642599 AUSTRALIA

SPRUSON & FERGUSON

PATENTS ACT 1990

PATENT REQUEST: STANDARD PATENT

I/We, the Applicant(s)/Nominated Person(s) specified below, request I/We be granted a patent for the invention disclosed in the accompanying standard complete specification.

[70,71] Applicant(s)/Nominated Person(s):

Wm. Wrigley Jr. Company, incorporated in Delaware, of 410 North Michigan Avenue, Chicago, Illinois, 60611, UNITED STATES OF AMERICA

[54] Invention Title:

Tocopherol Mixture for Use as a Mint Oil Antioxidant in Chewing Gum

[72] Inventor(s):

David G. Barkalow, Michael J. Greenberg and Gordon N. McGrew

[74] Address for service in Australia:

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Details of Basic Application(s):

[31] Appl'n No(s):	[33] Country:	[32] Application Date:
722,692	US	28 June 1991

DATED this TWENTY THIRD day of JUNE 1992

Wm. Wrigley Jr. Company

By: J. C. Hinde

Registered Patent Attorney

IRN: 214144

INSTR CODE: 55730

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5845/2

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Australia

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NOTICE OF ENTITLEMENT

I, Gregory Michael Turner, care of Spruson & Ferguson, St. Martins Tower, 31 Market Street, Sydney, New South Wales, 2000, Australia, being the patent attorney for the Applicant/Nominated Person in respect of an application entitled:

Tocopherol Mixture for Use as a Mint Oil Antioxidant in Chewing Gum

state the following:-

The Applicant/Nominated Person has entitlement from the actual inventor(s) as follows:-

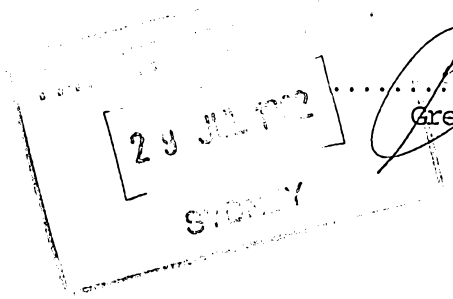
The Applicant/Nominated Person is the assignee of the actual inventor(s).

The Applicant/Nominated Person is entitled to rely on the basic application(s) listed on the Patent Request as follows:

The Applicant/Nominated Person is the assignee of the basic applicant(s).

The basic application(s) listed on the Patent Request is/are the application(s) first made in a Convention country in respect of the invention.

DATED this Twenty Seventh day of July 1992



.....
Gregory Michael Turner

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AU9218469

(12) PATENT ABRIDGMENT (11) Document No. AU-B-18469/92
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 642599

(54) Title
TOCOPHEROL MIXTURE FOR USE AS A MINT OIL ANTIOXIDANT IN CHEWING GUM

International Patent Classification(s)
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(56) Prior Art Documents
US 4977282
US 4489099
GB 2245812

(57)

Specific mixtures of tocopherol homologs exhibit surprisingly improved antioxidant effects in mint oil and mint-flavored chewing gum compositions. The mixtures of homologs fall within the range of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol. Homologue mixtures within this range are superior to the constituent homologs used alone, and to other homologue mixtures, in terms of antioxidant effects. The compositions of the invention facilitate the replacement of controversial synthetic antioxidants currently used in mint oil and mint-flavored chewing gum, with naturally occurring tocopherols.

CLAIM

1. A stabilized mint oil composition, comprising:

a mint oil selected from the group consisting of peppermint oil, spearmint oil, corn mint oil, Scotch spearmint oil and mixtures thereof; and

.../2

.05-3.0% by weight of a tocopherol mixture blended into the mint oil, the tocopherol mixture comprising 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

7. The stabilized mint oil composition of claim 1 in a chewing gum, in an amount of .5-3% of the weight of the chewing gum, wherein the mint oil comprises peppermint oil.

25. A mint flavored chewing gum composition, comprising a water soluble bulk portion, a water insoluble chewing gum base portion and a flavoring agent, the flavoring agent comprising corn oil and an antioxidant which consists primarily of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

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AUSTRALIA

PATENTS ACT 1990

COMPLETE SPECIFICATION

FOR A STANDARD PATENT

ORIGINAL

Name and Address of Applicant: Wm. Wrigley Jr. Company
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Actual Inventor(s): David G. Barkalow, Michael J. Greenberg and Gordon N. McGrew

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Sydney, New South Wales, 2000, Australia

Invention Title: Tocopherol Mixture for Use as a Mint Oil Antioxidant in Chewing Gum

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

TOCOPHEROL MIXTURE FOR USE AS A MINT OIL ANTIOXIDANT IN CHEWING GUM

FIELD OF THE INVENTION

The present invention relates to mint oil and mint flavored chewing gum compositions containing a specific blend of tocopherol homologs which provides improved antioxidant effects.

BACKGROUND OF THE INVENTION

As is known in the art, chewing gum compositions include a number of ingredients, some of which are subject to deterioration through oxidation during storage. Mint oils are important ingredients of chewing gum which require stabilization, both before and after the mint oils are incorporated into the chewing gum. The term "mint oil" as used herein includes, but is not limited to, peppermint oil, spearmint oil, corn mint oil and Scotch spearmint oil. The use of peppermint oil in chewing gum, enhanced with a minor amount of spearmint oil, is discussed in U.S. Patent 4,948,595 issued to Patel et al.

Several synthetic antioxidants are in widespread use for stabilizing mint oil and mint flavored chewing gum compositions. Among the most notable of these synthetic antioxidants are Butylated Hydroxyanisole (BHA), Butylated Hydroxytoluene (BHT) and tertiary butyl hydroquinone (TBHQ). While BHA, BHT and

TBHQ are effective in preventing and reducing oxidation of mint oil and mint flavored chewing gum compositions, these compounds have been found to be carcinogenic in animal testing.

Tocopherols are naturally occurring compounds which are known to have antioxidant properties. The most common tocopherol, alpha-tocopherol, is also known as vitamin E. The other common homologs are beta, gamma and delta tocopherols. All of these tocopherol homologs are FDA approved additives which have been shown to be safe in animal testing. Natural tocopherols are usually derived from vegetable oils, especially soybean oil. Tocopherols can also be found in many spices, fruits, nuts, seeds and other plant sources.

Unfortunately, tocopherols have been found to be generally less effective than BHA and BHT in preventing oxidation of mint oil and mint flavored chewing gum. Tocopherols also have upper usage level limits, above which oxidation of the mint oil will start to increase again. Tocopherols have been used successfully as chewing gum stabilizers when added in combination with certain synthetic stabilizers. U.S. Patent 4,489,099 issued to Shaheen et al. discloses the use of vitamin E tocopherol in combination with dilauryl thiodipropionate (DLTDP). However, it has previously been less effective to use tocopherols in mint oil, or in mint flavored (or other) chewing gum compositions, by themselves.

SUMMARY OF THE INVENTION

It has been discovered that certain mixtures of tocopherol homologs, in certain ratios, exhibit surprising and unexpected improvement in antioxidant properties when used in mint oil and mint flavored

chewing gum compositions. The homologue mixtures useful in the invention are more effective at preventing oxidation of mint oil than any of the constituent homologs used alone. The tocopherol mixtures useful in the present invention will contain at least three tocopherol homologs, alpha, gamma and delta, in specific ratios. Beta tocopherol and possibly other homologs may also be present in small quantities.

Expressed as percentage ranges of active ingredients (i.e. weight percentages based on total tocopherols) the preferred ranges are 7-15% alpha tocopherol, 45-70% gamma tocopherol and 20-35% delta tocopherol. The most preferred ranges are 9-13% alpha tocopherol, 55-63% gamma tocopherol and 27-32% delta tocopherol. The tocopherol mixtures used in the present invention may be naturally occurring products, mixtures of naturally occurring products, modifications of natural products, mixtures of component tocopherols or natural products with added component tocopherols.

Normally, the tocopherol mixture will be present in a diluent, most commonly a vegetable oil. The amount of the diluent is not important from an antioxidant standpoint. However, active levels of about 70% (reflecting about 70% by weight tocopherol and about 30% by weight diluent) are preferred for viscosity and handling reasons.

With the foregoing in mind, it is a feature and advantage of the invention to provide a stabilizer system for mint oils, especially peppermint oil, which minimizes the oxidation of the mint oils without the use of controversial synthetic antioxidants such as BHA and BHT.

It is also a feature and advantage of the invention to provide mint oil compositions for use in chewing gum, especially peppermint oil compositions, which are shelf stable and which do not contain controversial synthetic antioxidants.

It is also a feature and advantage of the invention to provide chewing gum compositions containing mint oil, which are stable and completely safe for human use, and which avoid the use of controversial BHA and BHT antioxidants.

These and other features and advantages of the invention will become apparent from the following description which, when read in conjunction with the accompanying examples and drawings, disclose presently preferred exemplary embodiments of the present invention. It should be understood that this description is illustrative rather than limiting, the scope of the invention being defined by the appended claims and equivalents thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1 shows oxidation as a function of time for peppermint oil samples which have been stabilized with Tenox GT-2, with substantially pure alpha, beta and delta tocopherols, and for an unstabilized peppermint oil sample. Tenox GT-2 is a commercially available tocopherol blend containing 10.2% alpha tocopherol, 61.6% gamma tocopherol, and 28.2% delta tocopherol (based on total tocopherols) in a soybean oil diluent (70% tocopherol mixture, 30% soybean oil). The experimental conditions are provided in Examples 1, 2, 3, 4, and 9 hereinafter.

FIGURE 2 shows oxidation as a function of time for peppermint oil samples which have been stabilized with Tenox GT-2 and with various vitamin E

mixtures, and for an unstabilized sample. FIGURE 2 reflects the test results of Examples 1, 5, 6, 7 and 9 hereinafter.

FIGURE 3 shows oxidation as a function of time for peppermint oil samples which have been stabilized with Tenox GT-2, Covi-Ox T-70 and vitamin E. Covi-Ox T-70 is a commercially available tocopherol mixture containing 12% alpha tocopherol, 2% beta tocopherol, 30% delta tocopherol and 56% gamma tocopherol diluted with 30% vegetable oil. FIGURE 3 reflects the experimental results from Examples 1, 5 and 8.

DETAILED DESCRIPTION OF THE PRESENTLY PREFERRED EMBODIMENTS

Oxidation causes undesirable alterations of flavor of mint oil and chewing gum compositions containing mint oil. It has always been a major objective of chewing gum manufacturers to maintain the quality of the flavor and prolong the shelf life of chewing gum, including mint-flavored chewing gum. Therefore, the oxidation of mint oil, and its adverse impact on flavor, is a central concern of chewing gum manufacturers.

Mint oils, and most particularly peppermint oil, contain several key components which readily oxidize. The following Table 1 is a list of peppermint oil components and percentages, and half lives in peppermint oil, in days, at 85°F and 100 psi oxygen.

Table 1

Composition Of Peppermint Oil And
Half Lives of Peppermint Oil Ingredients

Component	Percentage In Peppermint Oil*	Oxidation Half Life (Days, 85°F, 100 psi Oxygen)**
alpha-pinene	1.4	96.4
beta-pinene	1.8	
sabinene	.8	
myrcene	.3	111.0
alpha-terpinene	.4	16.5
limonene	2.5	103.4
1,8-cineole	7.3	
cis-ocimene	.4	25.8
gamma-terpinene	.4	19.5
trans-ocimene	.1	25.9
p-cymene	.1	
terpinolene	.1	
3-octanol	.3	34.7
1-octene-3-ol	.1	115.9
trans-sabinene hydrate	1.0	
menthone	18.7	
menthofuran	3.0	36.3
isomenthone	2.5	
linalool	.3	
cis-sabinene hydrate	.1	
beta-bourbonene	.5	
menthyl acetate	3.6	
neomenthol	3.1	
beta-caryophyllene	.5	34.1
neoisomenthyl acetate	.1	
neoisomenthol	.4	
menthol	40.0	
pulegone	.8	

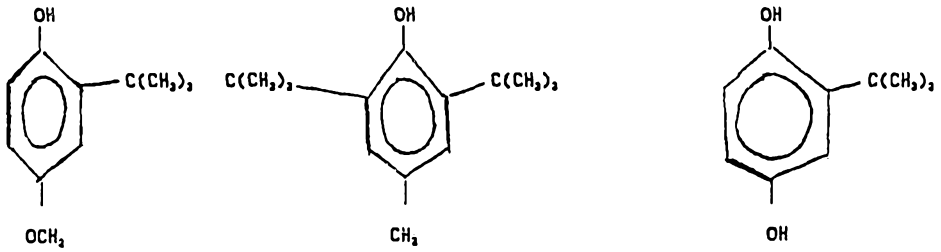
alpha-terpineol	.1	
germacrene D	1.3	24.4
piperitone	.3	
viridiflorol	.2	

* Source: Perfumer & Flavorist, Vol. 14, November/December 1989, p. 21, article entitled "Peppermint Oil Differentiation" by Shu and Harris.

** Data taken for twelve selected components.

Other mint oils also contain many of the oxidizable components that are present in peppermint oil. For instance, spearmint oil and Scotch spearmint oil contain alpha-pinene, limonene, 3-octanol and beta pinene along with other components. Corn mint oil contains alpha-pinene, myrcene, limonene, gamma-terpinene, 3-octanol, menthofuran, beta-caryophyllene, germacrene D, and beta-pinene along with other components. Therefore, an antioxidant which stabilizes peppermint oil will generally be useful for stabilizing other mint oils as well.

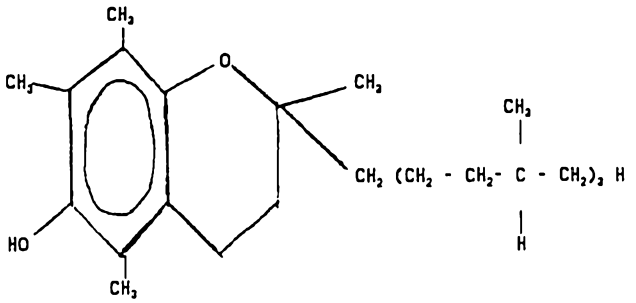
Previously synthetic antioxidants BHA, BHT and TBHQ have been primarily used for stabilizing peppermint oil and other mint-flavored oils. The common feature that renders these compounds effective as antioxidants is their phenolic-based structure, as shown below:



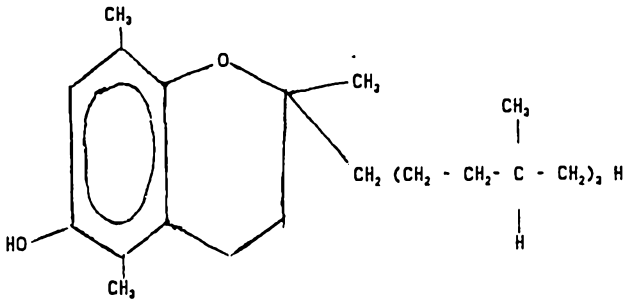
B H A B H T T B H Q

Tocopherols are natural substances found in vegetable and other plant seeds such as soybeans, sunflower seeds, and various fruits and nuts. The fact that tocopherols are naturally derived makes them more attractive to chewing gum manufacturers and consumers, than synthetic antioxidants. From an antioxidant standpoint, tocopherols are of interest because they contain phenolic-type groups similar to those found in BHA, BHT and TBHQ:

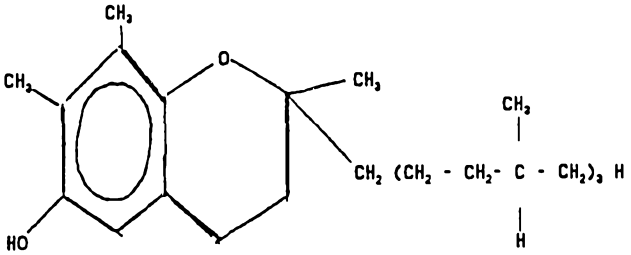
alpha tocopherol



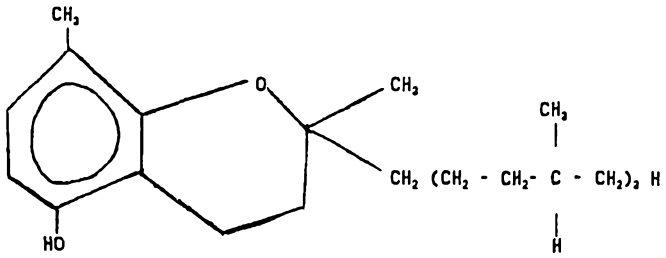
beta tocopherol



gamma tocopherol



delta tocopherol



When taken individually, the different tocopherol homologs are known to exhibit different levels of chemical activity, including different levels of antioxidant activity. For this reason, processes have been developed for isolating the individual homologs from mixtures, and for converting tocopherols from one homologue to another. Such processes are discussed in U.S. Patent 4,977,282 issued to Baldwin et al.

Prior to the current invention, however, it was not known that specific blends of tocopherol homologs exhibit levels of antioxidant activity in mint oil which are higher than each of the homologs taken individually. Most importantly, it was not previously known that specific mixtures of tocopherol homologs could be used successfully as antioxidants in applications where individual homologs had not proven effective, namely, mint oil and mint flavored chewing gum applications.

As previously stated, the present invention relates to the use of tocopherol mixtures which effect improved antioxidant activity in mint oil and chewing gum which contains mint oil. The preferred amounts of tocopherol mixture may vary depending on the specific mint oil and/or the specific chewing gum composition. Generally, the preferred amounts of tocopherol are higher when the mint oil is to be used in chewing gum.

This is because some of the antioxidant may migrate from the mint oil into the gum base.

The following Table 2 summarizes both the preferred composition of the tocopherol mixture of the invention, and the preferred amounts which are to be used in mint oil and mint-flavored chewing gum.

Table 2

Tocopherol Composition And Amounts Used

	(A)	(B)	(C)
	Tocopherol Composition (% by weight, based on total tocopherols)	Amount Used in Mint Oil (% by weight tocopherols in mint oil)	Amount Used In Mint Oil To Be Used In Chewing Gum (% by weight tocopherols based on mint oil weight)
Preferred Range	7-15% alpha tocopherol 45-70% gamma tocopherol 20-35% delta tocopherol	.05-.50% total tocopherols	Up to 3% but may vary considerably
Most Preferred Range	9-13% alpha tocopherol 55-63% gamma tocopherol 27-32% delta tocopherol	.07-.18% total tocopherols	Up to 2%

Column B in Table 2 reflects the amount of tocopherol which is necessary to stabilize unused mint oil, such as for storage of the mint oil. Column C indicates that the antioxidant level may have to be increased before or when the mint oil is mixed with the other chewing gum components. The amount of mint oil typically used in mint flavored chewing gum varies from about .5-3% based on the weight of the chewing gum product, and is most typically about 1%. The amount of tocopherol required to stabilize the mint oil in the chewing gum will vary depending on the amount and type of mint oil and other chewing gum components. Therefore, the antioxidant amounts in column C can only be stated generally, and should be optimized individually for individual chewing gums.

When the mint oil is to be used in chewing gum, it is preferred that the tocopherol mixture be preblended with the mint oil. However, the tocopherol mixture may alternatively be mixed into the chewing gum along with the mint oil. In either case, the tocopherol should be thoroughly blended with the product to produce a homogeneous mixture.

In general, a chewing gum composition includes a water soluble bulk portion, a water insoluble chewing gum base portion and typically water insoluble flavoring agents. The water soluble portion dissipates with a portion of the flavoring agents over a period of time during chewing. The gum base portion is retained in the mouth throughout the chewing process.

The insoluble gum base generally includes elastomers, resins, fats, oils, waxes, softeners and inorganic fillers. The elastomers may include polyisobutylene, isobutylene-isoprene copolymer, styrene butadiene rubber and natural latexes such as chicle. The resins may include polyvinyl acetate and terpene resins. Low molecular weight polyvinyl acetate is a preferred resin. Fats and oils may include animal fats such as lard and tallow, vegetable oils such as soybean and cottonseed oils, hydrogenated and partially hydrogenated vegetable oils, and cocoa butter. Commonly used waxes include petroleum waxes such as paraffin and microcrystalline wax, natural waxes such as beeswax, candellia, carnauba and polyethylene wax. Preferably, the waxes have a melting point between 95°F and 240°F.

The gum base typically also includes a filler component such as calcium carbonate, magnesium carbonate, talc, dicalcium phosphate and the like; elastomers, including glycerol monostearate and glycerol triacetate; and optional ingredients such as

antioxidants, colors and emulsifiers. The gum base constitutes between 5-95% by weight of the chewing gum composition, more typically 10-50% by weight of the chewing gum, and most commonly 25-35% by weight of the chewing gum.

The water soluble portion of the chewing gum may include softeners, bulk sweeteners, high intensity sweeteners and combinations thereof. Softeners are added to the chewing gum in order to optimize the chewability and mouth feel of the gum. The softeners, which are also known as plasticizers or plasticizing agents, generally constitute between about .5-15% by weight of the chewing gum. The softeners may include glycerin, lecithin, and combinations thereof. The softeners may also include aqueous sweetener solutions such as those containing sorbitol, hydrogenated starch hydrolysates, corn syrup and combinations thereof.

Bulk sweeteners constitute between 20-80% by weight of the chewing gum and may include both sugar and sugarless sweeteners and components. Sugar sweeteners may include saccharide containing components including but not limited to sucrose, dextrose, maltose, dextrin, dried invert sugar, fructose, levulose, galactose, corn syrup solids, and the like, alone or in combination. Sugarless sweeteners include components with sweetening characteristics but are devoid of the commonly known sugars. Sugarless sweeteners include but are not limited to sugar alcohols such as sorbitol, mannitol, xylitol, hydrogenated starch hydrolysates, maltitol, and the like, alone or in combination.

High intensity sweeteners may also be present. These may include but are not limited to sucralose, aspartame, salts of acesulfame, alitame, saccharin and its salts, cyclamic acid and its salts,

dihydrochalcones, thaumatin, monellin, and the like, alone or in combination.

Combinations of sugar and/or sugarless sweeteners may be used in chewing gum. The sweetener may also function in the chewing gum in whole or in part as a water soluble bulking agent. Additionally, the softener may also provide additional sweetness such as with aqueous sugar or alditol solutions.

One or more flavoring agents are generally present in the chewing gum in an amount within the range of about .1-10% by weight of the chewing gum, preferably between about .5-3% by weight of the chewing gum. The flavoring agents may include essential oils, synthetic flavors or mixtures thereof including but not limited to oils derived from plants and fruits such as citrus oils, fruit essences, peppermint oil, spearmint oil, other mint oils, clove oil, oil of wintergreen, anise and the like. Artificial flavoring agents and components may also be used. Natural and artificial flavoring agents may be combined in any sensorally acceptable fashion.

Optional ingredients such as colors, emulsifiers and pharmaceutical agents may also be included in chewing gum.

Chewing gum is generally manufactured by sequentially adding the various chewing gum ingredients to any commercially available mixer known in the art. After the ingredients have been thoroughly mixed, the gum mass is discharged from the mixer and shaped into the desired form such as by rolling into sheets and cutting into sticks, extruding into chunks, or casting into pellets. Generally, the ingredients are mixed by first melting the gum base and adding it to the running mixer. The gum base may alternatively be melted in the

mixer. Color and emulsifiers can be added at this time.

A softener such as glycerin can be added next along with syrup and part of the bulk portion. Further parts of the bulk portion may then be added to the mixer. The flavoring agent is typically added with the final part of the bulk portion. The entire mixing process typically takes from five to fifteen minutes, although longer mixing times are sometimes required. Those skilled in the art will recognize that variations of this mixing procedure, or other mixing procedures, may be followed.

A wide range of changes and modifications to the embodiments of the invention described above will be apparent to persons skilled in the art. The following examples are not to be construed as imposing limitations on the invention, but are included merely to illustrate preferred embodiments.

EXAMPLE 1

250 grams of peppermint oil was placed in a one pint glass jar. 0.25 grams of Kodak Tenox GT-2 (70% tocopherol mixture in 30% soybean oil) was added and mixed thoroughly to give an active tocopherol level of .07%. The glass jar containing the sample was placed in a Parr bomb reactor. The Parr bomb reactor was pressurized to 100 psi with oxygen and placed in an 85°F oven.

The lot of Kodak Tenox GT-2 used contained 10.3% alpha tocopherol, 61.4% gamma tocopherol and 28.3% delta tocopherol, based on total tocopherol weight, and contained 68.4% total active tocopherol and 31.6% diluent. Tenox GT-2 is useful in the present invention. Eight other samples were prepared for comparison with Example 1, as discussed below. In each

of Examples 2-8, a different tocopherol or tocopherol mixture was substituted for Tenox GT-2. The amount of tocopherol added to the 250 grams of peppermint oil corresponded, in each case, to an active tocopherol level of approximately .07% in the peppermint oil. Example 9, a control sample, did not contain tocopherol.

The tocopherols used in the remaining Examples, and the amounts added to peppermint oil, were as follows.

EXAMPLE 2

0.175 grams of Sigma Chemical Co. T-3251 was added to 250 grams of peppermint oil in a one pint glass jar. T-3251 contains virtually 100% alpha tocopherol and no diluent. The glass jar containing the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 3

0.175 grams of Sigma Chemical Co. T-2028 was added to 250 grams of peppermint oil in a one pint glass jar. T-2028 contains 83.3% delta tocopherol and 16.7% other tocopherols, mostly gamma tocopherol, with no diluent. The glass jar containing the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 4

0.175 grams of Kodak 1187962 was added to 250 grams of peppermint oil in a one pint glass jar. Kodak 1187962 contains 99.6% gamma tocopherol and .4% other tocopherols, with no diluent. The glass jar containing

the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 5

0.344 grams of Vitamin E 4-50 from Kodak was added to 250 grams of peppermint oil in a one pint glass jar. Vitamin E 4-50 contains 52.9% alpha tocopherol, 11.8% delta tocopherol and 35.3% gamma tocopherol in a vegetable oil diluent, the mixture containing 50.7% total active tocopherol and 49.3% diluent. The glass jar containing the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 6

0.260 grams of Vitamin E 4-80 from Kodak was added to 250 grams of peppermint oil in a one pint glass jar. Vitamin E 4-80 contains 83.0% alpha tocopherol, 0.8% delta tocopherol and 16.2% gamma tocopherol in a vegetable oil diluent, the mixture containing 71.7% total active tocopherol and 28.3% diluent. The glass jar containing the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 7

0.418 grams of Vitamin E 5-40 from Kodak was added to 250 grams of peppermint oil in a one pint glass jar. Vitamin E 5-40 contains 99.1% alpha tocopherol and 0.9% gamma tocopherol in a vegetable oil diluent, the mixture containing 43.6% total active tocopherol and 56.4% diluent. The glass jar containing

the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 8

0.25 grams of Covi-ox T-70 from Henkel was added to 250 grams of peppermint oil in a one pint glass jar. Covi-ox T-70 is useful in the present invention and, according to the manufacturer, contains 12% alpha tocopherol, 2% beta tocopherol, 30% delta tocopherol and 56% gamma tocopherol in a vegetable oil diluent, the mixture containing 70% total active tocopherol and 30% diluent. The glass jar containing the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1.

EXAMPLE 9

250 grams of peppermint oil was placed in a one pint glass jar for use as a Parr bomb control. No tocopherol was added. The glass jar containing the sample was placed in a Parr bomb reactor. The reactor was placed under the same pressure and temperature conditions as used for Example 1. A second control sample was placed in a one gallon amber glass jug with a polyethylene lined cap and stored in a refrigerator to serve as a reference standard to normalize results.

ANALYSIS AND RESULTS OF EXPERIMENTS

The Parr bomb reactors of Examples 1-9 were maintained at 100 psi oxygen and stored in the 85°F oven for 50 days. Samples were removed for analysis at 0, 7, 14, 21, 28, 35, 42 and 49 days. The samples were analyzed using Gas Chromatography for para-cymene, alpha-terpinene and gamma-terpinene. The terpinenes

are oxidizable components which are depleted over time. Para-cymene is the oxidation product of the two terpinenes which increases in concentration as oxidation proceeds.

The results were calculated as an oxidation ratio of products over reactants normalized to the refrigerated control:

$$\text{Oxidation ratio (after x days)} = \frac{\text{paracymene (Tx)/total terpinenes (Tx)}}{\text{paracymene (To)/total terpinenes (To)}}$$

where Tx represents concentrations after x days, in the Parr bomb samples, and

To represents concentrations in the refrigerated reference standard, measured at the same times as the Parr bomb samples.

The ratio would be 1/1 for an unoxidized Parr bomb sample with higher ratios occurring as the samples oxidize. The results are shown graphically in Figures 1-3.

Figure 1 compares the Tenox GT-2 samples useful in the invention (Example 1) with the three constituent tocopherol homologs (Examples 2-4), and with the unprotected Parr bomb control (Example 9). The Parr bomb control oxidized very quickly and all of the tocopherol samples (Examples 1-4) showed significant antioxidant effect. However, the tocopherol mixture useful in the invention (Example 1) exhibited significantly greater antioxidant activity than each of the constituent homologs (Examples 2-4), especially at longer time periods. Figure 1 clearly illustrates the existence of a surprising and beneficial synergistic relationship between the three homologs, when the

homologs are mixed in concentrations according to the invention.

Figure 2 compares the Tenox GT-2 samples (Example 1) with three vitamin E products (Examples 5-7) which are tocopherol mixtures falling outside the ranges of the invention. The Tenox GT-2 useful in the invention showed significantly greater antioxidant activity than each of the vitamin E products. In fact, the vitamin E products performed no better, and as a group were worse, than the individual homologs of Examples 2-4 plotted in Figure 1.

Figure 3 compares the Tenox GT-2 sample (Example 1) with the Covi-ox T-70 sample (Example 8) which is also useful in the invention. Both of the foregoing were compared with the vitamin E 4-50 sample (Example 5), for time periods of up to thirty-five days. The Tenox GT-2 and Covi-ox T-70 exhibited substantially identical antioxidant performance, and both outperformed the vitamin E 4-50 sample. This graph illustrates that the surprising and beneficial effects obtained with the invention are reproducible.

While the embodiments of the invention disclosed herein are at present considered to be preferred, various modifications and improvements can be made without departing from the spirit and scope of the invention. The scope of the invention is indicated in the appended claims, and all changes which come within the meaning and range of equivalency of the claims are intended to be embraced therein.

~~WE CLAIM:~~
The claims defining the invention are as follows:

1. A stabilized mint oil composition, comprising:

a mint oil selected from the group consisting of peppermint oil, spearmint oil, corn mint oil, Scotch spearmint oil and mixtures thereof; and

.05-3.0% by weight of a tocopherol mixture blended into the mint oil, the tocopherol mixture comprising 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

2. The stabilized mint oil composition of claim 1 comprising .05-2.0% by weight of the tocopherol mixture.

3. The stabilized mint oil composition of claim 1 comprising .05-.50% by weight of the tocopherol mixture.

4. The stabilized mint oil composition of claim 2 comprising .07-2.0% by weight of the tocopherol mixture.

5. The stabilized mint oil composition of claim 4 comprising .07-.18% by weight of the tocopherol mixture.

6. The stabilized mint oil composition of claim 1 wherein the tocopherol mixture comprises 9-13% alpha tocopherol, 55-63% gamma tocopherol and 27-32% delta tocopherol.

7. The stabilized mint oil composition of claim 1 in a chewing gum, in an amount of .5-3% of the weight of the chewing gum, wherein the mint oil comprises peppermint oil.

8. The stabilized mint oil composition of claim 1 in a chewing gum, in an amount of .5-3% of the weight of the chewing gum, wherein the mint oil comprises spearmint oil.

9. The stabilized mint oil composition of claim 1 in a chewing gum, in an amount of .5-3% of the weight of the chewing gum, wherein the mint oil comprises a blend of peppermint oil and spearmint oil.

10. The stabilized mint oil composition of claim 1 in a chewing gum, in an amount of .5-3% of the weight of the chewing gum, wherein the mint oil comprises corn mint oil.

11. The stabilized mint oil composition of claim 1 in a chewing gum, in an amount of .5-3% of the weight of the chewing gum, wherein the mint oil comprises Scotch spearmint oil.

12. A stabilized peppermint oil composition, which comprises peppermint oil and .05-3% by weight of an antioxidant consisting essentially of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

13. The stabilized peppermint oil composition of claim 12 in a chewing gum.

14. The stabilized peppermint oil composition of claim 13, comprising .07-2% by weight of the antioxidant.

15. A stabilized spearmint oil composition, which comprises spearmint oil and .05-3% by weight of an antioxidant consisting essentially of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

16. The stabilized spearmint oil composition of claim 15 in a chewing gum.

17. The stabilized spearmint oil composition of claim 16 comprising .07-2% by weight of the antioxidant.

18. A stabilized mint oil composition, which comprises peppermint oil and spearmint oil and .05-3% by weight of an antioxidant consisting essentially of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

19. A stabilized corn mint oil composition, which comprises corn mint oil and .05-3% by weight of an antioxidant consisting essentially of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

20. The stabilized corn mint oil composition of claim 19 in a chewing gum.

21. A stabilized Scotch spearmint oil composition, which comprises Scotch spearmint oil and .05-3% by weight of an antioxidant consisting essentially of

7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

22. The stabilized Scot spearmint oil of claim 21 in a chewing gum.

23. A mint flavored chewing gum composition, comprising a water soluble bulk portion, a water insoluble chewing gum base portion and a flavoring agent, the flavoring agent comprising peppermint oil and an antioxidant which consists primarily of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

24. A mint flavored chewing gum composition, comprising a water soluble bulk portion, a water insoluble chewing gum base portion and a flavoring agent, the flavoring agent comprising spearmint oil and an antioxidant which consists primarily of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

25. A mint flavored chewing gum composition, comprising a water soluble bulk portion, a water insoluble chewing gum base portion and a flavoring agent, the flavoring agent comprising corn oil and an antioxidant which consists primarily of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

26. A mint flavored chewing gum composition, comprising a water soluble bulk portion, a water insoluble chewing gum base portion and a flavoring agent, the flavoring agent comprising Scotch spearmint oil and an antioxidant which consists primarily of 7-15% by

weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol.

27. A stabilized mint oil composition substantially as hereinbefore described with reference to any one of the Examples.

28. A mint flavored chewing gum composition substantially as hereinbefore described with reference to any one of the Examples.

DATED this TWENTY-SECOND day of JUNE 1992

Wm. Wrigley Jr. Company

Patent Attorneys to the Applicant

SPRUSON & FERGUSON

Tocopherol Mixture for Use as a Mint Oil Antioxidant in Chewing Gum

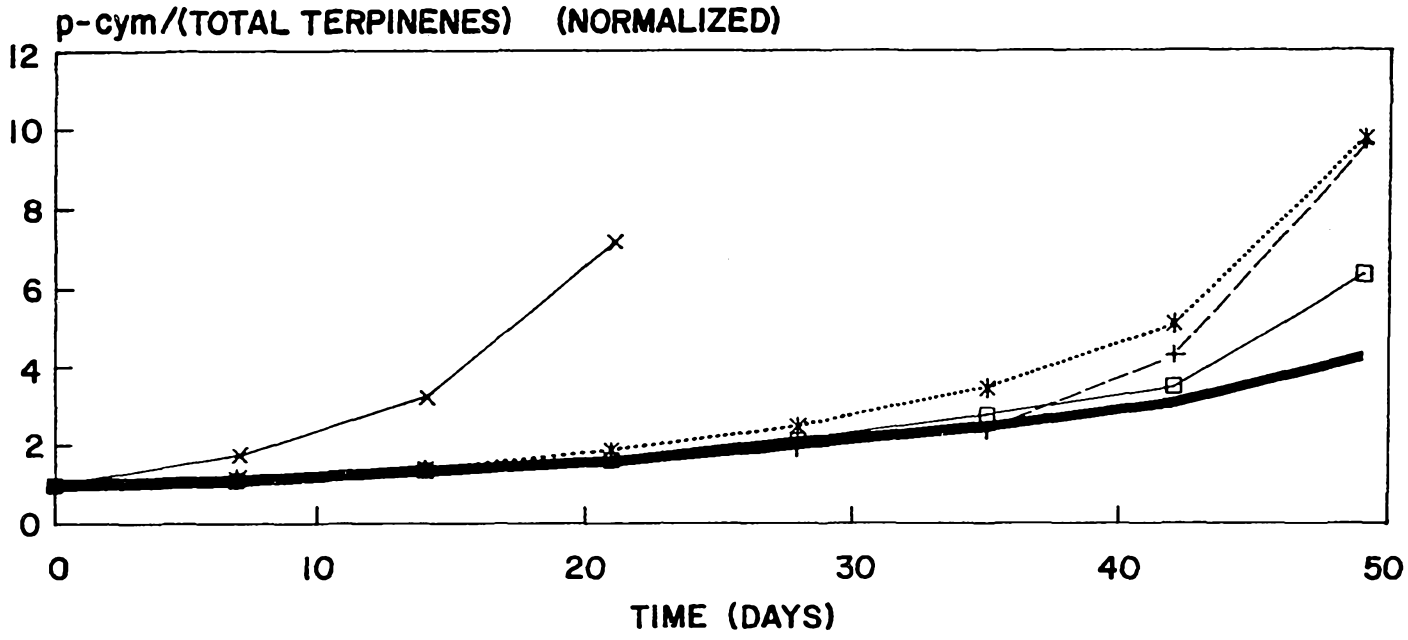
ABSTRACT OF THE DISCLOSURE

Specific mixtures of tocopherol homologs exhibit surprisingly improved antioxidant effects in mint oil and mint-flavored chewing gum compositions. The mixtures of homologs fall within the range of 7-15% by weight alpha tocopherol, 45-70% by weight gamma tocopherol and 20-35% by weight delta tocopherol. Homologue mixtures within this range are superior to the constituent homologs used alone, and to other homologue mixtures, in terms of antioxidant effects. The compositions of the invention facilitate the replacement of controversial synthetic antioxidants currently used in mint oil and mint-flavored chewing gum, with naturally occurring tocopherols.

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FIG. 1

PEPPERMINT OXIDATION RATIOS
TENOX GT-2 and PURE TOCOPHEROLS



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FIG. 2

PEPPERMINT OXIDATION RATIOS
TENOX GT-2 and VITAMIN E MIXTURES

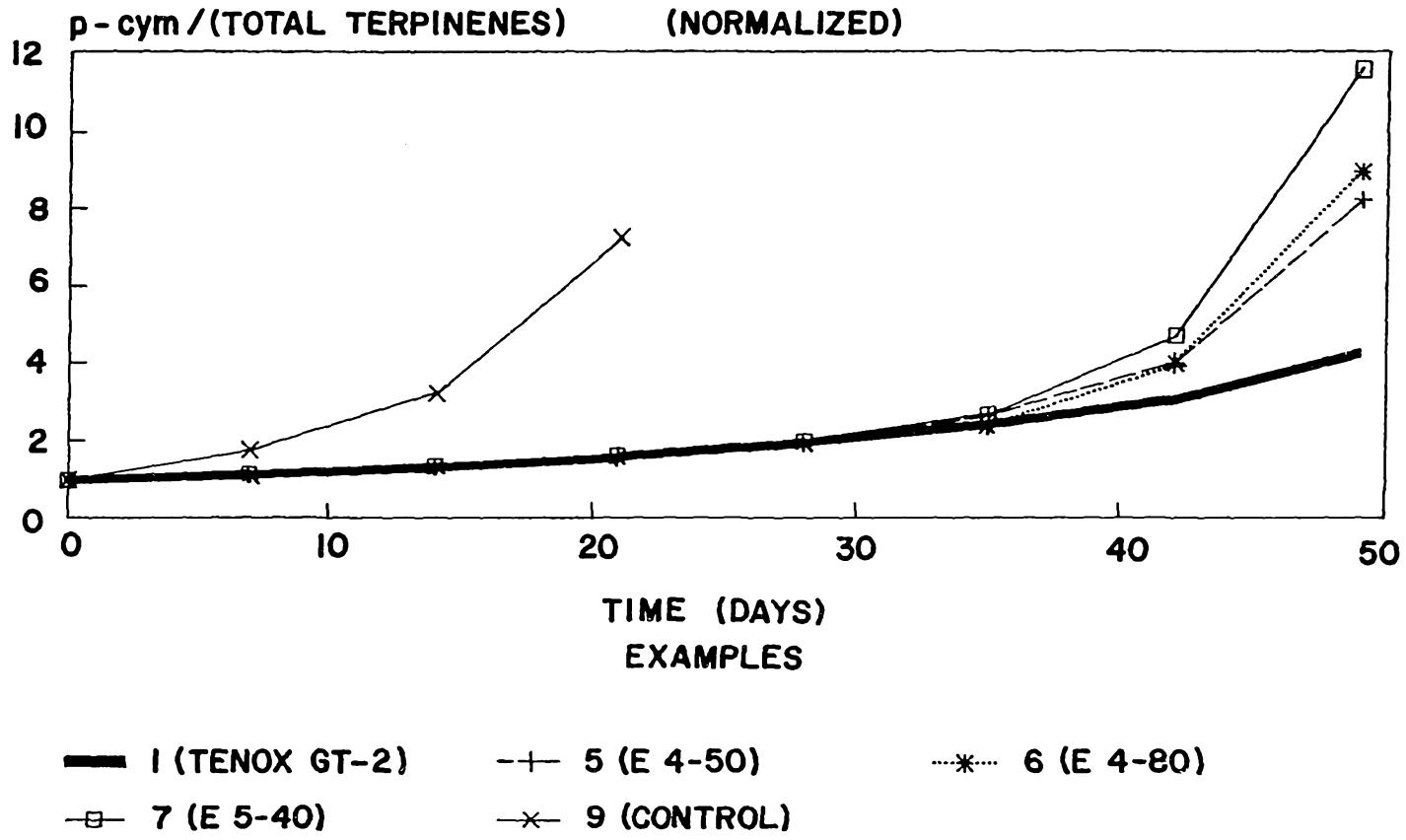
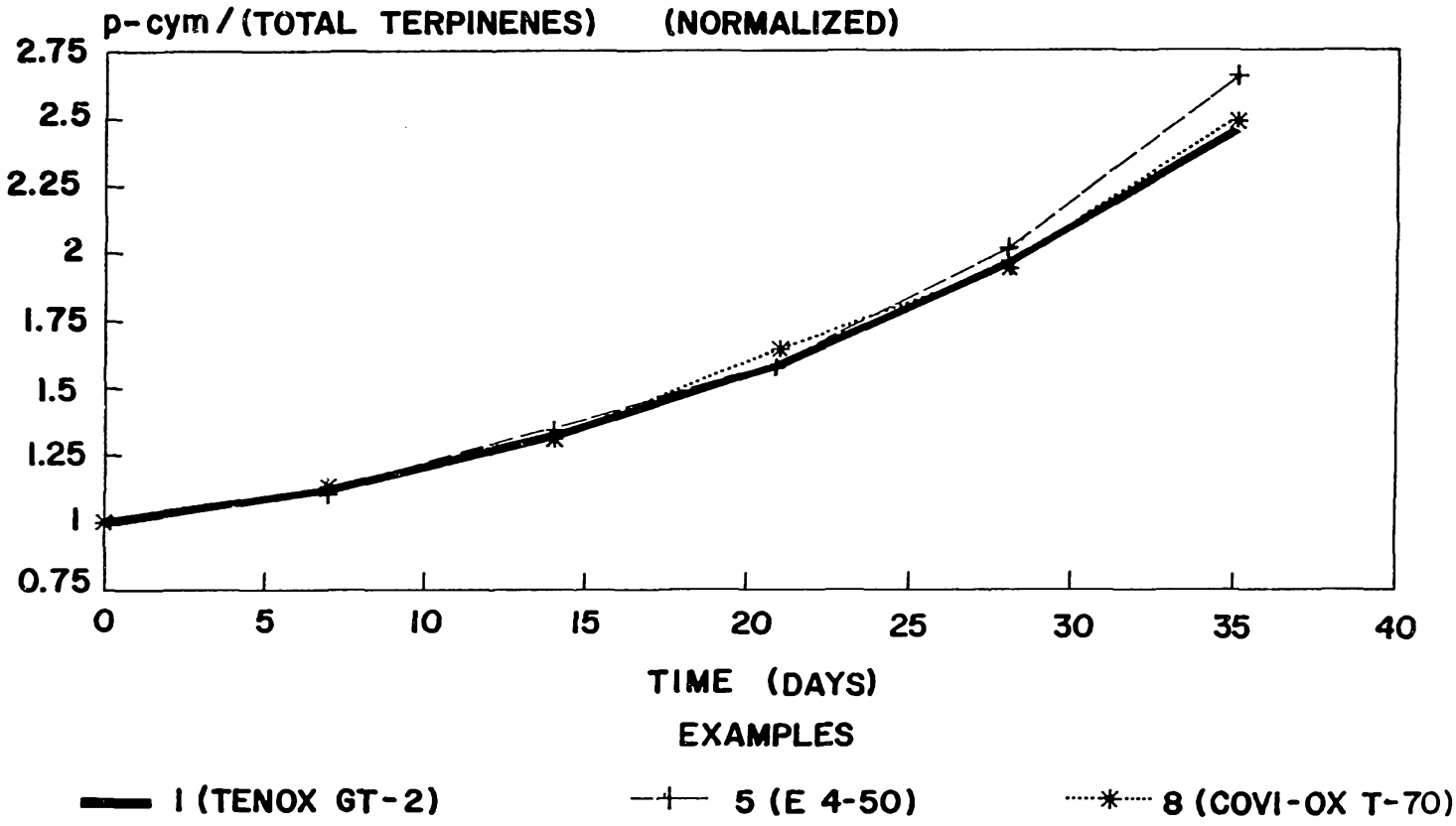


FIG. 3


PEPPERMINT OXIDATION RATIOS
TENOX GT-2, COVI-OX T-70, VITAMIN E
p-cym / (TOTAL TERPINENES) (NORMALIZED)
2.75
2.5
2.25
2
1.75
1.5
1.25
1
0.75
0
5
10
15
20
25
30
35
40
TIME (DAYS)
EXAMPLES
1 (TENOX GT-2)
5 (E 4-50)
8 (COVI-OX T-70)