

# Essential Oil Purification

On DAVISIL® Chromatographic Silica Media

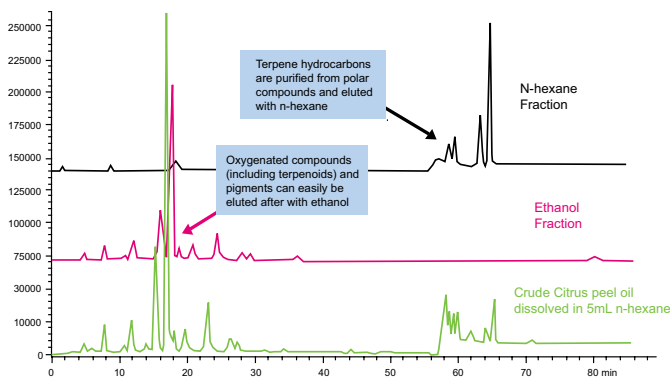


## Purification of Citrus Peel Oil by GRACE DAVISIL® LC60A 40-63µm silica

The purification of flavor and fragrance components from essential oils, such as lemon or orange oils, can be performed on DAVISIL® Chromatographic silica. This study details a procedure for purifying Limonene and  $\alpha$ -Terpinene, two non-polar hydrocarbons in the terpene class from their polar terpenoid analogs and other polar compounds found in citrus oil.

Citrus peel contains abundant essential oil, which can be used in many fields, such as medicine, foodstuff, beverages and cosmetics. Citrus oil consists of terpenes (over 95%) such as Limonene and Terpinene and oxygenated compounds (below 5%) like Sinensal, Neral, Octanal, Tridencanal, etc., and waxes and pigments. Both classes of compounds are valuable and are primarily used as flavor additives in the manufacture of soft drinks (lemon, lime and orange oils represent the largest segment of U.S. essential oil imports in both volume and value). Consistent purification of the components in citrus oils is important in ensuring the production of quality products with consistent flavors and aromas from batch to batch.

Terpenes are a class of naturally occurring compounds mainly found in plants as essential oil constituents whose carbon skeletons are composed exclusively of isoprene C5 units ( $\text{CH}_2=\text{C}(\text{CH}_3)-\text{CH}=\text{CH}_2$ ). Most terpenes have molecular formula ( $\text{C}_5\text{H}_8$ )<sub>n</sub> in a cyclic or acyclic, saturated or unsaturated structure, while the terpenoids are oxygen-containing analogues of the terpenes such as alcohols, aldehydes or ketones containing hydroxyl groups or carbonyl groups. Preparative separation of these components can be costly and time consuming. DAVISIL® granular silica was used in low pressure chromatography to enrich two terpene flavor components in an economical and effective purification step.



Column: Alltima™ C18, 5µm, 4.6 x 250mm (Part #: 88506)

Mobile Phase: A: Water; B: Methanol

Flow Rate: 1.0mL/min

Detection: UV@254nm

Column Temperature: 25°C

Gradient:	Time:	0	40	40	60
	%B:	70	70	100	100

Figure 1. Terpene hydrocarbons are enriched and eluted from the Grace Davison® LC60A 40-63 silica gel using n-hexane, thereafter, other components such as oxygenated compounds and pigments are easily washed off the column with ethanol.



## Experimental

### Extraction

180 grams of fresh Citrus peel with granularity, was extracted twice with sonication for 15 minutes. The extract was impregnated for 24 hours with 450mL petroleum ether (30-60°C). The crude citrus oil was obtained after the extracted liquor was concentrated under vacuum, and then dissolved with ethanol, frozen at 4°C for 48 hours and centrifugally separated and filtered.

### Purification

80 g of Grace Davisil® LC60A 40-63 silica gel was slurried with 120mL n-hexane and poured into a 25mm I.D.×300mm glass column.

3.2 grams crude citrus oil was dissolved in 5mL n-hexane. The sample was applied to the Grace Davisil® silica gel column and washed with n-hexane, and ethanol, to give two fractions. These fractions were then analyzed by RP-HPLC (figure 1) and the n-hexane fraction was compared to a standard to determine its constituents (figure 2).

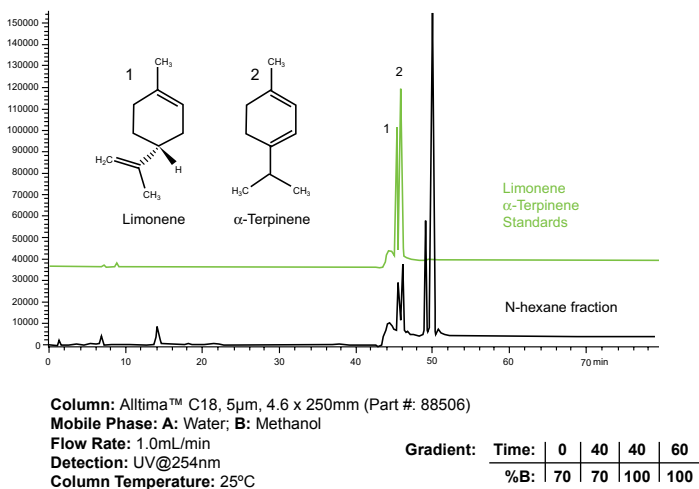


Figure 2. Limonene and  $\alpha$ -Terpinene are identified in the n-hexane fraction.

## Results and Discussion

Davisil® silica gel was used to effectively purify limonene and terpinene from the crude citrus oil extraction mixture. The effective retention of the oxygenated compounds and pigments allowed the target citrus flavor compounds, terpinene and limonene, to be easily eluted in the n-hexane fraction, purifying them from the crude material. Identification of the enriched material was easily performed on the high resolution, Alltima™ C18 analytical column. If desired, the polar compounds and pigments, including the terpenoid analogs, could be eluted with an ethanol fraction. Davisil® silica gel is an effective tool for simple separation and enrichment of polar and non-polar citrus flavor components from crude extraction mixtures because of its high purity, reproducibility, and good selectivity.

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