

## Honors Cup Synthetic Proposal

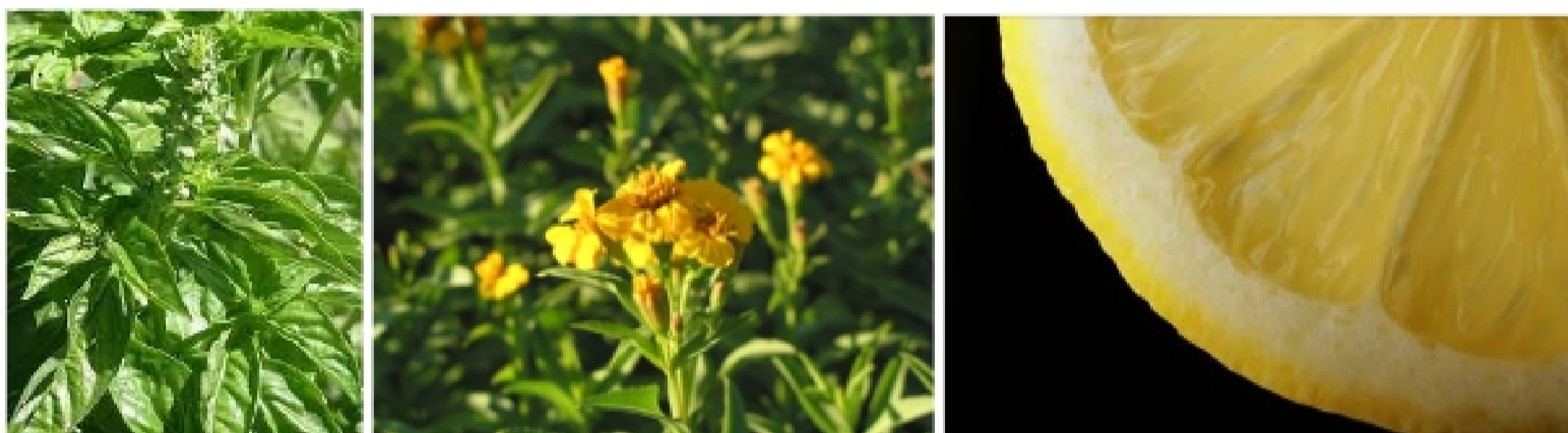
**Section:** 251

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**Title:** Synthesis of Linalool

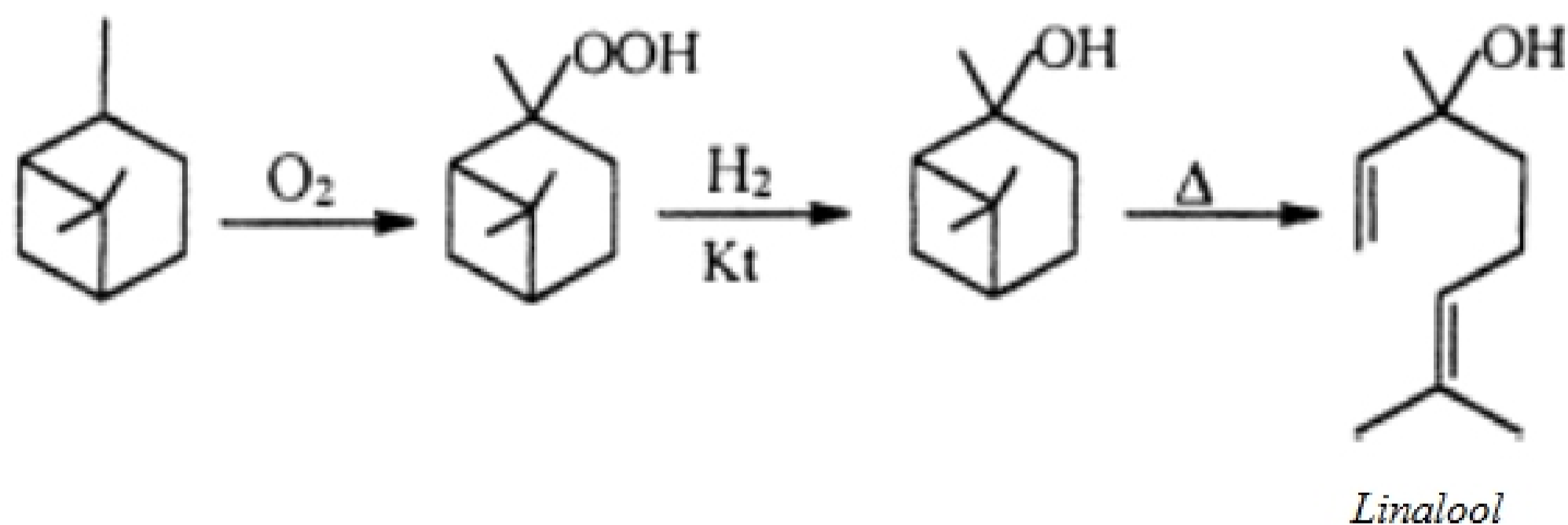
**Introduction:** Linalool is a fragrance used in many soaps, detergents, massage oils, creams, lotions, and shampoos. It is also a chemical intermediate in the formation of Vitamin E, and acts as an insecticide that is safe to humans.. Annually, approximately 12,000 tons of linalool are produced worldwide, both through natural sources (it is found in many flowers and spice plants) and chemical synthesis. The synthesis here is based on that of the following paper.

Semikolenov, V.A.; Ilyna, I.I.; Simakova, I.L. *Applied Catalysis* **2001**, *211*, 91-107.



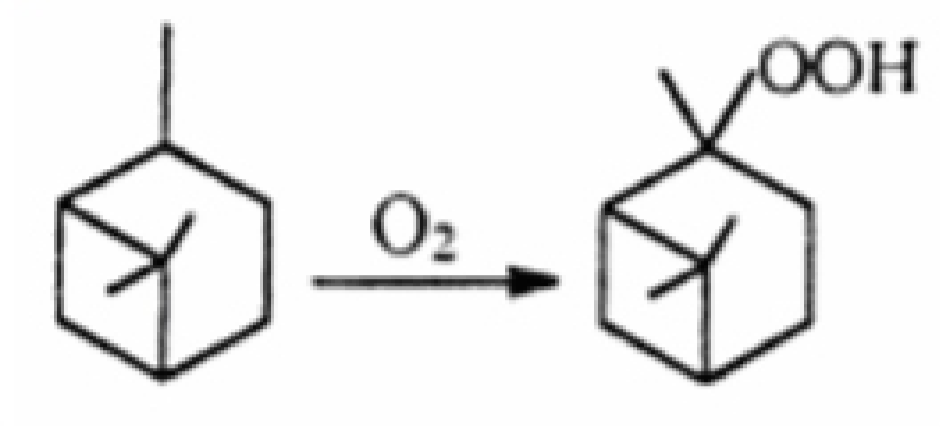
*Linalool is commonly found in basil, marigold, and citrus plants.*

**Overall synthetic reaction scheme:**



## Step 1

### Synthetic transformation 1:



### Experimental 1: Pinane Oxidation

Reaction conditions: pinane 50 cc,  $T = 100^{\circ}\text{C}$ ,  $P_{\text{O}_2} = 4 \text{ atm}$ .

Under 4 atm of pressure and at  $100^{\circ}\text{C}$ , 50 cc isomeric pinane and excess  $\text{O}_2(\text{g})$  were mixed to yield *cis*- and *trans*-pinane hydroperoxide.

**Expected Yield: 85 % 36 g**

### Safety, Disposal and Green Issues:

#### **Safety:**

Pinane-hydroperoxide is a stable hydroperoxide. It is recommended that it be stored in a tightly closed container at a temperature below  $35^{\circ}\text{C}$ . None of these chemicals should be ingested.

#### **Byproducts:**

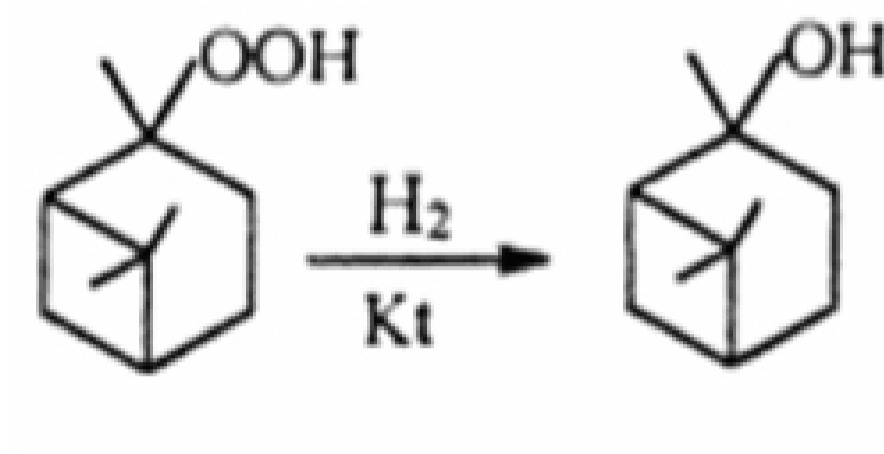
No byproducts other than untransformed pinane.

#### **Disposal and Green Issues:**

All chemicals are safe for basic organic or acid/base waste.

## Step 2

### Synthetic Transformation 2:



### Experimental 2: Pinane Hydroperoxide (PHP) Hydrogenation

Reaction conditions:  $T = 20\text{--}80^\circ\text{C}$ ,  $P_{\text{H}_2} = 1\text{--}11\text{ atm}$ , 5%\* Pd/C catalyst concentration 0.5—60.0 g/l

\*originally 4%, but changed to 5% due to commercial availability

Hydrogenation of PHP solution under the reaction conditions (see above) yielded *cis*- and *trans*- isomers of pinane-2-ol and the byproducts shown below.

Expected Yield: 88 % 31 g

### Safety, Disposal and Green Issues 2:

#### **Safety:**

Palladium can irritate the eyes, but shows no known irritation to the skin. After use, wash hands face thoroughly; protective gloves and glasses should be worn at all times. All of the byproducts can produce mild irritation to the skin, eyes, or lungs, and are flammable. Reaction should be performed in a hood with safety goggles and gloves. None of these chemicals should be ingested.

#### **Byproducts:**

1-acetyl-2,2-dimethyl-3-ethylcyclobutane  
Isopinocampnone  
Isopinocampheol + verbanol  
Isoverbanol + verbanone

#### **Waste and Green Issues:**

The palladium should be recovered for later lab reuse. All other chemicals are safe for basic organic or acid/base waste.