

## New techniques

# The Future of Citrus

## Developments in oil processing for citrus flavors and fragrances

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Citrus oils are some of the most commonly used flavor and fragrance ingredients. Containing characteristic properties, citrus oils impart fresh, zesty notes to many types of foods, beverages, household cleaning products, toiletries and fragrances. Perfumers often use grapefruit and orange together, for example, as a variation of the classical "eau de Cologne," while lemon and lime are being added to formulas to revitalize soft drink brands. Unfortunately, some commercially available citrus oils lack authentic natural notes, resulting in a disappointing flavor or fragrance. Recent advancements in processing techniques have been utilized to produce high-quality, well-balanced citrus oils.

### The Vital Components

Citrus oils are rich in nonpolar terpene hydrocarbons. The main terpene constituents are not always major contributors to the flavor or odor. It is the polar components (oxygenates) that usually contribute the most to flavor and odor. These include:

- Esters, such as ethyl butyrate
- Aldehydes, such as octanal, decanal, dodecanal, *trans*-2-hexenal, hexanal and citral
- Oxides, such as 1,8 and 1,4-cineole
- Alcohols, such as geraniol, nerol, borneol and terpinen-4-ol
- Acids, such as octanoic and decanoic acid

The odor and flavor of each of these natural molecules contribute to the complex interaction of components, creating a natural citrus odor and flavor profile. For example:

Component	Odor and flavor
ethyl butyrate	ripe fruit
<i>trans</i> -2-hexenal	leafy green
1,8-cineole	eucalyptus
borneol	earthy, camphoraceous
octanal	intense orange
citral	lemon

### Processing the Oil

Traditionally, vacuum distillation is used to concentrate citrus oils. During the process, heat is used to separate the complex mixture of natural components according to their volatility. This heat input can promote thermal degradation of the oil. To combat this problem, an alternative method using a low-temperature solvent-extraction process has been developed. This technique selectively separates components according to polarity differences. Volatile and nonvolatile flavor components, which may be lost during concentration by traditional distillation processes, therefore are retained. The result is a product with a rounder, smoother flavor profile.

Sometimes distilled products lack some of the non-volatile components that are reputed to provide stability to citrus oil products. These "back-end" materials are obtained specifically in the cold extraction process by the use of a proprietary solvent system.

### Extraction Steps

The cold extraction process includes a number of steps. First, oil is added to the solvent and mixed thoroughly. Each constituent of the oil has a different polarity, which affects its ability to dissolve in the solvent.

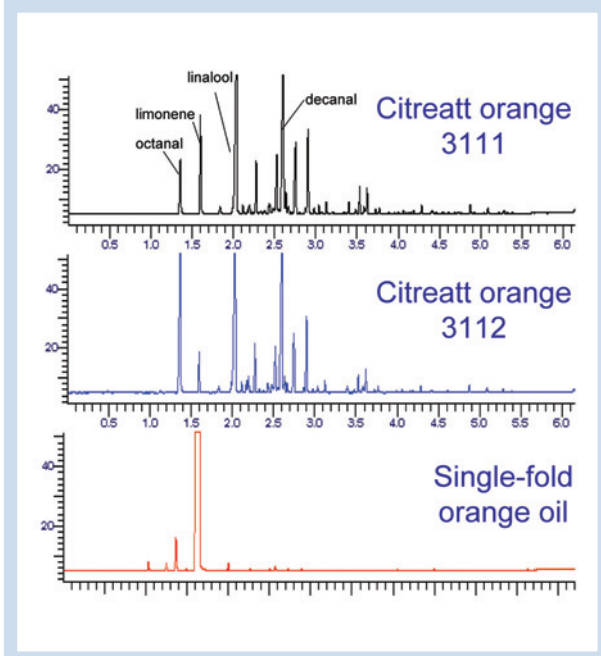
After a period of time, all components that will dissolve into the solvent have done so. The solvent is removed, and fresh solvent is added. Once again,

the components of the oils dissolve into the solvent at varying amounts according to their different polarities.

After several “washings” with solvent, the oil is exhausted. This byproduct of the process is known as washed oil. The different extracts in the solvent portion are combined, and the solvent is removed until only the concentrated oil is left. Compared to the starting oil, the concentration of the components is skewed in favor of the components that dissolve more readily in the solvent. The solvent system is, therefore, chosen to concentrate the organoleptically important

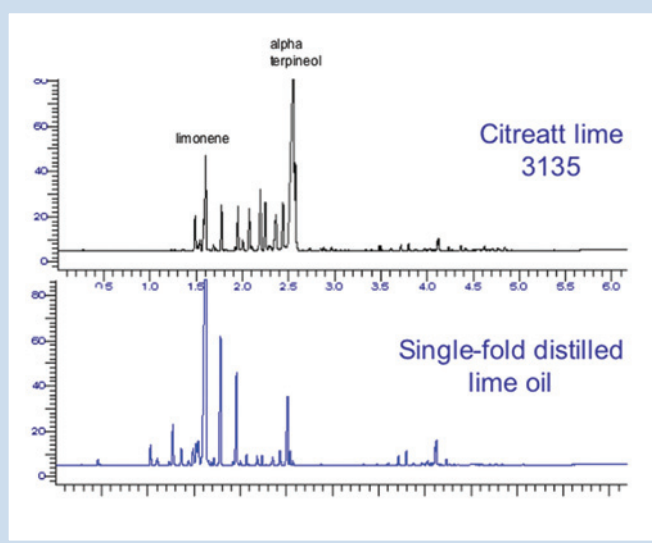
#### Citreatt orange versus single-fold orange oil

**F-1**



#### Citreatt lime versus single-fold distilled lime oil

**F-2**

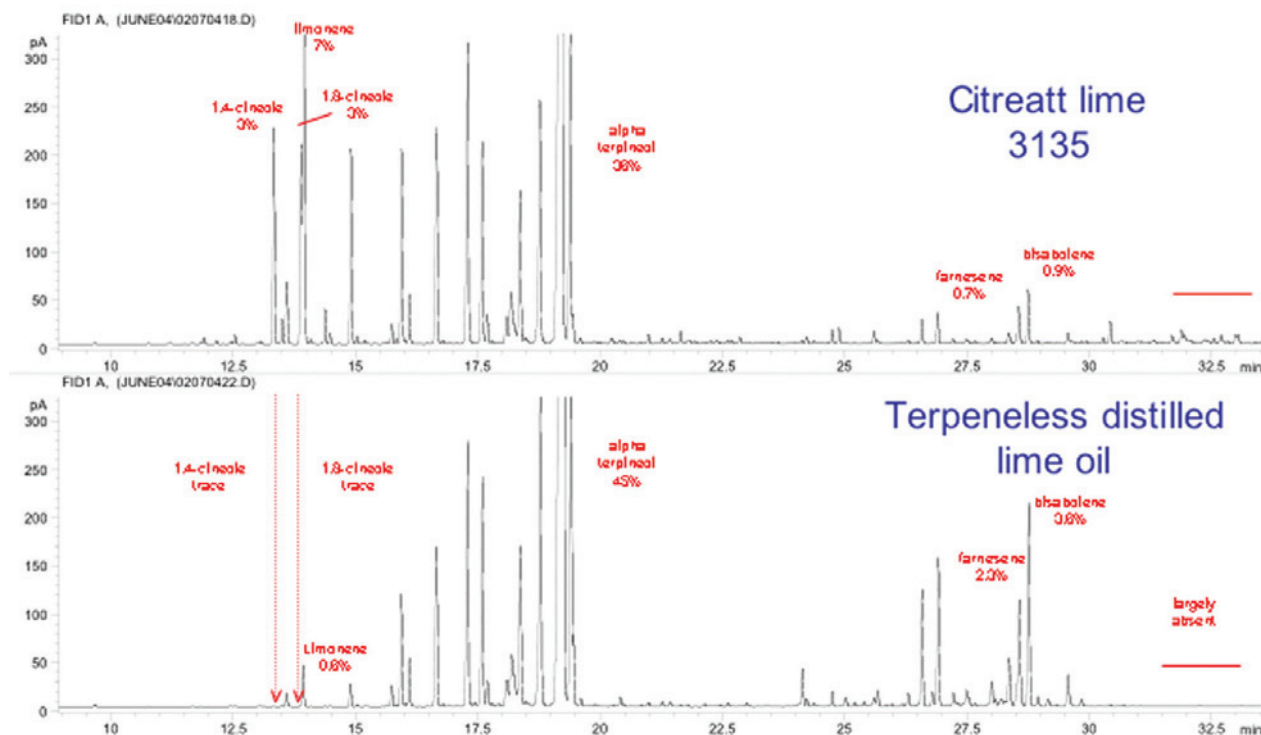


oxygenated compounds. The solvent-free product then is standardized and bulked ready for sale.

#### Natural Specialties

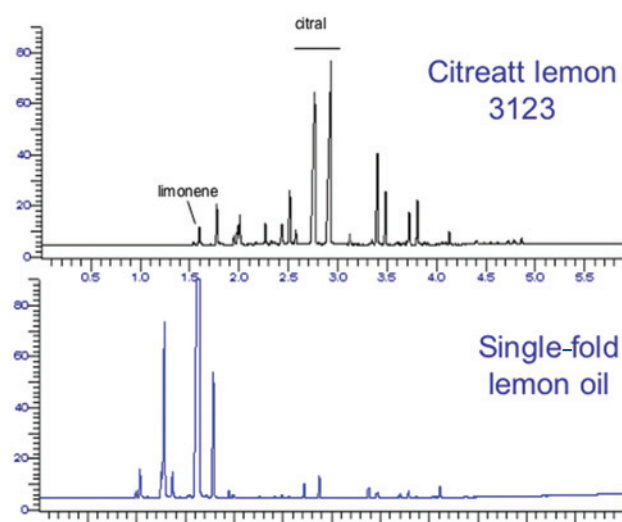
This method can be used to produce from the named food (FTNF) concentrated and terpeneless citrus oils. These specialty citrus oils retain their volatile flavor components, resulting in a natural flavor. The enhanced solubility of these oils in alcohol makes them particularly suitable for essence and beverage manufacture. In addition, the process is not as dependent on the quality of the starting oil, allowing component levels to be varied according to specific customer requirements.

The first products developed by my company using this technique were orange, lemon and lime oils. These were



Citreatt lemon versus single-fold lemon oil

F-4



followed by grapefruit and expressed lime. F-1 through F-4 show the gas chromatograms of Citreatt orange, lime and lemon oils compared to single-fold/terpeneless equivalents.

The bottom chromatogram in F-1 shows a very large peak due to limonene that is reduced significantly in the chromatograms above. In addition, the difference in octanal levels in the Citreatt orange 3111 and 3112 results in a different odor and flavor.

Citreatt lime 3135 is a highly concentrated distilled key lime oil. Note the low limonene and high  $\alpha$ -terpineol levels compared to those in the single-fold oil (F-2). These changes result in a product with enhanced alcohol solubility. Citreatt lime 3135 contains the volatile components 1,4- and 1,8-cineole and trace high boiling components lost in the traditionally produced terpeneless oil (F-3). Citreatt lemon 3123 shows a significant enhancement of the two isomers of citral and a corresponding reduction in limonene and other terpenes.

## Conclusion

Specialized processing techniques now allow the production of citrus oils that reflect the true character of the fresh fruit. Perfumers and flavorists can benefit from using these oils to create distinctive new citrus flavors and fragrances to add value and differentiate brands.

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