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Received for review July 27, 1978. Accepted November 2, 1978.

The following paper, presented at the Symposium on Lipoxygenase: Its Biochemistry, Products, and Role in Plant and Animal Chemistry, will appear in the next issue: "Recent Progress in the Study of Soybean Lipoxygenase", by J. F. G. Vliegenthart, G. A. Veldink, and J. Boldingh. Other papers presented at the Symposium but not printed in this issue are as follows: "Characterization and Occurrence of Cyclic Fatty Acids Produced by Plant Extracts", by D. Zimmerman, P. Feng, and B. Vick; "Lipoxygenase-Like Enzyme from Rat Testes Microsomes—Purification and Partial Characterization", by S. Grossman and I. Shahin; "Analysis of Lipid Oxidation Products by High-Performance Liquid Chromatography", by J. A. Singleton and H. E. Pattee.

REVIEW

Review of Quantitative Analyses of Citrus Essential Oils

Philip E. Shaw

A compilation of reported quantitative values for individual components of cold-pressed oils of sweet orange, grapefruit, mandarin, lemon, lime, bitter orange, bergamot, certain hybrid oils, and of distilled lime oil is presented. Different analytical methods used to determine these values are compared. Reasons for differences in quantitative values determined by gas chromatography (GLC) are ascribed to method of preliminary separation, method of calculating relative percent composition, type of column or detector used, decomposition during GLC separation, and sample history. Valid conclusions regarding chemotaxonomy of hybrids cannot be made from the available data because of variable sample histories and analytical techniques.

Quantitative values on a few individual components of citrus essential oils have been reported over many years, but it was not until the widespread use of gas chromatography that meaningful quantitative values for many components of each citrus essential oil became available. Attempts to correlate the presence of single components with the characteristic flavor of each fruit have been partly successful, but we now realize that several components are blended together in a specific proportion to create the unique full flavor of oil from each citrus species or hybrid (Braddock and Kesterson, 1976; Shaw, 1977, and references therein). Thus, the accuracy of the quantitative values that have been reported for each component becomes critical when we determine the mixture of components necessary for full citrus flavors, create synthetic or partially synthetic blends with flavor properties characteristic of each particular fruit, and study the chemotaxonomy of citrus hybrids.

Several reviews on quantitative analyses of citrus essential oils have been reported. Kefford and Chandler (1970) summarized the quantitative data available to them, but included data on terpeneless oils and individual fractions as well as total oil without relating quantities in various fractions to the level present in the total peel oil. Shaw (1977) supplemented the data of Kefford and Chandler by reporting quantitative values determined on a single sample of whole oil for each species of citrus so that the relative properties of components present would be more meaningful. In neither of these studies were all reported values for each component tabulated so that on a comparable basis the most meaningful values might be selected. No attempt to critically review the analytical methods to aid in selection of best probable quantitative values was made in either study.

The present review tabulates quantitative values for individual compounds as percent of total oil and compares

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analytical methods. It also tabulates values for several hybrids and relates certain analytical values to the chemotaxonomy of these hybrids.

DISCUSSION

Quantitative values for individual constituents of the various citrus oils are listed in Table I, and the source of each value is indicated. This format was chosen rather than the presentation of average values or ranges, so that the reader can better see the spread of values determined for each compound when more than one quantitative determination has been made. Only values that can be cited as percent of total oil are included; hence, some of those listed by Kefford and Chandler (1970) are not included. Most of the reported values omitted from this table could not be expressed as percentage of total oil because weights of total oil and individual fractions were not reported. Except for both distilled and cold-pressed lime oils, which are significant items of commerce, only cold-pressed peel oils are reported in Table I.

Comparison of Analytical Methods. Some wide variations exist in the analytical values reported for both major and minor components of the various citrus oils even though gas chromatography (GLC) was almost exclusively used for final separation into individual components and for quantitation. Possible sources of variation among gas chromatographic analyses include differences in: (1) method of preliminary separation, (2) method of calculating relative percent composition, (3) type of column or detector used, (4) decomposition of certain components due to conditions of the GLC separations, and (5) sample history, including method of preparation.

Variation in methods for preliminary separation of citrus oils prior to GLC analysis can be a major factor in variation of reported quantitative results (Stanley et al., 1961; Kefford and Chandler, 1970). Methods used for preliminary separation include distillation, extraction, liquid column chromatography, thin-layer chromatography, and preparative GLC, or a combination of several of these. No good method has yet been developed to quantitate the losses that occur during preliminary separation except to assume that all components are decreased by proportional amounts. This assumption probably causes only small errors in the final results unless the proportion of material lost during preliminary separation is large. Several workers have directly injected the whole oil into a GLC column to avoid the errors introduced by preliminary separation steps (Ziegler, 1971; Maekawa et al., 1967; Bernhard, 1960; Lund and Bryan, 1976; Moshonas and Shaw, 1974).

Methods for determining relative percent composition have usually involved calculation of peak areas in GLC chromatograms. Hand calculation of peak areas is seldom done anymore, because, generally, it is less accurate than mechanical integration (Keulemans, 1959). Some digital integrators are inaccurate in integrating small, broad peaks or groups of peaks because they are limited in slope sensitivity and time before a base line is reestablished, such as at the top of a small, broad peak (Shaw, 1978). Also, relative peak heights, rather than peak areas, have been used in a few studies to monitor changes in oil composition during fruit maturation (Attaway et al., 1967, 1968). Use of relative peak heights is more acceptable for such comparison studies than for the determination of absolute quantitative values. Because sharpness decreases with retention time, early-emerging compounds would tend to assay higher than late-emerging ones (Dal Nogare and Juvet, 1962; Keulemans, 1959). However, comparison of reported values in Table I shows that the amounts of the early-eluted compounds are not consistently higher when determined by the relative-peak-height method than by the peak-area method.

All cold-pressed citrus oils contain a significant amount of nonvolatile material that will not be eluted from a gas chromatographic column (Shaw, 1977). Unless some preliminary separation step is used to remove these materials, they need to be taken into account when the percentage of each GLC volatile compound present in the oil is determined. The percentage of nonvolatiles present in an oil sample depends on the species and the amount of winterizing (storage at cool temperatures) it has undergone (Kefford and Chandler, 1970; Wolford et al., 1971).

The type of GLC detector used in quantitating oils or oil fractions is a significant source of error unless response correction factors are determined. Thermal conductivity detectors afford a more representative chromatogram than do flame ionization detectors when a sample containing a variety of functional groups (such as are present in all citrus oils) is quantitated without the use of response factors (Shaw and Coleman, 1971). Response factors are most easily determined from a mixture of pure compounds shown to be present in the citrus oil. Known amounts of the components are mixed in the proportion they have been determined to be present in the oil, from peak area percentages, and the mixture is chromatographed under the same conditions used for the citrus oil. When a pure sample of a compound in the oil is not available, one of similar structure must be substituted. Use of gas density balance detector precludes the requirement of response factor determination, and weight percent can be calculated from GLC peak area percent (Dal Nogare and Juvet, 1962). No one has reported quantitative data on citrus oils using this detector even though it is commercially available and comparable in sensitivity to a thermal conductivity detector.

The history of a particular oil sample must be considered when quantitative results are interpreted, especially if they differ markedly from those previously reported for the same type of oil. Thus, if unusually high levels of known degradation products of some of the main oil constituents are present, the freshness of the oil sample should be questioned. Known degradation products shown present at high levels in some oil samples include *p*-cymene, α-terpineol, terpinen-4-ol, carvone, carveol, and *cis*- and *trans*-2,8-*p*-menthadien-1-ol (Ziegler, 1971; Shaw, 1977). It is likely that samples vary naturally from one source to another depending on area grown, cultivar, and extraction methods. Most of the oils examined to date (Table I) were commercial samples prepared by a variety of extraction methods.

As yet, not enough is known about quantitative composition of high-quality oil samples to exclude natural variation from causing the variations seen in Table I. Since absolute values for individual components of citrus oils have not been determined by unambiguous methods, the best method for quantitative analyses cannot be specified. However, the present knowledge about quantitative analysis of citrus oils suggests that one of the most accurate methods is to analyze them by direct injection onto a gas chromatograph, integrate the areas under the peaks, and use response factors and percentage of nonvolatiles (if present in the oil) to correct the peak area values to weight percent values.

Sweet Orange. Hydrocarbons and aldehydes are the two groups of compounds in orange oil most extensively quantified. Values for the major constituent, *d*-limonene, range from 83–97%. The lowest value, 83%, was reported by Attaway et al. (1968) and determined on the basis of

Table I. Quantities (%) of Components Found in Citrus Essential Oils

	sweet orange	grapefruit	mandarin	lemon	C.P. lime	distilled lime	bitter orange	bergamot	other species and hybrids
acids									
citronellic			0.006[a]						
decanoic			0.03[a]						
dodecanoic			0.006[a]						
heptanoic			0.004[a]						
nonanoic			0.01[a]						
octanoic			0.04[a]			0.02[b]			
undecanoic			0.003[a]						
alcohols									
benzyl alcohol			0.009[a]						
borneol						0.6[b]			
cis-carveol			0.01,[c] 0.02,[a] 0.04[d]						0.2[e,f]
trans-carveol			0.003,[d] 0.01,[c] 0.04[a]						0.4[e,f]
citronellol			0.01,[c] 0.03[a]	0.5[g,h]			0.2,[i] 0.9,[i] 1.1[i]		0.4[f,i,j]
decanol			0.04[a]			0.06[b]			
elemol		0.04[ww]							
fenchol						1.2[b]			
geraniol			0.01,[a] 0.04[k]						0.3[f,i]
heptanol			0.02[a]						
isopulegol			0.01,[c] 0.05[d]				0.3[i] 0.4[i]		0.05[f,i]
linalool	0.3,[l] 0.5,[e] 0.6,[m] 1.0,[n] 1.1,[k] 5.3[o]	0.3,[ww] 0.4[k]	0.03,[c] 0.07,[d] 0.2,[a] 0.9,[p] 1.0,[q] 1.5,[n] 1.6,[r] 2.1,[r] 4.2,[k] 6.1[r]	0.08,[g] 0.1,[s] 0.2,[t] 0.3[m]	0.09,[u] 0.2[n]	0.1[b]	0.5,[i] 0.6,[i] 1.0[i]	16,[i] 41[i]	0.24,[u,v] 0.4,[i,j] 1.0,[e,f] 1.38,[u,w] 1.9[n]
1,8-*p*-menthadien-4-ol									0.2[e]
cis-2,8-*p*-menthadien-1-ol									0.2[e]
trans-2,8-*p*-menthadien-1-ol									0.3[e]
nerol			0.05[a]				0.1,[i] 0.3[i]		1.1[f,i]
1-nonanol	0.1[n]		0.1[n]		0.1[n]	0.01[b]			
octanol		0.8[k]	0.09[a]			0.01[b]			
sabinene hydrate			0.2[a]						
terpinen-1-ol						0.7[b]			
terpinen-4-ol	0.06,[k] 0.2[o]	0.08[k]	0.06,[q] 0.1,[a] 0.3[r]	0.01,[x] 0.4[t]		1.6[b]	0.3,[r] 0.4[r]		
α-terpineol	0.1,[n] 0.2,[k] 0.3,[m] 0.5[o]	0.2[k]	0.03,[c] 0.05,[d] 0.06,[p] 0.2,[n] 0.9,[k] 1.1[a]	0.2,[t] 0.5[m]	0.3,[n] 1.05[u]	5.9[b]	0.2,[i] 0.3[i]	0.2,[i] 0.3,[i] 4.0[i]	0.05,[u,v] 0.08,[i,j] 0.3,[n] 0.6,[f,i]

Table I. (*Continued*)

								1.1[u,w]
β-terpineol			0.4[r]			0.7[b]		
tetrahydrogeraniol				0.1[g]				
thymol			0.04,[p] 0.08,[a] 0.2[k]					2.9,[u,x] 6[z]
α,α,p-trimethylbenzyl alcohol			0.03[a]			0.6[b]		
aldehydes								
citral	0.05,[aa,bb] 0.1,[aa,bb,cc] 0.2,[aa,cc]	0.05,[aa] 0.06[aa]		2.0-2.6,[aa] 2.1-13.2,[x] 2.9[dd]	3.1-3.7,[aa,ee] 5.3[aa,y]	0.3[aa]		
citronellal	0.1[l,m]	0.14[ww]	0.1,[n,p] 0.2[q]	0.03,[g] 0.1,[x] 0.2[m]	1.4[n]		0.3,[i] 0.4[i]	0.21,[u,w] 0.27,[u,v] 0.4[f,i,j]
decanal	0.1,[n] 0.2,[l,ff] 0.3,[ff] 0.4,[l,ff] 0.5,[dd,ff] 0.6,[cc] 0.7[m]	0.3,[ff] 0.6[ww]	0.04.[a,c] 0.2,[d,p] 0.3,[ff] 0.5[q] 0.6,[r] 0.9[r]	0.06,[g] 0.1,[dd] 0.2[m]	0.1[n]	0.09[b]		0.14,[ff] 0.2,[n,u,v] 0.3,[ff,gg] 0.6[dd,gg]
dodecanal	0.05,[l] 0.08,[cc] 0.09,[cc] 0.2[ff]	0.1,[dd] 0.2[dd,ff]	0.006,[a] 0.02,[d] 0.15[ff]	0.1[dd]		0.006[b]		0.07,[ff] 0.1,[m,dd,ff] 0.2[ff,gg]
furfural						0.005[b]		
geranial	0.06,[cc] 0.07,[ff] 0.09,[ff] 0.12,[ff] 0.14,[ff] 0.15,[l,cc] 0.2[k,m]	0.1,[ff,ww] 0.2[k]	0.06,[ff] 0.07,[p] 0.2,[q] 0.3[k]	0.6,[g] 0.9,[s] 1.6,[t] 2.3[m]	5.1,[y,hh] 6.8[u]	0.1[r]		0.03,[ff] 0.08,[ff,gg] 0.2[u,v]
heptanal	0.03,[dd] 0.05[dd]	0.05,[dd] 0.06[dd]		0.04[dd]				
hexanal	0.006,[ff] 0.02[ff]							
neral	0.01,[cc] 0.02,[ee,ff] 0.06,[ff] 0.07,[l,ff] 0.08,[ff] 0.2[m]	0.03[ff]	0.06[ff]	0.4,[s] 0.5,[g,h] 0.8,[t] 1.3[m]	0.4,[n] 3.2,[y,hh] 4.6[u]			0.05,[ff] 0.07,[ff,gg] 0.2[u,v]
nonanal	0.06,[cc,dd] 0.07,[dd] 0.09,[cc] 0.2[m]	0.04,[dd,ww] 0.1[dd]	0.03[q]	0.09,[g] 0.2,[dd] 0.3[m]	0.2[n]		0.1,[i] 0.3,[i] 0.4[i]	0.04,[dd,gg] 0.1,[f,i] 0.5[i,j]
octanal	0.2,[m,n,ff] 0.3,[cc,dd,ff] 0.4,[ff] 0.5,[cc,dd] 0.8,[k] 2.8[o]	0.3,[dd] 0.5,[ff] 0.6,[k,dd] 0.7[ww]	0.04,[a] 0.3[n,ff]	0.1,[dd] 0.15[g]	0.8[n]	0.03[b]	1.4,[i] 1.9,[i] 2.2[i]	0.15,[ff] 0.3,[f,i] 0.4,[n] 0.6,[ff,gg] 0.9,[dd,gg] 1.3[i,j]

Table I. (*Continued*)

	sweet orange	grapefruit	mandarin	lemon	C.P. lime	distilled lime	bitter orange	bergamot	other species and hybrids
perillaldehyde	0.02[l]	0.2[ww]	0.003,[c] 0.02,[d] 0.05,[a] 0.1[p]						
α-sinensal	0.03[l]		0.2[p]						
β-sinensal	0.06,[m,ii] 0.09[l]								
tetradecanal	0.05,[ff] 0.08[ff] 0.09[ff]	0.1[ff]	0.05[ff]						0.06,[ff] 0.1[ff,gg]
undecanal	0.01,[cc] 0.02,[cc,dd] 0.03,[dd] 0.2[m]								0.2[dd,gg]
esters									
citronellyl acetate			0.04,[c] 0.1[d]	0.04,[m] 0.2[g]			0.2,[i] 0.6[i]		0.08,[i,j] 0.4[f,i]
citronellyl formate							0.1,[i] 0.3,[i] 1.1[i]		0.08,[i,j] 0.4[f,i]
decyl acetate		0.15[ww]	0.002,[a] 0.003[c]	0.05[g]	0.1[n]				
geranyl acetate			0.003,[a] 0.04,[c] 0.1[d]	0.1,[s] 0.4,[g,m] 1[x]	0.55,[u] 3.1[y,hh,jj]	0.3[b]		0.6[r]	0.05,[u,v] 0.1[n]
geranyl formate							0.3,[i] 0.6[i]		0.2[f,i,j]
linalyl acetate							0.3,[i] 0.9,[i] 1[i]	11.4,[r] 38,[r] 44[r]	0.1[i,j]
1,8-*p*-menthadiene-9-yl acetate			0.003[c]						
1-*p*-menthen-9-yl acetate			0.001[c]						
neryl acetate	0.1[m]	0.2[ww]	0.06,[c] 0.1[d]	0.1,[s] 0.3,[t] 0.4[m]	2.5,[u] 3.1[y,hh,jj]	0.01[b]			0.4[u,v]
neryl formate	0.1[n]				0.2[n]				
octyl acetate	0.1[m]	0.1[ww]	0.02,[c] 0.04[d]	0.04[g]					
perillyl acetate			0.008,[c] 0.1[d]						
terpinyl acetate			0.002,[c] 0.05[d]						
hydrocarbons									
α-bergamotene	0.06[m]			0.4[m]	2.5[u,y]	0.5[b]			0.27,[u,v,y] 0.9[u,w,y]
β-bisabolene				0.14,[s] 0.8,[m] 1.4[x]	2.5,[y,hh] 4.0[u]	0.9[b]			0.3,[u,v] 0.4[u,w]
α-cadinene									0.1[e,f]
γ-cadinene			0.02[c]						0.1[e,f]

Table I. (*Continued*)

Δ-cadinene	0.1[m]	0.1[ww]	0.2[d]					
camphene		0.2[kk]	0.01,[kk] 0.02,[q,kk] 0.4[a]	0.2,[m] 0.5[kk]	0.5[kk]	0.8[b]	0.4,[i] 0.9[i]		0.3,[f,i] 0.9,[i,j] 1.2[gg,kk]
Δ[3]-carene			0.004,[kk] 0.06[kk]					0.02,[r] 0.1[r]	0.1[gg,kk]
caryophyllene		0.3[ww]	0.02,[a] 0.04,[c] 0.09[d]	0.3[m]	2.5[u,y]	0.3[b]			0.27,[u,v,y] 0.9[u,w,y]
α-copaene		0.06[ww]	0.008,[a] 0.06,[c] 0.2[d]						
β-copaene		0.01[ww]							
β-cubebene	0.1[m]								
p-cymene	0.2[n]	0.4[kk]	0.2,[n] 0.4,[kk] 0.5,[ll] 0.8,[kk] 1.6,[c] 1.9,[kk] 2.8,[d] 8.2[a]	0.6,[s] 0.9,[mm] 1.1[kk]	0.5,[mm] 1.3,[n] 1.9[kk]	11.6[b]			0.3,[n] 0.4,[gg,kk] 2.6[e]
α-*p*-dimethylstyrene						0.5[b]			
β-elemene	0.05[l]	0.06[ww]	0.07,[p] 0.3,[c] 0.8[d]						0.62[u,w]
γ-elemene			0.2[p]						
Δ-elemene			0.06,[c] 0.1[p]						0.1[e]
farnesene	0.02,[l] 0.07[m]								
α- + β-humulene			0.05[c]	0.2[m]					0.27[u,w]
limonene	83,[o] 88,[cc,mm] 89,[cc,mm] 93,[m] 95,[k,l] 97[h]	86,[mm,ww] 93,[k] 95[kk]	65,[ll] 68,[a] 79,[kk] 83,[q] 84,[r] 87,[k] 88,[b] 89,[r] 92,[r] 93,[c,kk] 94[d,n,kk]	54–76,[x] 58,[mm] 63,[m] 68,[mm] 72,[g,t] 74,[mm] 76,[kk] 80[s]	47,[mm] 48,[u,y,hh] 64,[h,kk]	60[b]	74,[i] 79,[i] 86[i]	25,[r] 27,[r] 32[r]	67,[gg,kk] 82,[u,w] 83,[i,j] 84,[f,i] 85,[e,f] 92,[u,v] 93[n]
longifolene			0.01[a]						
α-muurolene									0.1[e]
myrcene	0.9,[n] 1.1,[cc] 1.2,[mm] 1.3,[mm] 1.4,[k] 2.0,[l] 2.1[m]	1.4,[mm] 1.9,[k] 2.1[ww]	1.2,[a,k,ll] 2.0,[c] 2.1,[n] 2.3,[p] 2.6,[r] 2.8,[r] 2.9,[r] 7.6[q]	0.9,[mm] 1.1,[mm] 1.7,[mm] 2.1,[m] 12.7[g,nn]	0.7,[mm] 10.3[n]	0.8[b]			1.2,[n] 1.8,[f,i] 2.1,[u,w] 2.6,[i,j] 3.1[e,f]

Table I. (*Continued*)

	sweet orange	grapefruit	mandarin	lemon	C.P. lime	distilled lime	bitter orange	bergamot	other species and hybrids
nonane						0.06[b]			
ocimene		0.4[k]	0.08[k]						
α-phellandrene		0.8[kk]	0.03,[kk] 0.05,[kk] 0.06,[kk] 0.1,[n] 0.3[ll]	0.2[kk]	0.2[n,kk]				0.2[gg,kk]
β-phellandrene		0.7[kk]	0.4,[kk] 0.5[kk]	0.8[kk]	0.9[kk]				0.9[gg,kk]
α-pinene	0.1,[cc,mm] 0.2,[n,cc] 0.3,[mm] 0.5,[k,l] 0.6[m]	0.2,[mm] 0.5,[ww] 0.6,[k] 1.6[kk]	0.3,[n] 0.6,[kk] 0.8,[c,p,kk] 0.9,[r] 1.0,[kk] 1.5,[kk] 2.0,[q] 2.5[ll]	0.4-2.5,[x] 1.3,[kk] 1.5,[mm] 1.7,[mm] 2.5,[m] 5.0[s]	1.2,[kk] 1.5,[u] 1.7,[mm] 2.2,[n] 2.4[y,hh]	0.8[b]		0.8,[r] 2.0[r]	0.3,[n] 0.4,[u,w] 0.5,[u,v] 0.6,[i,j] 12.9 [gg,kk]
β-pinene			0.9,[r,oo] 1.0,[r,ll,oo] 1.3,[a] 1.5,[r,oo] 2.1[q,oo]	2.2-9.8,[x] 2.8,[mm] 5.5,[mm] 6.7,[s] 10.5,[mm] 12.4,[t] 12.7,[g,nn] 13.9[m,oo]	10.1,[mm] 11.4,[u] 11.9[y,hh]	0.9[b]	0.9,[i] 2.4,[i] 5.5[i]	4.1[r,oo]	1.0,[f,i] 1.3,[e] 4.9[u,v]
sabinene	0.1,[m,c,mm] 0.2,[cc,mm] 0.6[k]	0.7[k]	0.4,[ll] 0.9,[r,oo] 1.0,[r,oo] 1.5,[r,oo] 2.1[q,oo]	0.5,[mm] 0.8,[mm] 1.5[mm]	1.6[mm]			4.1,[r,oo] 4.2[r,oo]	
α-selinene			0.02[a]						
γ-selinene			0.008[a]						
sesquicitronellene						0.2[b]			
β-sesquiphellandrene			0.2[c]						
α-terpinene		0.1[kk]	0.1,[k,kk] 0.4[kk]	0.7[kk]	0.8[kk]				0.2[gg,kk]
γ-terpinene	0.1[o,mm]	0.5,[kk] 0.8[mm]	2.1,[q] 2.2,[p] 2.7,[kk] 3.4,[kk] 3.8,[kk] 9.1,[a] 13.4,[kk] 17.3[ll]	2.9,[s] 5-14,[x] 7.3,[mm] 7.9,[mm] 8.1,[mm] 8.3,[t] 8.5,[g] 9.8,[m] 11.8[kk]	7.3,[mm] 14.3,[u] 15.3,[n] 16.3,[y,hh] 21.7[kk]	0.6[b]		4.7,[r] 4.9,[r] 5.5[r]	0.08,[u,v] 0.14,[gg,kk] 0.6,[n] 2.6,[e] 6.8[u,w]
terpinolene	0.1[m]		0.2,[q] 0.3,[k] 0.6,[a] 0.8,[ll] 1.1[c]	0.6,[m] 0.9[s]	0.6,[y,hh] 1.2[u]	0.8[b]			

Table I. (*Continued*)

α-thujene			0.5[a,ll]		0.4[u]			0.09[u,w]
toluene						0.002[b]		
tridecane						0.02[b]		
undecane						0.03[b]		
valencene	0.1,[l] 0.2[m]							
ketones								
carvone	0.02,[dd] 0.1[dd]		0.005,[c] 0.006,[d] 0.03[a]	0.04[s]				0.2[e]
methylheptenone				0.06[g]		0.006[b]		
nootkatone	0.01[pp]	0.3,[pp] 0.8[qq]	0.01[pp]	0.01[pp]	0.01[pp]		0.01[pp]	
miscellaneous								
1,4-cineole						1.8[b]		
1,8-cineole			0.002[a]			0.7[b]	0.8,[i] 5.7,[i] 8.1[i]	6.3,[f,i] 8.9[i,j]
dimethyl anthranilate			0.9[a]					
2,2-dimethyl-5-(1-methyl-1-propenyl)-tetrahydrofuran						0.3[b]		
cis-linalool oxide		0.03[k]						
trans-linalool oxide		0.03[k]						
pinol						0.02[b]		
thymol methyl ether			0.1[p]					0.5[u,v]
α,α,*p*-trimethylbenzyl alcohol methyl ether						0.2[b]		
2,6,6-trimethyl-2-vinyl-tetrahydropyran						0.16[b]		
nonvolatiles	1.0,[l] 1.3-3.1,[rr] 4.1[ss]	1.37,[tt] 6.0,[uu] 6.2-9.5,[rr] 7.5[ww]	3.5-4.7,[rr] 4.2,[p] 4.6[a]	0.47,[tt] 1.8-2.2[rr]	6.7,[tt] 7.5[hh]		0.23[tt]	0.56[tt,vv]

[a] Kugler and Kovats (1963), Sicilian mandarin (*Citrus reticulata Blanco*). [b] Kovats (1963). [c] Kita et al. (1969), *Citrus unshiu*. [d] Yamanishi et al. (1968), *Citrus unshiu*. [e] Hiroi and Takaoka (1973). [f] *Citrus iyo tangor*. [g] Bernhard (1960). [h] Neral and citronellol. [i] Maekawa et al. (1967). [j] *Citrus natsudaidai Hayata*. [k] Attaway et al. (1967), Hamlin orange, Dancy tangerine, and Marsh grapefruit, most-mature sample. [l] Shaw and Coleman (1974). [m] Ziegler (1971). [n] Scora et al. (1968), Ruby orange, Clementine mandarin, Sukega orangelo, Rangpur lime. [o] Attaway et al. (1968), mature sample only. [p] Moshonas and Shaw (1974). [q] Calvarano et al. (1974). [r] Huet and DuPuis (1969), mature samples only reported. [s] Fincke et al. (1974), control sample only. [t] Lund and Bryan (1976). [u] Shaw and Wilson (1976). [v] Rough lemon. [w] Lemon-lime hybrid. [x] Gunther (1968). Ranges are for a total of 19 samples analyzed. [y] α-Bergamotene and caryophyllene combined. [z] Moshonas, Shaw and Veldhuis (1972), Meyer lemon. [aa] Yokoyama et al. (1961), vanillin–piperidine and barbituric acid condensation methods only. [bb] Bitter Seville orange. [cc] Lifshitz et al. (1970). [dd] Stanley et al. (1961), using the total percent aldehyde values of Braddock and Kesterson (1976) to calculate percent of each aldehyde in oil (except lemon). [ee] Mexican lime. [ff] Braddock and Kesterson (1976). [gg] Temple orange. [hh] Shaw et al. (1971), Persian lime. [ii] α- and β-sinensals combined. [jj] Neryl and geranyl acetates combined. [kk] Ashoor and Bernhard (1967). [ll] D'Amore and Calabro (1966), Sicilian mandarin. [mm] Ikeda et al. (1962), California, Arizona and Texas (Meyer) lemon, California and Florida orange, Texas grapefruit, and cold-pressed lime (calculated from data presented). [nn] β-Pinene and myrcene combined. [oo] β-Pinene and sabinene combined. [pp] MacLeod and Buigues (1964). [qq] Keterson et al. (1971). [rr] Wolford et al. (1971). [ss] Calculated from data of Hunter and Brogden (1966). [tt] Stanley (1963), coumarin-like nonvolatiles only. [uu] Calculated from data of Fisher and Nordby (1965). [vv] Bergamot oil. [ww] Wilson and Shaw (1978).

Table II. Comparison of Quantitative Aldehyde Values from Valencia Orange Oil

aldehyde	percent of total oil: DNPH-TLC[a]	percent of total oil: GLC[b]
octanal	0.36	0.29
decanal	0.41	0.42
dodecanal	0.13	0.06
neral	0.05	0.07
geranial	0.06	0.15

[a] Calculated from the dinitrophenylhydrazone-thin-layer chromatography (DNPH-TLC) method of Braddock and Kesterson (1976): total aldehyde percent determined from total aldehyde DNPH's (method A). [b] Shaw and Coleman (1974).

relative peak heights in a gas chromatogram of Valencia orange peel oil. Of the values they reported, I have included in Table I of this review, only those for the mature sample analyzed. The authors showed, however, that the level of limonene reached a maximum of 90% when the fruit was sampled 3 months before the mature sample was prepared. Other hydrocarbons for which three or more values have been reported include myrcene, the second most abundant component of orange oil, and α-pinene and sabinene. There is a more than twofold difference in the values reported for myrcene and a sixfold difference for α-pinene and sabinene. These wide differences may reflect the difficulty in cleanly separating these relatively minor hydrocarbons from the major constituent, *d*-limonene on most gas chromatographic columns.

Aldehydes quantitated and listed in Table I include citral, neral, and geranial. Although citral is composed of neral plus geranial, a separate listing is shown for citral, as total citral, determined colorimetrically by the vanillin–piperidine or barbituric acid methods (Yokoyama et al., 1961). Since the colorimetric procedure measures all α,β-unsaturated aldehydes the citral values should be slightly higher than the combined neral–geranial values. Such is not always the case, but the fact that the reported geranial values differ more than twofold and the neral values vary *20-fold* make such comparisons difficult.

Total aldehydes in orange oil are generally around 1.5% (Kesterson et al., 1971). In the total aldehyde procedure the aldehydes are measured as decanal since it is often the major aldehyde, and others in significant quantity have both higher and lower molecular weights than decanal. The two main aldehydes in orange oil, octanal and decanal, have been quantitated by many workers. Reported values for octanal vary from 0.2–2.8% and for decanal 0.1–0.7%. The higher values reported for decanal in Table I are probably more accurate than the lower ones. Generally, the octanal value averages approximately 70% of the decanal value in late-season orange oils (Shaw and Coleman, 1974). The one unusually high value for octanal (2.8%) was determined from relative peak heights in a GLC curve, but the reason for such a high value is not clear. No value for decanal or total aldehydes was reported in that study for comparison.

Quantitative data for aldehydes from orange oil determined by GLC analysis of whole oils using response factors and correcting for percent nonvolatiles present (Shaw and Coleman, 1974) compare closely with quantitative estimates made be Braddock and Kesterson (1976) on the basis of thin-layer chromatography of dinitrophenylhydrazone derivatives (see Table II).

Other aldehydes for which several quantitative values are reported include the other straight-chain aldehydes from C_6 through C_{14}. When quantitative values have been reported, saturated straight-chain aldehydes with even carbon numbers (e.g., octanal, decanal, dodecanal) are known to be present in greater quantities than adjacent members with odd carbon numbers (e.g., nonanal, undecanal), and this finding is reflected in the quantitative results shown in Table I. The two isomeric aldehydes α- and β-sinensal have been quantitated at less than 0.1% each, and the β isomer predominates.

The alcohols in orange oil believed most important to flavor that have been quantitated are linalool, α-terpineol, and terpinen-4-ol. Reported values for linalool range from 0.3–5.3%, and the unusually high value of 5.3% was determined from relative peak heights in a gas chromatogram (Attaway et al., 1968). In that study, the percentage of linalool in the peel oil from Valencia oranges was shown to decrease steadily from 36.6% in immature fruit to 5.3% in mature fruit 1 year later. Both α-terpineol and terpinen-4-ol are known degradation products of the main oil component, *d*-limonene (Slater and Watkins, 1964), and could be formed if the peel oil is allowed to remain in contact with the acidic juice for any length of time during processing (Kefford and Chandler, 1970). α-Terpineol is also a known product of microbial degradation of limonene (Murdock and Hunter, 1968) and a contributor to off-flavor in stored orange juice (Tatum et al., 1975). These two alcohols were found at low levels in most oils listed in Table I.

Other oxygenated compounds quantitated include three esters and two ketones. Ketones quantitated in orange oil were carvone, a known oxidation product of limonene (Verghese, 1968), found at 0.1% or less, and nootkatone, found at extremely low levels of <0.01% (MacLeod and Buigues, 1964).

The amount of nonvolatiles present, as determined from distillation residue, has been reported to be from 1.0–4.1%, depending on distillation method. Wolford et al. (1971) found a variation of 1.3–3.1% nonvolatiles in several samples of orange oil using a standard method of measuring evaporation residue for all samples. This variation emphasizes the need to determine percent nonvolatiles on each sample if nonvolatiles are to be accounted for in a quantitative determination. Extent of winterizing the oil may account for the variation observed since more nonvolatiles precipitate as winterizing is prolonged (Swisher and Swisher, 1977).

Grapefruit. Much less quantitative data exists for individual components in grapefruit oil than for orange, mandarin, or lemon oils. Only three components, *d*-limonene, α-pinene, and octanal, have more than two quantitative values reported for oil from mature fruit. Reported values for *d*-limonene range from 86–95%; for α-pinene, from 0.2–1.6%; and for octanal, from 0.3–0.6%. As in orange oil, myrcene is the second most abundant component, and the levels in both oils are about the same. γ-Terpinene is reported in grapefruit oil at 0.5–0.8%—levels significantly higher than those reported for orange oil. α-Phellandrene and β-phellandrene were found in one study to be present at about a 1% level (Ashoor and Bernhard, 1967) even though these two monoterpene hydrocarbons had not previously been reported as grapefruit oil constitutents (Shaw, 1977).

Individual aldehydes of grapefruit oil have not been extensively quantitated, although total aldehyde content is one measure of oil quality (Kesterson, et al., 1971). The combined levels of neral and geranial, determined individually, are approximately three times the level of total citral calculated from colorimetric determination (footnote *dd*, Table I). The limited quantitative data on the two

major aldehydes in grapefruit oil, octanal and decanal, suggest that octanal is present at slightly higher concentration than decanal (Braddock and Kesterson, 1976). The C_7, C_9, C_{12}, and C_{14} straight-chain aldehydes have also been quantitated.

Only four alcohols in grapefruit oil have been quantitated and that was by relative peak height determination in a single study that followed the change in oil composition with maturity (Attaway et al., 1967). The authors reported octanol to be the major alcohol present, with measurable quantities of α-terpineol and terpinen-4-ol also present. Linalool comprised 0.4% of the oil in that study. Kesterson and Hendrickson (1964) studied the composition of terpeneless oils prepared from red and white grapefruit oils and found measurable quantities of linalool in only the red grapefruit oil samples. In another study also on terpeneless oils, from white grapefruit, these same authors (1967) found that a measurable quantity of linalool in freshly prepared oil gradually disappeared during 1 year of storage. Thus, the quantity of linalool present in a grapefruit oil sample may depend on the length of time the oil has been stored.

Moshonas (1971) analyzed the carbonyl fraction from grapefruit oil and found six acetate esters to be major constituents of this fraction. Wilson and Shaw (1978) reported quantitative data on esters in grapefruit oil.

The level of nootkatone in a grapefruit oil is regarded as an indication of oil quality (Kesterson et al., 1971). Kesterson et al. (1965) noted an increase in nootkatone content in grapefruit oil as the fruit matured and reported a range of 0.3–0.8% nootkatone in mature oil samples.

The percentage of nonvolatiles in grapefruit oil is high relative to that in orange or mandarin oil. This is partially due to the coumarins and psoralens present; neither occurs in significant quantity in orange and mandarin oils. Stanley (1963) reported 1.37% coumarin-like compounds in grapefruit oil and stated that this figure was probably low. The precise nature of the other compounds that make up the approximately 7% nonvolatiles in grapefruit oil is not known, but carotenoids, tocopherols, flavonoids, and hydrocarbons are known to be present (Shaw, 1977).

Mandarin. Numerous quantitative values for individual components of mandarin oils have been determined, and a wider variation in results exists than with any of the other four major citrus cultivars. This variability probably reflects the large number of mandarin species (Reuther et al., 1967). The term "tangerine" is used interchangeably with "mandarin" in the United States, but the latter is an older term and much more widely used.

Such diverse quantitative values have been reported for mandarin oils that their interpretation must be based on the source of each oil. In particular, the major hydrocarbons differ widely among different samples. The major component, *d*-limonene, varies from 65–94% of the oil. The three oil samples which contained relatively low levels of limonene (65–79%) also contained abnormally high levels of γ-terpinene. Two of these oils were from Sicilian mandarin; Clementine (a hybrid) is a major variety grown in Sicily (Reuther et al., 1967). However, for Clementine oil, Calvarano et al. (1974) reported a much lower level of γ-terpinene and a relatively high level of myrcene, and Huet and DuPuis (1969) found a relatively high level of linalool, but no γ-terpinene. Thus, the reason for the different levels of monoterpenes in mandarin oils cannot be determined from the descriptions of the oils in the literature, but most likely the reason is varietal differences.

One of the most comprehensive studies on quantitative analyses of citrus oils was that on Sicilian mandarin oil by Kugler and Kovats (1963). They quantitated 46 components of the oil using a combination of distillation, extraction, column chromatography, and GLC techniques. Many compounds represented less than 0.1% of the total oil. Thymol and dimethyl anthranilate were cited as important to mandarin flavor, but no evidence was presented to support this claim.

Aldehydes are important to mandarin flavor, and they are the major class of oxygenated components in Dancy tangerine oil (Braddock and Kesterson, 1976). The two main aldehydes are octanal and decanal. A wide range of values for both octanal (0.04–0.3%) and decanal (0.04–0.9%) have been reported. The C_9, C_{11}, C_{12}, and C_{14} straight-chain aldehydes have also been quantitated, but their levels are generally lower than those of the C_8 and C_{10} analogues. α-Sinensal was found in one study at 0.2% of the oil. The β isomer was also present, but in only trace quantities. Thus, mandarin oil contrasts with orange oil, which contains more of the β isomer. Neral and geranial were both quantitated in one study at 0.06%, but in other studies geranial was found in levels up to 0.3%. Citronellal and perillaldehyde have been reported in amounts up to 0.1% each.

Eighteen alcohols in mandarin oils have been quantified and most were found in quantities of <0.1%. The major alcohol seems to be linalool. It has been quantitated ten times with values ranging from 0.07 to 6.1% of the oil from mature fruit. Huet and DuPuis (1969) showed that the linalool content decreased in Clementine oil during maturity from 33 to 6.1% of the oil. Thus, the maturity of the sample as well as the variety may be important in determining linalool content. Two possible indicators of oil quality, terpinen-4-ol and α-terpineol, varied markedly in content. The terpinen-4-ol level ranged from 0.06–0.3% and the α-terpineol level from 0.03–1.1%. The level of thymol, believed to be an important flavor compound in mandarin, varied from 0.04–0.2%. Whether these marked differences in concentration are due to variety, method of oil preparation, analytical procedures or other factors cannot be determined from literature descriptions of the oils and of processing and analytical techniques used.

Esters, ketones, and acids are present in only trace quantities in mandarin oil. Nine esters have been quantitated, but only three, citronellyl acetate, geranyl acetate, and neryl acetate, are reported at levels approaching 0.1%. The two ketones that have been quantitated are carvone and nootkatone, both at approximately 0.01%. The acids quantitated are all straight-chain hydrocarbon derivatives except one (citronellic); octanoic and decanoic acids predominate.

Other compounds of possible flavor importance that have been quantitated are dimethyl anthranilate and thymol methyl ether. Thymol methyl ether has an aroma similar to that of thymol, but its significance to mandarin flavor has not been studied.

The nonvolatile portion of mandarin oil ranges from 3.5–4.7% of the oil and consists mainly of flavonoids, especially tangeretin (Shaw, 1977). The percentage of nonvolatiles present would undoubtedly depend on the degree of winterizing the oil sample has undergone since the flavonoids precipitate readily from the oil upon standing at cool temperatures.

Lemon. The quantitative composition of lemon oil differs markedly from that of orange, grapefruit, and mandarin. Limonene is still the major hydrocarbon, but is in generally lower quantity than in the other oils. On the other hand, certain other hydrocarbons, especially β-pinene and γ-terpinene, are generally found in much

greater quantities. However, there is a wide variation in values reported for all three of these monoterpenes, which are the main components of most lemon oils. Other hydrocarbons that are reported to be present at 0.5% in at least one oil sample are the monoterpenes camphene, *p*-cymene, myrcene, β-phellandrene, α-pinene, sabinene, α-terpinene, and terpinolene and one sesquiterpene, β-bisabolene. Thus, a much wider variety of terpenes are present in lemon oil at levels approaching 1% than in orange, grapefruit, or mandarin oils.

The amount of total aldehydes in lemon oil is very important to oil quality, principally because of the citral content. Even though citral is comprised of two isomers, neral and geranial, the citral values (usually determined colorimetrically as total aldehydes) are listed separately in Table I from individual neral and geranial values (usually determined by gas chromatography). Total citral content ranges from 2.0–13.2%, but the better quality oils have citral values not exceeding ca. 4–5% (Ziegler, 1971; Gunther, 1968). Values for neral and geranial, determined individually, should total the citral value; but the wide range of values reported for citral and for neral (0.4–1.3%) and geranial (0.6–2.3%) and the difference in analytical methods used (colorimetric vs. gas chromatographic) make such a comparison difficult. Other aldehydes quantitated include the straight-chain derivatives from C_7 through C_{12}, with nonanal apparently present in greatest quantity. In contrast, octanal and decanal predominate in orange, grapefruit, and mandarin oils.

Alcohols present in lemon oils in the 0.1–0.5% range are citronellol, linalool, terpinen-4-ol, α-terpineol and tetrahydrogeraniol. Terpinen-4-ol and α-terpineol levels might be expected to vary considerably in lemon oil because the strongly acidic juice can catalyze the hydration of limonene and other monoterpenes to these two alcohols if the oil comes in contact with the juice during processing. Few quantitative values for these two alcohols have actually been reported, but the two reported values for terpinen-4-ol in Table I differ 40-fold (0.01 and 0.4%).

Esters present in lemon oil are believed important in providing the full-bodied desirable lemon flavor (Swisher, 1966). The predominant esters are neryl and geranyl acetates (Ziegler, 1971; Fincke et al., 1974).

The nonvolatile portion of lemon oil accounts for up to 2% of the total oil. It apparently contains mostly coumarins and psoralens, which act as natural antioxidants and are reported to stabilize the oil during storage.

Lime (Cold-Pressed and Distilled). The composition of cold-pressed lime oils is quite similar to that of cold-pressed lemon oil (Kefford and Chandler, 1970), with a few notable quantitative differences (see Table I): the citral content of good-quality lime oil seems to be higher; octanal, rather than nonanal, is the main straight-chain aldehyde; neryl and geranyl acetates are in much higher quantities; the nonvolatile portion is considerably higher (over 7% of the cold-pressed oil).

Differences between cold-pressed and distilled lime oils are pronounced. Distilled lime oil is obtained by distillation of a slurry of crushed whole limes; thus, the acidic juice and oil are in contact during heating, and artifacts form. Consequently, distilled lime oil has greatly reduced quantities of citral, β-pinene, and γ-terpinene and greatly increased quantities of *p*-cymene, terpinen-4-ol, and α-terpineol; and it has a more terpene-like flavor than that of the cold-pressed oil (Slater and Watkins, 1964). There are virtually no nonvolatile coumarins in the distilled oil to protect it from oxidation during storage, and the oil is already much changed by the distillation process.

Sour or Bitter Orange. Although relatively little quantitative data on bitter orange oil has been reported, the oil appears to contain higher quantities of the following compounds than sweet orange oil: myrcene and α-pinene (in one sample), nonanal, octanal, citronellyl acetate and formate, geranyl formate, linalyl acetate, and 1,8-cineol. The nonvolatile portion of the oil contains coumarins (0.23% of the oil) not found in sweet orange oil.

Bergamot. Although Bergamot is considered a variety of bitter orange (Reuther et al., 1967), the oil composition is listed separately in Table I because it is so dramatically different from that of the other citrus oils studied. The most striking differences are the extremely low limonene (25–32%) and extremely high linalool (16–41%) and linalyl acetate (11–44%) contents. Bergamot oil contains much more terpinen-4-ol, α-terpineol, sabinene, and γ-terpinene than sweet or bitter orange oil. Stanley (1963) found more nonvolatile coumarin-like compounds in Bergamot oil than in bitter orange oil.

Other Species and Hybrids. Oil composition of certain species of minor commercial importance and of hybrids is especially important for chemotaxonomic studies. The differences among the quantitative data suggest that chemotaxonomic conclusions are meaningful only if the quantitative data were obtained by the same procedure for fruits of the same maturity from the hybrid plant and its suspected parent plants. The following brief comments on other species and hybrids can be made on the basis of present qualitative data available.

Meyer lemon is believed to be a cross between *Citrus limon* and *C. sinensis* (Swingle, 1967) and the oil contains a high level (6%) of thymol (Moshonas et al., 1972). So far this compound has been found in mandarin and lime oils, but not in those of the suspected parents.

Rough lemon oil differs from the typical lemon oil of commerce in having a high limonene content and low levels of citral, β-pinene and γ-terpinene. This oil was analyzed by Shaw and Wilson (1976) in a study involving the composition of a cross between rough lemon and Persian lime. The lemon–lime cross had a higher content of linalool than either parent and also had large amounts of thymol and thymol methyl ether, neither of which is found in either parent.

Temple orange is believed to be a tangor (*Citrus reticulata* × *C. sinensis*) (Swingle, 1967). However, the oil contains less limonene and more α-pinene than either orange or mandarin oil. Another difference is the higher total aldehyde content (Braddock and Kesterson, 1976), but the quantitative data on individual aldehydes show no unusually high values except possibly for undecanal in one study. However, the amount of undecanal was not large enough to be quantitated in another study (Braddock and Kesterson, 1976).

Citrus iyo (tangor) was studied by Hiroi and Takaoka (1973), and 16 peel oil components were quantitated. In this tangor a higher level of limonene and a lower level of α-pinene were found than reported for Temple orange. Higher amounts of several alcohols and of myrcene, *p*-cymene, γ-terpinene, and carvone were found than had been reported for orange, mandarin, or Temple. When Maekawa et al. (1967) quantitated 19 oil components from *Citrus iyo*, they found limonene and β-pinene levels comparable to those found by Hiroi and Takaoka, but a higher level of linalool was detected as well as over 6% of 1,8-cineole, a compound not detected by the latter workers.

Orlando tangelo (hybrid of Duncan grapefruit and Dancy tangerine) was studied by Braddock and Kesterson (1976) with regard to aldehyde composition. Quantities

of aldehydes were found comparable to those reported for both grapefruit and mandarin oils.

Citrus natsudaidai Hayata exhibits characteristics of pummelo or bitter orange and mandarin (Reuther et al., 1967). Oil composition studies (Maekawa et al., 1967) showed high levels of 1,8-cineole and octanal, which were also present in bitter orange oils examined by the same workers.

Leaf oil chemotaxonomy was studied by Kesterson et al. (1964) and by Scora et al. (1976). The latter workers observed that quantities of components from hybrid leaf oils were not necessarily intermediate between those of the two parents. In a similar study on *Poncirus trifoliata* essential oils, Scora et al. (1966) observed that the maturity of the hybrid oil was a critical factor in correlating quantities of component to those present in the parents.

Quantitative analysis of citrus essential oils has reached a point where basic differences between oil samples, processing procedures, and analytical techniques—including methods of quantitation—need to be determined before truly reliable results can be obtained and compared. For meaningful conclusions in chemotaxonomic or maturity studies all samples must be analyzed by the same method. At present gas chromatography of whole oils without prior separation steps (using GLC response factors and correcting for percent nonvolatiles) seems to provide the most accurate data when varying classes of compounds need to be quantitated. When a single class (aldehydes) is quantitated, the procedure of Braddock and Kesterson (1976) seems the most accurate.

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Received for review June 12, 1978. Accepted October 16, 1978. Mention of a trademark or proprietary product is for information only and does not recommend its approval by the U.S. Department of Agriculture to the exclusion of others which may also be suitable.