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 (54) Title: POROUS, DISSOLVABLE SOLID SUBSTRATES AND SURFACE RESIDENT CYCLODEXTRIN PERFUME COMPLEXES

(57) **Abrégé/Abstract:**

The present invention relates to personal care compositions, especially those personal care compositions in the form of a personal care article that is a porous dissolvable solid substrate. The porous dissolvable solid substrate has a surface resident coating comprising a cyclodextrin perfume complex that can provide a consumer benefit.

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(57) Abstract: The present invention relates to personal care compositions, especially those personal care compositions in the form of a personal care article that is a porous dissolvable solid substrate. The porous dissolvable solid substrate has a surface resident coating comprising a cyclodextrin perfume complex that can provide a consumer benefit.



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## POROUS, DISSOLVABLE SOLID SUBSTRATES AND SURFACE RESIDENT CYCLODEXTRIN PERFUME COMPLEXES

### FIELD OF THE INVENTION

The present invention relates to personal care compositions, especially those personal care compositions providing personal care benefits in the form of an article comprising a porous dissolvable solid substrate and surface resident cyclodextrin perfume complexes.

### BACKGROUND OF THE INVENTION

Personal care compositions have traditionally been sold as liquid products. These liquid personal care products typically comprise a substantial amount of water in the formula. In addition to being liquids, many of the personal care products contain perfumes, as consumers typically desire perfumed personal care products. However, due to the water present in the personal care products, inclusion of perfume is limited to either the solubilization or emulsification within aqueous amphiphile assemblies (micelles, liquid crystals etc.). As a result, a significant portion of the perfume molecules are “trapped” within these aqueous assemblies which can limit the efficient delivery of the perfume to the nose of the consumer during product usage (e.g., lower perfume bloom) and to the target keratinous substrate (e.g., skin and/or hair). Moreover, choice of perfume is limited to those that deliver a single scent experience to the consumer (e.g., the scent of the product as packaged is the same and the only perfume experienced during use), and precludes delivery of a perfume that is activated by water during use.

Thus it is an object of the invention to provide a personal care product that can more efficiently deliver perfumes during consumer usage. It is also an object of the invention to provide a personal care product that delivers a perfume that can be water activated, i.e., the release of the perfume being maximally triggered via the addition of water to the product during usage. It is additionally an object of the invention to provide a personal care product that can deliver not only a primary perfume, but a secondary perfume that results in a second burst of perfume that is activated by water during use. For example the personal care product can have one perfume prior to being combined with water, and after exposure to water the personal care product can have a second perfume and/or a perfume bloom of the same original perfume.

### SUMMARY OF THE INVENTION

The present invention meets the aforementioned needs. In one aspect, the present invention provides a porous dissolvable solid substrate in the form of a unit dose personal care article that can be conveniently and quickly dissolved in the palm of the consumer's hand to reconstitute a liquid personal care composition for ease of application to the target keratinous substrate (e.g., skin and/or hair) while providing the consumer desired perfumes both before and during use.

In another aspect, the present invention provides a personal care article comprising: (i) a porous dissolvable solid substrate comprising: from about 10% to about 75% of a surfactant; from about 10% to about 50% water-soluble polymer; from about 1% to about 30% plasticizer; and (ii) a surface resident coating comprising from about 10% to about 100% of one or more cyclodextrin perfume complexes; wherein the ratio of the cyclodextrin to perfume in the complex is from about 100:1 to about 1:1, and wherein the ratio of the porous dissolvable solid substrate to the surface resident coating is from 110:1 to about 0.5:1.

In one embodiment, the present invention provides a method for making a personal care article, the method comprising applying a surface resident coating comprising the cyclodextrin perfume complex in powdered form to a porous dissolvable solid substrate comprising: from about 10% to about 75% of a surfactant, from about 10% to about 50% water-soluble polymer, and from about 1% to about 30% plasticizer.

In another embodiment, the present invention provides a method for making a personal care article. This method comprises: preparing a processing mixture comprising from about 5% to about 50% of a surfactant, from about 5% to about 35% water-soluble polymer, and from about 0.5% to about 20% plasticizer; aerating the processing mixture by introducing a gas into the processing mixture to form an aerated wet mixture; forming the aerated wet mixture into one or more desired shapes; drying the aerated wet mixture to form a porous dissolvable solid substrate; and applying a surface resident coating comprising the cyclodextrin perfume complex in powdered form to the porous dissolvable solid substrate.

### BRIEF DESCRIPTION OF THE DRAWINGS

While the specification concludes with claims which particularly point out and distinctly claim the present invention, it is believed that the present invention will be better understood from

the following description of embodiments, taken in conjunction with the accompanying drawings, in which like reference numerals identify identical elements and wherein:

FIG. 1 is a schematic view of a porous dissolvable solid substrate with a surface resident cyclodextrin perfume complex.

FIG. 2 is a schematic view of two porous dissolvable solid substrates with a surface resident cyclodextrin perfume complex layer in between the two substrates.

FIGS. 3A, and 3B are a schematic view of a dimpled porous dissolvable solid substrate with a surface resident cyclodextrin perfume complex inside the dimples.

FIG. 4 is a schematic view of a porous dissolvable solid substrate that is folded over to enclose a surface resident cyclodextrin perfume complex.

FIG. 5 shows the GC/MS headspace ratio of perfume raw materials (PRM) from a dissolved shampoo pad (7:1 water dilution) vs. the original dry shampoo pad

FIG. 6 shows GC headspace abundance of the more volatile perfume raw materials from a dissolved shampoo pad (7:1 water dilution) vs. the original dry shampoo pad.

FIG. 7 shows GC headspace abundance of the less volatile perfume raw materials from a dissolved shampoo pad (7:1 water dilution) vs. the original dry shampoo pad.

FIG. 8 shows the GC/MS headspace ratio of perfume raw materials (PRM) from a dissolved shampoo pad (7:1 water dilution) vs. the neat liquid shampoo control.

FIG. 9 shows the GC/MS headspace ratio of perfume raw materials (PRM) from a dissolved and fully diluted shampoo pad vs. the fully diluted liquid shampoo control (10 parts de-ionized water to 1 part liquid shampoo or dissolved pad shampoo).

FIG. 10 shows GC headspace abundance of perfume raw materials (PRM) from a dissolved shampoo pad (7:1 water dilution) vs. the neat liquid shampoo control.

#### DETAILED DESCRIPTION OF THE INVENTION

In all embodiments of the present invention, all percentages are by weight of the total composition, unless specifically stated otherwise. All ratios are weight ratios, unless specifically stated otherwise. All ranges are inclusive and combinable. The number of significant digits conveys neither a limitation on the indicated amounts nor on the accuracy of the measurements. All numerical amounts are understood to be modified by the word "about" unless otherwise specifically indicated. Unless otherwise indicated, all measurements are understood to be made at 25°C and at ambient conditions, where "ambient conditions" means conditions under about one atmosphere of pressure and at about 50% relative humidity. All such weights as they pertain to

listed ingredients are based on the active level and do not include carriers or by-products that may be included in commercially available materials, unless otherwise specified.

## **I. Definitions**

As used herein, the term “personal care composition” means a composition that may be applied to mammalian hair and skin without undue undesirable effects.

The term, “surface resident cyclodextrin perfume complex,” as used herein, refers to a surface resident coating comprising a cyclodextrin perfume complex that is adsorbed to at least a portion of the solid/air interface of the porous dissolvable solid substrate. The resulting surface resident coating minimizes the physical interactions between the cyclodextrin perfume complex and the bulk of the dissolvable porous solid over the shelf life of the product, and before the personal care article is put in contact with water during consumer use.

As used herein, “personal care article” means the porous dissolvable solid substrate comprising a surfactant, water-soluble polymer, and plasticizer, along with the surface resident coating of the cyclodextrin perfume complex. The personal care article may be referred to herein as “the article.”

As used herein, “dissolvable” means that the porous dissolvable solid substrate has a dissolution rate that satisfies the Hand Dissolution Method Test described herein.

As used herein “porous dissolvable solid substrate” means a solid polymer-containing matrix that defines an interconnected network of spaces or cells that contain the gas of the surrounding atmosphere, typically air. The interconnectivity of the structure may be described by a Star Volume, a Structure Model Index (SMI) or a Percent Open Cell Content.

As used herein, “Group I” means anionic surfactants that are suitable for use in personal care compositions.

As used herein, “Group II” means a surfactant selected from the group consisting of amphoteric, zwitterionic, and combinations thereof, that are suitable for use in personal care compositions.

## **II. Personal Care Article**

The personal care article of the present invention delivers a unique perfume experience to the consumer from a lathering/cleansing product by enabling the more efficient delivery of perfumes as well as the water activated release of a perfume during consumer usage. The water activated perfume may encompass a secondary perfume such that the personal care product can have one perfume prior to being combined with water, and after exposure to water the personal care product can have a second perfume and/or a perfume bloom of the same original perfume.

This is achieved by incorporating a surface resident coating comprising a cyclodextrin perfume complex on the porous dissolvable solid substrate. Any suitable application method can be used to apply the surface resident coating comprising the cyclodextrin perfume complex to the porous dissolvable solid substrate to form a surface resident coating that is adsorbed to at least a portion of the solid/air interface of the porous dissolvable solid substrate. In one embodiment a surface resident coating comprising the cyclodextrin perfume complex is in the form of a powder coating, which is applied to the surface of the porous dissolvable solid substrate.

Traditionally, perfumes are delivered from aqueous liquid consumer products via the solubilization or emulsification of the perfume within stabilized aqueous amphiphile assemblies (e.g., micelles, liquid crystals, etc.). This may result in the less efficient delivery of the perfume to the nostrils of the consumer during product usage (i.e., lower perfume bloom) and to the target keratinous substrate (i.e., skin and/or hair). This may be due to the “entrapment” of the perfume molecules within the stabilized aqueous amphiphile assemblies which may hinder the diffusion of the hydrophobic perfume molecules through the aqueous phase to the water/air and water/substrate interfaces during product usage, thereby reducing the efficient delivery of the perfume molecules to the intended targets (consumer nostrils and keratinous substrates) and even resulting in greater amounts of the perfume molecules being wasted (e.g., rinsed down the drain). While this may still occur in the present inventive personal care articles as they are dissolved in water immediately prior to application to the hair or skin, such physical “entrapment” interactions are minimized when the personal care articles are made and/or used according to the present invention.

The present invention also enables the delivery of a perfume that is activated by water, i.e., the release of the perfume being maximally triggered via the addition of water to the product during usage. This occurs due to the cyclodextrin perfume complexes of the present invention being water releasable, i.e., the cyclodextrin dissolves with the perfume physically released or liberated as the personal care article is dissolved in water.

This present invention additionally enables the unique and optional delivery of both a primary perfume and a secondary perfume during the product usage experience by the consumer. For example, the personal care product can have one perfume prior to being combined with water, and after exposure to water the personal care product can have a second perfume and/or a more intense perfume bloom of the same original perfume. This can be achieved by incorporating a secondary perfume within the cyclodextrin perfume complex with a perfume distinct from the primary perfume. Alternatively, the same perfume may be employed within the



cyclodextrin perfume complex, but enabling a more intense perfume bloom upon the dissolution of the personal care article.

#### **A. The Porous Dissolvable Solid Substrate**

The porous dissolvable solid substrate comprises a surfactant, a water-soluble polymer, and a plasticizer. The porous dissolvable solid substrate can be prepared such that it can be conveniently and quickly dissolved in the palm of the consumer resulting in a liquid personal care composition. Once dissolved, this personal care composition can be used in a manner similar to conventional liquid personal care compositions, i.e. applied to the scalp and/or hair. The porous dissolvable solid substrate has a maximum Cell Wall Thickness. The porous dissolvable solid substrate has a Cell Wall Thickness of from about from about 0.02 mm to about 0.15 mm, in one embodiment from about 0.025 mm to about 0.12 mm, in another embodiment from about 0.03 mm to about 0.09 mm, and in still another embodiment from about 0.035 mm to about 0.06 mm. The porous dissolvable solid substrate has a minimum level of interconnectivity between the cells, which is quantified by the Star Volume, the Structure Model Index (SMI), and the Percent Open Cell Content. The porous dissolvable solid substrate has a Star Volume of from about 1 mm<sup>3</sup> to about 90 mm<sup>3</sup>, in one embodiment from about 1.5 mm<sup>3</sup> to about 60 mm<sup>3</sup>, in another embodiment from about 2 mm<sup>3</sup> to about 30 mm<sup>3</sup>, and in still another embodiment from about 2.5 mm<sup>3</sup> to about 15 mm<sup>3</sup>. The porous dissolvable solid substrate has a non-negative Structure Model Index of from about 0.0 to about 3.0, in one embodiment from about 0.5 to about 2.75, and in another embodiment from about 1.0 to about 2.50. The porous dissolvable solid substrate has a Percent Open Cell Content of from about 80% to 100%, in one embodiment from about 85% to about 97.5%, and in another embodiment from about 90% to about 95%. The porous dissolvable solid substrate also has a minimum Specific Surface Area. The porous dissolvable solid substrate has a Specific Surface Area of from about 0.03 m<sup>2</sup>/g to about 0.25 m<sup>2</sup>/g, in one embodiment from about 0.035 m<sup>2</sup>/g to about 0.22 m<sup>2</sup>/g, in another embodiment from about 0.04 m<sup>2</sup>/g to about 0.19 m<sup>2</sup>/g, and in still another embodiment from about 0.045 m<sup>2</sup>/g to about 0.16 m<sup>2</sup>/g. The porous dissolvable solid substrate has a basis weight of from about 125 grams/m<sup>2</sup> to about 3,000 grams/m<sup>2</sup>, in one embodiment from about 300 grams/m<sup>2</sup> to about 2,500 grams/m<sup>2</sup>, in another embodiment from about 400 grams/m<sup>2</sup> to about 2,000 grams/m<sup>2</sup>, in another embodiment from about 500 grams/m<sup>2</sup> to about 1,500 grams/m<sup>2</sup>, and in another embodiment from about 600 grams/m<sup>2</sup> to about 1,200 grams/m<sup>2</sup>, and in another embodiment from about 700 to about 1,000 grams/m<sup>2</sup>. The porous dissolvable solid substrate has a solid density of from about 0.03 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup>, in one embodiment from about 0.05 g/cm<sup>3</sup> to about 0.35 g/cm<sup>3</sup>,

in another embodiment from about 0.08 g/cm<sup>3</sup> to about 0.30 g/cm<sup>3</sup>, in another embodiment from about 0.10 g/cm<sup>3</sup> to about 0.25 g/cm<sup>3</sup>, and in another embodiment from about 0.12 g/cm<sup>3</sup> to about 0.20 g/cm<sup>3</sup>.

In one embodiment, the porous dissolvable solid substrate of present invention is a flat, flexible substrate in the form of a pad, a strip, or tape, and having a thickness of from about 0.5 mm to about 10 mm, in one embodiment from about 1 mm to about 9 mm, in another embodiment from about 2 mm to about 8 mm, and in a further embodiment from about 3 mm to about 7 mm as measured by the methodology below.

### **1. Surfactants**

The porous dissolvable solid substrates of the present invention may be lathering or non-lathering under consumer relevant usage instructions. The porous dissolvable substrates include at least one surfactant as a processing aid to generate a stable foam solid prior to drying (solidification) and in the case of a lathering substrate the surfactant may also serve dual functions as a foaming and/or cleansing agent.

#### **a. Lathering Porous Dissolvable Solid Substrates**

Lathering porous dissolvable solid substrates for the purposes of lathering and/or cleaning comprise from about 10% to about 75%, in one embodiment from about 30% to about 70%, and in another embodiment from about 40% to about 65% by weight of the personal care article of surfactant; wherein the surfactant comprises one or more surfactants from Group I, wherein Group I means anionic surfactants which are suitable for use in hair care or other personal care compositions, and optionally one or more surfactants from Group II, wherein Group II means a surfactant selected from the group consisting of amphoteric, zwitterionic and combinations thereof suitable for use in hair care or other personal care compositions; wherein the ratio of Group I to Group II surfactants is from about 100:0 to about 30:70. In another embodiment of the present invention the ratio of Group I to Group II surfactants is from about 85:15 to about 40:60. In yet another embodiment of the present invention the ratio of Group I to Group II surfactants is from about 70:30 to about 55:45.

Non limiting examples of anionic surfactants are described in U.S. Pat. Nos. 2,486,921; 2,486,922; and 2,396,278. The anionic surfactant can be selected from the group consisting of alkyl and alkyl ether sulfates, sulfated monoglycerides, sulfonated olefins, alkyl aryl sulfonates, primary or secondary alkane sulfonates, alkyl sulfosuccinates, acid taurates, acid isethionates, alkyl glycerylether sulfonate, sulfonated methyl esters, sulfonated fatty acids, alkyl phosphates,

acyl glutamates, acyl sarcosinates, alkyl lactylates, anionic fluorosurfactants, sodium lauroyl glutamate, and combinations thereof.

Non limiting examples of suitable zwitterionic or amphoteric surfactants are described in U.S. Pat. Nos. 5,104,646 (Bolich Jr. et al.) and 5,106,609 (Bolich Jr. et al.).

Additional suitable Group I and Group II surfactants include those disclosed in U.S. Patent Application No. 61/120,765 and those surfactants disclosed in McCutcheon's Detergents and Emulsifiers, North American Edition (1986), Allured Publishing Corp.; McCutcheon's, Functional Materials, North American Edition (1992), Allured Publishing Corp.; and U.S. Patent 3,929,678 (Laughlin et al.). Other non-limiting examples of suitable surfactants are included in U.S. Serial No. 61/120,790.

**b. Non – lathering porous dissolvable solid substrates**

The non-lathering porous dissolvable solid substrates comprise from about 10% to about 75%, in another embodiment from about 15% to about 60%, and in another embodiment from about 20% to about 50% by weight of the personal care article of surfactant; wherein the surfactant comprises one or more of the surfactants described below.

(i) Anionic Surfactants

If the porous dissolvable solid substrate of the present invention is non lathering, the substrate may comprise a maximum level of 10% (or less than 10%) of anionic surfactants to be used primarily as a process aid in making a stable foam solid.

(ii) Non-ionic surfactants

In one embodiment non-ionic surfactants are included as a process aid in making a stable porous dissolvable solid substrate. Suitable nonionic surfactants for use in the present invention include those described in McCutcheon's Detergents and Emulsifiers, North American edition (1986), Allured Publishing Corp., and McCutcheon's Functional Materials, North American edition (1992). Suitable nonionic surfactants for use in the personal care compositions of the present invention include, but are not limited to, polyoxyethylenated alkyl phenols, polyoxyethylenated alcohols, polyoxyethylenated polyoxypropylene glycols, glyceryl esters of alkanolic acids, polyglyceryl esters of alkanolic acids, propylene glycol esters of alkanolic acids, sorbitol esters of alkanolic acids, polyoxyethylenated sorbitol esters of alkanolic acids, polyoxyethylene glycol esters of alkanolic acids, polyoxyethylenated alkanolic acids, alkanolamides, N-alkylpyrrolidones, alkyl glycosides, alkyl polyglucosides, alkylamine oxides, polyoxyethylenated silicones, and combinations thereof.

(iii) Polymeric surfactants

Polymeric surfactants can also be surfactants employed as a process aid in making the porous dissolvable solid substrate of the present invention, either alone or in combination with ionic and/or nonionic surfactants. Suitable polymeric surfactants for use in the personal care compositions of the present invention include, but are not limited to, block copolymers of ethylene oxide and fatty alkyl residues, block copolymers of ethylene oxide and propylene oxide, hydrophobically modified polyacrylates, hydrophobically modified celluloses, silicone polyethers, silicone copolyol esters, diquatary polydimethylsiloxanes, co-modified amino/polyether silicones, and combinations thereof.

## **2. Water-Soluble Polymer (“Polymer Structurant”)**

The porous dissolvable solid substrate comprises water-soluble polymers that function as a structurant. As used herein, the term “water-soluble polymer” is broad enough to include both water-soluble and water-dispersible polymers, and is defined as a polymer with a solubility in water, measured at 25°C, of at least about 0.1 gram/liter (g/L). In some embodiments, the polymers have solubility in water, measured at 25°C, of from about 0.1 gram/liter (g/L) to about 500 grams/liter (g/L). (This indicates production of a macroscopically isotropic or transparent, colored or colorless solution). The polymers for making these solids may be of synthetic or natural origin and may be modified by means of chemical reactions. They may or may not be film-forming. These polymers should be physiologically acceptable, i.e., they should be compatible with the skin, mucous membranes, the hair and the scalp.

The one or more water-soluble polymers may be present from about 10% to about 50% by weight of the porous dissolvable solid substrate, in one embodiment from about 15% to about 40% by weight of the porous dissolvable solid substrate, and in yet another embodiment from about 20% to about 30% by weight of the porous dissolvable solid substrate.

The one or more water-soluble polymers of the present invention are selected such that their weighted average molecular weight is from about 40,000 to about 500,000, in one embodiment from about 50,000 to about 400,000, in yet another embodiment from about 60,000 to about 300,000, and in still another embodiment from about 70,000 to about 200,000. The weighted average molecular weight is computed by summing the average molecular weights of each polymer raw material multiplied by their respective relative weight percentages by weight of the total weight of polymers present within the porous dissolvable solid substrate.

In one embodiment, at least one of the one or more water-soluble polymers is chosen such that about 2% by weight solution of the water-soluble polymer gives a viscosity at 20°C of from

about 4 centipoise to about 80 centipoise; in an alternate embodiment from about 5 centipoise to about 70 centipoise; and in another embodiment from about 6 centipoise to about 60 centipoise.

The water-soluble polymer(s) of the present invention can include, but are not limited to, synthetic polymers as described in U.S. Serial No. 61/120,786, including polymers derived from acrylic monomers such as the ethylenically unsaturated carboxylic monomers and ethylenically unsaturated monomers as described in US 5,582,786 and EP-A-397410. The water-soluble polymer(s) which are suitable may also be selected from naturally sourced polymers including those of plant origin examples which are described in U.S. Serial No. 61/120,786. Modified natural polymers are also useful as water-soluble polymer(s) in the present invention and are included in U.S. Serial No. 61/120,786. In one embodiment, water-soluble polymers of the present invention include polyvinyl alcohols, polyacrylates, polymethacrylates, copolymers of acrylic acid and methyl acrylate, polyvinylpyrrolidones, polyalkylene oxides, starch and starch derivatives, pullulan, gelatin, hydroxypropylmethylcelluloses, methycelluloses, and carboxymethylcelluloses. In another embodiment, water-soluble polymers of the present invention include polyvinyl alcohols, and hydroxypropylmethylcelluloses. Suitable polyvinyl alcohols include those available from Celanese Corporation (Dallas, TX) under the CELVOL® trade name. Suitable hydroxypropylmethylcelluloses include those available from the Dow Chemical Company (Midland, MI) under the METHOCEL® trade name.

In a particular embodiment, the above mentioned water-soluble polymer(s) may be blended with any single starch or combination of starches as a filler material in such an amount as to reduce the overall level of water-soluble polymers required, so long as it helps provide the personal care article with the requisite structure and physical/chemical characteristics as described herein.

In such instances, the combined weight percentage of the water-soluble polymer(s) and starch-based material generally ranges from about 10% to about 50%, in one embodiment from about 15% to about 40%, and in a particular embodiment from about 20% to about 30% by weight relative to the total weight of the porous dissolvable solid substrate. The weight ratio of the water-soluble polymer(s) to the starch-based material can generally range from about 1:10 to about 10:1, in one embodiment from about 1:8 to about 8:1, in still another embodiment from about 1:7 to about 7:1, and in yet another embodiment from about 6:1 to about 1:6.

Typical sources for starch-based materials can include cereals, tubers, roots, legumes and fruits. Native sources can include corn, pea, potato, banana, barley, wheat, rice, sago, amaranth, tapioca, arrowroot, canna, sorghum, and waxy or high amylase varieties thereof. The starch-

based materials may also include native starches that are modified using any modification known in the art, including those described in U.S. Serial No. 61/120,786.

### **3. Plasticizer**

The porous dissolvable solid substrate of the present invention comprises a water soluble plasticizing agent suitable for use in personal care compositions. In one embodiment, the one or more plasticizers may be present from about 1% to about 30% by weight of the porous dissolvable solid substrate; in another embodiment from about 3% to about 25%; in another embodiment from about 5% to about 20%, and in yet another embodiment, from about 8% to about 15%. Non-limiting examples of suitable plasticizing agents include polyols, copolyols, polycarboxylic acids, polyesters and dimethicone copolyols. Examples of useful polyols include, but are not limited to, glycerin, diglycerin, propylene glycol, ethylene glycol, butylene glycol, pentylene glycol, cyclohexane dimethanol, hexane diol, polyethylene glycol (200-600), sugar alcohols such as sorbitol, manitol, lactitol and other mono- and polyhydric low molecular weight alcohols (e.g., C2-C8 alcohols); mono di- and oligo-saccharides such as fructose, glucose, sucrose, maltose, lactose, and high fructose corn syrup solids and ascorbic acid. Suitable examples of polycarboxylic acids for use herein are disclosed in U.S. Serial No. 61/120,786.

In one embodiment, the plasticizers include glycerin or propylene glycol and combinations thereof. European Patent Number EP283165B1 discloses other suitable plasticizers, including glycerol derivatives such as propoxylated glycerol.

### **4. Optional Ingredients**

The porous dissolvable solid substrate may further comprise other optional ingredients that are known for use or otherwise useful in personal care compositions, provided that such optional materials are compatible with the selected essential materials described herein, or do not otherwise unduly impair the performance of the personal care composition.

Such optional ingredients are most typically those materials approved for use in cosmetics and that are described in reference books such as the CTFA Cosmetic Ingredient Handbook, Second Edition, The Cosmetic, Toiletries, and Perfume Association, Inc. 1988, 1992. Examples of such optional ingredients are disclosed in U.S. Serial No. 12/361,634, 10/392422 filed 3/18/2003; and US Publication 2003/0215522A1, dated 11/20/2003.

Other optional ingredients include organic solvents, especially water miscible solvents and co-solvents useful as solubilizing agents for polymeric structurants and as drying accelerators. Examples of suitable organic solvents are disclosed in U.S. Serial No. 12/361,634. Other optional ingredients include: latex or emulsion polymers, thickeners such as water soluble polymers, clays,

silicas, ethylene glycol distearate, deposition aids, including coacervate forming components. Additional optional ingredients include anti-dandruff actives including but not limited to zinc pyrithione, selenium sulfide and those actives disclosed in US Publication 2003/0215522A1.

**B. Surface Resident Coating comprising a Cyclodextrin perfume complex**

In one embodiment, the porous dissolvable solid substrates provide a continuous and accessible high surface area “scaffold” (a 3-D network of “struts”) for the surface resident coating comprising the cyclodextrin perfume complex to be adsorbed or distributed across creating a high surface area thin coating. This location puts the coating in position to immediately contact water during use, releasing the perfumes from the encapsulate.

In one embodiment the surface resident coating comprises from about 10% to about 100% of one or more cyclodextrin perfume complexes; in another embodiment from about 25% to about 100%, and in yet another embodiment from about 40% to about 100%. In one embodiment the ratio of the porous dissolvable solid substrate to the surface resident coating comprising the cyclodextrin perfume complex is from about 110:1 to about 0.5:1, in another embodiment from about 20:1 to about 1:1, in another embodiment from about 10:1 to about 1.5:1, and in yet another embodiment from about 7:1 to about 3:1.

**Cyclodextrin perfume complexes**

The ratio of the cyclodextrin to perfume in the cyclodextrin perfume complex is in one embodiment from about 100:1 to about 1:1, in another embodiment from about 50:1 to about 3:1, and in yet another embodiment from about 20:1 to about 5:1.

As used herein, the term “cyclodextrin” includes any of the known cyclodextrins such as unsubstituted cyclodextrins containing from about six to about twelve glucose units, especially alpha-cyclodextrin, beta-cyclodextrin, gamma-cyclodextrin and/or their derivatives and/or mixtures thereof. For example, the present invention may use cyclodextrins selected from the group consisting of beta-cyclodextrin, hydroxypropyl alpha-cyclodextrin, hydroxypropyl beta-cyclodextrin, methylated-alpha-cyclodextrin, methylated-beta-cyclodextrin, and mixtures thereof. In a highly preferred embodiment, the cyclodextrin is a beta-cyclodextrin.

Cyclodextrins and/or mixtures thereof are useful to the present invention since they are particularly known to absorb body odors. Therefore, an added benefit of using cyclodextrins as complexing aids for the secondary fragrance is that once the matrix solubilizes and the fragrance is released, the cyclodextrin may then become available to absorb malodor.

The release of the secondary fragrance from the complex between cyclodextrin and the secondary fragrance occurs rapidly when wetted (e.g., in the shower). This is convenient for use

within the present invention since the secondary fragrance should initially remain substantially odorless until aqueous activation and solubilization of the matrix. For example, cyclodextrins having a small particle size may complex with the secondary fragrance of the present invention and remain odorless within the composition until the article contacts water in the shower. Cyclodextrins having a small particle size may aid in providing higher cyclodextrin surface availability for activation. As used herein, the particle size refers to the largest dimension of the particle. Small particle cyclodextrins useful in the present invention may have a particle of less than about 50 microns, less than about 25 microns, or less than about 10 microns. A more complete description of the cyclodextrins, cyclodextrin derivatives and cyclodextrin particle sizes useful in the matrices of the present invention may be found in U.S. Pat. No. 5,429,628, issued to Trinh et al. on July 4, 1995.

Perfumes suitable for use herein include any perfumes suitable for use in a personal care product including the use of primary and secondary perfumes as defined herein below.

(i) Primary Perfume

Personal care compositions of the present invention comprise a primary perfume which provides the article with the desired perfume or unscented/neutral aroma prior to use (i.e., prior to contacting the article with water). The scented primary perfume may include any perfume or perfume chemical suitable for topical application to the skin and suitable for use in personal care compositions.

The concentration of the primary perfume in the personal care compositions should be effective to provide the desired aroma including, but not limited to, unscented. Generally, the concentration of the scented primary perfume is from about 0% to about 30%, in one embodiment from about 1% to about 20%, in yet another embodiment from about 2% to about 10%, and in yet another embodiment from about 3% to about 8%, by weight of the solid article. The primary perfume may be included in the personal care compositions of the present invention as a free perfume.

(ii) Secondary Perfume

In order to provide a secondary perfume shift (e.g., a change from one perfume to another), the secondary perfume of the present invention should be substantially different and distinct from the composition of the primary perfume in order to overcome the effect of perfume habituation and to make the second perfume noticeable over the primary perfume. Alternatively, where the same perfume is desired throughout the article's usage, the secondary perfume should



be substantially the same and indistinct from the composition of the primary perfume in order to provide a single, continuous, long-lasting perfume experience.

Any perfume or perfume chemical suitable for topical application to the skin and suitable for use in personal care compositions may be used as the secondary perfume, however, it will not be included within the composition as a free perfume. The secondary perfume will be included in a surfactant-free and water-releasable matrix. The secondary perfume may be selected from the group consisting of perfumes, highly volatile perfume materials having a boiling point of less than about 250°C, and mixtures thereof. Such perfumes will be included within a water-releasable matrix formed from an encapsulating material, as described herein.

In one embodiment, the secondary perfume is selected from high impact accord perfume ingredients having a ClogP of greater than about 2 and odor detection thresholds of less than or equal to 50 parts per billion (ppb).

The cyclodextrin perfume complexes according to the present invention are anhydrous. However, water remnants are likely to be present even immediately after manufacture as a result of processing limitations. It may typically occur that water will re-enter the particulate complexes subsequently, for example during storage. The aqueous phase may not only comprise water, but may also comprise additional water-soluble components, such as alcohols; humectants, including polyhydric alcohols (e.g. glycerine and propylene glycol); active agents such as d-panthenol, vitamin B<sub>3</sub> and its derivatives (such as niacinamide) and botanical extracts; thickeners and preservatives. The aqueous phase does not represent more than 20% by weight of the encapsulate and may comprise from about 0.001% but no more than about 20%, no more than about 10%, no more than about 5%, or no more than about 2%, by weight of the particulate complex.

The cyclodextrin perfume complexes according to the invention may have a particle size from about 1µm to about 200µm, in another embodiment from about 2 µm to about 100µm, and in yet another embodiment from about 3 µm to about 50µm.

The surface resident coating is applied to the porous dissolvable solid substrate. In one embodiment, the surface resident coating is in the form of a fine powder. As seen in FIG. 1, in certain embodiments of the present invention, the personal care article 10 contains a surface resident coating 12 that is located on at least a portion of the surface of the porous dissolvable solid substrate 14. It will be appreciated that the surface resident coating 12 may not always be adjacent to the porous dissolvable solid substrate 14. In certain embodiments, the surface resident coating 12 may permeate the porous dissolvable solid substrate 14 in whole or in part.

Alternatively, the surface resident coating can be included (e.g., sandwiched or encased) within the personal care article or parts thereof. Such a surface resident coating can be sprayed, dusted, sprinkled, coated, surface-printed (e.g., in the shape of a desired adornment, decoration, or pattern), poured on, injected into the interior, dipped, or by any other suitable means, such as by use of a depositor, sifter, or powder bed. In the embodiments depicted by FIGS. 3A, 3B, and 4, the personal care article 10 contains a surface resident coating that can be situated below the surface of the porous dissolvable solid substrate. As seen in FIG. 3B which is a cross sectional view of the personal care article 10, the surface resident coating 24 is located within the dimples 22 of the porous dissolvable solid substrate 26.

Referring now to FIG. 2, in certain embodiments the powder is sandwiched between two porous dissolvable solid substrate which are then joined together (e.g., via sealing the adjoining surfaces or edges with a thin layer of water and/or plasticizer so as to not substantially dissolve the porous dissolvable solid substrate and applied pressure to induce adhesion). In this embodiment, the personal care article 10 comprises two porous dissolvable solid substrates 16, 18 in between which a surface resident coating 20 is located.

Alternatively, in certain embodiments, the powder may be on one personal care article which is folded over to form a pouch, encasing the powder. As depicted in FIG. 4, the personal care article 10 comprises a surface resident coating 32 that is enclosed within a folded porous dissolvable solid substrate 34.

The surface resident cyclodextrin perfume complex coatings of the present invention may also impart other desirable attributes to the personal care article including, but not limited to, improved visual appearance. Moreover, the surface resident cyclodextrin perfume complex coatings of the present invention may provide additional benefits such as imparting anti-stick properties to minimize sticking of the article to the product packaging or to other articles in the case to where they are delivered as a stack of articles.

### **III. Product Form of the Personal Care Article**

The personal care article can be produced in any of a variety of product forms, including porous dissolvable solid substrates along with the surface resident coating comprising the cyclodextrin perfume complex used alone or in combination with other personal care components. Regardless of the product form, the product form embodiments contemplated herein include the selected and defined personal care article that comprises a combination of a porous dissolvable solid substrate and a surface resident coating comprising a cyclodextrin perfume complex.

In one embodiment, the personal care article is in the form of one or more flat sheets or pads of an adequate size to be able to be handled easily by the user. It may have a square, rectangle or disc shape or any other suitable shape. The pads can also be in the form of a continuous strip including delivered on a tape-like roll dispenser with individual portions dispensed via perforations and/or a cutting mechanism. Alternatively, the personal care articles are in the form of one or more cylindrical objects, spherical objects, tubular objects or any other shaped object.

The personal care article may comprise one or more textured, dimpled or otherwise topographically patterned surfaces including letters, logos or figures. The textured substrate can result from the shape of the substrate, in that the outermost surface of the substrate contains portions that are raised with respect to other areas of the surface. The raised portions can result from the formed shape of the personal care article, for example the personal care article can be formed originally in a dimpled or waffle pattern. The raised portions can also be the result of creping processes, imprinted coatings, embossing patterns, laminating to other layers having raised portions, or the result of the physical form of the porous dissolvable solid substrate itself. The texturing can also be the result of laminating one porous dissolvable solid substrate to a second porous dissolvable solid substrate that is textured. In a particular embodiment, the personal care article can be perforated with holes or channels penetrating into or through the porous solid.

#### **IV. Method of Manufacture**

The personal care article can be prepared by the process comprising: (1) Preparing a processing mixture comprising surfactant(s), dissolved polymer structurant, and plasticizer; (2) Aerating the processing mixture by introducing a gas into the processing mixture to form an aerated wet mixture; (3) Forming the aerated wet mixture into one or more desired shapes; (4) Drying the aerated wet mixture to form a porous dissolvable solid substrate; and (5) Applying the surface resident coating comprising a cyclodextrin perfume complex in powdered form to the porous dissolvable solid substrate.

##### **A. Preparation of Processing Mixture**

The processing mixture is generally prepared by dissolving the polymer structurant in the presence of water, plasticizer, surfactant and other optional ingredients by heating followed by cooling. This can be accomplished by any suitable heated batch agitation system or via any suitable continuous system involving either single screw or twin screw extrusion or heat exchangers together with either high shear or static mixing. Any process can be envisioned such

that the polymer is ultimately dissolved in the presence of water, the surfactant(s), the plasticizer, and other optional ingredients including step-wise processing via pre-mix portions of any combination of ingredients.

The processing mixtures of the present invention comprise: from about 15% to about 60% solids, in one embodiment from about 20% to about 55% solids, in another embodiment from about 25% to about 50% solids, and in yet another embodiment from about 30% to about 45% solids by weight of the processing mixture before drying; and have a viscosity of from about 2,500 cps to about 150,000cps, in one embodiment from about 5,000 cps to about 100,000 cps, in another embodiment from about 7,500 cps to about 75,000 cps, and in still another embodiment from about 10,000 cps to about 60,000 cps.

The % solids content is the summation of the weight percentages by weight of the total processing mixture of all of the solid, semi-solid and liquid components excluding water and any obviously volatile materials such as low boiling alcohols. The processing mixture viscosity values are measured using a TA Instruments AR500 Rheometer with 4.0 cm diameter parallel plate and 1,200 micron gap at a shear rate of 1.0 reciprocal seconds for a period of 30 seconds at 23°C.

#### **B. Aeration of Processing Mixture**

The aeration of the processing mixture is accomplished by introducing a gas into the mixture. In one embodiment this is done by mechanical mixing energy. In another embodiment this may be achieved via chemical means. The aeration may be accomplished by any suitable mechanical processing means, including but not limited to: (i) batch tank aeration via mechanical mixing including planetary mixers or other suitable mixing vessels, (ii) semi-continuous or continuous aerators utilized in the food industry (pressurized and non-pressurized), or (iii) spray-drying the processing mixture in order to form aerated beads or particles that can be compressed such as in a mould with heat in order to form the porous solid.

In a particular embodiment, it has been discovered that the personal care article can be prepared within continuous pressurized aerators that are conventionally utilized within the foods industry in the production of marshmallows. Suitable continuous pressurized aerators include the Morton whisk (Morton Machine Co., Motherwell, Scotland), the Oakes continuous automatic mixer (E.T. Oakes Corporation, Hauppauge, New York), the Fedco Continuous Mixer (The Peerless Group, Sidney, Ohio), and the Preswhip (Hosokawa Micron Group, Osaka, Japan).

Aeration can also be accomplished with chemical foaming agents by *in-situ* gas formation (via chemical reaction of one or more ingredients, including formation of carbon dioxide (CO<sub>2</sub>

(g)) by an effervescent system. An additional option is aeration via volatile blowing agents such as low boiling hydrocarbons or alcohols including, but not limited to, isopentane, pentane, isobutene, ethanol etc.

In one embodiment, the pre-mixture is pre-heated immediately prior to the aeration process at above ambient temperature but below any temperatures that would cause undesirable degradation of any of the components. In one embodiment, the pre-mixture is kept at above about 40°C and below about 99°C, in another embodiment above about 50°C and below about 95°C, in another embodiment above about 60°C and below about 90°C. In one embodiment, when the viscosity at ambient temperature of the pre-mix is from about 20,000 cps to about 150,000 cps, the optional continuous heating should be utilized before the aeration step. In another embodiment, additional heat is applied during the aeration process to try and maintain an elevated temperature during the aeration. This can be accomplished via conductive heating from one or more surfaces, injection of steam, a surrounding hot water bath, or other processing means.

In one embodiment the wet density range of the aerated pre-mixture ranges from about 0.12 g/cm<sup>3</sup> to about 0.50 g/cm<sup>3</sup>, in another embodiment from about 0.15 g/cm<sup>3</sup> to about 0.45 g/cm<sup>3</sup>, in another embodiment from about 0.20 g/cm<sup>3</sup> to about 0.40 g/cm<sup>3</sup>, and in yet another embodiment from about 0.25 g/cm<sup>3</sup> to about 0.35 g/cm<sup>3</sup>.

### **C. Forming the Aerated Wet Processing Mixture**

The forming of the aerated wet processing mixture may be accomplished by any suitable means to form the mixture in a desired shape or shapes including, but not limited to: (i) depositing the aerated mixture to moulds of the desired shape and size comprising a non-interacting and non-stick surface including aluminium, Teflon, metal, HDPE, polycarbonate, neoprene, rubber, LDPE, glass and the like; (ii) depositing the aerated mixture into cavities imprinted in dry granular starch contained in a shallow tray, otherwise known as starch moulding forming technique; and (iii) depositing the aerated mixture onto a continuous belt or screen comprising any non-interacting or non-stick material Teflon, metal, HDPE, polycarbonate, neoprene, rubber, LDPE, glass and the like which may be later stamped, cut, embossed or stored on a roll.

### **D. Drying the Aerated Wet Processing Mixture into a Porous Dissolvable Solid**

#### **Substrate**

The drying of the formed aerated wet processing mixture may be accomplished by any suitable means including, but not limited to: (i) drying room(s) including rooms with controlled temperature and pressure or atmospheric conditions; (ii) ovens including non-convection or

convection ovens with controlled temperature and optionally controlled humidity; (iii) truck/tray driers, (iv) multi-stage inline driers; (v) impingement ovens; (vi) rotary ovens/driers; (vii) inline roasters; (viii) rapid high heat transfer ovens and driers; (ix) dual plenum roasters, and (x) conveyor driers, and combinations thereof. Any suitable drying means that does not comprise freeze-drying can be used.

The drying temperature may range from about 40°C to about 200°C. In a one embodiment, the drying environment is heated to a temperature between 100°C and 150°C. In one embodiment, the drying temperature is between 105°C and 145°C. In another embodiment, the drying temperature is between 110°C and 140°C. In a further embodiment, the drying temperature is between 115°C and 135°C.

Other suitable drying environments include “volumetric heating” techniques using high frequency electromagnetic fields such as Microwave Drying and Radio Frequency (RF) Drying. With these techniques, the energy is transferred electromagnetically through the aerated wet pre-mixture rather than by conduction or convection.

Optional ingredients may be imparted during any of the above described four processing steps or even after the drying process.

#### **E. Preparing the Surface Resident Coating Comprising the Cyclodextrin perfume complex**

The preparation of the surface resident coating comprising the cyclodextrin perfume complex may include any suitable mechanical or otherwise physical means to produce a particulate composition comprising the cyclodextrin perfume complex as described herein. In one embodiment, the cyclodextrin perfume complexes of the present invention are prepared by spray drying wherein the perfume is dispersed or emulsified within an aqueous composition comprising the dissolved cyclodextrin based material under high shear (with optional emulsifying agents) and spray dried into a fine powder. The optional emulsifying agents can include gum arabic, specially modified starches, or other tensides as taught in the spray drying art (See Flavor Encapsulation, edited by Sara J. Risch and Gary A. Reineccius, pages 9, 45-54 (1988)). The preparation of cyclodextrin perfume complexes via a spray drying process according to the present invention can be referenced in application filed by Deckner, et. al. on May 19, 2005, entitled “Oil Encapsulation”.

Alternatively, the perfume oil can be adsorbed or absorbed into or combined with a cyclodextrin based powder that has been previously produced via a variety of mechanical mixing means (spray drying, paddle mixers, grinding, milling etc.). In one embodiment, the cyclodextrin

based material in solid-based form (and comprising any minor impurities as supplied by the supplier including residual solvents and plasticizers) may be ground or milled into a fine powder in the presence of the perfume via a variety of mechanical means, for instance in a grinder or hammer mill.

In one embodiment the surface resident coatings of the present invention may have a particle size from about 1 $\mu$ m to about 200 $\mu$ m, in another embodiment from about 2  $\mu$ m to about 100 $\mu$ m, and in yet another embodiment from about 3  $\mu$ m to about 50 $\mu$ m.

In some embodiments, inert fillers can be included within the particulate complex forming process or particulate complex after forming, for instance aluminum starch octenylsuccinate under the trade name DRY-FLO® PC and available from Akzo Nobel, at a level sufficient to improve the flow properties of the powder and to mitigate inter-particle sticking or agglomeration during powder production or handling. Other optional excipients or cosmetic actives, as described herein, can be incorporated into the powder during the particulate complex forming process. The resulting powder may also be blended with other powders, either of inert materials or other powder-active complexes as described herein.

#### **F. Combining Surface Resident Coating comprising the Cyclodextrin perfume complexes with the Porous Dissolvable Solid Substrate**

Any suitable application method can be used to apply the surface resident coating comprising the cyclodextrin perfume complex to the personal care article such that it forms a part of the personal care article. For instance, the porous dissolvable solid substrate can have a tacky surface by drying the porous dissolvable solid substrate's surface to a specific water content before application of powder to facilitate the adherence of the surface resident coating comprising the cyclodextrin perfume complex to the porous solid. In one embodiment, the porous dissolvable solid substrate is dried to a moisture content of from about 0.1 % to about 25%, in one embodiment from about 3% to about 25%, in another embodiment from about 5% to about 20%, and in yet another embodiment from about 7% to about 15%. Alternatively, a previously dried dissolvable porous solid substrate's surface can be made to reversibly absorb a desired level of atmospheric moisture prior to application of the powder within a controlled humidity environment for a specific period of time until equilibrium is achieved. In one embodiment, the humidity environment is controlled from about 20% to about 85% relative humidity; in another embodiment, from about 30% to about 75% relative humidity; and in yet another embodiment, from about 40% to about 60% relative humidity.

In another embodiment, the porous dissolvable solid substrate is placed in a bag, tray, belt, or drum containing or otherwise exposed to the powder and agitated, rolled, brushed, vibrated or shaken to apply and distribute the powder, either in a batch or continuous production manner. Other powder application methods may include powder sifters, electrostatic coating, tribo charging, fluidized beds, powder coating guns, corona guns, tumblers, electrostatic fluidized beds, electrostatic magnetic brushes, and/or powder spray booths. The surface resident coating comprising the cyclodextrin perfume complex can be applied over portions or entire regions of the porous dissolvable solid substrate's exterior surface, and can be applied in a manner to adorn, decorate, form a logo, design, etc.

## **V. Test Methods**

### **A. Dissolution Rate**

The personal care article of present invention has a Dissolution Rate that allows the personal care article to rapidly disintegrate during use with water. The Dissolution Rate of the personal care article is determined in accordance with the methodology described below.

Hand Dissolution Method: 0.5 to 1.5 g (approximately 10 to 20 square centimeters if in a 3 to 10mm thick sheet/pad form) of the personal care article (as described in the examples herein) is placed in the palm of the hand while wearing nitrile gloves. 7.5 cm<sup>3</sup> of warm tap water (from about 30°C to about 35°C) is quickly applied to the personal care composition via syringe. Using a circular motion, palms of hands are rubbed together 2 strokes at a time until dissolution occurs (up to 30 strokes). The hand dissolution value is reported as the number of strokes it takes for complete dissolution or as 30 strokes as the maximum. For the latter scenario, the weight of the undissolved material is also reported.

The personal care articles of the present invention have a hand dissolution value of from about 1 to about 30 strokes, in one embodiment from about 2 to about 25 strokes, in another embodiment from about 3 to about 20 strokes, and in still another embodiment from about 4 to about 15 strokes.

### **B. Thickness**

The thickness of the personal care article and/or the porous dissolvable solid substrate is obtained using a micrometer or thickness gage, such as the Mitutoyo Corporation Digital Disk Stand Micrometer Model Number IDS-1012E (Mitutoyo Corporation, 965 Corporate Blvd, Aurora, IL, USA 60504). The micrometer has a 1 inch diameter platen weighing about 32 grams, which measures thickness at an application pressure of about 40.7 phi (6.32 gm/cm<sup>2</sup>).



The thickness of the personal care article and/or the porous dissolvable solid substrate is measured by raising the platen, placing a section of the sample on the stand beneath the platen, carefully lowering the platen to contact the sample, releasing the platen, and measuring the thickness of the sample in millimeters on the digital readout. The sample should be fully extended to all edges of the platen to make sure thickness is measured at the lowest possible surface pressure, except for the case of more rigid samples which are not flat. For more rigid samples which are not completely flat, a flat edge of the sample is measured using only one portion of the platen impinging on the flat portion of the sample. In the case of cylindrical, spherical, or other objects with more of a third dimension versus a pad or strip, the thickness is taken as the maximum distance of the shortest dimension, i.e., the diameter of a sphere or cylinder for instance, and the thickness ranges are the same as described above.

### **C. Basis Weight**

The Basis Weight of the personal care article and/or the porous dissolvable solid substrate is calculated as the weight of the personal care article and/or the porous dissolvable solid substrate per area of the selected personal care article and/or the porous dissolvable solid substrate ( $\text{grams/m}^2$ ). The area is calculated as the projected area onto a flat surface perpendicular to the outer edges of the personal care article and/or the porous dissolvable solid substrate. For a flat object, the area is thus computed based on the area enclosed within the outer perimeter of the sample. For a spherical object, the area is thus computed based on the average diameter as  $3.14 \times (\text{diameter}/2)^2$ . For a cylindrical object, the area is thus computed based on the average diameter and average length as diameter  $\times$  length. For an irregularly shaped three dimensional object, the area is computed based on the side with the largest outer dimensions projected onto a flat surface oriented perpendicularly to this side.

### **D. Solid Density**

The porous dissolvable solid substrate of the personal care compositions described herein can be characterized in terms of a solid density determination.

The solid density of the porous dissolvable solid substrate can be determined by dividing the weight of the solid by the known volume of the solid. The latter can be determined by a number of techniques including producing the solid within a mold of known x-y dimensions and measuring the resulting thickness to account for any shrinkage or expansion during the drying process. The solid can also be cut to known x-y dimensions, i.e., by using a circular or square cutting die of known diameter or dimensions and then by measuring the thickness. Alternatively, in the instances where there are not any significant thickness variations, the density can be

calculated by the equation: Calculated Density = Basis Weight of porous solid / (Average porous Solid Thickness x 1,000).

#### **E. Cell Inter-connectivity**

The personal care article and/or the porous dissolvable solid substrate of the present invention have a high degree of cell inter-connectivity, i.e., are predominantly open-celled solid foams as opposed to being predominantly closed-cell solid foams. The cell inter-connectivity can be assessed by light microscopy, scanning electron microscopy, micro computed tomography imaging parameters (Star Volume and SMI Index), gas pycnometry parameters (% Open Cells), or other suitable methodology.

A qualitative method of determining cell inter-connectivity is via light microscopy. This is performed by cutting a 2-3 mm wide sliver of the personal care article and/or the porous dissolvable solid substrate in the z-direction using scissors or a sharp blade, measured across the normal x-y largest surface, and turning the resulting sliver by 90 degrees to reveal the internal cellular structure of the freshly cut cross-sectional area. This cross-sectional area can be assessed by close visual inspection or, more accurately, by employing magnification under a stereo microscope such as the SZX12 Stereo microscope available from Olympus America Inc., Center Valley, PA. The open-celled personal care article and/or the porous dissolvable solid substrate of the present invention can easily be identified by examining the inner portion of the cross-sectional area which will comprise a predominantly three dimensional network of struts with open void spaces surrounding the struts that are inter-connected to one another including in the third dimension through the depth of the cross-section. In contrast, the inner cross-section of a closed-cell foam will appear as discrete bubbles that are cut across and then only being inter-connected at the cross-sectional surface in two dimensions by virtue of the cutting process employed to generate the exposed cross-sectional area.

Another means to determine the cell interconnectivity is via the Star Volume and the Structure Model Index. Disk-like samples, approximately 4 cm in diameter and 3 to 7 mm high, are scanned using a micro computed tomography system ( $\mu$ CT80, SN 06071200, Scanco Medical AG). Each sample is imaged while sitting flat on the bottom of a cylindrical tube. Image acquisition parameters are 45 kVp, 177  $\mu$ A, 51.2 mm field of view, 800 ms integration time, 1000 projections. The number of slices is adjusted to cover the height of the sample. The reconstructed data set consisted of a stack of images, each 2048x2048 pixels, with an isotropic resolution of 25  $\mu$ m. For data analysis, a volume of interest is selected to be fully within the sample, avoiding the surface region. A typical volume of interest is 1028x772x98 voxels.

Structure Model Index (SMI) is measured using Scanco Medical's Bone Trabecular Morphometry evaluation with a threshold of 17. With this index the structural appearance of trabecular bone is quantified (*see* T. Hildebrand, P. Rüegsegger. Quantification of bone microarchitecture with the structure model index. *Comp Meth Biomech Biomed Eng* 1997;1:15-23). The triangulated surface is dilated in normal direction by an infinitesimal amount, and the new bone surface and volume is calculated. By this, the derivative of the bone surface (dBS/dr) can be determined. The SMI is then represented by the equation:

$$SMI = 6 \cdot \frac{BV \cdot \frac{dBS}{dr}}{BS^2}$$

SMI relates to the convexity of the structure to a model type. Ideal (flat) plates have an SMI of 0 (no surface change with dilation of the plates), whereas ideal cylindrical rods have an SMI of 3 (linear increase in surface with dilation of rods). Round spheres have an SMI of 4. Concave structure gives negative dBS/dr, resulting in negative SMI values. Artificial boundaries at the edge of the volume of interest are not included in the calculation and thus suppressed.

In addition to the Scanco Medical Analysis, StarVolume measurements are made. Star Volume is a measure of the "openness" of the void space in a two phase structure. By choosing a random uniformly distributed set of points in the phase of interest (in our case this is the background), we can extend lines in random directions from each of these points. The lines are extended until they touch the foreground phase. The length of each of these lines is then recorded. The random points have a sampling of 10 in each direction (x/y/z) and at each point 10 random angles are chosen. If the line extends to the border of the ROI of interest that line is discarded (we only want to accept lines that actually intersect with the foreground phase). The final equation is based upon the research published in Star volume in bone research. A histomorphometric analysis of trabecular bone structure using vertical sections; Vesterby, A.; *Anat Rec.*; 1993 Feb;235(2):325-334.:

$$StarVolume = \frac{4}{3} \pi \cdot \frac{\sum dist^3}{N}$$

where "dist" is the individual distances and N is the number of lines examined.

The Percent Open Cell Content is measured via gas pycnometry. Gas pycnometry is a common analytical technique that uses a gas displacement method to measure volume accurately.

Inert gases, such as helium or nitrogen, are used as the displacement medium. The sample is sealed in the instrument compartment of known volume, the appropriate inert gas is admitted, and then expanded into another precision internal volume. The pressure before and after expansion is measured and used to compute the sample volume. Dividing this volume into the sample weight gives the gas displacement density.

ASTM Standard Test Method D2856 provides a procedure for determining the percentage of open cells using an older model of an air comparison pycnometer. This device is no longer manufactured. However, you can determine the percentage of open cells conveniently and with precision by performing a test which uses Micromeritics' AccuPyc Pycnometer. The ASTM procedure D2856 describes 5 methods (A, B, C, D, and E) for determining the percent of open cells of foam materials.

For these experiments, the samples can be analyzed using an Accupyc 1340 using nitrogen gas with the ASTM foampyc software. Method C of the ASTM procedure is to be used to calculate to percent open cells. This method simply compares the geometric volume as determined using calipers and standard volume calculations to the true volume as measured by the Accupyc. It is recommended that these measurements be conducted by Micromeritics Analytical Services, Inc. (One Micromeritics Dr, Suite 200, Norcross, GA 30093). More information on this technique is available on the Micromeritics Analytical Services web sites ([www.particletesting.com](http://www.particletesting.com) or [www.micromeritics.com](http://www.micromeritics.com)), or published in a book, "Analytical Methods in Fine particle Technology", by Clyde Orr and Paul Webb.

#### **F. Cell Wall Thickness**

The Cell Wall Thickness of the personal care article and/or the porous dissolvable solid substrate is computed from the scanned images via a micro computed tomography system ( $\mu$ CT80, SN 06071200, Scanco Medical AG) as described herein. The Cell Wall Thickness is determined according to the method defined for the measurement of Trabecular Thickness using Scanco Medical's Bone Trabecular Morphometry evaluation. The definition of Trabecular Thickness as taken from the Scanco User's manual: Trabecular Thickness uses a Euclidean distance transformation (EDM), which calculates the Euclidean distance from any point in the foreground to the nearest background point. The Trabecular Thickness measure represents twice the centerline values associated with the local maxima of the EDM, which represents the distance to the center of the object (twice this distance will yield the thickness).

### G. Specific Surface Area

The Specific Surface Area of the personal care article and/or the porous dissolvable solid substrate is measured via a gas adsorption technique. Surface Area is a measure of the exposed surface of a solid sample on the molecular scale. The BET (Brunauer, Emmet, and Teller) theory is the most popular model used to determine the surface area and is based upon gas adsorption isotherms. Gas Adsorption uses physical adsorption and capillary condensation to measure a gas adsorption isotherm. The technique is summarized by the following steps; a sample is placed in a sample tube and is heated under vacuum or flowing gas to remove contamination on the surface of the sample. The sample weight is obtained by subtracting the empty sample tube weight from the degassed sample + sample tube weight. The sample tube is then placed on the analysis port and the analysis is started. The first step in the analysis process is to evacuate the sample tube, followed by a measurement of the free space volume in the sample tube using helium gas at liquid nitrogen temperatures. The sample is then evacuated a second time to remove the helium gas. The instrument then begins collecting the adsorption isotherm by dosing krypton gas at user specified intervals until the requested pressure measurements are achieved.

**Sample Preparation (Degassing):** A sample not adequately cleaned of adsorbed contaminants will outgas during an analysis and some portion of the surface will be inaccessible to measurement. The purpose of degassing is to remove these adsorbed molecules from the surface of the sample prior to analysis. Adsorptive molecules must reach all parts of the surface for the true surface area to be revealed. Samples are prepared by heating the sample while simultaneously evacuating the sample tube.

For these experiments, the samples are outgassed under evacuation at room temperature overnight. Samples may then analyzed using an ASAP 2420 with krypton gas adsorption. Krypton gas is preferred over nitrogen gas as it has a saturation pressure approximately 1/300 that of nitrogen at liquid nitrogen temperature (krypton: 2.5 torr; nitrogen: 760 torr). Therefore, compared to nitrogen, there is in the free space above the sample about 1/300 the number of krypton molecules present at the same relative pressure. Since about the same number of krypton and nitrogen molecules are required to form a monolayer, this number represents a far greater proportion of the quantity dosed than in the case of nitrogen. These measurements can be conducted by Micromeritics Analytical Services, Inc. (One Micromeritics Dr., Suite 200, Norcross, GA 30093). More information on this technique is available on the Micromeritics Analytical Services web sites ([www.particletesting.com](http://www.particletesting.com) or [www.micromeritics.com](http://www.micromeritics.com)), or

published in a book, "Analytical Methods in Fine particle Technology", by Clyde Orr and Paul Webb.

#### **H. Evaluation of Surface Resident Coating**

The presence of a surface resident coating comprising a cyclodextrin-perfume complex of the present invention can be determined by a number of techniques. For detection of a particulate or powder coating, the surface of application as well as the cross-sections perpendicular to the larger surfaces of the porous dissolvable solid substrate can be examined by microscopic techniques. These microscopic techniques may include light microscopy and scanning electron microscopy (SEM). The light microscopy may include but are not necessarily limited to bright field, dark field, or confocal microscopy techniques. Other techniques for mapping unique elements such as silicon or distinctive functional groups such as quaternary ammonium groups on the cross-sectional surface include: time of flight secondary ion mass spectroscopy (ToF-SIMS), or infrared microscopy.

Potential methods for looking at the distribution of particles from the surface to the interior of the porous dissolvable solid substrate without sectioning the samples include: micro-Computed Tomography (micro-CT), Magnetic Resonance Imaging (MRI), Acoustic Imaging, Confocal Fluorescence Microscopy, Confocal Raman Spectroscopy, and Confocal Infrared Reflectance Spectroscopy.

The determination of surface-resident coating particles on cross-sectioned porous dissolvable solid substrate can be performed by comparing the distribution of the particles across the cut cross-section of the porous solid. Specifically, the surface resident coating particles should be present at the original solid/air interfaces, but not within the exposed cross sectioned interior of the solid cell walls as can be ascertained by analyzing the exposed freshly cut cross-sectional interiors of the solid. It should be noted that some contamination of the freshly cut cross-sectional solid cell wall interiors may occur as a consequence of the cutting process of the porous solid. However, the preponderance (in one embodiment, from about 50% to about 100%) of the surface resident coating particle distribution will occur at the original solid/air interfaces and not within the exposed cut cross-sectional interiors of the cell walls.

It should also be noted that the surface resident coating particles of the present invention generally do not spread uniformly across all exposed solid/air interfaces. Rather, it has been found that the surface resident coatings of the present invention typically spread, from the point of coating application, into cavities down to about 0.5 to about 3.0 mm according to gravity. Accordingly, the determination of surface resident particles of cosmetic actives of the present

invention (as described above), should be conducted across many different cross sections from top-to-bottom and from edge-to-edge of the porous solid. If present, the surface resident cosmetic active particle will generally be within the regional vicinity (to within about 0.5 to about 3.0 mm from the surface) of the surface to where the coating was first applied.

### **I. Expert Perfume Panel**

The solid polymeric dissolvable porous solids of the present invention provide a novel perfume benefit versus liquid hair care products. An expert perfume sensory panel is conducted to quantify this benefit within a normal shampoo protocol regimen with three expert perfumers evaluating the odor character and odor intensity on a 1 to 100 scale (no odor to the most intense odor possible) as described below.

The expert perfume panel assessment is performed on 15g/10" flat Oriental virgin hair switches. The hair switch is rinsed with 100°F tap water at 1.5 to 2.0 gallons/min for 20 seconds with a shower nozzle. For testing the liquid control products, the liquids are first smelled in an open glass jar for the neat product odor. 5 cm<sup>3</sup> of liquid product is then applied to the palm of a pre-wetted hand for the initial perfume bloom assessment in the hand. Next, the liquid is applied to the center of the switch with the palm and lathered for 40 seconds by repeatedly rubbing and squeezing the hair switch with both hands in a downward motion for the lather on switch odor assessment. The switch is then thoroughly rinsed for 45 seconds and assessed for wet hair odor. Additional assessments include the 4 hour damp hair odor, the 24 hour dry hair odor and the 24 hour re-wet odor.

When testing the dissolvable porous solids of the present invention, 1 pad (approximately 1.0 to 1.1 grams) is substituted for the 5 cm<sup>3</sup> of liquid. For testing the dissolvable porous solids, the pad is first smelled in a freshly opened wrapper for the neat product odor. The pad is then applied to the palm of a pre-wetted hand and diluted with 7.5 ml of 100°F tap water with rubbing of the palms (4 to 8 strokes) until the solid is fully dissolved for the initial perfume bloom assessment in the hand. Next, the resulting liquid mixture is applied to the center of the switch with the palm and lathered for 40 seconds by repeatedly rubbing and squeezing the hair switch with both hands in a downward motion for the lather on switch odor assessment. The switch is then thoroughly rinsed for 45 seconds and assessed for wet hair odor, 4 hour damp hair odor, 24 hour dry hair odor and 24 hour re-wet odor as described above.

## **J. Perfume Headspace Analysis Using Dynamic Collection and Gas Chromatography/Mass Spectrometry**

All laboratory instruments should be operated according to manufacturers' instructions, as set forth in the instrument operation manuals and instructional materials, unless otherwise indicated.

- a) Shampoo pad “dry” (solid neat product odor): A single shampoo pad (about 1.0 to 1.1 grams) is placed into a clean 125 ml I-Chem headspace jar. The sample is allowed to equilibrate for at least 1 hour at room temperature. The headspace was purged with a flow of helium (20 ml/min) for 10 minutes (200mLs total volume collected). The purged headspace was collected onto a desorption tube packed with about 120 mg of Tenax-TA. The collected sample is then analyzed using a thermal desorption-GC-MS system.
- b) Shampoo pad “wet” (simulating initial solid perfume bloom upon dissolution): A single shampoo pad (about 1.1 grams) is placed into a clean 125 ml I-Chem headspace jar and 7 grams of DI water is added to the jar. A stir bar is added to help the pad to dissolve. The sample is allowed to equilibrate for at least 1 hour at room temperature. The remaining procedure is the same as above.
- c) Shampoo control (liquid neat product odor): 5 grams of shampoo is placed into a clean 125 ml I-Chem headspace jar. The remaining procedure is the same as above.
- d) 1:10 Dilution in DI water on the shampoo pads (solid fully diluted odor assessment): A shampoo pad “wet” solution was made, then 80 grams of DI water were added to the solution. Following 10 minutes of stirring, a 10 g aliquot is transferred to another clean 125 ml I-Chem headspace jar. The remaining procedure is the same as above.
- e) Shampoo 1:10 Dilution (liquid fully diluted odor assessment): 50 grams of DI water were added to 5 grams of neat shampoo control. Following 10 minutes of stirring, a 10 g aliquot is transferred to another clean 125 ml I-Chem headspace jar. The remaining procedure is the same as above.

Materials: Tenax-TA, 60/80 Mesh, Alltech.

Apparatus:

1. 125 ml I-Chem headspace jar.
2. SIS-desorption Tubes with Tenax TA
3. Timer.
4. Gas Chromatograph (GC): Agilent model 6890 with a SIS thermal desorption unit.



5. GC column: Agilent DB-5MS, 60 M x 0.32 mm ID, 1.0  $\mu$ m film thickness obtained from Agilent Technologies, Inc. Wilmington, DE., USA.
6. Carrier gas, helium, 0.8 ml/min. flow rate.
7. The Detector is a model Agilent 5973 Mass Selective Detector obtained from Agilent Technologies, Inc., Wilmington, DE, USA having a source temperature of about 230 °C, and a MS Quad temperature of about 150 °C.

Analysis procedure:

1. Transfer sample to proper sample tray and proceed with GC-MS analysis.
2. Start sequence of sample loading and analysis. In this step, a cryogenic trap is cooled to -120°C, A helium flow (flow rate about 30 ml/min) is used to purge the trap. The desorption temperature is set at 250°C and the tube is purged for 7 minutes. Then cryo-trap is then heated to remove the trapped perfume compounds (up to 280°C and held for 8 minutes), followed by the start of the GC/MS analysis run in splitless mode. The following temperature program is used:
  - i) an initial temperature of about 40 °C which is held for 2 minutes,
  - ii) increase the initial temperature at a rate of about 5 °C/min until a temperature of about 220 °C is reached, then 25 °C/min to 280 °C, hold for about 10 minutes.
3. Perfume compounds are identified using the MS spectral libraries of John Wiley & Sons and the National Institute of Standards and Technology (NIST), purchased and licensed through Agilent Technologies, Inc., Wilmington, DE, USA.
4. Chromatographic peaks for specific ions are integrated using the Chemstation software obtained from Agilent Technologies, Inc., Wilmington, DE, USA.

#### **IV. Methods of Use**

The compositions of the present invention may be used for treating mammalian keratinous tissue such as hair and/or scalp, and provide rapid rinse-ability. The method for conditioning the hair may comprise the steps of: a) applying an effective amount of the personal care product to the hand, b) wetting the personal care product with water and rubbing to dissolve the solid, c) applying the dissolved material to either the hair or scalp such as to treat, and d) rinsing the diluted treatment from the hair or scalp using water. These steps can be repeated as many times as desired to achieve the desired treatment benefit.

**V. Examples**

The following examples further describe and demonstrate embodiments within the scope of the present invention. The examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention, as many variations thereof are possible without departing from the spirit and scope of the invention. All exemplified amounts are concentrations by weight of the total composition, i.e., wt/wt percentages, unless otherwise specified.

**Comparative Example 1: Liquid Shampoo comprising a primary fragrance 1a**

A conventional liquid hair shampoo is prepared for comparative purposes only according to methods known to those skilled in the art. The liquid shampoo comprises base ingredients (non-perfume) similar to the retail Herbal Essences® Drama Clean Shampoo which is distributed by Procter and Gamble Company, Cincinnati, OH and with the addition of 0.8% by weight of a primary fragrance 1a. The ingredients include: Water, Sodium Laureth Sulfate, Sodium Lauryl Sulfate, Cocamidopropyl Betaine, Sodium Chloride, Cocamide MEA, Sodium Benzoate, Citric Acid, Sodium Citrate, Tetrasodium EDTA, Guar Hydroxypropyltrimonium Chloride, Linoleamidopropyl PG-Dimonium Chloride Phosphate, Propylene Glycol, Methylchloroisothiazolinone, Methylisothiazolinone, Sodium Xylenesulfonate, Yellow 10, and Blue 1.

**Comparative Example 2: Liquid Shampoo comprising a primary fragrance 1b**

A conventional liquid hair shampoo is prepared for comparative purposes only according to methods known to those skilled in the art. The liquid shampoo comprises base ingredients (non-perfume) similar to the retail Pantene® Pro-V Classic Clean Shampoo which is distributed by Procter and Gamble Company, Cincinnati, OH and with the addition of 0.4% by weight of a primary fragrance 1b. The ingredients include: Water, Ammonium Laureth Sulfate, Ammonium Lauryl Sulfate, Sodium Chloride, Glycol Distearate, Citric Acid, Cocamide MEA, Panthenol, Panthenyl Ethyl Ether, Dimethicone, Fragrance, Cetyl Alcohol, Sodium Citrate, Sodium Benzoate, Guar Hydroxypropyltrimonium Tricaprylate/Tricaprate, Ammonium Xylenesulfonate, Methylchloroisothiazolinone, and Methylisothiazolinone.

**Example 3: Dissolving porous solid shampoo with a primary fragrance 1a and a surface-added beta cyclodextrin complex of a secondary fragrance 2a**

The following dissolving porous solid is prepared in accordance to the present invention as described below.

Component	Wt%
Distilled water	30.7
Glycerin <sup>1</sup>	3.0
Polyvinyl alcohol <sup>2</sup>	7.4
Sodium Lauroamphoacetate (25% activity) <sup>3</sup>	14.6
Ammonium Laureth-3 sulfate (25% activity) <sup>4</sup>	6.0
Ammonium C11 alkyl sulfate (24% activity) <sup>5</sup>	28.0
Ammonium Laureth-1 sulfate (70% activity) <sup>6</sup>	9.7
Citric Acid <sup>7</sup>	0.6
Total	100.0

<sup>1</sup> Superol K, USP FCC EP Glycerin, supplier: Procter & Gamble Chemicals

<sup>2</sup> Sigma-Aldrich Catalog No. 363081, MW 85,000-124,000, 87-89% hydrolyzed

<sup>3</sup> MACKAM HPL-28ULS, supplier: McIntyre Group Ltd, University Park, IL,

<sup>4</sup> Ammonium Laureth-3 Sulfate at 25% active with an estimated C8/C9/C10/C11/C12/C13/C14/C15/C16/C18 alkyl chain length distribution of 0/0.3/0.9/0.5/71.5/1/20/1.9/3.9/0 and an average of 2.0 moles of ethoxylation, supplier: Procter & Gamble Chemicals

<sup>5</sup> Ammonium Undecyl Sulfate at 24% active with an estimated C8/C9/C10/C11/C12/C13/C14/C15/C16/C18 alkyl chain length distribution of 0/0/0.6/93.6/5.1/0.7/0/0/0, supplier: Procter & Gamble Chemicals

<sup>6</sup> Ammonium Laureth-1 Sulfate at 70% active with an estimated C8/C9/C10/C11/C12/C13/C14/C15/C16/C18 alkyl chain length distribution of 0/0.3/1.3/0.5/69.6/1.1/21.8/1.2/4.2/0 and an average of 0.82 moles of ethoxylation, supplier: Procter & Gamble Chemicals

<sup>7</sup> Citric Acid Anhydrous Fine Granular 51N, supplier: S.A. Citrique Belge N.V. Pastorijstraat 249, B-3300 Tienen, Belgium

A target weight of 300 grams of the above composition is prepared with the use of a conventional overhead stirrer (IKA® RW20DZM Stirrer available from IKA® Works, Inc., Wilmington, DE) and a hot plate (Corning Incorporated Life Sciences, Lowell, MA). Into an appropriately sized and cleaned vessel, the distilled water, glycerin and polyvinyl alcohol is added with stirring until homogenous. The mixture is slowly heated to 75 to 80C after which the surfactants are added. The mixture is then re-heated to 85C with stirring and then allowed to cool to room temperature. Additional distilled water is added to compensate for water lost to evaporation (based on the original tare weight of the container). The final pH is between 5.2 - 6.6 and adjusted with citric acid or diluted sodium hydroxide if necessary. The resulting processing mixture viscosity is approximately 13,000 cps.

250 grams of the above mixture is transferred into a 5 quart stainless steel bowl of a KitchenAid® Mixer Model K5SS (available from Hobart Corporation, Troy, OH) and fitted with a flat beater attachment. The mixture is vigorously aerated at a speed setting of 6 (out of a maximum setting of 10) until a wet density of approximately 0.24-0.25 grams per milliliter is

achieved (approximately 75 seconds). The wet foam density is measured by weighing a filling a cup with a known volume and evenly scraping off the top of the cup with a spatula. The resulting aerated mixture is then spread with a spatula into square 160 mm x 160 mm aluminum molds with a depth of 6.5 mm with the excess wet foam being removed with the straight edge of a large metal spatula that is held at a 45 degrees angle and slowly dragged uniformly across the mold surface. The aluminum molds are then placed into a 130C convection oven until the weight loss due to evaporation is between 67% and 69% of the original foam weight within each mold (approximately 30 to 45 minutes). The molds are allowed to cool to room temperature with the substantially dry porous solids removed from the molds with the aid of a thin spatula and tweezers.

Each of the resulting 160mm x 160mm square pads is cut into nine 43mm x 43mm squares (with rounded edges) using a cutting die and a Samco SB20 cutting machine. The resulting smaller pads are then equilibrated overnight in a constant environment room kept at 70 degrees Fahrenheit and 50% relative humidity. Each pad is then weighed and placed on an individual weight boat with the original mold side facing downward. The average pad weight is approximately 0.95 +/- 0.07 grams. The resulting porous solids are determined by the methodology described herein to be predominantly open-celled, to have thickness of between 5 and 6 mm, to have a basis weight of approximately 560 grams per square meter, and to have a dry density of approximately 0.10 grams per cubic centimeter.

With the pad sitting on the weigh boat, 40 microliters of a primary fragrance 1a is applied across the surface of the pad with a micropipette with a plastic tip (100 microliter calibrated Gilson Pipetman at a setting of 40 microliters). This is accomplished by dragging the plastic tip along the surface of the pad while dispensing the primary fragrance. The weigh boat comprising the primary fragrance is then immediately stored within a smaller zip-lock bag which is sealed to minimize the evaporation of the primary fragrance. This procedure is repeated for the remaining pads.

A beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2a (high impact perfume accord complexed at 8.50% by weight of the inclusion complex and prepared via a spray drying process according to the application filed by Deckner, et. al. on May 19, 2005, entitled "Oil Encapsulation") is then applied to the surface of each pad. The pad is coated with the perfume inclusion complex powder by gently shaking the pad in a tray (or other suitable container) containing an excess of the perfume inclusion complex in a side-to-side manner ten times (the process is repeated for the other side). The resulting powder coated pad is then picked up (with gloved hands) and gently shaken and tapped several times to remove any excess powder that is not sufficiently adhered to the pad. This coating procedure is repeated for the remaining pads. The weight of beta cyclodextrin complex of the secondary fragrance high impact accord 2a applied per pad is approximately 0.13 +/- 0.01 grams.

The resulting pads comprising a surface applied primary fragrance 1a and a beta cyclodextrin complex of a secondary fragrance high impact accord 2a are then sealed within clear 1.5 MIL LDPE tubing using the lowest setting of a PFS-200 impulse sealer.

**Example 4: Dissolving porous solid shampoo with a primary fragrance 1b and a surface-added beta cyclodextrin complex of a secondary fragrance 2b**

Dissolving porous solid shampoo pads are prepared according to the identical formulation and procedures as described in Example 3, but with the replacement of primary fragrance 1a with different primary fragrance 1b and with the replacement of surface added beta cyclodextrin complex of a secondary fragrance 2a with a different surface added beta cyclodextrin complex of a secondary fragrance 2b (high impact perfume accord complexed at 9.7% by weight of the inclusion complex and prepared via a spray drying process according to the application filed by Deckner, et. al. on May 19, 2005, entitled "Oil Encapsulation"). The resulting porous solids are determined by the methodology described herein to be predominantly open-celled, to have thickness of between 5 and 6 mm, to have a basis weight of approximately 500 grams per square meter, and to have a dry density of approximately 0.09 grams per cubic centimeter. 40 microliters of the primary fragrance 1b is added to each pad as described in example 3. The weight of beta cyclodextrin complex of the secondary fragrance high impact accord 2b applied per pad is approximately 0.14 +/- 0.02 grams.

**Example 5: Dissolving porous solid shampoo with a primary fragrance 1a and an internally loaded beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2a**

Dissolving porous solid shampoo pads are prepared according to the identical formulation and procedures as described in Example 3, but with the replacement of the 160mm x 160mm aluminum molds with individual circular 42 mm diameter teflon molds with a depth of 3.0 mm with the loading of the surface added beta cyclodextrin complex of a secondary fragrance 2a in-between two thinner identical circular pads which are sealed at the edges as described below.

An individual circular pad is placed on a weight boat with the original mold side facing upward. 0.35 grams of the beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2a powder as described in Example 3 is then applied onto the central region of the circular pad with special care to not apply powder to the edges. A second pad is then placed on top of the first pad comprising the powder with the mold side facing downward and with the edges closely aligned. The edges are then sealed by slightly wetting the edges of the two pads and squeezing together along the edges with pre-wetted gloved fingers until a seal is formed. Special care must be taken so as to not over-wet the edges causing the partial dissolution and collapse of the solid foam material. 40 microliters of primary fragrance 1a is then applied across the surface of the upper pad using a micropipette with a plastic tip (100 microliter calibrated Gilson Pipetman at a setting of 40 microliters) by dragging the plastic tip along the surface of the

upper pad while dispensing the primary fragrance. The weigh boat comprising the is then immediately stored within a smaller zip-lock bag which is sealed to minimize the evaporation of the primary fragrance.

The resulting pad comprising the beta cyclodextrin complex of a secondary fragrance high impact accord and primary fragrance is then sealed within clear 1.5 MIL LDPE tubing using the lowest setting of a PFS-200 impulse sealer. The above procedure is repeated for the remaining pads.

**Example 6: Dissolving porous solid shampoo with a primary fragrance 1b and an internally loaded beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2b (MVF1480-017A-1b).**

Dissolving porous solid shampoo pads are prepared according to the identical formulation and procedures as described in Example 5, but with the replacement of primary fragrance 1a with different primary fragrance 1b and with the replacement of surface added beta cyclodextrin complex of a secondary fragrance 2a with a different surface added beta cyclodextrin complex of a secondary fragrance 2b (high impact perfume accord complexed at 9.7% by weight of the inclusion complex and prepared via a spray drying process according to the application filed by Deckner, et. al. on May 19, 2005, entitled "Oil Encapsulation").

#### **Porous Solid Structural Measurements**

The following structural measurements were made of the porous solid produced in Example 3 (before the addition of the primary fragrance and the beta cyclodextrin complex of a secondary fragrance high impact perfume accord) according to the methods as described herein.

#### **Structural Characterization**

**(Porous Solid from Example 3 before the addition of the primary fragrance and the beta cyclodextrin complex of a secondary fragrance high impact perfume accord)**

<b>Structural Parameter</b>	<b>Value</b>
Kr BET Surface Area	0.047 m <sup>2</sup> /g
Pycnometry % Open Cells	90.7%
Micro-CT Cell Wall thickness	0.050 mm
Micro-CT Star Volume	11.1 mm <sup>3</sup>
Micro-CT SMI Index	2.4

The above structural measurements are indicative of a predominantly open-celled porous solid and within the structural ranges of the present invention.

**Dissolution Performance:**

The below table summarizes the conductivity dissolution and hand dissolution performance data taken from the prepared dissolvable porous solid shampoos comprising both a primary fragrance and a beta cyclodextrin complex of a secondary fragrance as described in examples 3 through 6. The data was collected by the methods as described herein.

	Conductivity Dissolution	Hand Dissolution
Dissolving porous solid shampoo with a primary fragrance 1a and a surface-added beta cyclodextrin complex of a secondary fragrance 2a (Ex. 3)	250 seconds	8 strokes
Dissolving porous solid shampoo with a primary fragrance 1b and a surface-added beta cyclodextrin complex of a secondary fragrance 2b (Ex. 4)	170 seconds	10 strokes
Dissolving porous solid shampoo with a primary fragrance 1a and an internally loaded beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2a (Ex. 5)	140 seconds	9 strokes
Dissolving porous solid shampoo with a primary fragrance 1b and an internally loaded beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2b (Ex. 6)	220 seconds	8 strokes

The above data on the porous solids comprising both a primary fragrance and a surface applied or internally applied beta cyclodextrin complex of a secondary fragrance indicate conductivity dissolutions within the ranges specified by the present invention. It can also be seen that these dissolution characteristics translate into a minimum number of strokes (8 to 10 strokes) for complete dissolution (typically less than 10 to 20 seconds) under the more consumer relevant applied shear conditions afforded by the hand dissolution methodology as described herein.

**Lather Performance:**

The below table summarizes the hair switch lather performance data comparing the shampoo liquid controls to the prepared dissolvable porous solid shampoos comprising both a primary fragrance and a beta cyclodextrin complex of a secondary fragrance as described in examples 1 through 6. The data was collected by the methods as described herein.

	Lather Volume
Liquid Shampoo comprising a primary fragrance 1a (Ex. 1)	140 ml
Liquid Shampoo comprising a primary fragrance 1b (Ex. 2)	110 ml
Dissolving porous solid shampoo with a primary fragrance 1a and a surface-added beta cyclodextrin complex of a secondary fragrance 2a (Ex. 3)	100 ml
Dissolving porous solid shampoo with a primary fragrance 1b and a surface-added beta cyclodextrin complex of a secondary fragrance 2b (Ex. 4)	100 ml
Dissolving porous solid shampoo with a primary fragrance 1a and an internally loaded beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2a (Ex. 5)	110 ml
Dissolving porous solid shampoo with a primary fragrance 1b and an internally loaded beta cyclodextrin complex of a secondary fragrance high impact perfume accord 2b (Ex. 6)	100 ml

The above data on the porous solids comprising both a primary fragrance and a surface applied or internally applied beta cyclodextrin complex of a secondary fragrance indicate good lather performance comparable to the performance from conventional liquid shampoos.

### GC/MS Headspace Analysis

GC/MS headspace data was taken from the prepared dissolvable porous solid shampoo comprising primary fragrance 1a and a beta cyclodextrin complex of a secondary fragrance 2a of example 3 as well as the conventional shampoo liquid comprising primary fragrance 1a of example 1. It should be noted that both the primary fragrance 1a and the secondary fragrance 2a comprise many of the same perfume raw materials, though they have distinct overall perfume characters from one another.

Figure 5 gives the relative concentration of 31 measured perfume components in the headspace for the shampoo pad “wet” (simulating initial perfume bloom from the pad upon dissolution) relative to the shampoo pad “dry” (solid neat product odor), expressed as a ratio of the amount of the individual perfume raw materials measured under the “wet” conditions divided by the “dry” conditions. Here it can be seen that despite the approximate 7:1 solid dilution with de-ionized water, 17 out of the 31 measured perfume raw materials exhibited a ratio of greater than 1.0 for the dissolved shampoo pad “wet” vs. the undiluted shampoo pad “dry” and with a mean ratio of 1.56. This data clearly demonstrates a water-activated perfume release (perfume bloom).



Figures 6 and 7 contrast the measured abundance of the more volatile and less volatile perfume raw materials, respectively, of the shampoo pad “wet” (simulating initial perfume bloom from the pad upon dissolution) relative to the shampoo pad “dry” (solid neat product odor). Despite the dilution conditions, here again one can clearly see an increase in the concentration of several highlighted perfume raw materials upon wetting (7:1 dilution of the solid with de-ionized water to dissolve it) relative to the original undiluted solid. These increased or new PRMs in the headspace are primarily due to the release of each respective PRM from the secondary fragrance in the  $\beta$ -cyclodextrin complex upon water addition.

Figure 8 gives the relative abundance of 31 measured perfume components of the shampoo pad “wet” (simulating initial perfume bloom from the pad upon dissolution) relative to the neat shampoo liquid, expressed as a ratio of the abundance of the individual perfume raw materials from the shampoo pad “wet” divided by the shampoo liquid. Here it can be seen that 22 out of the 31 measured perfume raw materials exhibited a ratio of greater than 1.0 for the dissolved shampoo pad “wet” vs. neat shampoo liquid and with a mean ratio of 2.05. This data clearly demonstrates approximately a 2 fold increase on average in concentration among these measured perfume raw materials in the headspace above the dissolved shampoo solid relative to the neat shampoo liquid.

Figures 9 gives the relative abundance of 31 measured perfume components of the fully diluted shampoo pad (after an initial 7:1 water dilution for dissolution and a further 10:1 water dilution to simulate in-use shampooing conditions) relative to the fully diluted shampoo liquid (after a 10:1 water dilution to simulate in-use shampooing conditions), expressed as a ratio of the concentration of the individual perfume raw materials from the shampoo pad “wet” diluted divided by the shampoo liquid diluted. Here it can be seen that 17 out of the 31 measured perfume raw materials exhibited a ratio of greater than 1.0 for the dissolved and fully diluted shampoo pad vs. the fully diluted neat shampoo liquid and with a mean ratio of 1.88. This data demonstrates a potential in-use (water dilution) perfume bloom benefit for the shampoo pad versus liquid shampoo control.

Figure 10 contrasts the measured abundance of perfume raw materials in the headspace above the dissolved shampoo pad “wet” (7:1 water dilution) comprising primary fragrance 1a and a beta cyclodextrin complex of a secondary fragrance 2a relative to the shampoo liquid control comprising the primary fragrance 1a (neat liquid product). In congruence with Figures 5 through 7, one can observe the release of several distinct or increased perfume raw materials from the addition of water to the shampoo solid (during dissolution) relative to the shampoo liquid control product.

### **Expert Perfume Sensory Panel**

The below table summarizes the Expert Perfume Sensory Panel data comparing the shampoo liquid controls comprising a primary fragrance to the prepared dissolvable porous solid

shampoos comprising both a primary fragrance and a beta cyclodextrin complex of a secondary fragrance as described in examples 1 through 4. The data was collected by the methods as described herein.

<b>Products</b>	<b>Neat Product Odor</b>	<b>Bloom (In hand)</b>	<b>Lather (On switch)</b>
Liquid Shampoo comprising a primary fragrance 1a (Example 1)	Peachy, fruity, citrus (Fragrance 1a character)	75 Peachy, fruity, citrus (Fragrance 1a character)	80 Peachy, fruity, citrus (Fragrance 1a character)
Dissolving porous solid shampoo with a primary fragrance 1a and a surface-added beta cyclodextrin complex of a secondary fragrance 2a (Example 3)	Peachy fruity, green, citrus (mostly Fragrance 1a character)	85/90 Peachy fruity, aldehydic green citrus (Mix of Fragrance 1a and Fragrance 2a characters)	90/95 aldehydic green citrus (Fragrance 2a character dominant)
Liquid Shampoo comprising a primary fragrance 1b (Example 2)	Green, apple-y, floral (Fragrance 1b character)	60 Apple-y, floral (Fragrance 1b character)	65/70 Apple-y, floral (Fragrance 1b character)
Dissolving porous solid shampoo with a primary fragrance 1b and a surface-added beta cyclodextrin complex of a secondary fragrance 2b (Example 4)	Green fruity floral (mix of Fragrance 1b & Fragrance 2b characters)	75/80 very strong, more fruity, green (Mix of Fragrance 1b and mostly Fragrance 2b characters)	80/85 More fruity, green (Mix of Fragrance 1b and mostly Fragrance 2b characters)

The above data demonstrates a significant and panel perceptible shifting of the fragrance of the solid shampoo pads upon dissolution in water (initial dissolution and full dilution) compared to the shampoo liquid controls. Moreover, the resulting fragrance of the dissolved shampoo pads is perceived as being more intense relative to the liquid shampoo controls. This novel result can be attributed to the water activated release of the secondary fragrance perfume raw materials upon dilution of the solid shampoos as is technically supported by the aforementioned GCMS headspace data and conclusions.

All documents cited are incorporated herein by reference in their entirety; the citation of any document is not to be construed as an admission that it is prior art with respect to the present invention. To the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the term in a document incorporated herein by reference, the meaning or definition assigned to the term in this document shall govern.

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

Note that any actives and/or compositions disclosed herein can be used in and/or with the articles, and in particular the household care articles, disclosed in the following U.S Patent Applications, including any publications claiming priority thereto: US 61/229981; US 61/229986; US 61/229990; US 61/229996; US 61/230000; and US 61/230004. Such articles may comprise one or more of the following: detergent surfactant; plasticizer; enzyme; suds suppressor; suds booster; bleach; bleach stabilizer; chelant; cleaning solvent; hydrotrope; divalent ion; fabric softener additive (e.g. quaternary ammonium compounds); nonionic surfactant; perfume; and/or a perfume delivery system. Such articles may be utilized in methods including, but not limited to: dosing into a washing machine to clean and/or treat fabric; dosing into a dishwasher to clean and/or treat cutlery; and dosing into water to clean and/or treat fabric and/or hard surfaces.

## CLAIMS

What is claimed is:

1. A personal care article comprising:

a.) a porous dissolvable solid substrate comprising:

- i. from 10% to 75% surfactant;
- ii. from 10% to 50% water-soluble polymer;
- iii. from 1% to 30% plasticizer; and

b.) a surface resident coating comprising from 10% to 100% of one or more cyclodextrin perfume complexes;

wherein the ratio of the cyclodextrin to perfume in the complex is from 100:1 to 1:1, and

wherein the ratio of the porous dissolvable solid substrate to the surface resident coating is from 110:1 to 0.5:1.

2. The personal care article of Claim 1, wherein the cyclodextrin is selected from alpha-cyclodextrin, beta-cyclodextrin, gamma-cyclodextrin, derivatives thereof, and mixtures thereof.

3. The personal care article of claim 1 or 2, wherein the surfactant comprises at least one Group I surfactant, and preferably additionally comprises a Group II surfactant, wherein the Group II surfactant is selected from the group consisting of amphoteric, zwitterionic, and combinations thereof.

4. The personal care article of claim 3, wherein the Group I surfactant is an anionic surfactant selected from the group consisting of alkyl and alkyl ether sulfates, sulfated monoglycerides, sulfonated olefins, alkyl aryl sulfonates, primary or secondary alkane sulfonates, alkyl sulfosuccinates, acid taurates, acid isethionates, alkyl glycerylether sulfonate, sulfonated methyl esters, sulfonated fatty acids, alkyl phosphates, acyl glutamates, acyl sarcosinates, alkyl lactylates, anionic fluorosurfactants, sodium lauroyl glutamate, and combinations thereof.

5. The personal care article of Claim 1, wherein the surfactant is selected from the group consisting of anionic surfactant, non-ionic surfactant, polymeric surfactant, and combinations thereof.

6. The personal care article of any of claims 1-5, wherein the porous dissolvable solid substrate has a basis weight of from 125 grams/m<sup>2</sup> to 3,000 grams/m<sup>2</sup> and a thickness of from 0.5 mm to 10 mm, and further wherein said porous dissolvable solid substrate preferably has a surface area from 0.03 m<sup>2</sup>/gram to 0.25 m<sup>2</sup>/gram.

7. The personal care article of any of claims 1-6, wherein the porous dissolvable solid substrate has a percent open cell content of from 80% to 100%, and further wherein said porous dissolvable solid substrate preferably has a cell wall thickness of from 0.02 mm to 0.15 mm.

8. A method for making the personal care article of any of claims 1-7, the method comprising applying a surface resident coating comprising a cyclodextrin perfume complex in powdered form to a porous dissolvable solid substrate, wherein said porous dissolvable solid substrate comprises:

- (a) from 10% to 75% surfactant;
- (b) from 10% to 50% water-soluble polymer; and
- (c) from 1% to 30% plasticizer.

9. A method for making the personal care article of any of claims 1-8, the method comprising:

- (a) preparing a processing mixture comprising from 5% to 50% surfactant, from 5% to 35% water-soluble polymer, and from 0.5% to 20% plasticizer;
- (b) aerating the processing mixture by introducing a gas into the processing mixture to form an aerated wet mixture;
- (c) forming the aerated wet mixture into one or more desired shapes;
- (d) drying the aerated wet mixture to form a porous dissolvable solid substrate; and
- (e) applying a surface resident coating comprising a cyclodextrin perfume complex in powdered form to the porous dissolvable solid substrate.

10. The method according to claims 8 or 9, wherein the surface resident coating comprising the cyclodextrin perfume complex is applied to the porous dissolvable solid substrate at a humidity of 20% to 70%, preferably from 30% to 60%.

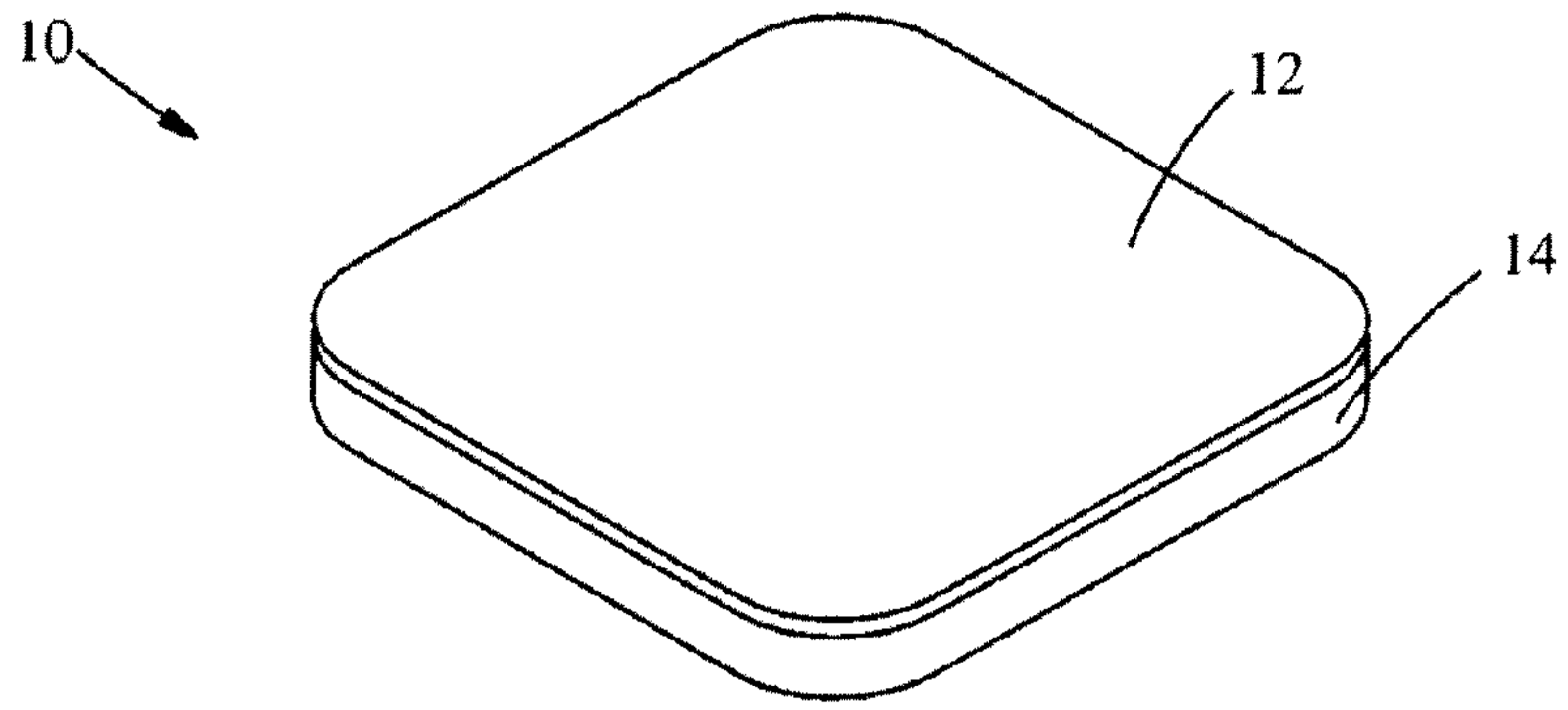


Fig. 1

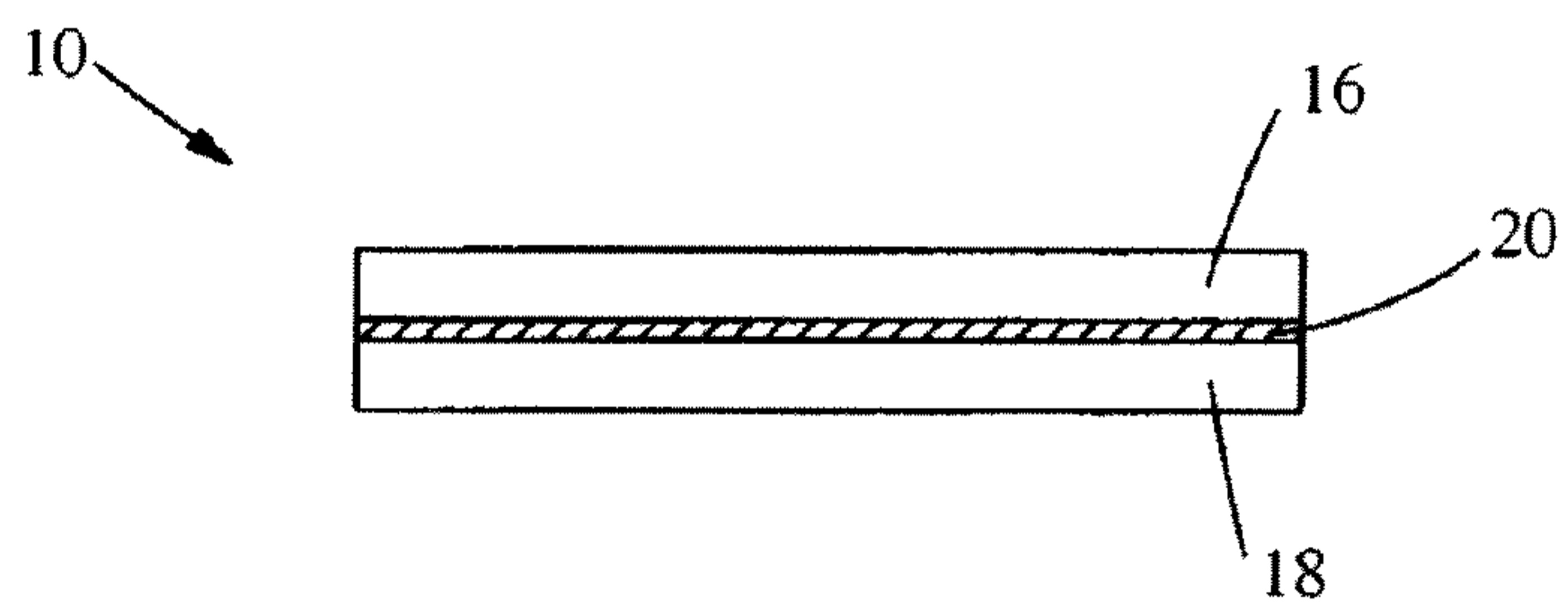
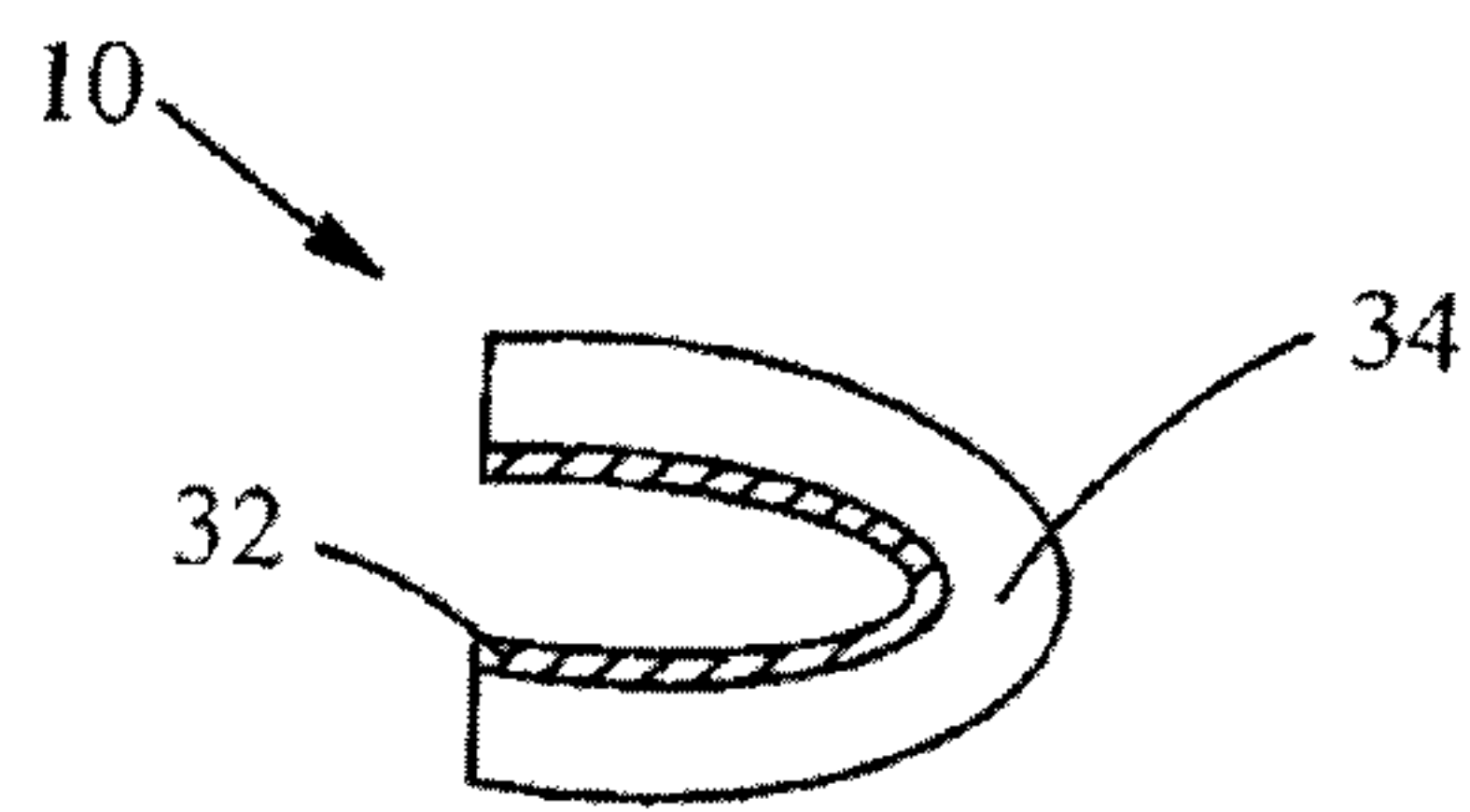
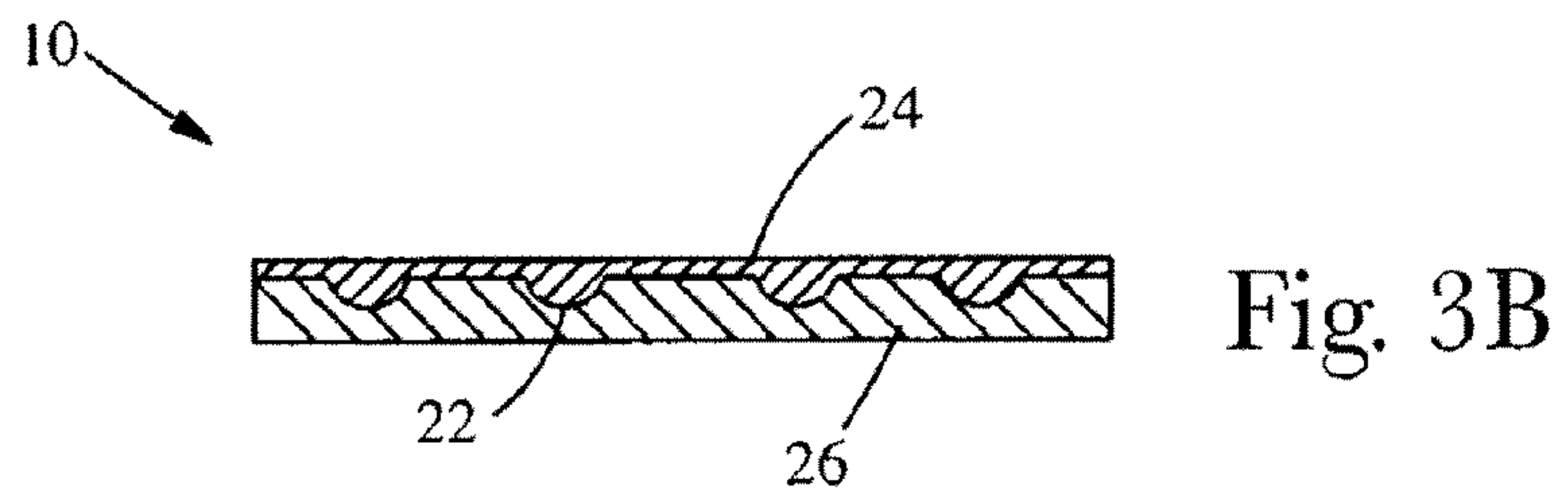
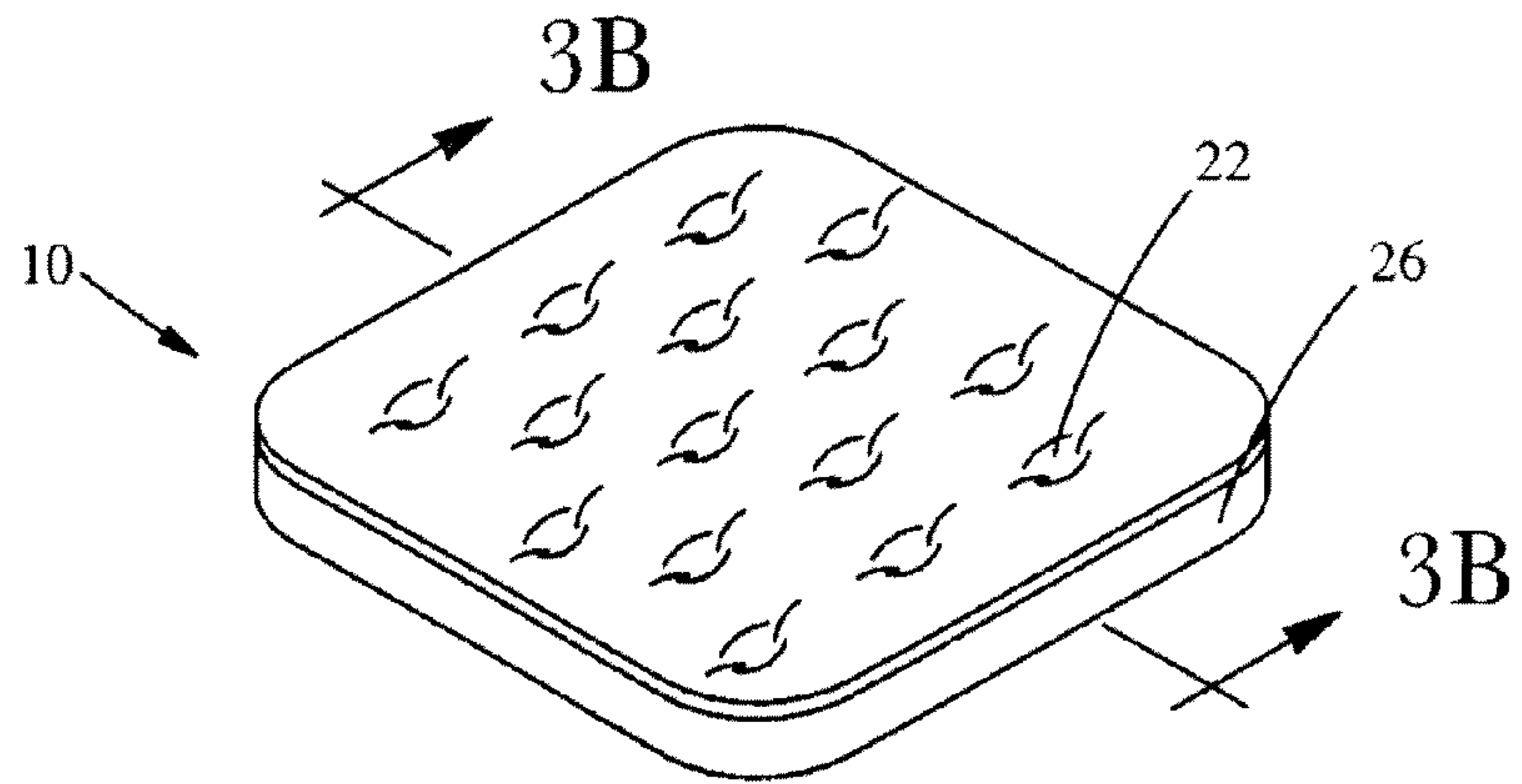
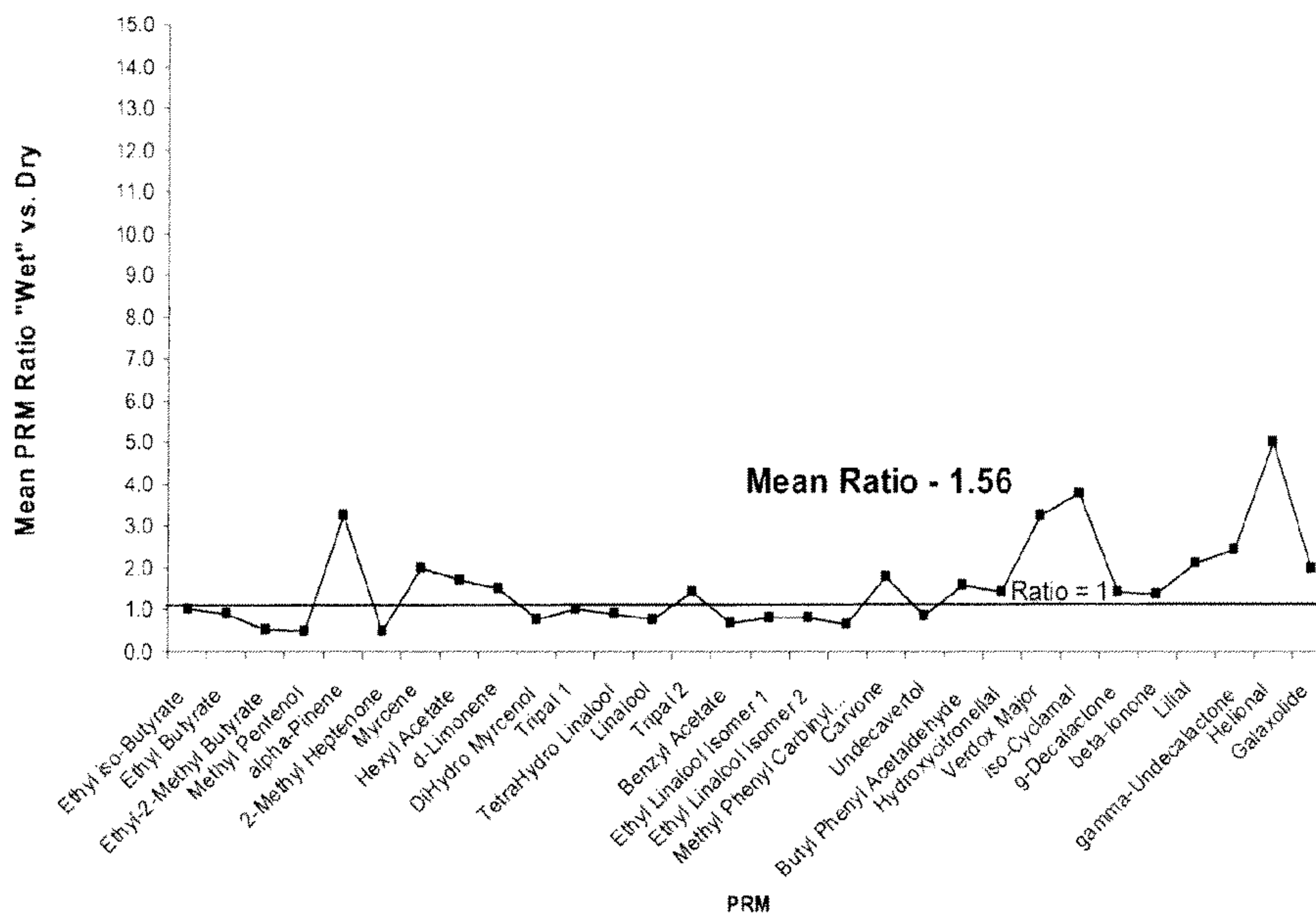


Fig. 2

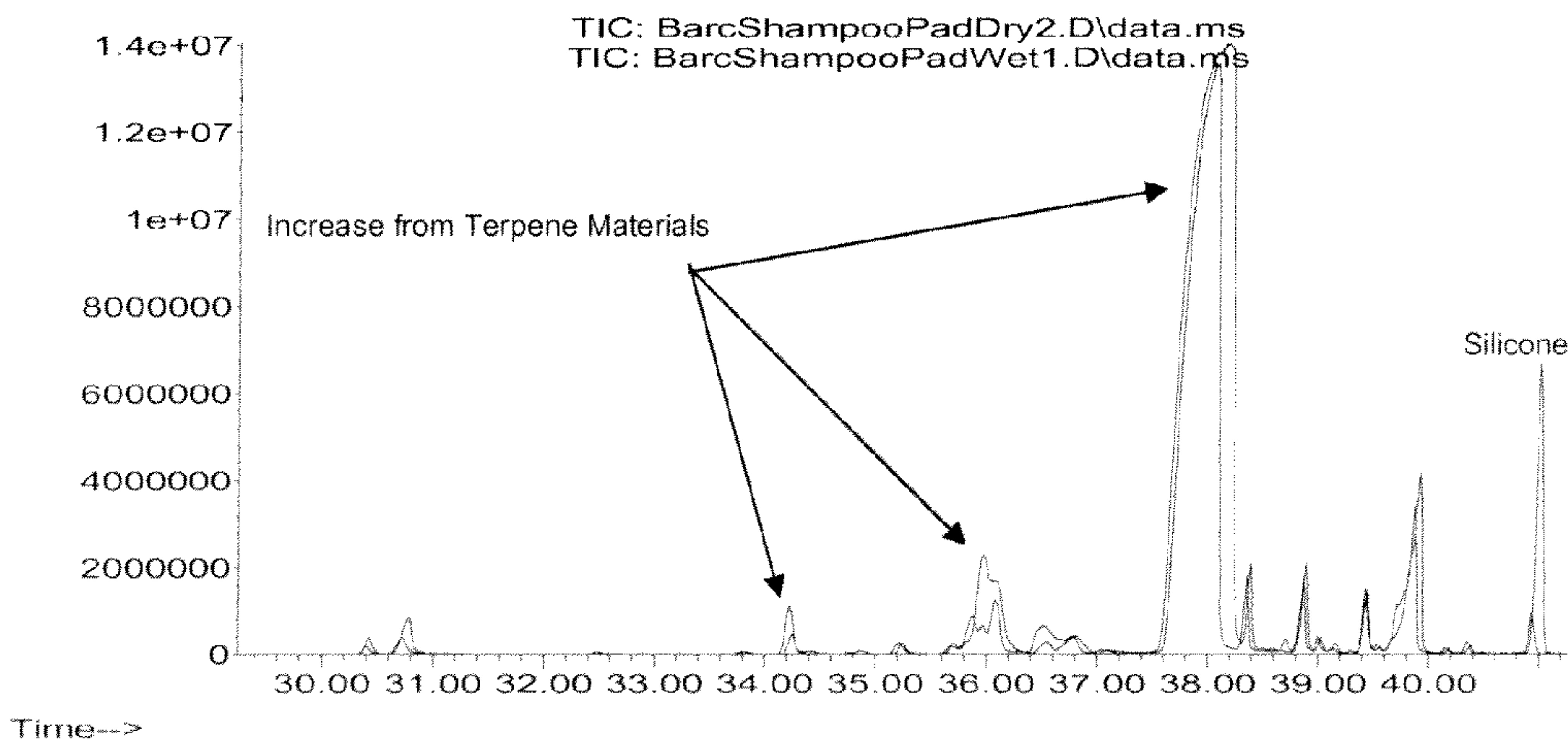


**Figure 5. GC/MS Headspace ratio of perfume raw materials (PRM) from the dissolved shampoo pad (7:1 water dilution) vs. the original dry shampoo pad**



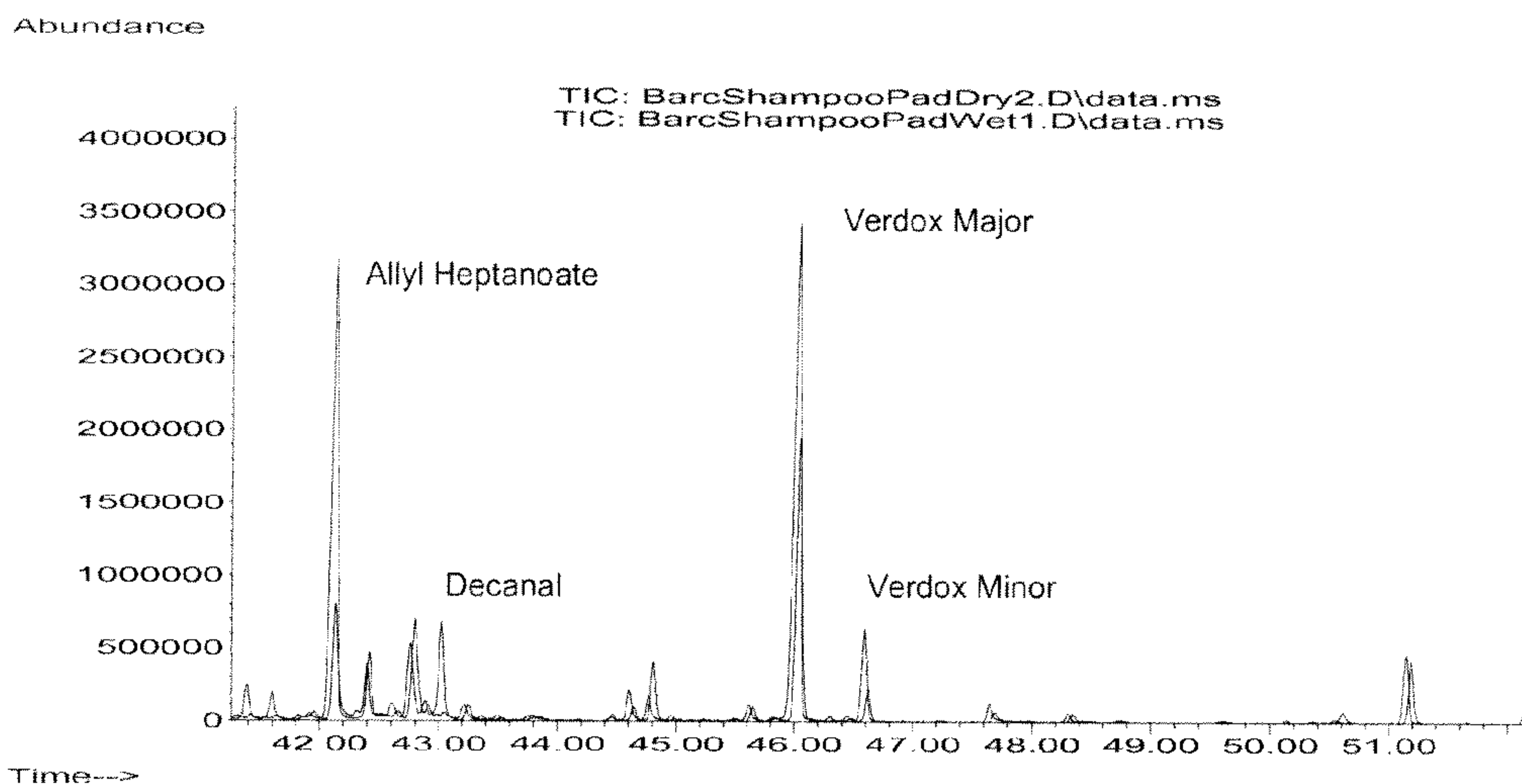
**Figure 6. GC headspace abundance of the more volatile perfume raw materials from the dissolved shampoo pad (7:1 water dilution) vs. the original dry shampoo pad**

Abundance

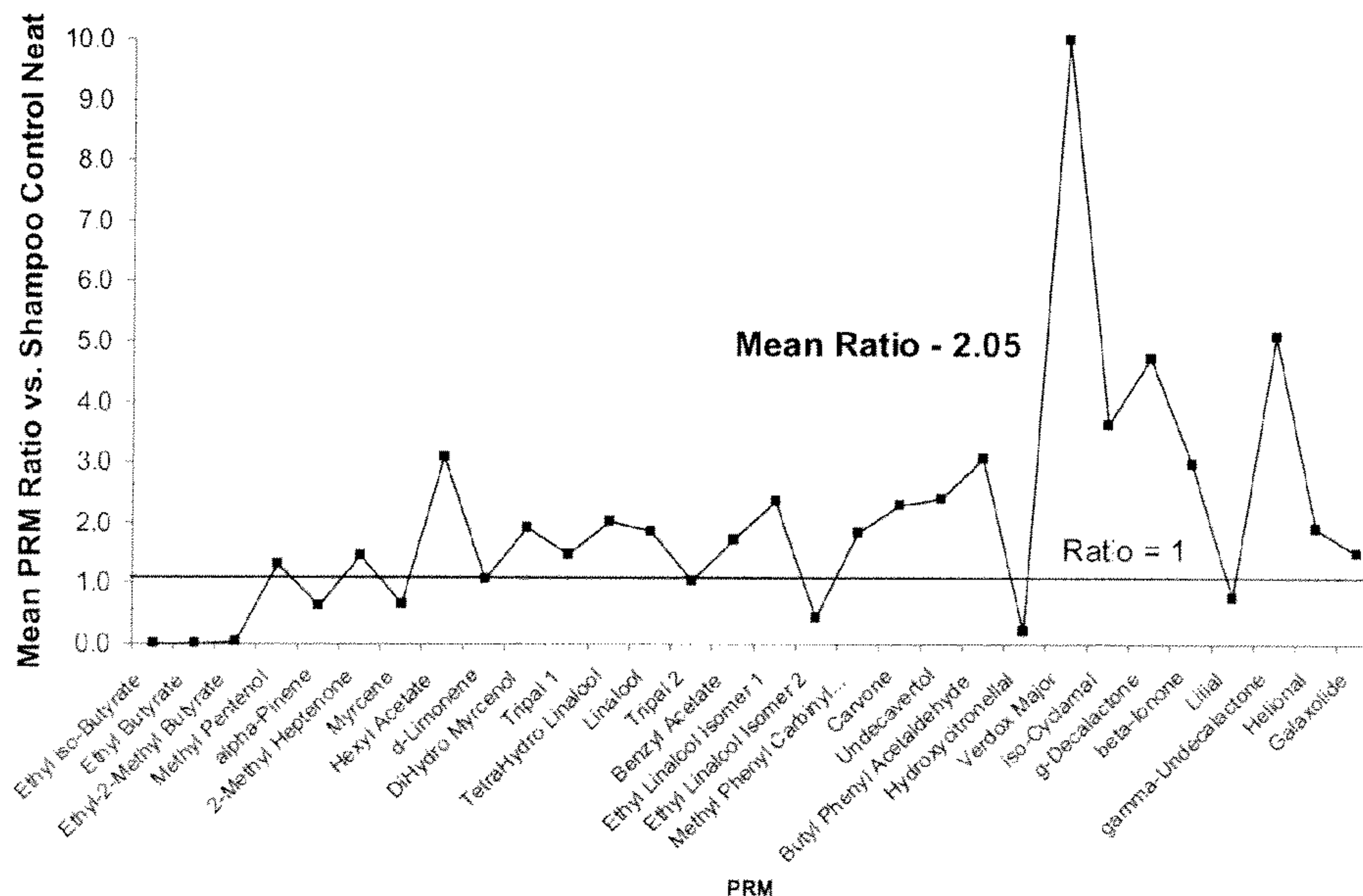




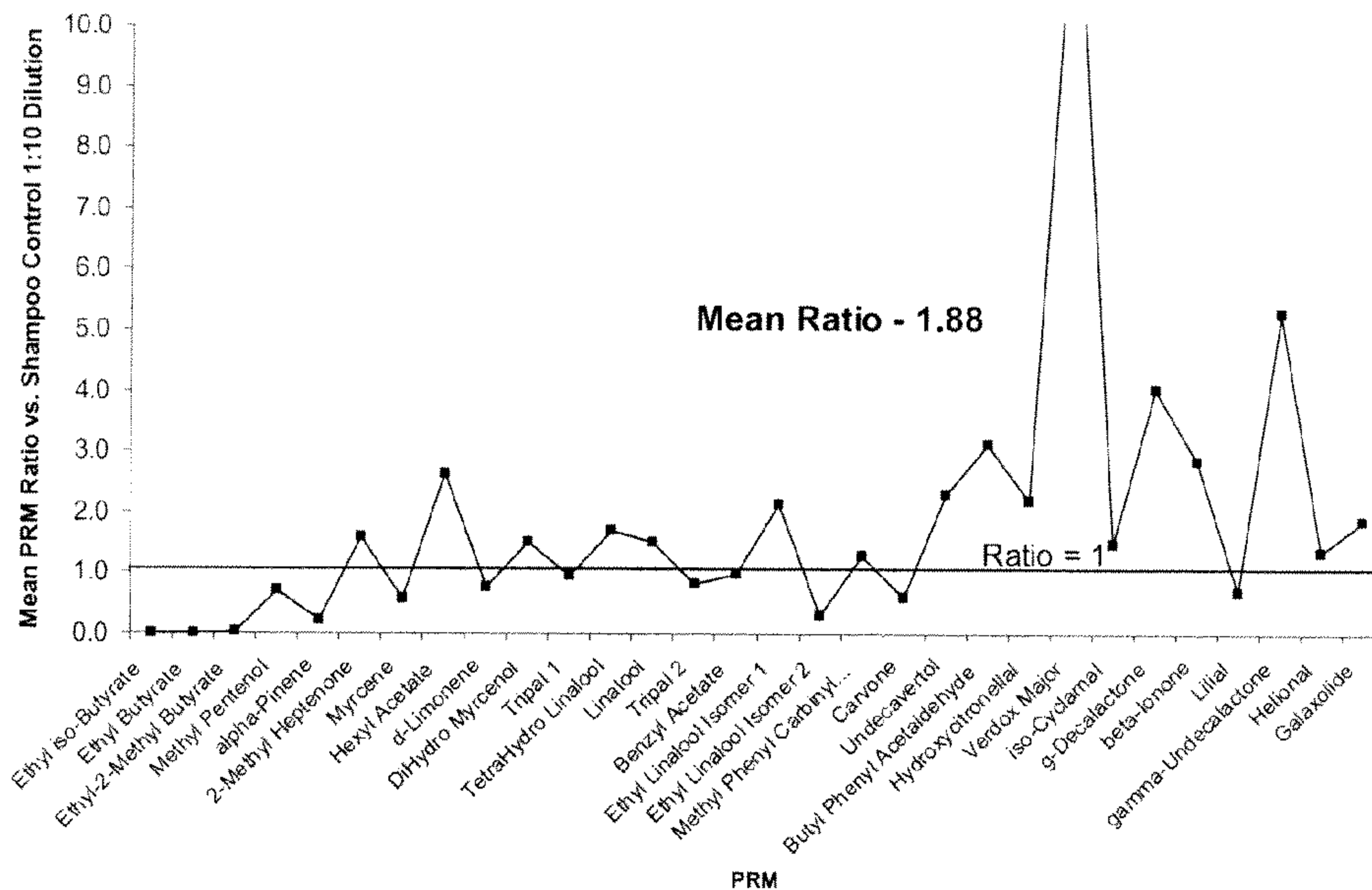
**Figure 7.** GC headspace abundance of the less volatile perfume raw materials from the dissolved shampoo pad (7:1 water dilution) vs. the original dry shampoo pad



**Figure 8.** GC/MS Headspace ratio of perfume raw materials (PRM) from the dissolved shampoo pad vs. the neat liquid shampoo control



**Figure 9. GC/MS Headspace ratio of perfume raw materials (PRM) from the dissolved and fully diluted shampoo pad vs. the fully diluted liquid shampoo control (10 parts de-ionized water to 1 part liquid shampoo or dissolved pad shampoo)**



**Figure 10. GC headspace abundance of perfume raw materials from the dissolved shampoo pad (7:1 water dilution) vs. the neat liquid shampoo control**

