified as lupen-3-one and friedlan-3-one.

Lupen-3-one (R_{f_1} 0.20) shows a positive color reaction with carbazole and could be reduced with $NaBH_4$ to lupeol. MS data of the isolated lupen-3-one are identical with those from authentic samples and are in agreement with MS data reported by Budzikiewicz et al. (1963).

Friedlan-3-one (R_{f_1} 0.16) showed no reaction with carbazole but could be reduced with $NaBH_4$ to the corresponding alcohol friedelanol. MS data of the isolated friedelan-3-one were identical with those from authentic samples and in agreement with MS data published by Budzikiewicz et al. (1963) (see the Experimental Procedures and Table IV).

Sterols. In the alcohol fraction, sterols (R_{f_2} 0.18) could also be identified. They showed a positive color reaction with carbazole and four peaks in the GC chromatograms. Comparison with authentic samples by GC and GC/MS data indicated cholesterol, campesterol, stigmasterol, and β-sitosterol.

In the genus *Citrus*, β-sitosterol is apparently the principal steroid. It has been reported from the juice and peel

oil of *Citrus paradisi*, the grapefruit, by Kefford (1959), orange juice by Tatum et al. (1965), lemon juice by Vandercook and Yokoyama (1965), lemon peel by Chopin et al. (1964), mandarin peel by Chaliha et al. (1967), and the rags of the calamondin, *Citrus mitis*, by Row and Sastry (1962). Böhme and Völcker (1959) reported that cholesterol was found to accompany β-sitosterol in the nonvolatiles of orange peel oils.

Pentacyclic triterpenes are common in the Rutales, occurring mainly as alcohols, α- and β-amyrins, and lupeol, as well as the ketone friedlin. α- and β-amyrins were found to be constituents of the Burseraceous genera *Aucouma*, *Bursera*, and *Canarium* and α-amyrin additionally of *Commiphora* and *Dacryoides*. Lupeol has been isolated from Boswellia and several *Bursera* species in the Burseraceae (Khalid, 1983), as well as from several species of *Myrtopsis* in the Rutaceae (Hegnauer, 1983). The saturated triterpenoid Friedelin has been reported from the peel oil of the Duncan grapefruit, *C. paradisi*, only once (Weizman et al., 1955).

Such triterpenes, however, have not been reported from any leaves of the genus *Citrus*, nor from any of its closely related Aurantioid genera. Therefore, the high amount of triterpenes in the leaf wax of *C. halimii* was surprising to us. While Baker et al. (1975) reported the alkane content of surface waxes to range from 42 to 66% in some horticultural *Citrus* varieties and Leece (1976) states such paraffins to constitute 40% of the epicuticular wax of orange leaves, we found only 6% in *C. halimii*. Primary alcohols of mandarin and sweet orange varieties ranging from 23 to 38% were a major part of the leaf wax as reported by Baker et al. (1975). We found *C. halimii* leaves to contain only 3.7% of these substances. The fatty acid content of 2.6% of total wax in *C. halimii* fits well with the 2% reported by Baker et al. (1975) from lemon leaves, less so with the 19% in Valencia orange leaves.

These differences, however, are easily acceptable if one considers the only partially known effects of geography and climate upon such components and the tropical mountainous nature of *C. halimii*, which makes this species rather unique within its genus.

ACKNOWLEDGMENT

We thank W. Clerx for procuring the crude waxes.

Registry No. β-Amyrin, 559-70-6; α-amyrin, 638-95-9; lupeol, 545-47-1; lupen-3-one, 1617-70-5; friedelan-3-one, 559-74-0; cholesterol, 57-88-5; campesterol, 474-62-4; stigmasterol, 83-48-7; β-sitosterol, 83-46-5; tetracosanyl benzoate, 103569-99-9; hexacosanyl benzoate, 109334-04-5; octacosanyl benzoate, 54288-58-3; hentriacontane, 630-04-6; palmitic acid, 57-10-3; stearic acid, 57-11-4; oleic acid, 112-80-1; linoleic acid, 60-33-3; octacosanol, 557-61-9; triacontanol, 593-50-0; dotriacontanol, 6624-79-9.

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