# Comparative Study of the Essential Oils of Key and Persian Limes

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Within the acid lime classification there are two groups whose essential oils are of commercial interest: the Key, Mexican or West Indian lime (*Citrus aurantifolia* Swingle) whose fruit is small and seeded and the Persian or Tahiti lime (*Citrus latifolia* Tanaka) with larger seedless fruit. According to the literature, the Persian lime is a hybrid, one of the parents being the Key lime and the other most likely a lemon or a citron with the latter being the more probable (Sacco and Calvarano, 1981).

The most important areas for Key lime production and processing are Mexico, Peru and Haiti. Florida and Brazil are the major producers of the Persian lime.

Two common methods are used for oil production: distillation and centrifugation. To obtain the distilled oil the whole fruit is fed into a screw press where the oil cells within the peel are ruptured by pressure resulting in a juice-oil emulsion. This emulsion is then steam distilled to recover the oil. The cold-pressed oil is obtained by passing the fruit through machinery which ruptures the oil cells either by puncturing or rasping. The oil is then washed away with water, forming an oil-water emulsion which can be separated by centrifugation. Cold-pressed Persian lime and the Type B Key lime oils are obtained by this method. A different kind of oil, type A Key lime is obtained when the oil-juice emulsion obtained in the screw press is centrifuged instead of distilled.

It was considered of interest to carry out a comparative study on Key and Persian lime oils since they exhibit significant organoleptic differences. The Persian lime is often described as "weaker" and "flatter" when compared with Key lime oils. Sometimes these oils are sold in the market or presented in papers using the term "lime oil" without any further distinction regarding the variety used to obtain the oil.

## Experimental

*Samples.* Key lime oil. Three commercial samples of oil produced in Mexico were obtained: distilled; cold-pressed type A (from oil-juice emulsion) and cold-pressed type B (from oil-water emulsion).

Persian lime oil. Two commercial samples of oil produced in Florida were obtained: distilled and cold-pressed (from oil-water emulsion).

*Physical and chemical characteristics.* Specific gravity, optical rotation and refractive index were determined using the E.O.A. methods (Essential Oil Association of U.S.A., 1975). Aldehyde content was determined using the hydroxylamine/tertbutyl alcohol method as described in the Food Chemicals Codex (1981).

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**Fig.1 Cold-pressed lime oils on SE-30**

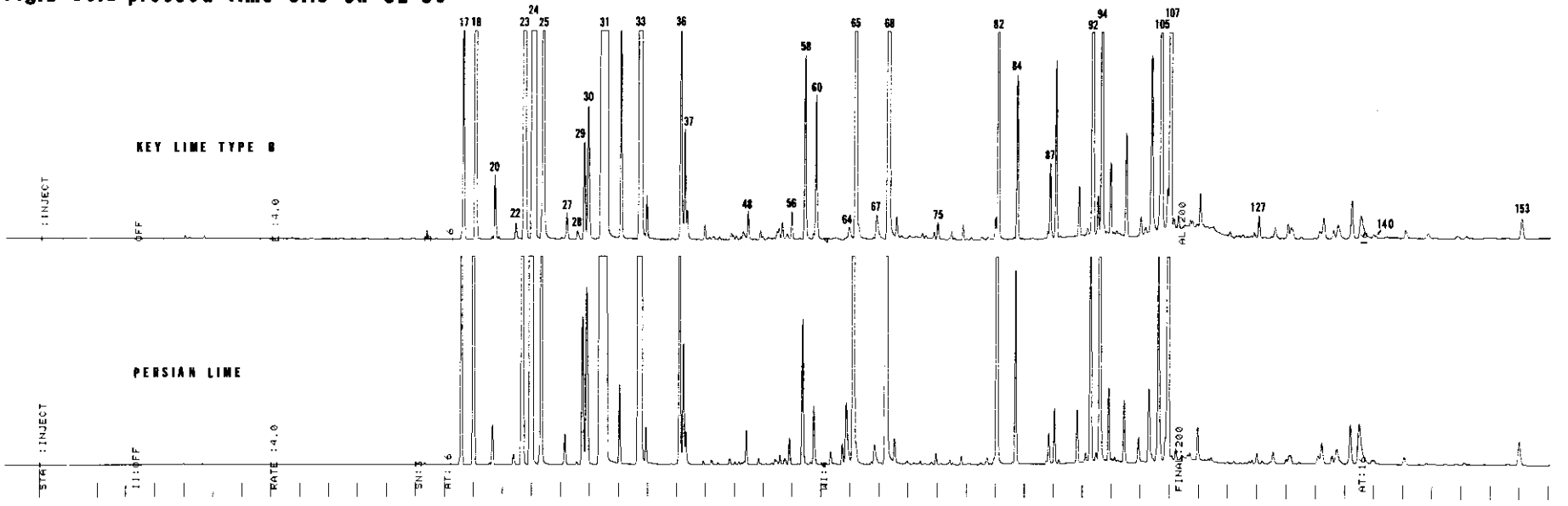
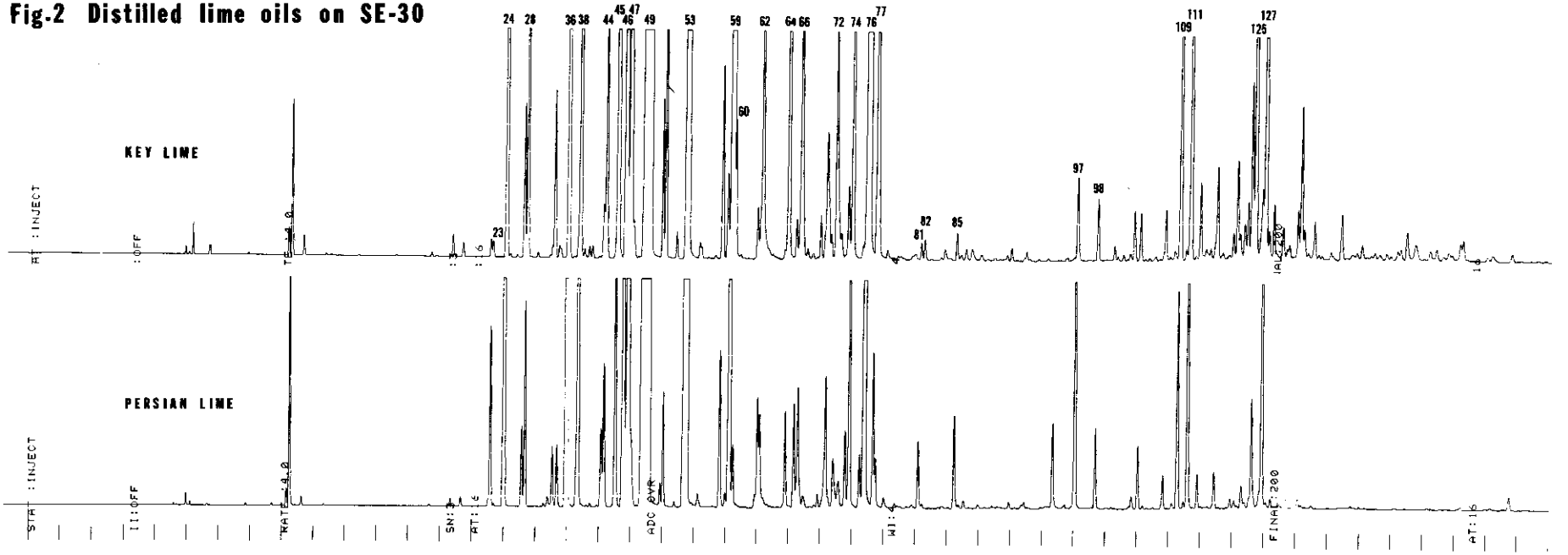



**Fig.2 Distilled lime oils on SE-30**



*Column chromatography.* The oil was fractionated into oxygenated compounds and hydrocarbons by column chromatography on silical-gel impregnated with 0.7% Carbowax 1540 (Tapanes et al., 1971) using hexane and ethyl acetate to elute hydrocarbons and oxygenated compounds respectively.

*Isolation of carbonyl compounds.* Carbonyl compounds were isolated using Girard T reagent as described by Ikeda et al., (1961).

*Gas chromatography.* Gas chromatograms were run on a Varian Vista 6000 gas chromatograph with flame ionization detector using two different columns: Fused silica capillary, 60 m. long, 0.25 mm. I.D., 0.25 um. film of SE-30; and Glass capillary, 60 m. long, 0.25 mm. I.D., 0.25 um. film of SP-1000. The linear gas rate was 23 cm/sec and the temperature programming: 75°C for 8 min., then 4° C/min up to 200°C, final isothermal hold 25 min. Sample size: 0.2 ul, split: 150:1.

Peak identification was made based on the compounds reported in the literature as being present in lime oils (Azzouz et al., 1971; Calvarano and Gallino, 1975; Huet et al., 1978; Kovats, 1963; McHale, 1980; Moshonas and Shaw, 1980; Shaw et al., 1971; Shaw and Wilson, 1976; Tapanes et al., 1971 and Ziegler, 1971) and comparing their Kovats indices with those of the peaks in the chromatograms on both polar and non-polar columns. Peak identity was corroborated by their appearance in the chromatograms of the isolated fractions (hydrocarbons, oxygenated and/or carbonyl compounds).

## Results and Discussion

### Cold-Pressed Oils

The differences in the three kinds of oils' physical and chemical characteristics can be seen in Table I.

Chromatograms of the three samples were made and although no qualitative differences were found, important quantitative differences are shown in Table II. The chromatograms of Key type B and Persian cold-pressed lime oils are shown in figure 1.

*Key lime A vs. B.* Certain labile components such as sabinene, citronellal, neral and geranial show a lower percentage in type A oil. This is because type A oil has been exposed to the acidic

**Table I. Physical and Chemical Characteristics of Cold-Pressed Oils**

| | Key Lime Type A | Key Lime Type B | Persian Lime |
|---|---|---|---|
| Specific gravity 25/25 C | 0.878 | 0.879 | 0.864 |
| Optical rotation at 20 C | +36.5 | * | +38.0 |
| Refractive index at 20 C | 1.4854 | 1.4857 | 1.4795 |
| Aldehydes as citral | 4.6% | 5.3% | 5.2% |

* Non obtainable due to the dark color of the oil

juice for several minutes during processing. p-Cymene formed by the acid catalyzed cyclization of citral is higher in type A.

*Key lime B vs. Persian lime.* We chose Key lime type B to compare with Persian as both have been obtained by a similar method.

The largest quantitative differences are found in camphene, sabinene, beta-pinene, decanal, undecanal, dodecanal, beta-caryophyllene and beta-sesquiphellandrene which are much smaller in Persian lime. On the other hand d-limonene, gamma-terpinene, terpinolene, nerol and neryl acetate are considerably higher in Persian lime.

The components responsible for the largest differences are beta-pinene (7.3%), gamma-terpinene (6.2%) and d-limonene (5.1%).

*Carbonyl compounds fraction.* From the point

**Table II. Volatile Compounds Identified in Cold-Pressed Lime Oils**

| Peak # | Component | Key Lime Type A Area % | Key Lime Type B Area % | Persian Lime Area % |
|---|---|---|---|---|
| 17 | alpha-thujene | 0.355 | 0.369 | 0.525 |
| 18 | alpha-pinene | 2.230 | 2.164 | 2.013 |
| 20 | camphene | 0.100 | 0.094 | 0.057 |
| 22 | methylheptenone | 0.008 | 0.025 | 0.016 |
| 23 | sabinene | 3.044 | 3.282 | 2.071 |
| 24 | beta-pinene | 19.948 | 19.535 | 12.199 |
| 25 | myrcene + octanal | 1.173 | 1.222 | 1.353 |
| 27 | alpha-phellandrene | 0.043 | 0.046 | 0.049 |
| 28 | delta-3-carene + 1,4 cineol | 0.017 | 0.017 | 0.005 |
| 29 | alpha-terpinene | 0.166 | 0.162 | 0.241 |
| 30 | p-cymene | 0.616 | 0.268 | 0.370 |
| 31 | d-limonene + 1,8 cineol | 50.010 | 47.868 | 52.947 |
| 33 | gamma-terpinene + octanol | 7.098 | 8.227 | 14.454 |
| 36 | terpinolene + nonanal | 0.411 | 0.451 | 0.626 |
| 37 | linalool | 0.153 | 0.164 | 0.166 |
| 48 | citronellal | 0.026 | 0.042 | 0.047 |
| 56 | terpinen-4-ol | 0.223 | 0.043 | 0.040 |
| 58 | alpha-terpineol | 0.260 | 0.299 | 0.231 |
| 60 | decanal | 0.198 | 0.238 | 0.091 |
| 64 | nerol | 0.023 | 0.036 | 0.143 |
| 65 | neral | 1.432 | 1.821 | 0.731 |
| 67 | geraniol | 0.030 | 0.072 | 0.052 |
| 68 | geranial | 2.362 | 2.991 | 2.769 |
| 75 | undecanal | 0.028 | 0.026 | 0.018 |
| 82 | neryl acetate + unknown hydrocarbon | 0.471 | 0.509 | 1.533 |
| 84 | geranyl acetate | 0.275 | 0.254 | 0.269 |
| 87 | dodecanal | 0.113 | 0.123 | 0.047 |
| 92 | beta-caryophyllene | 0.935 | 1.164 | 0.571 |
| 94 | alpha-bergamotene | 1.271 | 1.203 | 1.144 |
| 105 | beta-sesquiphellandrene | 1.554 | 1.888 | 0.445 |
| 107 | beta-bisabolene | 2.107 | 1.966 | 1.749 |
| 127 | tetradecanal | 0.040 | 0.040 | 0.018 |
| 140 | pentadecanal | 0.006 | 0.008 | 0.007 |
| 153 | hexadecanal | 0.059 | 0.065 | 0.060 |

**Table III. Carbonyl Compounds Isolated from Cold-Pressed Lime Oils**

| Peak # | Component | Key Lime Type A Area % x C | Key Lime Type B Area % x C | Persian Lime Area % x C |
|---|---|---|---|---|
| 22 | methylheptenone | 0.004 | 0.018 | 0.008 |
| 25 | octanal | 0.041 | 0.065 | 0.023 |
| 31 | unknown | 0.033 | 0.048 | 0.026 |
| 36 | nonanal | 0.036 | 0.033 | 0.018 |
| 37 | unknown | 0.030 | 0.034 | 0.030 |
| 48 | citronellal | 0.015 | 0.033 | 0.030 |
| 56 | unknown | 0.039 | 0.021 | 0.017 |
| 58 | unknown | 0.066 | 0.099 | 0.056 |
| 60 | decanal | 0.242 | 0.290 | 0.113 |
| 63 | unknown | 0.006 | - | 0.036 |
| 65 | neral | 1.332 | 1.604 | 1.547 |
| 68 | geranial | 2.317 | 2.805 | 2.850 |
| 75 | undecanal | 0.031 | 0.031 | 0.021 |
| 87 | dodecanal + ? | 0.104 | 0.112 | 0.056 |
| 94 | alpha-bergamotene | 1.271 | 1.203 | 1.144 |
| 104 | tridecanal | 0.007 | - | 0.006 |
| 127 | tetradecanal | 0.027 | 0.030 | 0.021 |
| 140 | pentadecanal | 0.005 | - | 0.011 |
| 153 | hexadecanal | 0.034 | 0.043 | 0.067 |
| 163 | unknown | 0.030 | - | 0.011 |
| | straight chain aldehydes | 0.528 | 0.604 | 0.336 |
| | citral | 3.649 | 4.409 | 4.397 |
| | aldehydes by titration (C) | 4.6% | 5.3% | 5.2% |

of view of odor the carbonyl compounds, which are mostly aldehydes, are the most important class of compounds found in lime oil. In the chromatogram of the whole oil they are often unresolved from the terpenes, rendering their quantitation very difficult. Consequently, it was decided to isolate them.

The carbonyl containing fraction of each oil was run on the gas chromatograph and the results are shown in Table III. The area percent found for each component was corrected by the total aldehydes content found for each oil (by titration) to obtain a figure closer to the percent of each compound in the total oil.

*Key lime A vs. B.* In general type A oil exhibits a lower content of aldehydes than type B. Methylheptenone is remarkably lower in type A.

*Key type B vs. Persian lime.* A comparison between Key lime type B and Persian lime shows that with the exception of hexadecanal all other carbonyl compounds are found at a lower level in Persian lime.

The citral (neral + geranial) content in Persian lime is about the same as in Key lime, but Persian lime has only one half the amount of straight chain aldehydes. Citral accounts for 79.3% of the total carbonyl compounds in Key lime type A, 83.2% in type B, and 84.6% in Persian lime.

## Distilled Oils

The physical and chemical characteristics of distilled lime oils are shown in Table IV.

The chromatograms of the two samples were made (figure 2) and as in the case of cold-pressed oils, only quantitative differences were found. The area percent of the identified peaks are shown in Table V.

It can be seen that Persian lime contains a higher content of alpha-thujene, alpha-pinene, beta-pinene, d-limonene, gamma-terpinene, neral, geranial and neryl acetate. In contrast the

**Table IV. Physical and Chemical Characteristics of Distilled Lime Oils**

| | Key Lime | Persian Lime |
|---|---|---|
| Specific gravity 25/25 C | 0.860 | 0.851 |
| Optical rotation at 20 C | +37.3 | +52.2 |
| Refractive index at 20 C | 1.4763 | 1.4744 |
| Aldehydes as citral | 0.6% | 1.0% |

**Table V. Volatile Compounds Identified in Distilled Lime Oils**

| Peak # | Component | Key Lime Area % | Persian Lime Area % |
|---|---|---|---|
| 23 | alpha-thujene | 0.020 | 0.212 |
| 24 | alpha-pinene | 1.194 | 2.245 |
| 28 | camphene | 0.477 | 0.245 |
| 36 | beta-pinene | 2.229 | 6.035 |
| 38 | myrcene | 1.281 | 1.562 |
| 44 | alpha-phellandrene | 0.371 | 0.203 |
| 45 | 1,4 cineol + delta-3-carene | 1.963 | 0.391 |
| 46 | alpha-terpinene | 2.526 | 1.046 |
| 47 | p-cymene | 1.792 | 1.507 |
| 49 | d-limonene + 1,8 cineol | 48.850 | 59.016 |
| 53 | gamma-terpinene | 10.908 | 16.089 |
| 59 | terpinolene + nonanal | 7.759 | 2.692 |
| 60 | linalool | 0.049 | 0.045 |
| 62 | alpha-fenchol | 0.663 | 0.296 |
| 64 | terpinen-1-ol | 0.837 | 0.124 |
| 66 | beta-terpineol | 0.691 | 0.189 |
| 72 | borneol | 0.419 | 0.095 |
| 74 | terpinen-4-ol | 0.799 | 0.461 |
| 76 | alpha-terpineol | 6.260 | 2.145 |
| 77 | gamma-terpineol (?) | 0.938 | 0.194 |
| 81 | neral | 0.023 | 0.091 |
| 82 | carvone | 0.017 | 0.007 |
| 85 | geranial | 0.036 | 0.121 |
| 97 | neryl acetate + unknown hydrocarbon | 0.115 | 0.542 |
| 98 | geranyl acetate | 0.076 | 0.094 |
| 109 | beta-caryophyllene | 0.616 | 0.293 |
| 111 | alpha-bergamotene | 0.830 | 0.563 |
| 125 | beta-sesquiphellandrene | 1.114 | 0.182 |
| 127 | beta-bisabolene | 1.416 | 0.626 |

proportion of 1,4 cineol + delta-3-carene, alpha-terpinene, terpinolene + nonanal, terpinen-1-ol, beta-terpineol, alpha-terpineol, gamma-terpineol (?) and beta-sesquiphellandrene in Persian lime are lower.

In both cases (distilled and cold-pressed) the oils obtained from Key and Persian limes show remarkable quantitative differences, large enough to explain those found in odor and flavor.

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