

CLARY SAGE PRODUCTION IN THE SOUTHEASTERN UNITED STATES

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Clary Sage (*Salvia sclarea* L.) production has long been extremely important for use in both perfumery and flavoring⁽¹⁾. The aromatic portion of Clary Sage originates in the flowering top and foliage portion of this tall perennial botanical. Depending on climatic and soil conditions, Clary Sage plantings are treated as annuals or are carried over for several years with the summer harvest season in any given area being limited to about three to six weeks⁽²⁾. Although a second cutting of Clary Sage can sometimes be achieved in late fall, the oil or concrete obtained from processing the second overcut is usually inferior in both yield and quality since the plant rarely has time to recover to full maturity before, winter arrives.



Mature Clary sage plant (White Variety)



Mature Clary sage plant (Pink Variety)

The oil of Clary Sage is valued by perfumers for use in conjunction with Lavandin, Lavender, and Bergamot and is now also used extensively in trace quantities for rounding off the aroma of perfume oils containing quality synthetic linalool and linalyl acetate chemicals produced either from the Roche acetylenic route or the Glidden pinene process. It is used in Oriental perfume creations and "tabac" type fragrances and possesses real value in the so-called "Herbal" compounds.

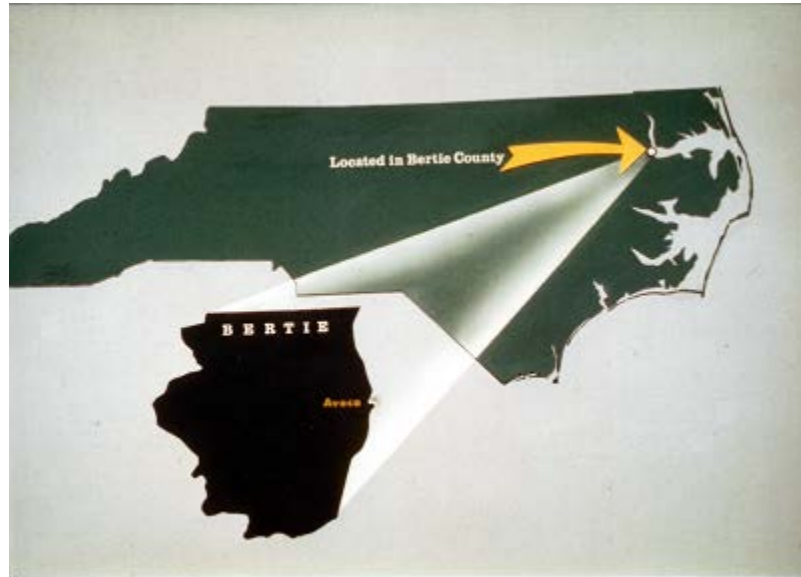
Clary Sage oil is considered by many perfumers as possessing a characteristic note of sexuality so indispensable in the most expensive perfume creations.

Although only a relatively small quantity of the Oil of Clary Sage is used on the flavoring side, it is well known that certain European wines and liqueurs (Rhine types and vermouths, for example) owe their characteristic individuality to the trace addition of this essential oil. In fact, the German name for this oil (Muskateller Salbeiöl) is literally translated as "muscatel sage oil"⁽³⁾.

The tobacco-like dry out note of this oil, which is of so much value in perfumery, is also very interesting in the highly specialized tobacco flavor field⁽⁴⁾.

Historically, Clary Sage appears to have its origin in the countries bordering the northern Mediterranean Sea⁽¹⁾. In the last forty years, this plant has been commercially cultivated in France, Russia, Hungary, Bulgaria, Italy, Morocco, Romania, England, and Yugoslavia. In the 1950's a serious attempt was made in the far western peppermint growing areas of Oregon and Washington in the United States, but these efforts met with little success. Today, only the south of France and the Crimean and Caucasus districts of the Union of Soviet Socialist Republics bordering the Black Sea represent a major factor in the world production of Clary Sage.

Over ten years ago, R. J. Reynolds undertook a program of adapting the cultivation of Clary Sage and other essential oil bearing crops to the rich and fertile coastal plain near the old, and historically important, town of Edenton which was the capital of the original British Royal Crown Colony of North Carolina.



Avoca location



View of Facility from Albemarle Sound

Our Avoca plantation, which is located at the western end of Albemarle Sound in Bertie County, is situated in an area where the altitude and climatic conditions contrast sharply with the higher and dryer regions such as are found in the Departments of Var, Basses-Alpes, Drome, etc. in the South of France. Because of this, an extensive agronomy program was required to adapt and select strains of *Salvia sclarea*

L. which would produce good yields both of the essential oil and of Clary Sage Concrete - not only from an economic viewpoint, but just as important, from the standpoint of quality.



Experimental Clary Sage Plots

In order to provide you with an idea of our Clary Sage plantings, these aerial photographs of some of our sage fields present a colorful picture of the plantation fields just prior to harvesting.



An Aerial View of the Sage Fields



Another Aerial View of the Sage Fields

The foreign production of Sage and other essential oils is usually carried out on plant material grown by local farmers, which is cut, allowed to air dry, and then hauled to central distilling centers where it is placed into large stationary stills for steam distillation.

The plant material is ordinarily suspended in the body of the still on top of a large perforated grate to which chains are attached. The top of the still is then fixed in place and distillation is begun with low pressure steam⁽⁵⁾. Following distillation, the spent plant material may be removed with a hoist, for disposal.

Our initial experiments were conducted in a similar, but considerably smaller system simply constructed from a 55 gallon stainless steel drum. This portable field still followed a design kindly provided by Professor C. W. Shoppee and is modeled after a unit he has used in investigations of various indigenous oil bearing plants in the "outback" of Australia and New Zealand.



Experimental Distillation Apparatus



Experimental Distillation Apparatus in Operation

Upon successful completion of our agronomic program, we evaluated the various possible commercial methods for essential oil production. From the beginning, we felt that the use of fixed, permanent, single batch stationary stills was inefficient for large scale production. Therefore, we investigated both distillation through a continuous horizontal screw conveyor steam distillation system

(similar to the type which has been reported to be successful in the USSR), and the portable truck distillation system which is currently in widespread use for the production of peppermint and spearmint oils in the northwest United States⁽⁶⁾. Although both distillation systems possess considerable merit, we now feel that the efficiency of the portable truck system is best suited to our operation in that it allows us greater flexibility for expansion of oil production. Since 1971, when we first produced commercial quantities of Clary Sage oil of sage, we have been increasing our oil production at a rate of more than 400% per year and expect within the next few years to be able to supply reasonable quantities of North Carolina (Avoca) Clary Sage oil which may be required by the trade.



Mechanical Harvesting of Clary Sage



Mechanical Harvesting of Clary Sage into Distillation Bin



Delivering Truck to the Distillation Unit



Docking a Truck Bin to the Distillation Unit

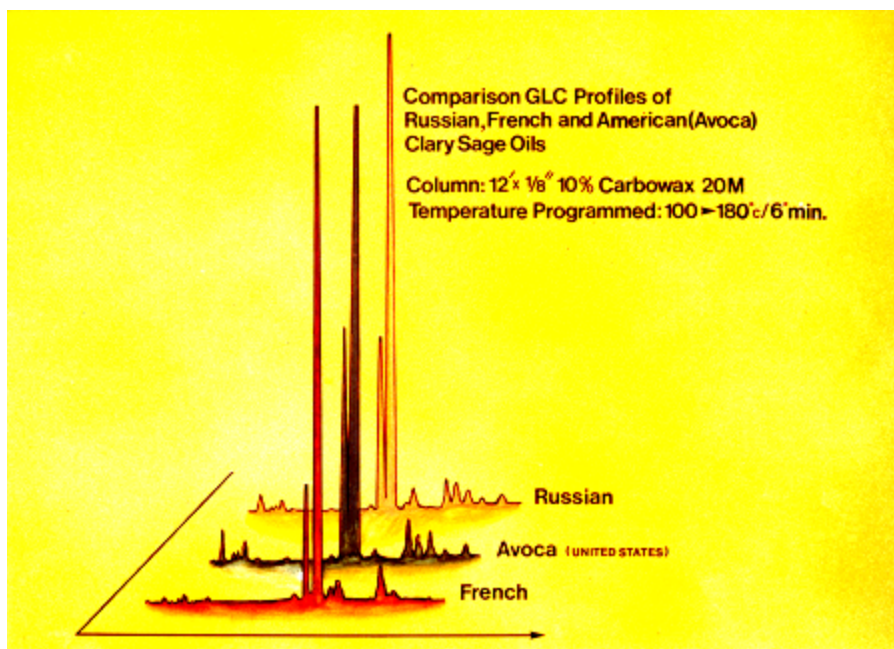


Truck Bins at Distillation Units



Frank Kane taking Clary Sage Oil Sample for Q.C.

The physical and organoleptic characteristics of our oil are more similar to those of Russian Clary Sage than French.



Comparison of Three Sources of Clary Sage Oil

This is best illustrated by examination of typical gas chromatograms (temperature programmed on a Carbowax 20M column) from samples of our Avoca oil compared to those of authentic samples of French and Russian oils. These chromatographic profiles which were obtained from our quality control laboratory provide clues that the major variations between types of Clary Sage oils occur primarily in the terpenoid and sesquiterpenoid portions with higher retention times. The slight variations in the low boiling monoterpene hydrocarbon fractions are probably indicative only of the age of cut Sage before it is processed. Organoleptically, this is of less importance to oil quality.

Examination of a series of authentic Clary Sage oils, obtained at our facility, and from France and Russia, by glass capillary gas chromatography-mass spectrometry allowed us to rapidly identify more than 94% by weight of all eluting volatile components. The twenty-six components pinpointed in this chromatogram are listed in the following table along with the identification method and source oil analyzed. It should be pointed out that, in addition to these few components, Dr. R. A. Lloyd of our laboratory estimates that the total number of trace volatile constituents may exceed 300.

The structural relationships between the monoterpenes present are all rather obvious to terpene chemists, and no particular surprises are found in the reported constituents. The monoterpene ratios observed from sample to sample and source to source are somewhat a variable of the steam distillation process and, at least to some degree, are a result of this processing. For example, it is well known that acid rearrangements of the two major Clary Sage components (linalool and linalyl acetate) can provide a rather impressive series of terpenoids, almost all of which are present in the monoterpene fraction of these oils. However, one cannot assume that such components are simply processing artifacts since Teisseire

TABLE
COMPONENTS OF CLARY SAGE ESSENTIAL OILS¹

Peak	Component Name	Identification Method	American - 1973	French - 1972	French - 1972	French - 1972	Russian - 1972
			Avoca	Apt	St. Pantaleon	Commercial	Commercial
1	α -Pinene	GC, MS	X	X	X	X	X
2	Camphene	GC	X	X	X	X	X
3	β -Pinene	GC, MS	X	X	X	X	X
4	Myrcene	GC, MS	X	X	X	X	X
5	α -Terpinene	GC, MS	X	X	X	X	X
6	Limonene	GC, MS	X	X	X	X	X
7	Ocimene (isomer I)	GC, MS	X	X	X	X	X
8	Ocimene (isomer II)	GC, MS	X	X	X	X	X
9	p-Cymene	GC, MS	X	X	X	X	X
10	Terpinolene	GC	X	X	X	X	X
11	<i>cis</i> -3-Hexen-1-ol	GC	X	--	--	--	X
12	Linalool	GC, MS	X	X	X	X	X
13	Linalyl acetate	GC, MS	X	X	X	X	X
14	Terpinen-4-ol	GC, MS	X	X	X	X	X
15	β -caryophyllene	GC, MS	X	X	X	X	X
16	Citronellol acetate	GC	X	X	X	X	X
17	<i>trans</i> - β -Terpineol	GC	X	X	X	X	X
18	α -Terpineol and	GC, MS	X	X	X	X	X
18a	Terpinyl acetate	GC, MS	X	X	X	X	X
19	β -Gurjunene	MS	X	X	X	X	X
20	Neryl acetate	GC,MS	X	X	X	X	X
21	Geranyl acetate	GC	X	X	X	X	X
22	Citronellol	GC	--	X	X	X	--
23	Nerol	GC,MS	X	X	X	X	X
24	Geraniol	GC,MS	X	X	X	X	X
25	Caryophyllene Oxide	GC,MS	X	X	X	X	X
Total Volatiles relative to tridecane			71.66%	66.46%	65.17%	67.26%	73.14%
Percent of Volatiles identified			96.5	95.1	95.6	94.4	97.0

¹ FFAP glass capillary column, FID, temperature programed, 60-205° @ 2°/min.

and Bernard⁽⁷⁾ have shown that the optical activity of the linalool and α -terpineol present are in differing enantiomeric (chiral) forms than that which would arise via hydrolysis and artifact formation during distillation.

Combining our findings with those published in the literature, I would now like to present a series of Tables listing the known constituents of Clary Sage oils⁽⁷⁻⁹⁾.

REPORTED COMPONENTS OF CLARY SAGE OILS

I. MONOTERPENOIDS

α -PINENE	LINALOOL
CAMPHENE	GERANIOL
MYRCENE	NEROL
β -PINENE	CITRONELLOL
LIMONENE	α -TERPINEOL
α -OCIMENE	TERPINEN-4-OL*
β -OCIMENE	<i>trans</i> - β -TERPINEOL**
CINEOLE	
p-CYMENE	LINALYL ACETATE
<i>cis</i> -ALLOOCIMENE	GERANYL ACETATE
<i>trans</i> -ALLOOCIMENE	NERYL ACETATE
α -TERPINENE*	CITRONELLYL ACETATE**
γ -TERPINENE	α -TERPINYL ACETATE*
TERPINOLENE	
δ -3-CARENE	

*NEW COMPOUNDS IDENTIFIED IN THIS WORK (GC, MS)

**NEW COMPOUNDS TENTATIVELY IDENTIFIED IN THIS WORK (GC ONLY)

***NEW COMPOUNDS TENTATIVELY IDENTIFIED IN THIS WORK (MS ONLY)

REPORTED COMPONENTS OF CLARY SAGE OILS

II. SESQUITERPENOIDS

α -CUBEENE	γ -MURULENE
α -COPAENE	EREMOPHILENE
β -COPAENE	ϵ -MURULENE
YLANGENE	δ -CADINENE
β -ELEMENE	γ -CADINENE
β -CARYOPHYLLENE	α -CALACORENE
AROMADENDRENE	β -GURJUNENE***
α -MURULENE	β -CARYOPHYLLENE OXIDE*
α -HUMULENE	NEROLIDOL (?)
β -SANTALENE	NEROLIDYL ACETATE (?)

*NEW COMPOUNDS IDENTIFIED IN THIS WORK (GC, MS)

**NEW COMPOUNDS TENTATIVELY IDENTIFIED IN THIS WORK (GC ONLY)

***NEW COMPOUNDS TENTATIVELY IDENTIFIED IN THIS WORK (MS ONLY)

REPORTED COMPONENTS OF CLARY SAGE OILS

III. MISCELLANEOUS

n-PENTANOL	ACETALDEHYDE
n-HEXANOL	PROPIONALDEHYDE
3-OCTANOL	BUTYRALDEHYDE
1-OCTEN-3-OL	n-PENTANAL
<i>cis</i> -3-HEXENOL	n-HEXANAL
	n-HEPTANAL
SCLAREOL	n-OCTANAL
	n-NONANAL
	BENZALDEHYDE
	FURFURAL

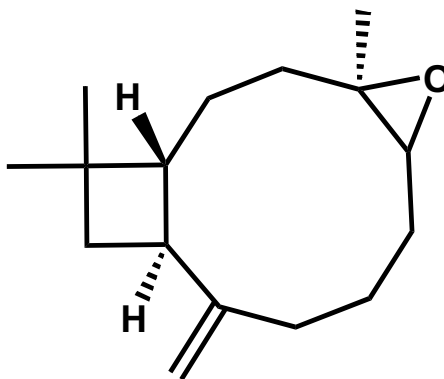
*NEW COMPOUNDS IDENTIFIED IN THIS WORK (GC, MS)

**NEW COMPOUNDS TENTATIVELY IDENTIFIED IN THIS WORK (GC ONLY)

***NEW COMPOUNDS TENTATIVELY IDENTIFIED IN THIS WORK (MS ONLY)

You will note that we have placed question marks beside several of the reported constituents because we were unable to confirm their presence in our studies even though we had authentic synthetic samples available for comparison.

We now wish to report for the first time the presence of *trans*- β -terpineol, terpinen-4-ol, α -terpinene, β -gurjunene (tentative), and of particular interest, a sesquiterpene epoxide present as 0.2-1.0% of the volatiles which we presume to be the portion of the sesquiterpene sage oil fraction described by Teisseire⁽⁷⁾ in 1957 as a part of the amber odor fraction possessing an epoxide linkage. β -Caryophyllene epoxide, which indeed contributes an amber fixative note, was confirmed by us to be present in six separate Clary Sage oils obtained from various regions and sources. This interesting oxide has also been recently identified by Enzell and co-workers⁽¹⁰⁾ in Oriental tobacco and undoubtedly is one of the more important contributors to the amber/tobacco-like dry out note of Clary Sage.



β -CARYOPHYLLENE OXIDE

The interesting results obtained in this brief look at sage oil constituents only scratch the surface of what we are sure will prove to be a worthwhile challenge to those who desire to carry out a more extensive analysis of Clary Sage oil.

Let me now turn to the subject of specialty perfume and flavor products arising from the processing of Clary Sage Concrete.

As you will note from this series of photographs, we have constructed the largest stainless steel extraction facility in the world for the continuous extraction of botanical materials.

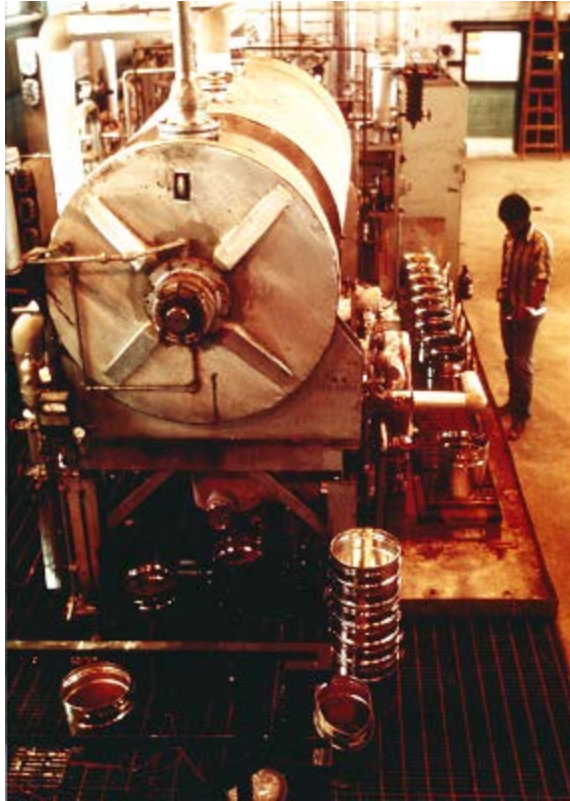


Rotocell in Operation to Extract Sclareol from Plant Material after Distillation



Conveying Plant Material into Rotocell for Extraction

In this way, we process very large quantities of Clary Sage Concrete for the main chemical constituent, "Sclareol"⁽¹¹⁾. Sclareol, in its own right, possesses very little aroma but is extremely useful in perfumery for its "fixing" power.

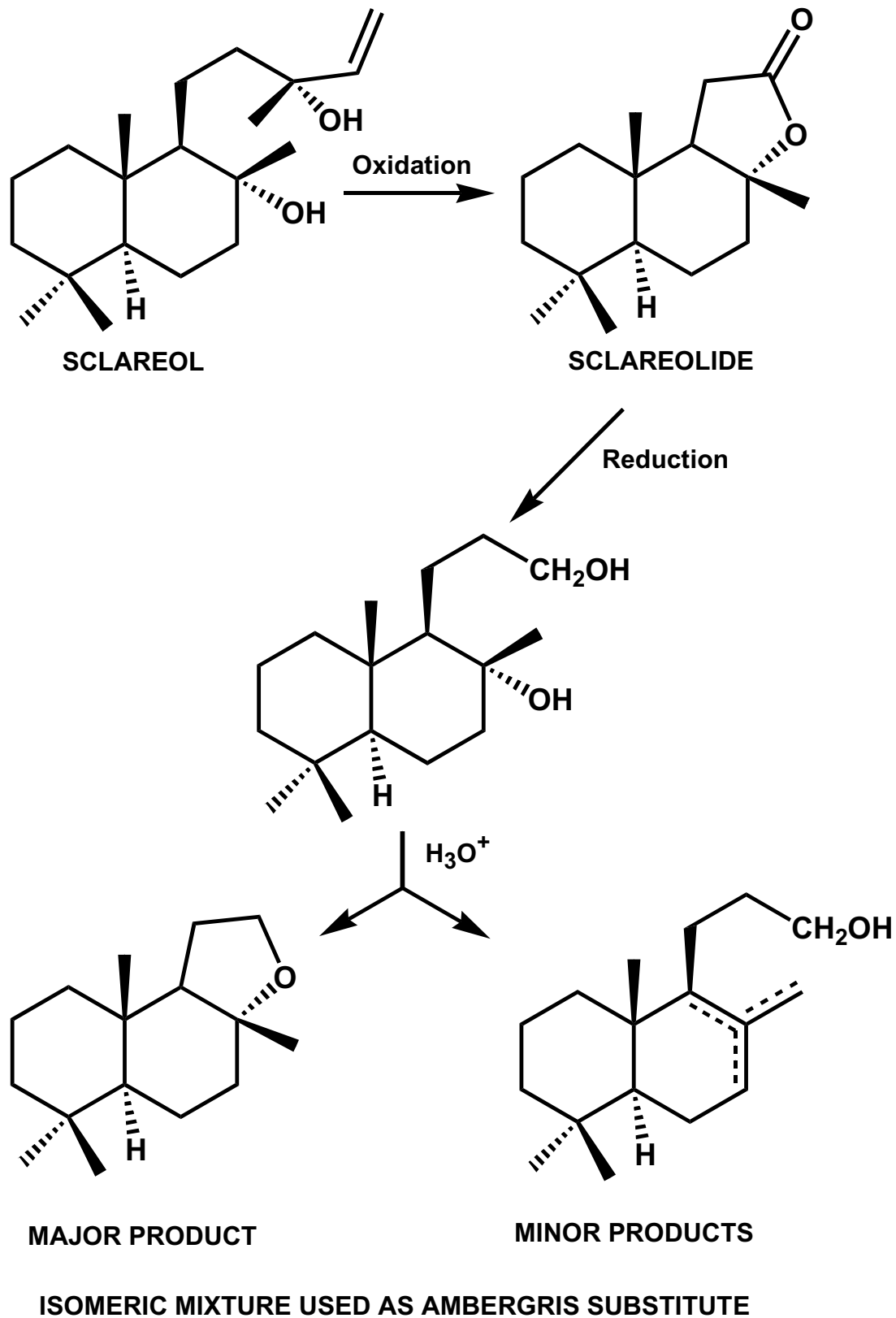


Sclareol Filling Line after Purification



This versatile diterpene alcohol is also the starting material for an important series of perfume and flavor chemicals⁽¹²⁾.

Controlled oxidation and cyclization of sclareol affords a material (Norambrienolide), commonly referred to as "Sclareol-lactone" or "Sclareolide". Sclareolide, an oxidative degradation product of Ambriene from ambergris⁽¹³⁾, is also a well-known tobacco constituent, having been isolated by Schumacher from Oriental tobacco⁽¹⁴⁾ and by Japanese workers from cigar tobacco⁽¹⁵⁾. Contrary to numerous published reports⁽¹⁶⁾ that this substance is odorless, we reported in 1973⁽¹⁷⁾ that, on vaporization, this material possesses a marked cedar/amber-like note. In solution, it has remarkable value for perfume oils - providing not only a fixative effect, but also a long lasting faint amber character. This material, produced in our chemical manufacturing facility at Avoca, is now widely used throughout the world in both fine perfumes and tobacco. Sclareolide is also the intermediate for production of an important ambergris substitute and the next sequence illustrates this chemical conversion⁽¹⁸⁾.



In summary, we have shown only one facet of our continuing interest in botanicals for the perfume and flavor industry.



Capsicum Pepper (Hot Chiles) Field



Mechanical Harvesting of Capsicum Peppers

A commercial program of agronomics, processing, and extraction of Capsicum peppers has also been developed at our Avoca facility over the last few years, and experimental programs with such essential oil bearing plants as peppermint, spearmint, and others, have provided the base for a viable new agri-business complex in North Carolina.

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