

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of: Beetz et al.
U.S. Patent No.: 9,332,776 Attorney Docket No. 61529-0003IP1
Issue Date: May 10, 2016
Appl. Serial No.: 14/606,771
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Title: METHODS AND APPARATUS FOR LOW HEAT SPRAY
DRYING

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**PETITION FOR *INTER PARTES* REVIEW OF UNITED STATES PATENT
NO. 9,332,776 PURSUANT TO 35 U.S.C. §§ 311–319, 37 C.F.R. § 42**

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LIST OF EXHIBITS

- EX-1001 U.S. Patent No. 9,332,776 B1 to Beetz et al., “Methods and Apparatus for Low Heat Spray Drying,” issued May 10, 2016 (“776Patent”).
- EX-1002 Prosecution History of U.S. Patent No. 9,332,776 B1 (“Prosecution History”).
- EX-1003 Declaration of Dr. Dave A. Miller, Ph.D (“Miller Declaration”).
- EX-1004 Subramaniam et al., European Patent No. EP 1,435,797 B1, “Spray-Dried Compositions and Method for Their Preparation,” granted Aug. 22, 2007 (“Subramaniam”).
- EX-1005 Renata Baranauskienė, Eglė Bylaitė, Jūratė Žukauskaitė & Rimantas P. Venskutonis, “Flavor Retention of Peppermint (*Mentha piperita* L.) Essential Oil Spray-Dried in Modified Starches during Encapsulation and Storage,” 55 J. Agric. Food Chem. 3027–3036 (2007) (“Baranauskiene”).
- EX-1006 Watanabe et al., U.S. Patent Application Publication No. US 2004/0228959 A1, “Edible Powder Material Having Excellent Shelf Stability,” published Nov. 18, 2004 (“Watanabe”).
- EX-1007 R.P. Singh, *Scientific Principles of Shelf Life Evaluation, in Shelf Life Evaluation of Foods 3* (C.M.D. Man & A.A. Jones eds., 1st ed. 1994) (“Singh”).
- EX-1008 ICH Harmonised Tripartite Guideline, Stability Testing of New Drug Substances and Products, Q1A(R2), Step 4 version (Feb. 6, 2003) (“ICH Q1A(R2)”).
- EX-1009 U.S. Food & Drug Admin., Guidance for Industry: Q1A(R2) Stability Testing of New Drug Substances and Products (Nov. 2003) (“FDA Q1A(R2)”).

- EX-1010 Hagemann et al., U.S. Patent No. 4,663,068, “Bleach-Stable Deodorant Perfumes in Detergent Powders,” issued May 5, 1987 (“Hagemann”).
- EX-1011 RESERVED
- EX-1012 Aurélie Bechoff, Claudie Dhuique-Mayer, Manuel Dornier, Keith I. Tomlins, Renaud Boulanger, Dominique Dufour & Andrew Westby, “Relationship between the kinetics of β -carotene degradation and formation of norisoprenoids in the storage of dried sweet potato chips,” 121 Food Chemistry 348–357 (2010) (“Bechoff”).
- EX-1013 Theodore P. Labuza, Shelf-Life Dating of Foods (Food & Nutrition Press, Westport, Conn. 1982) (“Labuza”).
- EX-1014 Manuel Jesús Palma Astudillo, “Extraction of Capsanthin from Paprika Flakes (*Capsicum annuum* L.) and Stabilization by Microencapsulation” (M.S. thesis, Univ. of Chile, Mar. 2013) + certificate of translation (“Palma”).
- EX-1015 Jennings, U.S. Patent No. 5,723,424, “Concrete Cleaning Mixture,” issued Mar. 3, 1998 (“Jennings”).
- EX-1016 EMEA/HMPC, Community Herbal Monograph on *Mentha × piperita* L., aetheroleum, Doc. Ref. EMEA/HMPC/349466/2006 (Final, Oct. 31, 2007) (“EMA Monograph”).
- EX-1017 “Aetheroleum Menthae Piperitae,” in WHO Monographs on Selected Medicinal Plants, Vol. 2, at 188 (World Health Organization, Geneva 2002) (“WHO Monograph”).
- EX-1018 József Deli, Péter Molnár, Zoltán Matus & Gyula Tóth, “Carotenoid Composition in the Fruits of Red Paprika (*Capsicum annuum* var. *lycopersiciforme rubrum*) during Ripening; Biosynthesis of Carotenoids in Red Paprika,” 49 J. Agric. Food Chem. 1517–1523 (2001) (“Deli”).
- EX-1019 Ayhan Topuz & Feramuz Özdemir, “Assessment of carotenoids, capsaicinoids and ascorbic acid composition of some selected pepper

cultivars (*Capsicum annuum* L.) grown in Turkey,” 20 J. Food Composition & Analysis 596–602 (2007) (“Topuz 2007”).

- EX-1020 H.-G. Grigoleit & P. Grigoleit, “Pharmacology and preclinical pharmacokinetics of peppermint oil,” 12 *Phytomedicine* 612–616 (2005) (“Grigoleit”).
- EX-1021 Dámaso Hornero-Méndez & M. Isabel Mínguez-Mosquera, “Rapid Spectrophotometric Determination of Red and Yellow Isochromic Carotenoid Fractions in Paprika and Red Pepper Oleoresins,” 49 J. Agric. Food Chem. 3584–3588 (2001) (“Hornero-Méndez”).
- EX-1022 G. Britton, S. Liaaen-Jensen & H. Pfander (eds.), *Carotenoids*, Volume 1B: Spectroscopy (Birkhäuser Verlag 1995) (“Britton”).
- EX-1023 Manuel Jarén-Galán & M. Isabel Mínguez-Mosquera, “Quantitative and Qualitative Changes Associated with Heat Treatments in the Carotenoid Content of Paprika Oleoresins,” 47 J. Agric. Food Chem. 4379–4383 (1999) (“Jarén-Galán”).
- EX-1024 Antonio Pérez-Gálvez & M. Isabel Mínguez-Mosquera, “Structure-Reactivity Relationship in the Oxidation of Carotenoid Pigments of the Pepper (*Capsicum annuum* L.),” 49 J. Agric. Food Chem. 4864–4869 (2001) (“Pérez-Gálvez 2001”).
- EX-1025 Antonio Pérez-Gálvez & M. Isabel Mínguez-Mosquera, “Degradation of non-esterified and esterified xanthophylls by free radicals,” 1569 *Biochimica et Biophysica Acta* 31–34 (2002) (“Pérez-Gálvez 2002”).
- EX-1026 Ayhan Topuz & Feramuz Özdemir, “Influences of γ -Irradiation and Storage on the Carotenoids of Sun-Dried and Dehydrated Paprika,” 51 J. Agric. Food Chem. 4972–4977 (2003) (“Topuz 2003”).
- EX-1027 Ayhan Topuz, “A novel approach for color degradation kinetics of paprika as a function of water activity,” 41 *LWT – Food Science and Technology* 1672–1677 (2008) (“Topuz 2008”).
- EX-1028 J.H. Shin, H.L. Chung, J.K. Seo, J.H. Sim, C.S. Huh, S.K. Kim & Y.J.

Baek, “Degradation Kinetics of Capsanthin in Paprika (*Capsicum annuum* L.) as Affected by Heating,” 66 J. Food Sci. 15–19 (2001) (“Shin”).

- EX-1029 M. Isabel Mínguez-Mosquera & Manuel Jarén-Galán, “Kinetics of the Decolouring of Carotenoid Pigments,” 67 J. Sci. Food & Agric. 153–161 (1995) (“Mínguez-Mosquera”).
- EX-1030 Antonio Pérez-Gálvez, José J. Rios & María Isabel Mínguez-Mosquera, “Thermal Degradation Products Formed from Carotenoids during a Heat-Induced Degradation Process of Paprika Oleoresins (*Capsicum annuum* L.),” 53 J. Agric. Food Chem. 4820–4826 (2005) (“Pérez-Gálvez 2005”).
- EX-1031 José J. Rios, Elisabet Fernández-García, María Isabel Mínguez-Mosquera & Antonio Pérez-Gálvez, “Description of volatile compounds generated by the degradation of carotenoids in paprika, tomato and marigold oleoresins,” 106 Food Chemistry 1145–1153 (2008) (“Ríos”).
- EX-1032 David Pybus & Charles Sell (eds.), *The Chemistry of Fragrances* (Royal Society of Chemistry 1999) (“Pybus”).
- EX-1033 “Paprika Extract,” in *Compendium of Food Additive Specifications*, FAO JECFA Monographs 5, at 51 (69th meeting, Rome 2008) (“JECFA Monograph”).
- EX-1034 Richard Cantrill, “Paprika Extract: Chemical and Technical Assessment (CTA),” prepared for the 69th JECFA (2008) (“JECFA CTA”).
- EX-1035 James T. Scanlon & Donald E. Willis, “Calculation of Flame Ionization Detector Relative Response Factors Using the Effective Carbon Number Concept,” 23 J. Chromatogr. Sci. 333–340 (1985) (“Scanlon & Willis”).
- EX-1036 Thomas E. Furia & Nicolò Bellanca (eds.), *Fenaroli’s Handbook of Flavor Ingredients*, Vol. II (2d ed., CRC Press 1975) (“Fenaroli’s”).

- EX-1037 ACS Record (American Chemical Society Publications Record for Baranauskiene et al., 55 J. Agric. Food Chem. 3027 (2007), DOI 10.1021/jf062508c (published online Mar. 24, 2007; published in issue Apr. 1, 2007), available at <https://pubs.acs.org/doi/10.1021/jf062508c>)
- EX-1038 PubMed Record (U.S. National Library of Medicine, MEDLINE/PubMed Record for Baranauskiene et al. (PMID 17381102), available at <https://pubmed.ncbi.nlm.nih.gov/17381102/>)
- Ex-1039 Food & Agriculture Organization of the United Nations, Document Repository Record for Compendium of Food Additive Specifications, FAO JECFA Monographs 5 (Rome 2008), FAO Doc. No. i0345e, available at <https://www.fao.org/4/i0345e/i0345e00.htm> (“FAO Repository Record”)
- EX-1040 Food & Agriculture Organization of the United Nations, JECFA Online Database of Food Additive Specifications, available at <https://www.fao.org/food/food-safety-quality/scientific-advice/jecfa/jecfa-additives/en/> (“JECFA Database”)
- EX-1041 Baranauskiene Cited-By Bibliography (bibliography of select publications citing Baranauskiene et al., 55 J. Agric. Food Chem. 3027 (2007))
- EX-1042 Baranauskiene Cited-By Bibliography on Google Scholar (bibliography of publications citing Baranauskiene et al., 55 J. Agric. Food Chem. 3027 (2007) (available at https://scholar.google.com/scholar?cites=12531058245374932528&as_sdt=5,33&sciodt=0,33&hl=en))
- EX-1043 FAO guidelines on the structure and content of the document called “Chemical and Technical Assessment (CTA)” (available at https://www.fao.org/fileadmin/templates/agns/pdf/jecfa/2003-02-24_CTA.pdf)

LISTING OF CLAIMS

Claim 1	
[1pre]	A spray dried powder, comprising:
[1a]	a plurality of dried particles, which individually contain an amount of final active ingredient encapsulated within a carrier resulting from drying a slurry containing an initial active ingredient, a liquid solvent and the carrier, wherein:
[1b]	the initial active ingredient includes one or more constituent components, at least one of which is among one or more principle molecular types from which at least one of a desirable food, flavor, fragrance, medicament, and pigment is obtained;
[1c]	the final active ingredient includes one or more of the constituent components corresponding with those of the initial active ingredient as modified by the drying of the slurry; and
[1d]	a weight percentage of at least one of the one or more principle molecular types in the final active ingredient is within about 5% of a weight percentage of the corresponding principle molecular types in the initial active ingredient.
Claim 2	
[2]	The spray dried powder of claim 1, wherein the one or more principle molecular types are molecular types of a food composition.
Claim 3	
[3]	The spray dried powder of claim 1, wherein the one or more principle molecular types are molecular types of a flavor composition.
Claim 4	
[4]	The spray dried powder of claim 1, wherein the one or more principle molecular types are molecular types of a fragrance composition.

Claim 5	
[5]	The spray dried powder of claim 1, wherein the one or more principle molecular types are molecular types of a medicament composition.
Claim 7	
[7]	The spray dried powder of claim 1, wherein the weight percentage of at least one of the one or more principle molecular types in the final active ingredient is within about 3% of a weight percentage of the corresponding principle molecular types in the initial active ingredient.
Claim 8	
[8]	The spray dried powder of claim 1, wherein the weight percentage of at least one of the one or more principle molecular types in the final active ingredient is within about 2% of a weight percentage of the corresponding principle molecular types in the initial active ingredient.
Claim 9	
[9]	The spray dried powder of claim 1, wherein the weight percentage of at least one of the one or more principle molecular types in the final active ingredient is within about 1% of a weight percentage of the corresponding principle molecular types in the initial active ingredient.
Claim 10	
[10]	A spray dried powder, comprising:
[10a]	a plurality of dried particles, which individually contain an amount of active ingredient encapsulated within a carrier, wherein:
[10b]	the active ingredient includes one or more constituent components, at least one of which is among one or more principle molecular types from which at least one of a desirable food, flavor, fragrance, medicament, and pigment is obtained; and

[10c]	a weight percentage of at least one of the one or more principle molecular types in the active ingredient does not vary by more than about 5% during aging of the dried powder during any period of elevated temperature of about 95° F. up to about 1000 hours.
Claim 11	
[11]	The spray dried powder of claim 10, wherein the one or more principle molecular types are molecular types of a food composition.
Claim 12	
[12]	The spray dried powder of claim 10, wherein the one or more principle molecular types are molecular types of a flavor composition.
Claim 13	
[13]	The spray dried powder of claim 10, wherein the one or more principle molecular types are molecular types of a fragrance composition.
Claim 14	
[14]	The spray dried powder of claim 10, wherein the one or more principle molecular types are molecular types of a medicament composition.
Claim 15	
[15]	The spray dried powder of claim 10, wherein the one or more principle molecular types are molecular types of a pigment composition.
Claim 16	
[16]	The spray dried powder of claim 10, wherein the weight percentage of at least one of the one or more principle molecular types in the active ingredient does not vary by more than about 3% during aging of the dried [powder] during any period of elevated temperature of about 95° F. up to about 1000 hours.

Claim 17	
[17]	The spray dried powder of claim 10, wherein the weight percentage of at least one of the one or more principle molecular types in the active ingredient does not vary by more than about 2% during aging of the dried [powder] during any period of elevated temperature of about 95° F. up to about 1000 hours.
Claim 18	
[18]	The spray dried powder of claim 10, wherein the weight percentage of at least one of the one or more principle molecular types in the active ingredient does not vary by more than about 1% during aging of the dried [powder] during any period of elevated temperature of about 95° F. up to about 1000 hours.
Claim 19	
[19]	The spray dried powder of claim 10, wherein the carrier comprises starch.

I. INTRODUCTION

McCormick & Company, Inc. (“Petitioner”) petitions for IPR of claims 1-5, 7-19 (“Challenged Claims”) of U.S. Patent 9,332,776 (“’776Patent”). A reasonable likelihood exists that Petitioner will prevail on at least one Challenged Claim.

II. IPR REQUIREMENTS

A. Standing

Petitioner certifies the ’776Patent is available for IPR, and Petitioner is not barred/estopped from requesting this IPR.

B. Challenge / Relief Requested

1. Prior Art

Petitioner’s grounds are based on a September 27, 2010, priority date.¹

The references are analogous to the ’776Patent. First, each is in the same field of endeavor as the ’776Patent: spray drying active ingredients (e.g., flavors, fragrances, pigments) encapsulated within a carrier to form powders. *See, e.g.*, EX-1004, Title, [0002], [0005]-[0006], [0060]-[0061] (Example 5), [0064]-[0067], Table 1; EX-1005, [3027], [3029]-[3031], Tables 1-2 ; EX-1006, Title, [0001]-[0002], [0008], [0053], [0062]-[0064], Table 4; EX-1003, ¶¶13-14, 71-83.

Second, each reference is reasonably pertinent to the problems faced by the

¹ Petitioner does not concede entitlement to this date.

inventors: improving retention (measured after spray-drying) and/or stability (measured after long-term storage at elevated temperatures) of the powder's active ingredient. *See* EX-1004, *e.g.*, [0060]–[0061], Example 5 and [0067]–[0068], Table 1 ; EX-1005, [3028], Abstract ; EX-1006, [0053]–[0058], Table 3 ; EX-1003, ¶¶13-14, 56-63.

Prior Art	Publication Date	Basis ²
Subramaniam (EX-1004)	August 22, 2007	102(b)
Baranauskiene (EX-1005)	March 24, 2007 (EX-1003, ¶¶19-23)	102(b)
Watanabe (EX-1006)	November 18, 2004	102(b)
JECFA Monograph (EX-1033)	2008 (EX-1003, ¶¶24-28)	102(b)
JECFA CTA (EX-1034)	2008 (EX-1003, ¶¶29-33)	102(b)

2. Grounds

² Citations are to pre-AIA statute; analysis would be identical under post-AIA statute.

Ground	Claims	Prior Art and Basis
1	1-5, 7-9	§103: Baranauskiene
2	10-11, 15-17, 19	§103: Watanabe in view of JECFA References
3	1-5, 7-14, 16-19	§103: Subramaniam

C. Level of Ordinary Skill in the Art

A person of ordinary skill in the art (a “POSITA”) as of the priority date would have had (1) at least a bachelor’s degree in chemical engineering, chemistry, pharmaceutical science, food science, or a related field, and (2) at least three years of experience in the formulation, spray-drying, or encapsulation of pharmaceutical, food, flavor, fragrance, or pigment compositions. EX-1003, ¶¶15-17, 4-7. Additional education could substitute for professional experience, and vice versa. *Id.*

D. Claim Construction

Claim terms are construed herein using the standard used in civil actions under 35 U.S.C. §282(b), in accordance with the ordinary and customary meaning as understood by a POSITA and the patent’s prosecution history. 37 C.F.R. §42.100(b). The Board need only construe terms to the extent necessary “to resolve [a] controversy.” *Nidec Motor Corp. v. Zhongshan Broad Ocean Motor Co.*, 868 F.3d

1013, 1017 (Fed. Cir. 2017). Petitioners are not conceding that each Challenged Claim satisfies all statutory requirements, nor waiving arguments that can only be raised in district court.

III. '776PATENT

A. Description

The '776Patent claims a spray-dried powder of the type long known in the encapsulation art: dried particles in which an active ingredient (food, flavor, fragrance, medicament, or pigment) is encapsulated within a carrier. EX-1001, Abstract, 1:25–67. The claims purport to provide a powder with a weight percentage of a “principle molecular type” of the active ingredient measured either (i) before and after drying of a slurry of the active ingredient, carrier, and liquid solvent (final active ingredient within about 5% of the initial active ingredient) (measuring “*retention*”) or (ii) after aging of the resulting powder during any period of an elevated temperature up to about 1000 hours (variation within about 5% during aging at about 95°F) (measuring “*stability*”). EX-1001, claims 1, 10. EX-1003, ¶¶61-65.

B. Prosecution History

The application for the '776Patent is a continuation-in-part of an earlier non-provisional application, which claims the benefit of a provisional application filed on September 27, 2010. EX-1001, 1:7-21. EX-1003, ¶¶66-69.

In a non-final rejection, the application’s pending claims were rejected based

on a single reference, Jennings (EX-1015), which relates to a clay/cat litter concrete-cleaning mixture. EX-1002, 60-65. Applicant overcame that rejection in a response filed in March 2016. EX-1002, 30-38. EX-1003, ¶¶66-69.

The Examiner only substantively considered Jennings during prosecution. While Subramaniam (EX-1004) was cited among the references in the References Considered by the examiner (EX-1002 at 102), it was not substantively considered or applied in a rejection. Barauskiene (EX-1005), Watanabe (EX-1006),³ and the JECFA References (EX-1033, EX-1034) were not cited or considered during prosecution. EX-1003, ¶70.

IV. CHALLENGED CLAIMS ARE UNPATENTABLE

A. GROUND 1: Barauskiene

1. Barauskiene

Barauskiene (EX-1005) evaluates the effect of different food starch carriers on the flavor retention of peppermint essential oil (“EO”) during spray drying. EX-1005, [3027] (Abstract), [3028]. Barauskiene evaluated these effects on eleven constituent components of EO, which was emulsified into aqueous solutions of eight starch carriers, creating eight different emulsions that were then spray dried. EX-

³ Patent Owner (ZoomEssence, Inc.) has known about Barauskiene and Watanabe since at least 2017.

1005, [3028]. This resulted in eight spray-dried powders in which the EO was encapsulated by eight carriers, shown in Table 2. EX-1005, [3031], Table 2. EX-1003, ¶71.

Baranauskiene (EX-1005), Table 2
(annotated)
Boxes 1-8 = the eight commercial modified starch carriers

Table 2. Changes in the Total Oil Composition after Processing in Both Liquid-Emulsified and Spray-Dried Encapsulated Products as Compared with Pure Initial EO^a

no.	compound	KI on DB5	pure EO	1 HI-CAP 100		2 N-LOK		3 CAPSUL	
				emulsion	powder	emulsion	powder	emulsion	powder
1	limonene ^{c,e}	1032	0.6 bc	0.5 a	0.5 abc	0.5 a	0.4 a	0.5 abc	0.5 abc
2	menthone ^{c,u}	1166	19.5 cde	18.8 abc	19.0 bcde	19.0 bcde	18.8 abc	19.3 bcde	19.3 bcde
3	iso-menthone ^{b,d}	1176	16.0 bc	15.8 abc	16.0 bc	15.8 abc	15.3 a	16.4 c	15.8 abc
4	menthol ^{c,e}	1194	47.5 a	49.5 cde	49.1 bcde	49.3 bcde	50.0 de	48.5 bc	48.5 bc
5	iso-menthol ^{c,e}	1197	0.8 abc	0.9 bcde	0.9 abcde	0.9 abcde	0.7 a	1.0 cde	0.9 abcde
6	α-terpineol ^{c,e}	1201	0.9 c	0.8 b	0.8 b	0.8 b	0.9 cd	0.8 b	0.9 cd
7	cis-carveol ^d	1241	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a
8	pulegone ^{c,e}	1249	2.2 e	1.9 a	2.1 d	2.1 bcd	2.1 d	2.1 cd	2.1 d
9	piperitone ^d	1264	1.5 g	1.0 a	1.3 de	1.2 bc	1.3 cd	1.2 bc	1.4 f
10	menthyl acetate ^{c,e}	1300	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.2 a
11	β-caryophyllene ^{c,e}	1433	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.0 a	1.5 abcd
	% RSD ^f		3.2	2.4	4.5	5.5	2.4	2.4	2.7

no.	4 ENCAPSUL 855		5 CRYSTAL TEX627		6 CiEmCap 12633		7 CiEmCap 12634		8 CiEmCap 12635	
	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder
1	0.5 abc	0.4 a	0.5 abc	0.5 abc	0.5 a	0.6 bc	0.5 abc	0.6 bc	0.5 abc	0.6 c
2	19.8 cde	18.5 ab	18.1 a	18.8 abc	18.5 ab	20.0 e	18.8 abc	19.3 bcde	19.0 bcde	19.5 cde
3	16.1 bc	15.8 abc	15.8 abc	16.2 bc	15.5 abc	16.1 bc	16.2 bc	15.8 abc	16.0 bc	15.3 a
4	48.3 ab	50.0 e	49.1 bcde	49.0 bcde	50.1 de	48.3 ab	49.0 bcde	49.3 bcde	49.0 bcde	49.5 cde
5	0.8 abc	0.9 bcde	1.1 e	0.8 abc	0.8 ab	0.8 ab	0.9 bcde	0.80 abc	0.8 abc	0.8 abc
6	0.8 b	0.9 cd	0.8 b	0.9 cd	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.1 a
7	0.9 bc	0.8 abc	0.8 a	0.8 abc	0.8 a	0.9 bc	0.8 a	0.8 abc	0.8 a	0.9 c
8	2.1 d	2.0 bc	2.0 ab	2.1 d	2.0 bc	2.1 cd	2.1 cd	2.1 d	2.1 d	2.1 d
9	1.2 b	1.3 cd	1.2 bc	1.3 cd	1.2 cd	1.2 cd	1.2 bc	1.2 bc	1.3 ef	1.3 ef
10	4.3 abc	4.2 a	4.4 bc	4.2 a	4.3 ab	4.4 bc	4.3 abc	4.4 c	4.3 ab	4.3 ab
11	1.3 ab	1.0 a	2.0 d	1.0 a	1.8 bcd	1.3 ab	1.5 abcd	1.0 a	1.5 abcd	1.0 a
	1.9	3.6	2.5	3.0	4.4	2.3	1.2	2.0	1.3	2.4

Legend: = "emulsion" column (pre-dried) = "powder" column (spray-dried, encapsulated)

Each carrier (boxes 1-8) has both an emulsion and a powder column; "pure EO" is the initial oil before processing.

11 constituent components of peppermint EO ('EO')

(same 11 components)

The compositions of the various EOs—pure starting EO (“pure EO”), EO recovered from each liquid emulsion (“emulsion”), and EO recovered from each spray-dried encapsulated powder (“powder”)—were determined for the eleven identified peppermint constituents. EX-1005, [3028], [3031], Table 2. The peppermint EO composition (i.e., the eleven compounds) was essentially preserved through spray drying, as the pure, emulsified, and encapsulated oils were “quite

similar” across these eleven constituents, with only minor changes observed in a few individual compounds. EX-1005, [3027], [3031], Table 2; *id.*, 3027 (Abstract). EX-1003, ¶72.

2. Claim 1

(a) [1pre]

If the preamble is limiting, Baranauskiene discloses ***a spray-dried powder***.⁴ EX-1003, ¶¶85-87.

Baranauskiene’s title is “Flavor Retention of Peppermint (*Mentha piperita* L.) Essential Oil *Spray-Dried* in Modified Starches during Encapsulation and Storage.” EX-1005, [3027] (Title). Baranauskiene reports that its disclosed “[e]mulsions were spray-dried,” and the resulting product is repeatedly described as a “*spray-dried encapsulated powder*”⁵ or “*spray-dried powder*.” See EX-1005, [3028], [3031] (Table 2 caption: “Spray-Dried Encapsulated Products EX-1003, ¶¶86-87.

(b) [1a]

Baranauskiene discloses ***a plurality of dried particles*** (the spray-dried encapsulated peppermint EO powder particles), ***which individually contain an amount of final active ingredient*** (peppermint EO recovered from the powder)

⁴ ***Bold italics*** are used herein to identify claimed features.

⁵ *Underlined italics* are used herein to show added emphasis in the cited disclosures.

encapsulated within a carrier (modified food starch matrix) *resulting from drying a slurry* (spray-dried emulsion) *containing an initial active ingredient* (pure peppermint EO emulsified into the matrix), *a liquid solvent* (deionized water), *and the carrier* (the modified food starch). EX-1003, ¶¶88-94.

Baranauskiene describes the preparation of the spray-dried particles (*plurality of dried particles*) as follows: “The solutions of coating matrices [] were prepared by dispersing dried powders in [] water,” and after the EO “was emulsified into the hydrated coating material. Homogenization was accomplished by [a] ... basic homogenizer ... Emulsions were spray-dried” EX-1005, [3028]; EX-1003, ¶89.

Baranauskiene’s peppermint EO is an *active ingredient* contained in the plurality of dried particles, consistent with how this term is used in the ’776Patent. The ’776Patent describes an active ingredient as “any desirable single constituent component or a combination of constituent components” imparting a desirable characteristic, such a flavor, to an end product. EX-1001, 6:10–15; *see id.*, 1:40–43, 4:30–34. Further, the ’776Patent treats flavor and fragrance oils as active ingredients. EX-1001, 13:27–29; 14:48–50, Example 1, FIG. 11. Similarly, Baranauskiene’s EO has several constituent components (described in [1b]) that contribute to peppermint flavor. EX-1003, ¶90.

Baranauskiene discloses a *slurry* per the ’776Patent. *See* EX-1001, 1:60-67

(describing the making of “a slurry of ingredients,” wherein a “water 1 and carrier 2 are added into the solution tank 4 while stirring,” and then the “active ingredient 3 is ...added to the tank 4 and stirred into the slurry”; the active ingredient is either emulsified in the carrier fluid system or dissolved” therein). Baranauskiene’s emulsion (described above) is a *slurry* because it contains (i) peppermint EO (*initial active ingredient*), (ii) deionized water (*liquid solvent*), and (iii) the modified food starch (*carrier*). EX-1003, ¶91.

The spray-dried powder *results from drying* this *slurry*. EX-1005, [3028]. A POSITA understood the spray-dried powder includes a plurality of dried particles. EX-1003, ¶92.

Baranauskiene further discloses the *plurality of dried particles* each/*individually contain an amount of the final active ingredient* (EO) that is *encapsulated within* the modified-starch *carrier*. Baranauskiene calls its spray-dried products “*microencapsulated peppermint EO* products” and reports (in Table 1) the “total oil” content (encapsulated EO) and “surface oil” content (non-encapsulated EO) for each product, confirming that EO is encapsulated within the carrier in each particle. EX-1005, [3027] (Abstract); [3028]; [3030], Table 1; EX-1003, ¶93-94.

Baranauskiene (EX-1005), Table 1 (annotated)

Boxes 1-8 = the eight commercial modified-starch carriers

Table 1. Properties of Liquid Emulsion and Spray-Dried Microencapsulated Peppermint EO Products

	1 HI-CAP 100	2 N-LOK	3 CAPSUL	4 ENCAPSUL 855	5 CRYSTAL TEX 627	6 CIEmCAP 12633	7 CIEmCAP 12634	8 CIEmCAP 12635
	liquid microencapsulated product							
total oil content (g 100 g ⁻¹)	15.03 cd	14.44 bcd	14.73 bcd	13.54 a	14.14 ab	15.21 d	14.44 bcd	14.81 bcd
emulsification efficiency ^a (%)	98.56 cd	94.69 bcd	96.59 bcd	88.78 a	92.72 ab	99.74 d	94.69 bcd	97.11 bcd
	spray-dried microencapsulated product							
total oil content (g 100 g ⁻¹)	13.49 b	12.15 e	13.96 c	6.10 a	9.41 d	14.94 g	13.67 bc	14.48 f
surface oil content (g 100 g ⁻¹)	0.20 cd	0.18 bc	0.25 def	0.13 ab	0.23 cdef	0.08 a	0.17 bc	0.29 f
encapsulation efficiency ^a (%)	87.15 b	78.49 e	89.90 c	39.15 a	60.19 d	97.44 g	88.52 bc	93.05 f
moisture (mL 100 g ⁻¹)	1.83 a	2.08 ab	3.34 cd	1.49 a	3.39 cd	2.35 ab	2.85 bcd	3.75 d
water activity (a _w)	0.39 ab	0.43 abc	0.35 a	0.45 bc	0.44 bc	0.44 bc	0.49 cde	0.55 e
particle size weighted mean diameter d ₃ (μm)	27.19 d	34.99 f	30.50 e	11.9 a	23.52 b	137.63 h	56.88 g	24.82 c
specific surface area (m ² g ⁻¹)	17.7 e	16.6 d	16.8 d	0.7 a	10.5 a	4.3 b	19.4 f	17.4 e

^a Computed on the basis of a theoretical oil content of 15.25% of the solids. For letters a-h, values within rows followed by the same letter do not differ statistically at P = 0.05.

□ = 8 modified-starch carriers (boxes 1-8) ■ = EMULSION: liquid product (pre-dried) ■ = ENCAPSULATED: spray-dried product
 □ Each dried particle holds EO encapsulated in the starch carrier — Total oil = encapsulated EO • Surface oil = non-encapsulated EO

(c) [1b]

Baranauskiene discloses *the initial active ingredient* (pure peppermint EO) *includes one or more constituent components* (at least one of eleven peppermint EO compounds in Table 2), *at least one of which is among one or more principle molecular types* (menthol, menthone, iso-menthone + neo-menthol, menthyl acetate, and pulegone) *from which at least one of a desirable flavor* (peppermint flavor) *is obtained*. EX-1003, ¶¶95-99.

Baranauskiene's *initial active ingredient*—pure peppermint EO—is a complex mixture of 57 identified volatile compounds. EX-1005, [3031]. Table 2 reports on eleven of these compounds. EX-1005, [3028]; [3031], Table 2. Each compound is a *constituent component* of the pure EO. EX-1003, ¶96.

Baranauskiene (EX-1005), Table 2

(annotated)

Boxes 1-8 = the eight commercial modified starch carriers

11 constituent
components
of peppermint
EO ('EO')

(same 11
components)

Table 2. Changes in the Total Oil Composition after Processing in Both Liquid-Emulsified and Spray-Dried Encapsulated Products as Compared with Pure Initial EO^a

no.	compound	KI on DB5	1 HI-CAP 100			2 N-LOK		3 CAPSUL	
			pure EO	emulsion	powder	emulsion	powder	emulsion	powder
1	limonene ^{c,e}	1032	0.6 bc	0.5 a	0.5 abc	0.5 a	0.4 a	0.5 abc	0.5 abc
2	menthone ^{c,e}	1166	19.5 cde	18.8 abc	19.0 bcde	19.0 bcde	18.8 abc	19.3 bcde	19.3 bcde
3	iso-menthone ^{b,d}	1176	16.0 bc	15.8 abc	16.0 bc	15.8 abc	15.3 a	16.4 c	15.8 abc
4	menthol ^{c,e}	1194	47.5 a	49.5 cde	49.1 bcde	49.3 bcde	50.0 de	48.5 bc	48.5 bc
5	iso-menthol ^{c,e}	1197	0.8 abc	0.9 bcde	0.9 abcde	0.9 abcde	0.7 a	1.0 cde	0.9 abcde
6	α-terpineol ^{c,e}	1201	0.9 c	0.8 b	0.8 b	0.8 b	0.9 cd	0.8 b	0.9 cd
7	cis-carveol ^f	1241	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a
8	pulegone ^{c,e}	1249	2.2 e	1.9 a	2.1 d	2.1 bcd	2.1 d	2.1 cd	2.1 d
9	piperitone ^d	1264	1.5 g	1.0 a	1.3 de	1.2 bc	1.3 cd	1.2 bc	1.4 f
10	menthyl acetate ^{c,e}	1300	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.2 a
11	β-caryophyllene ^{c,e}	1433	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.0 a	1.5 abcd
	% RSD ^f		3.2	2.4	4.5	5.5	2.4	2.4	2.7

no.	4 ENCAPSUL 855		5 CRYSTAL TEX627		6 ClEmCap 12633		7 ClEmCap 12634		8 ClEmCap 12635	
	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder
1	0.5 abc	0.4 a	0.5 abc	0.5 abc	0.5 a	0.6 bc	0.5 abc	0.6 bc	0.5 abc	0.6 c
2	19.8 cde	18.5 ab	18.1 a	18.8 abc	18.5 ab	20.0 e	18.8 abc	19.3 bcde	19.0 bcde	19.5 cde
3	16.1 bc	15.8 abc	15.8 abc	16.2 bc	15.5 abc	16.1 bc	16.2 bc	15.8 abc	16.0 bc	15.3 a
4	48.3 ab	50.0 e	49.1 bcde	49.0 bcde	50.1 de	48.3 ab	49.0 bcde	49.3 bcde	49.0 bcde	49.5 cde
5	0.8 abc	0.9 bcde	1.1 e	0.8 abc	0.8 ab	0.8 ab	0.9 bcde	0.80 abc	0.8 abc	0.8 abc
6	0.8 b	0.9 cd	0.8 b	0.9 cd	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.1 a
7	0.9 bc	0.8 abc	0.8 a	0.8 abc	0.8 a	0.9 bc	0.8 a	0.8 abc	0.8 a	0.9 c
8	2.1 d	2.0 bc	2.0 ab	2.1 d	2.0 bc	2.1 cd	2.1 cd	2.1 d	2.1 d	2.1 d
9	1.2 b	1.3 cd	1.2 bc	1.3 cd	1.2 cd	1.2 cd	1.2 bc	1.2 bc	1.2 bc	1.3 ef
10	4.3 abc	4.2 a	4.4 bc	4.2 a	4.3 ab	4.4 bc	4.3 abc	4.4 c	4.3 ab	4.3 ab
11	1.3 ab	1.0 a	2.0 d	1.0 a	1.8 bcd	1.3 ab	1.5 abcd	1.0 a	1.5 abcd	1.0 a
	1.9	3.6	2.5	3.0	4.4	2.3	1.2	2.0	1.3	2.4

□ = "emulsion" column (pre-dried)

□ = "powder" column (spray-dried, encapsulated)

□ = "pure EO" column (initial oil)

Each carrier (boxes 1-8) has both an emulsion and a powder column; "pure EO" is the initial oil before processing.

Baranauskiene discloses that the initial active ingredient includes *one or more principle molecular types from which at least one of a desirable food, flavor, fragrance, medicament, and pigment is obtained*. In the '776Patent, a "principle molecular type" from which a desirable characteristic (here, flavor) of the active ingredient is obtained encompasses a constituent component that contributes to the desired characteristic (here, flavor), such that when the component's concentration decreases in the overall spray-dried composition, the desired characteristic (here, flavor) is likewise undesirably altered. EX-1001, 2:31–37. For example, the '776Patent identifies D-limonene as the "principle molecular type" of orange oil,

describing D-limonene as “[t]he most abundant and readily perceived molecule in citrus flavors,” and further explaining that “[a] decrease of d-limonene concentration in the citrus flavor composition dramatically alters the citrus flavor in an undesirable manner.” EX-1001, 12:18–21, Fig. 11; 15:18–23; *see also* EX-1001, 2:18–30 (using “principle” and “principal” interchangeably: “[s]imilar results were found for all other principal constituents of other flavor oils”); EX-1001, 15:53–54 (describing “Gamma-terpinene” as “contribut[ing] to the top note of the flavor” in “lemon and lime oils”), 16:22–24 (describing terpineols as “a small constituent of citrus oils, which contribute to heavier, less fresh tastes”); EX-1002, 43 (characterizing the limitation as “a molecular type for obtaining a food, flavor, fragrance, medicament, and/or pigment”). Additionally, the claim recitation of “one or more”/“at least one of” confirms an active ingredient may have multiple principle molecular types and, thus, is not limited to a single most abundant/dominant constituent component of an active ingredient. EX-1001, claims 1[1d], 10[10c]. EX-1003, ¶97.

Consistent with these disclosures, Baranauskiene discloses that at least one of the eleven constituent components (in Table 2) of the *initial active ingredient* (peppermint EO) is a *principle molecular type from which the desirable flavor* (peppermint flavor) is obtained. EX-1003, ¶98. Baranauskiene identifies five peppermint EO constituent components — “[m]enthol (47.5%), menthone (19.5%),

iso-menthone + neo-menthol (1.1:1) (16.0%), menthyl acetate (4.3%), and pulegone (2.2%)”—as “the major compounds among 57 volatiles identified in pure peppermint EO used as the initial material for the encapsulation (Table 2).” EX-1005, [3031]. One or more of these five “major” constituent components are “flavor compounds” of the EO that contribute to its desirable flavor. EX-1005, [3027], [3031], [3033]; EX-1003, ¶98. The claim’s “one or more”/“at least one of” language confirms that the peppermint EO may have multiple principle molecular types and is not limited to the single most abundant/dominant constituent. EX-1001, 17:66–18:21, 18:47–65, 20:7–21; EX-1003, ¶¶98-99. Nevertheless, even if the constituent component’s abundance is relevant for purposes of this limitation, Baranauskiene confirms that one or more of these “major” compounds (e.g., menthone, menthol) are relatively more abundant in peppermint EO. EX-1005, [3031]. EX-1003, ¶98-99.

(d) [1c]

Baranauskiene discloses that *the final active ingredient* (the peppermint EO recovered from the spray-dried encapsulated powder) *includes one or more of the constituent components* (eleven components in Table 2 for each spray-dried product) *corresponding with those of the initial active ingredient* (same eleven components of the pure peppermint EO) *as modified by the drying of the slurry*.

EX-1003, ¶¶100-106.

Baranauskiene discloses *the final active ingredient* as the peppermint EO encapsulated in the spray-dried powder. EX-1003, ¶¶100-102. The peppermint EO that is the *initial active ingredient* is emulsified in aqueous modified-starch carrier solutions and then spray dried to obtain dry powders in which the peppermint EO is encapsulated within the starch matrix. EX-1005, [3031], Table 2 (reporting the composition of each emulsion and the resulting spray-dried powder). Baranauskiene confirms that this encapsulated EO is recovered from the finished powders. EX-1005, [3031] (“volatiles from liquid-emulsified and spray-dried encapsulated products were isolated by hydrodistillation and analyzed by GC and GC-MS.”). EX-1003, ¶101.

The peppermint EO recovered from the spray-dried powder *includes one or more of the constituent components corresponding with those of the initial active ingredient*. Baranauskiene identifies, in the recovered EO of the encapsulated powders, the same constituent compounds present in the initial pure peppermint EO—reporting their peak-area percentages. EX-1005, [3030]–[3031], Table 2. As shown below, the eleven constituents tabulated for the pure EO are present in every spray-dried encapsulated powder, including each of the initial EO’s major constituents: menthol, menthone, iso-menthone + neo-menthol, menthyl acetate, and

pulegone. *Id.* Each recovered EO constituent component thus corresponds to an initial pure EO constituent component. EX-1005, [3031], Table 2; EX-1003, ¶102.

Baranauskiene (EX-1005), Table 2

(annotated)

Boxes 1-8 = the eight commercial modified starch carriers

Table 2. Changes in the Total Oil Composition after Processing in Both Liquid-Emulsified and Spray-Dried Encapsulated Products as Compared with Pure Initial EO^a

no.	compound	KI on DB5	pure EO	1 HI-CAP 100		2 N-LOK		3 CAPSUL		4 ENCAPSUL 855		5 CRYSTAL TEX627		6 ClEmCap 12633		7 ClEmCap 12634		8 ClEmCap 12635	
				emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder
1	limonene ^{c,e}	1032	0.6 bc	0.5 a	0.5 abc	0.5 a	0.4 a	0.5 abc	0.5 abc	0.5 abc	0.4 a	0.5 abc	0.5 abc	0.5 a	0.6 bc	0.5 abc	0.6 bc	0.5 abc	0.6 c
2	menthone ^{c,e}	1166	19.5 cde	18.8 abc	19.0 bcde	19.0 bcde	18.8 abc	19.3 bcde	19.3 bcde	18.8 abc	19.3 bcde	18.8 abc	19.3 bcde	19.0 e	18.8 abc	19.3 bcde	19.3 bcde	19.5 cde	
3	iso-menthone ^{b,d}	1176	16.0 bc	15.8 abc	16.0 bc	15.8 abc	15.3 a	16.4 c	15.8 abc	16.1 bc	16.2 bc	15.8 abc	16.0 bc	16.1 bc	16.2 bc	15.8 abc	16.0 bc	15.3 a	
4	menthol ^{c,e}	1194	47.5 a	49.5 cde	49.1 bcde	49.3 bcde	50.0 de	48.5 bc	48.5 bc	48.3 ab	50.1 de	49.0 bcde	50.1 de	48.3 ab	49.0 bcde	49.3 bcde	49.0 bcde	49.5 cde	
5	iso-menthol ^{c,e}	1197	0.8 abc	0.9 bcde	0.9 abcde	0.9 abcde	0.7 a	1.0 cde	0.9 abcde	0.8 abc	0.9 bcde	0.8 abc	0.8 abc	0.9 bcde	0.8 abc	0.8 abc	0.8 abc	0.8 abc	
6	α-terpineol ^{c,e}	1201	0.9 c	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.1 a
7	cis-carveol ^d	1241	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.9 c
8	pulegone ^{c,e}	1249	2.2 e	1.9 a	2.1 d	2.1 bcd	2.1 d	2.1 cd	2.1 d	2.1 d	2.0 bc	2.1 d	2.1 d	2.1 cd	2.1 d	2.1 d	2.1 d	2.1 d	
9	piperitone ^d	1264	1.5 g	1.0 a	1.3 de	1.2 bc	1.3 cd	1.2 bc	1.4 f	1.2 b	1.3 cd	1.2 bc	1.3 cd	1.2 cd	1.2 bc	1.2 bc	1.2 bc	1.3 ef	
10	menthyl acetate ^{c,e}	1300	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.2 a	4.3 ab	4.4 bc	4.3 ab	4.4 bc	4.3 ab	4.4 c	4.3 ab	4.3 ab	4.3 ab	
11	β-caryophyllene ^{c,e}	1433	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.0 a	
	% RSD ^f		3.2	2.4	4.5	5.5	2.4	2.4	2.7	1.9	3.6	2.5	3.0	4.4	2.3	1.2	2.0	1.3	2.4

emulsion" column (pre-dried)

"powder" column (spray-dried, encapsulated)

"pure EO" column (initial oil)

Each carrier (boxes 1-8) has both an emulsion and a powder column; "pure EO" is the initial oil before processing.

11 constituent components of peppermint EO ('EO')

(same 11 components)

The recovered EO constituent components are present *as modified by the drying of the slurry* consistent with the '776Patent. There, the modifications described result from drying the slurry during the spray-drying itself (EX-1001, 2:31–37), not as a separate, quantified requirement that any particular modification be present in the final active ingredient. EX-1001, 1:30–46. EX-1003, ¶103.

Similarly, Baranauskiene's initial active ingredient is modified by drying the slurry because it is subjected to spray drying and recovered from the resulting

powder in encapsulated form. EX-1003, ¶104. Additionally, or alternatively, the initial active ingredient is modified by drying the slurry because the percentages of its individual constituents shift slightly from that drying. *Id.*

In particular, Baranauskiene confirms that its spray drying both encapsulates and slightly alters the EO. EX-1003, ¶¶105-106; EX-1005, [3031] (the “changes in the composition of complex aroma mixtures during encapsulation, i.e., emulsification and especially spray drying, are unavoidable;” the “compositions of pure, emulsified, and encapsulated in different starches matrices peppermint EO were quite similar; however, some changes in the percentages of some individual compounds were observed (Table 2).”). Table 2 documents these drying-induced changes constituent-by-constituent. *Id.*, Table 2. The composition of the EO recovered from the spray-dried powder is therefore the composition of the initial pure EO *as modified by the drying of the slurry*. EX-1003, ¶¶105-106.

(e) [1d]

Baranauskiene discloses *a weight percentage of at least one of the one or more principle molecular types* (menthone) *in the final active ingredient* (peppermint EO recovered from Baranauskiene’s spray-dried encapsulated powder) *is within about 5% of a weight percentage of the corresponding principle molecular types in the initial active ingredient* (the pure peppermint EO before

emulsification and spray drying). EX-1003, ¶¶107-122.

As to *weight percentage*, Baranauskiene measured *the principle molecular types* of the pure peppermint EO and the recovered, spray-dried EO using gas chromatography. Table 2 reports each constituent's results as a *peak-area percentage*. EX-1003, ¶108.

Baranauskiene (EX-1005), Table 2

(annotated)

Boxes 1-8 = the eight commercial modified starch carriers

Table 2. Changes in the Total Oil Composition after Processing in Both Liquid-Emulsified and Spray-Dried Encapsulated Products as Compared with Pure Initial EO^a

no.	compound	KI on DB5	1 HI-CAP 100		2 N-LOK		3 CAPSUL		4 ENCAPSUL 855		5 CRYSTAL TEX627		6 CIEmCap 12633		7 CIEmCap 12634		8 CIEmCap 12635		
			pure EO	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder
1	limonene ^{c,e}	1032	0.6 bc	0.5 a	0.5 abc	0.5 a	0.4 a	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc	0.5 abc
2	menthone ^{c,e}	1166	19.5 cde	18.8 abc	19.0 bcde	19.0 bcde	18.8 abc	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde	19.3 bcde
3	iso-menthone ^{b,d}	1176	16.0 bc	15.8 abc	16.0 bc	15.8 abc	15.3 a	16.4 c	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc	15.8 abc
4	menthol ^{c,e}	1194	47.5 a	49.5 cde	49.1 bcde	49.3 bcde	50.0 de	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc	48.5 bc
5	iso-menthol ^{c,e}	1197	0.8 abc	0.9 bcde	0.9 abcde	0.9 abcde	0.7 a	1.0 cde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde	0.9 abcde
6	α-terpineol ^{c,e}	1201	0.9 c	0.8 b	0.8 b	0.8 b	0.9 cd	0.8 b	0.9 cd	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b
7	cis-carveol ^d	1241	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a
8	pulegone ^{c,e}	1249	2.2 e	1.9 a	2.1 d	2.1 d	2.1 d	2.1 cd	2.1 d	2.1 cd	2.1 d	2.1 cd	2.1 d	2.1 cd	2.1 d	2.1 cd	2.1 d	2.1 cd	2.1 d
9	piperitone ^d	1264	1.5 g	1.0 a	1.3 de	1.2 bc	1.3 cd	1.2 bc	1.4 f	1.2 bc	1.2 bc	1.2 bc	1.2 bc	1.2 bc	1.2 bc	1.2 bc	1.2 bc	1.2 bc	1.2 bc
10	menthyl acetate ^{c,e}	1300	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab
11	β-caryophyllene ^{c,e}	1433	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a
	% RSD ^f		3.2	2.4	4.5	5.5	2.4	2.4	2.7										

emulsion = "emulsion" column (pre-dried)

powder = "powder" column (spray-dried, encapsulated)

pure EO = "pure EO" column (initial oil)

Each carrier (boxes 1-8) has both an emulsion and a powder column; "pure EO" is the initial oil before processing.

11 constituent components of peppermint EO ('EO')

(same 11 components)

In a gas chromatograph, each oil constituent produces a separate signal, or *peak*, and that peak's size reflects how much of the constituent is present. EX-1003, ¶¶109-111. Baranauskiene reports each constituent as its share of the combined area of all peaks. EX-1005, [3028] (constituents "expressed as a GC peak area percent");

[3031], Table 2. Table 2 reports these peak-area percentages for eleven identified constituents in the pure starting oil (*initial active ingredient*) and in the spray-dried encapsulated powder (*final active ingredient*) for each of eight carrier products. *Id.* EX-1003, ¶109.

Although element [1d] is written in terms of *weight* percentage, a constituent's peak-area percentage reliably tracks the change in its weight percentage for [1d]'s contemplated comparison. EX-1003, ¶¶110-111. Element [1d] compares the same molecular type in the initial and final active ingredients, and Baranauskiene analyzed that compound by the same GC-FID method, on the same instrument, in both the pure initial oil and the spray-dried product. EX-1005, [3028]; EX-1003, ¶¶110-121. Because the same compound is measured the same way in both samples, its detector response is common to the initial and final values and cancels when the two are compared—so the relationship between that compound's peak area and its actual weight is the same before and after drying. EX-1003, ¶¶110-111. Thus, as explained (and quantified) in Dr. Miller's declaration (EX-1003, ¶¶112-121 ((citing EX-1035))), a POSITA understood or found obvious the change in a constituent's peak-area percentage from the initial to the final oil tracks the change in its weight percentage, such that where a constituent's peak-area percentage is unchanged before and after drying, the change in its weight percentage

is correspondingly small. EX-1003, ¶110.

Applying that comparison, Baranauskiene discloses that *a weight percentage of at least one of the principle molecular types* (menthone) *in the final active ingredient is within about 5% of* the weight percentage of *the corresponding principle molecular type in the initial active ingredient*. The peppermint essential oil (“EO”) was emulsified into aqueous solutions of eight commercial starch carriers, and then each emulsion was spray dried. EX-1005, [3028]. Table 2 sets forth the compositions of the pure starting EO, of the EO recovered from each liquid emulsion, and of the EO recovered from each of the eight spray-dried encapsulated powders, specifically reporting the peak-area percentage of the eleven constituent components. For menthone in the CIEmCap 12635 spray-dried product, Table 2 reports the same peak-area percentage—19.5%—in both the pure EO and the spray-dried powder. EX-1005, [3031], Table 2. EX-1003, ¶111.

Baranauskiene (EX-1005), Table 2 (annotated)									
Table 2. Changes in the Total Oil Composition after Processing in Both Liquid-Emulsified and Spray-Dried Encapsulated Products as Compared with Pure Initial EO ^a									
no.	compound	KI on DB5	pure EO	HI-CAP 100		N-LOK		CAPSUL	
				emulsion	powder	emulsion	powder	emulsion	powder
1	limonene ^{c,e}	1032	0.6 bc	0.5 a	0.5 abc	0.5 a	0.4 a	0.5 abc	0.5 abc
2	menthone ^{c,e}	1166	19.5 cde	18.8 abc	19.0 bcde	19.0 bcde	18.8 abc	19.3 bcde	19.3 bcde
3	iso-menthone ^{b,d}	1176	16.0 bc	15.8 abc	16.0 bc	15.8 abc	15.3 a	16.4 c	15.8 abc
4	menthol ^{c,e}	1194	47.5 a	49.5 cde	49.1 bcde	49.3 bcde	50.0 de	48.5 bc	48.5 bc
5	iso-menthol ^{c,e}	1197	0.8 abc	0.9 bcde	0.9 abcde	0.9 abcde	0.7 a	1.0 cde	0.9 abcde
6	α-terpineol ^{c,e}	1201	0.9 c	0.8 b	0.8 b	0.8 b	0.9 cd	0.8 b	0.9 cd
7	cis-carveol ^d	1241	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a	0.8 a
8	pulegone ^{c,e}	1249	2.2 e	1.9 a	2.1 d	2.1 bcd	2.1 d	2.1 cd	2.1 d
9	piperitone ^d	1264	1.5 g	1.0 a	1.3 de	1.2 bc	1.3 cd	1.2 bc	1.4 f
10	menthyl acetate ^{c,e}	1300	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.3 ab	4.2 a
11	β-caryophyllene ^{c,e}	1433	1.3 ab	1.0 a	1.5 abcd	1.3 ab	1.0 a	1.0 a	1.5 abcd
	% RSD ^f		3.2	2.4	4.5	5.5	2.4	2.4	2.7

no.	ENCAPSUL 855		CRYSTAL TEX627		CIEmCap 12633		CIEmCap 12634		CIEmCap 12635	
	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder	emulsion	powder
1	0.5 abc	0.4 a	0.5 abc	0.5 abc	0.5 a	0.6 bc	0.5 abc	0.6 bc	0.5 abc	0.6 c
2	19.8 cde	18.5 ab	18.1 a	18.8 abc	18.5 ab	20.0 e	18.8 abc	19.3 bcde	19.0 bcde	19.5 cde
3	16.1 bc	15.8 abc	15.8 abc	16.2 bc	15.5 abc	16.1 bc	16.2 bc	15.8 abc	16.0 bc	15.3 a
4	48.3 ab	50.0 e	49.1 bcde	49.0 bcde	50.1 de	48.3 ab	49.0 bcde	49.3 bcde	49.0 bcde	49.5 cde
5	0.8abc	0.9 bcde	1.1 e	0.8 abc	0.8 ab	0.8 ab	0.9 bcde	0.80 abc	0.8 abc	0.8 abc
6	0.8 b	0.9 cd	0.8 b	0.9 cd	0.8 b	0.8 b	0.8 b	0.8 b	0.8 b	0.1 a
7	0.9 bc	0.8 abc	0.8 a	0.8 abc	0.8 a	0.9 bc	0.8 a	0.8 abc	0.8 a	0.9 c
8	2.1 d	2.0 bc	2.0 ab	2.1 d	2.0 bc	2.1 cd	2.1 cd	2.1 d	2.1 d	2.1 d
9	1.2 b	1.3 cd	1.2 bc	1.3 cd	1.2 cd	1.2 cd	1.2 bc	1.2 bc	1.2 bc	1.3 ef
10	4.3 abc	4.2 a	4.4 bc	4.2 a	4.3 ab	4.4 bc	4.3 abc	4.4 c	4.3 ab	4.3 ab
11	1.3 ab	1.0 a	2.0 d	1.0 a	1.8 bcd	1.3 ab	1.5 abcd	1.0 a	1.5 abcd	1.0 a
	1.9	3.6	2.5	3.0	4.4	2.3	1.2	2.0	1.3	2.4

■ = no. 2 menthone
■ = "pure EO" (initial oil)
■ = "powder" (spray-dried, encapsulated)
□ = starch carrier

Because menthone's peak-area percentage is unchanged, its weight percentage is likewise essentially unchanged for the reasons noted earlier in this limitation's analysis. EX-1003, ¶¶112-121. Thus, the change in menthone's weight percentage from the initial to final oil is well under 1%, below the claimed "about 5%" tolerance. EX-1003, ¶¶107-122.

3. Claim 2

Baranauskiene discloses or renders obvious that *the one or more principle*

molecular types (at least menthol, menthone, iso-menthone + neo-menthol, menthyl acetate, and pulegone of Baranauskiene’s peppermint EO, from which the flavor is obtained)) are molecular types *of a food composition* (used for flavoring food compositions). EX-1003, ¶¶123-124.

Baranauskiene teaches that “the encapsulation of flavor ingredients is among the most important applications in the food industry” (EX-1005, [3027]) and “[t]he controlled release of ingredients ...can improve the effectiveness of food additives...”). The ’776Patent likewise contemplates that the same molecular type can be of a food or flavor composition, depending on end-use. EX-1001, 12:19–30, 15:18–24, 18:13–18, FIG. 11 (treating D-limonene of orange oil as simultaneously a flavor and a food principle molecular type). A POSITA thus understood at least one of the peppermint EO’s constituents (*one or more principle molecular types*)—being a constituent from which peppermint flavor is obtained and which Baranauskiene teaches is encapsulated for flavoring food—is a *molecular type of a food composition*. EX-1003, ¶¶123-124.

4. Claim 3

Baranauskiene discloses or renders obvious that *the one or more principle molecular types* (at least menthol, menthone, iso-menthone + neo-menthol, menthyl acetate, and pulegone of Baranauskiene’s peppermint EO, from which the flavor is

obtained) are molecular types *of a flavor composition* (used for flavoring food compositions). EX-1003, ¶¶125-126.

Baranauskiene seeks to test EO's flavor retention. EX-1005, [3027] (Abstract: "The effect of different commercial modified food starch carrier materials on the flavor retention of the essential oil (EO) of peppermint ... during spray drying and storage was evaluated."); [3027]; [3031]. A POSITA thus understood at least one of the peppermint EO's constituents (*one or more principle molecular types*)—being a constituent from which peppermint flavor is obtained, and which Baranauskiene spray-dries and evaluates for flavor retention as a flavoring—is a *molecular type of a flavor composition*. EX-1003, ¶¶125-126.

5. Claim 4

Baranauskiene discloses or renders obvious that *the one or more principle molecular types* (at least menthol, menthone, iso-menthone + neo-menthol, menthyl acetate, and pulegone of Baranauskiene's peppermint EO, from which fragrance is obtained) are molecular types *of a fragrance composition* (used for perfuming a product). EX-1003, ¶¶127-128.

Baranauskiene describes the encapsulated EO in terms of its *aroma* properties—*i.e.*, its fragrance. EX-1005, [3027] (Abstract: "aroma binding capacity of different modified starch matrices to lock EO droplets"); [3031] ("The changes in

the composition of complex aroma mixtures during encapsulation, *i.e.*, emulsification and especially spray drying, are unavoidable.”). A POSITA thus understood at least one of the peppermint EO’s constituents (*one or more principle molecular types*)—being a constituent from which the EO’s aroma, *i.e.*, its fragrance, is obtained—is itself a *molecular type of a fragrance composition*. EX-1003, ¶¶127-128.

6. Claim 5

Baranauskiene discloses or renders obvious that *the one or more principle molecular types* (at least menthol, menthone, iso-menthone + neo-menthol, menthyl acetate, and pulegone of Baranauskiene’s peppermint EO, from which therapeutic (pharmacological) activity is obtained) are molecular types *of a medicament composition* (used as/in a medicament). EX-1003, ¶¶129-130.

As of the priority date, peppermint EO (the active ingredient) and some of its major components—menthone and menthol—were well-known as pharmacologically active and having therapeutic uses. EX-1003, ¶130 (citing EX-1016; EX-1017; EX-1020). A POSITA therefore understood peppermint EO is a medicament composition and one or more of the peppermint EO principle molecular types are *molecular types of a medicament composition*. EX-1003, ¶130; EX-1005, [3031], Table 2. EX-1003, ¶¶129-130.

7. Claim 7

Baranauskiene discloses claim 7 for the reasons in §IV.A.2 ([1d]). EX-1003, ¶¶131-132. Menthone—one of peppermint EO principle molecular types—is present at the same weight percentage in the spray-dried EO (*final active ingredient*) as in the pure EO (*initial active ingredient*), reflecting a change of well under 1%. EX-1003, ¶¶131-132.

8. Claim 8

Baranauskiene discloses claim 8 for the reasons in §IV.A.2 ([1d]). EX-1003, ¶¶133-134. Menthone—one of peppermint EO principle molecular types—is present at the same weight percentage in the spray-dried EO (*final active ingredient*) as in the pure EO (*initial active ingredient*), thus reflecting a change of well under 1%. EX-1003, ¶¶133-134.

9. Claim 9

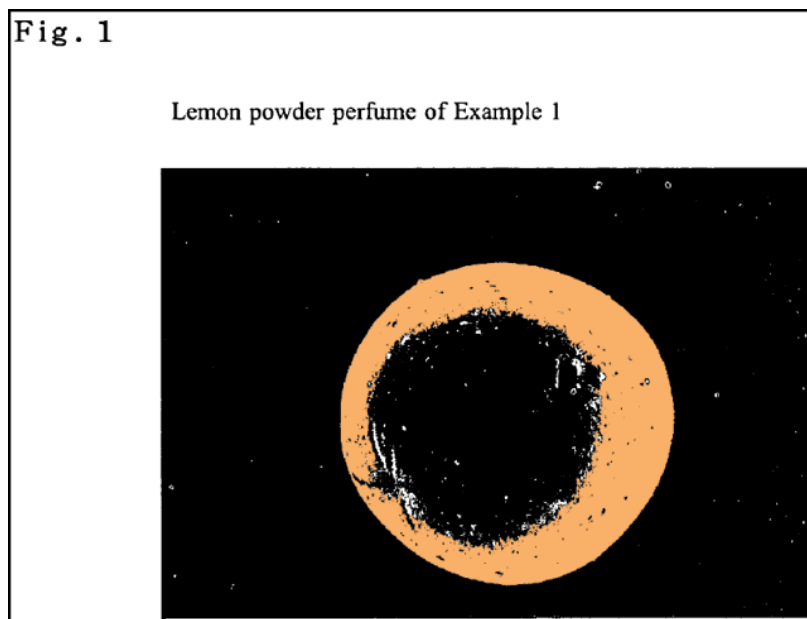
Baranauskiene discloses claim 9 for the reasons in §IV.A.2 ([1d]). EX-1003, ¶¶135-136. Menthone—one of peppermint EO principle molecular types—is present at the same weight percentage in the spray-dried EO (*final active ingredient*) as in the pure EO (*initial active ingredient*), thus reflecting a change of well under 1%. EX-1003, ¶¶135-136.

B. GROUND 2: Watanabe-JECFA References

1. Watanabe

Watanabe relates to edible powder compositions comprising at least one component selected from perfumes, coloring agents, and functional substances (*e.g.*, vitamins), in combination with trehalose and water-soluble hemicellulose, prepared by spray drying an aqueous emulsion of those components. EX-1006, Abstract; [0001], [0006], [0008]–[0009], [0019]. Watanabe teaches that the resulting spray-dried powders “exhibit excellent shelf stability of the active component contained therein” and can be incorporated into drinks, foods, cosmetics, and the like to “impart a desired fragrance, flavor, color and/or function stably thereto for a long period of time.” EX-1006, [0001], [0006]; EX-1003, ¶73.

The spray-dried particles take the form of “dense spherules having a stiff wall surface” and this morphology produces excellent stability of the active component contained therein. EX-1006, [0044], FIG. 1. Watanabe’s FIG. 1 (below) shows a scanning electron micrograph of a particle of Example 1’s spray-dried lemon powder perfume, highlighting this lemon powder perfume’s dense-spherule, stiff-walled morphology. EX-1006, Fig. 1. The orange annotation highlights the continuous, intact carrier wall enclosing/encapsulating the active ingredient in the particle’s interior. EX-1006, [0043]–[0044], FIG. 1; EX-1003, ¶74.



EX-1006, FIG. 1 (color added).

Per Watanabe, the emulsion-and-spray-drying process used to prepare the lemon powder perfume, is used to prepare Example 5's paprika-containing powder. EX-1006, [0023], [0053]. In Example 5, water-soluble hemicellulose and trehalose were dissolved in water, paprika oil was emulsified into the solution, and the emulsion was spray-dried to yield a paprika-containing powder ("inventive product 5"). EX-1006, [0053]; EX-1003, ¶75.

After storage at 50°C for 4 weeks (672 hours) in a low-density polyethylene bag in the dark, the paprika-containing powder retained 97.5% of its "paprika pigment," as measured by spectrophotometry against the content of paprika pigment immediately after preparation. EX-1006, [0055]–[0058], Table 3 (below). EX-1003, ¶76.

TABLE 3	
<u>Retention of Paprika Pigment</u>	
After storage at 50° C. for 4 weeks	
Example 5	97.5%
Comparative Example 5	32.8%

2. JECFA References

JECFA References are two companion documents on paprika extract issued by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) in connection with its 69th meeting (2008): the JECFA specification monograph for paprika extract (“JECFA Monograph,” EX-1033), and the paprika-extract Chemical and Technical Assessment prepared as background documentation for that meeting (“JECFA CTA,” EX-1034). EX-1033⁶, 51; EX-1034. Together, they describe paprika color’s composition and measurement. EX-1003, ¶77.

JECFA Monograph discloses that paprika extract derives its color from a mixture of carotenoids in which capsanthin and capsorubin are the principal red compounds (not less than 30% of total carotenoids). EX-1033, 51–52. JECFA Monograph also discloses that this color is quantified as a single bulk total-carotenoid value by visible spectrophotometry at about 462 nm, expressed in

⁶ EX-1033 citations reference page number at the top of the page.

capsanthin (capsanthin/capsorubin) equivalents. EX-1033, 52, 54–55; EX-1034, 3–4. The JECFA CTA describes corresponding compositional detail, reporting a representative carotenoid profile and confirming that capsanthin is the predominant carotenoid, contributing up to 60% of total carotenoids. EX-1034, 3–4. JECFA References thus establish that “paprika pigment” is a mixture of carotenoid components, of which capsanthin is the dominant component, and that paprika pigment is measured in bulk as capsanthin equivalents. EX-1003, ¶77.

3. Watanabe-JECFA References Combination

A POSITA would have found it obvious to combine the teachings of Watanabe and the JECFA References. EX-1003, ¶¶138-142.

For context, three terms are used herein that are related: Watanabe’s “paprika oil” (Example 5’s active ingredient) is a commercial, oil-form “paprika extract” of the type JECFA characterizes, and “paprika pigment” is that ingredient’s carotenoid color-bearing fraction—principally capsanthin and capsorubin, of which capsanthin is predominant—which Watanabe measures spectrophotometrically and reports as retained. EX-1006, [0053], [0055]–[0056]; EX-1033, 51; EX-1034, 3–4; EX-1003, ¶137.

Watanabe prepares and measures a paprika-containing spray-dried powder and reports its stability in terms of retained “paprika pigment” measured

spectrophotometrically (describing the pigment only in the aggregate). EX-1006, [0053], [0055]–[0056]. While Watanabe does not expressly enumerate the pigment’s constituents, a POSITA working with paprika-colored material would have consulted recognized compositional and analytical standards for paprika extract. EX-1003, ¶138. A POSITA would have thus sought out an authoritative statement of paprika-extract composition and assay—such as the JECFA References—to identify the carotenoids that make up paprika color, their representative proportions, and the spectrophotometric, capsanthin-equivalents basis on which paprika color is quantified. EX-1033, 51–52, 54–56; EX-1034, 3–4; EX-1003, ¶138.

A POSITA would have been motivated to seek out the composition behind a bulk paprika-color figure: paprika color was a commodity colorant that the art specified and stability-assessed by carotenoid content, and resolving it into its individual carotenoids was routine and conventional. EX-1003, ¶¶138-139. A POSITA given Watanabe’s aggregate “paprika pigment” measurement would thus have consulted the recognized compositional and analytical authority for paprika extract—the JECFA References—to learn what that pigment is and how its measured value behaves. EX-1033, 51–52, 54–56; EX-1034, 3–4; EX-1018; EX-1019; EX-1021; EX-1003, ¶139.

A POSITA would have had a reasonable expectation of success in combining

the teachings of Watanabe and JECFA References. First, Watanabe discloses “paprika pigment” and the JECFA References disclose that pigment’s specific components and their measurements, providing a component level measurement breakdown relative to the overall measurement of that ingredient in Watanabe. EX-1003, ¶140. Second, the combination calls for no modification of Watanabe; the JECFA References confirm the composition of the paprika pigment Watanabe already discloses and measures. EX-1006, [0053], [0055]–[0056]; EX-1033, 51–52; EX-1034, 3–4; EX-1003, ¶140.

In summary, Watanabe provides a spray-dried, stored paprika-pigment powder and a spectrophotometric figure showing that pigment was retained after storage, and the JECFA References disclose the ordinary knowledge of what that pigment is—a carotenoid mixture dominated by capsanthin—and of the capsanthin-equivalents basis on which the figure is measured. EX-1003, ¶142. In combination, a POSITA would have found obvious Watanabe’s retained “paprika pigment” is a capsanthin-dominated carotenoid pool measured by the standard assay, such that Watanabe’s retention figure reflects the behavior of capsanthin as its principal constituent. EX-1003, ¶¶141-142.

4. Claim 10

(a) [10pre]

If the preamble is limiting, Watanabe-JECFA References render obvious *a*

spray-dried powder (inventive product 5's paprika-containing powder). Watanabe provides a powder composition prepared by spray drying an aqueous emulsion containing trehalose, water-soluble hemicellulose, and at least one component selected from perfumes, coloring agents, and functional substances. EX-1006, [0008]–[0009], [0019], [0053]; EX-1003, ¶¶143-144.

(b) [10a]

Watanabe-JECFA References render obvious *a plurality of dried particles* (paprika containing powder's spray-dried spherules), *which individually contain an amount of active ingredient* (paprika oil) *encapsulated within a carrier* (trehalose/water-soluble hemicellulose matrix). EX-1003, ¶¶145-152.

Watanabe's spray-dried powder composition comprises trehalose, water-soluble hemicellulose, and at least one component selected from perfumes, coloring agents, and functional substances. EX-1006, [0008]–[0009], [0017]; EX-1003, ¶146.

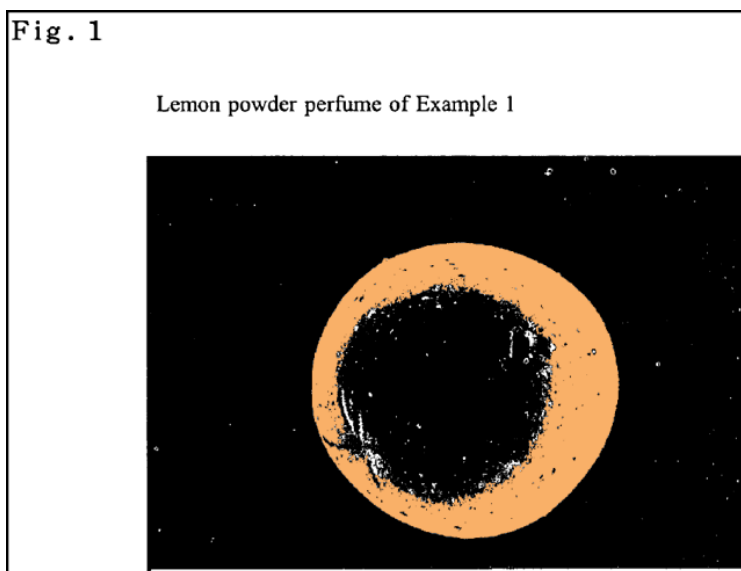
In Example 5, paprika oil is the perfume/coloring agent component, and trehalose and water-soluble hemicellulose together form the *carrier* into which the paprika oil is incorporated (*encapsulated*) by emulsification and subsequently spray dried. EX-1006, [0053]; EX-1003, ¶147.

Paprika oil is the *active ingredient*. The '776Patent describes an active ingredient as “any desirable single constituent component or a combination of

constituent components, at least one of which is among one or more principle molecular types from which at least one of a desirable food, flavor, fragrance, medicament, pigment ...is obtained.” EX-1001, 6:10–16. The patent thus contemplates a pigment-imparting component as an active ingredient, and dependent claim 15 confirms that an active ingredient’s principle molecular types may be “of a pigment composition.” EX-1001, 19:4–6. Consistently, Watanabe’s paprika oil is an *active ingredient* because it is a coloring agent that imparts a desirable color—paprika pigment—to an end product, and it has several constituent components (carotenoid pigments described in [10b], below) from which that desirable pigment is obtained. EX-1006, [0011], [0053]; EX-1003, ¶148. The trehalose/water-soluble hemicellulose matrix is the *carrier*. EX-1006, [0008]-[0009], [0053]; EX-1003, ¶148.

Additionally, spray drying the emulsion yields a paprika powder composed of discrete particles (*a plurality of dried particles*). EX-1006, [0009], [0019], [0044]. Scanning electron micrographs at 2000× magnification show that the inventive product particles (Example 1, FIG. 1) form “dense spherules having a stiff wall surface,” and Watanabe teaches that this dense-spherule, stiff-walled morphology produces the contained active component’s excellent stability. EX-1006, [0044], FIG. 1. The same emulsion-and-spray-drying process used in Example 1

(trehalose/water-soluble hemicellulose carrier, Mobile Minor spray dryer, inlet 150–160°C, outlet 80°C) is used in Example 5 to produce the paprika-containing powder. EX-1006, [0023], [0053]; EX-1003, ¶149.



A POSITA understood Example 5's paprika-containing powder has the same dense-spherule, stiff-walled morphology as Example 1 in FIG. 1, with paprika oil held within each spherule's carrier shell. That is because Watanabe prepares Example 1's lemon-perfume powder and Example 5's paprika-containing powder by the same emulsion-and-spray-drying process, using the same trehalose/water-soluble hemicellulose carrier composition, the same Mobile Minor type spray dryer, and the same outlet temperature of 80°C. EX-1006, [0023], [0053]. Further, Watanabe attributes the dense-spherule, stiff-walled morphology in FIG. 1 to the carrier composition and spray-drying process—not to the active ingredient—and

identifies that morphology as the structural basis for the powder's excellent shelf stability of the active component contained therein. EX-1006, [0017], [0044]. Finally, Watanabe's Reference Example 3 reports that Example 5's paprika-containing powder exhibits the same shelf stability of its active component that Watanabe attributes to Example 1's dense-spherule, stiff-walled morphology. EX-1006, [0044], [0055]–[0058], Table 3. EX-1003, ¶150.

Moreover, Watanabe's emulsion-and-spray-drying process is similar to the '776 Patent's encapsulation mechanism. *See* EX-1001, 1:45-57 (describing Watanabe's spray drying process, including the slurry being fed into a drying chamber... into which heated air is also introduced"; "the heated air suppl[ying] energy for evaporation of volatile components of the liquid (the water) from the droplets"—such that carrier "forms a hardened shell around the load, producing a dried powder"). Watanabe's Example 5 process performs a similar encapsulation mechanism: the carrier components form a hardened shell around the paprika oil load as the water evaporates, producing a dried powder of encapsulated particles. EX-1006, [0053]. EX-1003, ¶151.

In summary, each *dried particle* of the Example 5 powder *contain[s] an amount of active ingredient* (paprika oil) *encapsulated within a carrier* (trehalose/water-soluble hemicellulose shell of the dense spherule). EX-1006,

[0044], [0053], FIG. 1; EX-1003, ¶152.

(c) [10b]

Watanabe-JECFA References render obvious that *the active ingredient* (paprika oil) *includes one or more constituent components at least one of which is among one or more principle molecular types* (capsanthin, paprika oil’s principal carotenoid constituent) *from which at least one of a desirable food, flavor, fragrance, medicament, and pigment is obtained* (paprika oil’s desirable red pigment, obtained from capsanthin). EX-1003, ¶¶153-158.

Paprika oil is Example 5’s *active ingredient*. At least one desirable property that paprika oil contributes is its color, and Watanabe identifies that color property as the powder’s “paprika pigment”—the attribute whose retention is measured in Watanabe’s Reference Example 3 to evaluate the powder’s storage stability. EX-1006, [0053], [0055]–[0056], Table 3. EX-1003, ¶154.

Although Watanabe does not itself enumerate the chemical constituents of its “paprika pigment,” it was well known and obvious to a POSITA that “paprika pigment” is a mixture of carotenoid constituents that together give paprika oil its characteristic red color — that is, the constituent components of the active ingredient from which its pigment property is obtained. As explained in §IV.B.3 above, a POSITA would have found obvious to combine the teachings of Watanabe and the

JECFA References to determine the carotenoid composition of Watanabe's paprika pigment. EX-1033, 51–52; EX-1034, 3–4; EX-1003, ¶155.

In combination, the JECFA References provide that the paprika extract derives its color from a mixture of carotenoids in which capsanthin and capsorubin are the principal red, *Capsicum*-specific compounds (together not less than 30% of total carotenoids), and that capsanthin is the single predominant carotenoid, contributing up to 60% of total carotenoids and the largest single share of paprika's red color. EX-1033, 51–52; EX-1034, 3–4; EX-1003, ¶156.

Among those carotenoid constituents, capsanthin is *the principle molecular type from which* paprika oil's desirable *pigment is obtained*: it is the *Capsicum*-specific red ketocarotenoid that is the single most abundant individual carotenoid in ripe red paprika and the largest single contributor to paprika's characteristic red color. EX-1033, 51–52; EX-1034, 3–4. Capsanthin is therefore a *principle molecular type* within the meaning of [10b] at least because it is a principal, characteristic, and defining molecular constituent of paprika oil (active ingredient), and the molecule from which the desirable pigment (red color) of paprika oil is obtained. EX-1006, [0053], [0055]–[0056]. Watanabe confirms this in view of its teaching that its powder compositions are “incorporated into various drinks, foods, cosmetics and the like” to “impart a desired...color...stably thereto for a long period

of time”—i.e., the capsanthin-derived red pigment is a desirable property of the active ingredient that the powder is designed to deliver, including in *food* products. EX-1006, [0001], [0006]. The JECFA References further corroborate that color is paprika extract’s defining, intended property: the JECFA Monograph lists paprika extract’s functional use as “Colour” (INS No. 160c), and the JECFA CTA describes its extensive use in the food industry as a natural colouring agent. EX-1033, 51–52; EX-1034, 6. EX-1003, ¶¶157-158.

(d) [10c]

Watanabe-JECFA References render obvious *a weight percentage of at least one of the one or more principle molecular types* (capsanthin) *in the active ingredient* (paprika oil) *does not vary by more than about 5% during aging of the dried powder* (2.5% variation; 97.5% retention) *during any period of elevated temperature of about 95°F up to about 1000 hours* (672 hours at 122°F (50°C), an elevated temperature in excess of about 95°F). EX-1003, ¶¶159-188.

Capsanthin is a *principle molecular type* of paprika oil (*active ingredient* of Example 5’s spray-dried powder) and the molecule from which the powder’s desirable red pigment is obtained. §IV.B.4 ([10b]); EX-1006, [0053]. Watanabe’s Reference Example 3 measured that powder’s stability: Watanabe stored the Example 5 powder in a low-density polyethylene bag, in the dark, at 122°F (50°C)

for four weeks (672 hours), and measured its residual “paprika pigment” by spectrophotometry against the powder’s own pigment content measured immediately after preparation, which Watanabe sets at 100%. EX-1006, [0055]–[0058], Table 3. On that basis, Table 3 reports 97.5% retention—a 2.5% variation in paprika pigment over 672 hours at 122°F (50°C). EX-1006, Table 3. EX-1003, ¶160.

A POSITA understood the weight percentage of capsanthin—one of *the principle molecular types* in the active ingredient—*does not vary by more than about 5% during aging of the dried powder*. EX-1003, ¶161. Paprika pigment is a mixture of several carotenoid constituent components, which fall into two color groups: a red group (chiefly capsanthin, together with capsorubin, the Capsicum-specific carotenoids that give paprika its characteristic red color); and a yellow group (such as β -carotene and zeaxanthin). EX-1033, 51, 56; EX-1034, 3–4. Capsanthin is the most abundant carotenoid and the largest contributor to paprika oil’s red color. EX-1034, 3–4. EX-1003, ¶161.

With this context, Watanabe’s spectrophotometric assay does not measure any one of these carotenoids separately; it measures the light absorbed by all of them together and reports that combined signal as a single paprika-pigment number. EX-1006, [0055]–[0056]. Such a measurement is conventionally expressed in capsanthin equivalents. EX-1033, 51–52, 54–56; EX-1003, ¶162. In other words, the total color

is stated as though it came from capsanthin, the largest contributor and the standard reference point for reporting total paprika color. EX-1003, ¶162.

Because Watanabe's reported figure is a combined signal, the measured 97.5% retention is a weighted average of how well the individual carotenoids were retained—i.e., each carotenoid counts toward the 97.5% in proportion to its share of the original color. Two consequences follow from this. First, if the weighted average is 97.5%, a POSITA understood at least one carotenoid would be retained at 97.5% or more, because an average cannot be 97.5% if every contributor to the average falls below 97.5%. Second, because the red group (including capsanthin) makes up roughly half of the total color signal (*see* EX-1022; EX-1018), that group must have been retained at approximately 95% or more (since the whole sample lost only 2.5%, even assigning that entire loss to the red group alone amounts to just a 5% loss within a group that is half the signal). EX-1003, ¶163.

Capsanthin's own variation, in turn, would not have exceeded the measured 2.5% loss. EX-1003, ¶¶164-170. The red carotenoids, including capsanthin, degrade more slowly than the yellow carotenoids under these dry, dark storage conditions at temperatures below 60°C; the faster-degrading yellow carotenoids therefore would account for more than their proportional share of the 2.5% loss, leaving capsanthin's own loss at or below 2.5%. *Id.* (citing EX-1018; EX-1019; EX-1021 EX-1022; EX-

1023; EX-1024; EX-1025; EX-1026; EX-1029; EX-1033). Nevertheless, even setting aside the relative stability of the red and yellow carotenoids, the arithmetic establishes that the red group—of which capsanthin is the dominant member—was retained at approximately 95% or more. *Id.* That is because assigning the entire 2.5% total loss to a red group comprising roughly half the signal yields only about a 5% loss within that group. *Id.* Capsanthin’s variation thus falls within the ***about 5%*** ceiling of [10c] under either analysis: at or below about 2.5% accounting for the slower red carotenoid degradation, and no more than about 5% on the arithmetic alone. *Id.*

Watanabe further discloses or at least renders obvious that this stability occurred ***during aging of the dried powder during an elevated temperature of about 95°F***. Watanabe aged the dried Example 5 powder at 122°F (50°C)—an elevated temperature that meets and substantially exceeds the ***about 95°F*** recited in the claim. EX-1006, [0055]–[0058], Table 3. Although that temperature is hotter than the “about 95°F” recited in [10c], it would have been obvious to a POSITA that a powder exhibiting only about 2.5% capsanthin variation at the harsher 122°F would exhibit no greater variation at the milder claimed 95°F. That is because the rate of carotenoid degradation increases with temperature and falls as temperature falls. EX-1007 (Singh) at 14–15, 19, Fig. 1.9; EX-1012 (Bechoff), at 354; EX-1003, ¶¶171-172.

Thus, a powder losing no more than about 2.5% of its capsanthin over 672 hours at 122°F cannot lose more than that amount over the same period at the cooler claimed 95°F; the lower temperature can slow the degradation—not accelerate it. *Id.* A POSITA would thus recognize that the claimed condition is gentler than the condition Watanabe tested. *Id.* And because the Example 5 powder a POSITA would assess at 95°F is the same powder Watanabe tested at 122°F, no modification of the powder is required—only the storage temperature differs. EX-1003, ¶¶171-172.

Indeed, elevated-temperature stability testing of the kind Watanabe performed is the established, routine technique formulation scientists used as of the priority date to bound an ingredient's stability at the lower temperatures of ordinary storage. EX-1007, 14–19; EX-1013, Ch. 16. So a stability test executed at a much higher temperature of 50°C would conservatively bound the loss the same powder will experience at lower temperatures. *Id.* Thus, in view of Watanabe, a POSITA would have been motivated by the recognized purpose of accelerated testing, to recognize that result as establishing the powder's stability at the gentler about 95°F condition. EX-1003, ¶172. Moreover, because degradation rate decreases as temperature decreases for the same tested powder, a POSITA would have had a reasonable expectation of success that capsanthin's variation at about 95°F would be no greater than the about 2.5% Watanabe measured at 122°F. *Id.* This would have amounted to

applying a known technique (accelerated stability testing) to a known powder (Example 5) to obtain the expected outcome (at least as much retention at the lower temperature). *Id.* This assessment is confirmed by the three analyses presented below, each of which independently projects capsanthin's variation at 95°F well within the about 5% ceiling recited in the claim. *Id.* Accordingly, Watanabe discloses or at least renders obvious that the stability occurs or would have occurred **during aging of the dried powder during an elevated temperature of about 95°F.** EX-1003, ¶172.

Finally, Watanabe-JECFA References renders obvious that the weight percentage of capsanthin would not vary by more than about 5% **during any period** of elevated temperature of about 95°F **up to about 1000 hours.** Watanabe measured capsanthin retention over 672 hours at 122°F (50°C), finding variation of no more than about 2.5%. EX-1006, [0055]–[0058], Table 3. While Watanabe did not test for the about 1,000 hours that [10c] recites, it would have been obvious to a POSITA, applying the routine predictive tools of the formulation art to Watanabe's data, that the same powder's capsanthin variation remains within about 5% out to about 1,000 hours. EX-1003, ¶173.

A POSITA developing or evaluating a spray-dried flavor or pigment powder would have been motivated to confirm that the encapsulated active remains stable

across a commercially meaningful warm-storage shelf life on the order of about 1,000 hours (roughly six weeks). EX-1006, [0011]; EX-1013, Ch. 3; EX-1003, ¶¶173-174. Projecting longer-term storage stability from a shorter elevated-temperature test is the recognized purpose of accelerated stability testing—the standard method a formulator uses to predict shelf life. EX-1007, 14–19; EX-1013, Ch. 3; EX-1003, ¶¶173-174. A POSITA presented with Watanabe’s 672-hour, 50°C result would thus, in the ordinary course, have been motivated to project it to the storage durations of practical interest, including about 1,000 hours. *Id.*

A POSITA would have had a reasonable expectation in that projection and expected the result of that projection to fall within the claimed about 5% ceiling. EX-1003, ¶175. Capsanthin degrades in spray-dried, dark-stored matrices by first-order kinetics, losing a roughly constant fraction of whatever remains per unit time. EX-1027 (Topuz 2008), 1672, 1674; EX-1012 (Bechoff), at 354; EX-1014 (Palma). Because the loss rate does not accelerate over the relevant window, extending Watanabe’s measured 672-hour loss to 1,000 hours conservatively bounds the variation a POSITA would expect, rather than understating it. EX-1003, ¶175. Extending a known first-order rate from 672 to 1,000 hours is the predictable application of a known kinetic model to known data: the claimed ~1,000-hour durability is what a POSITA would have predicted for this powder using ordinary

tools. *Id.*

A POSITA would have confirmed that expectation quantitatively, and three independent analyses each place capsanthin's variation over 1,000 hours at 95°F well within the about 5% ceiling. EX-1003, ¶¶176, 182-185.

First, the simplest of the three analyses takes the loss rate Watanabe actually measured and projects it forward to the full 1,000-hour period, without accounting for the fact that the claim's 95°F condition is cooler (and therefore a gentler test) than Watanabe's 122°F (50°C) test temperature. Even so, the projected variation is only about 3.7%, within the ***about 5%*** requirement recited in [10c]. EX-1003, ¶¶177, 182-185.

Second, applying the standard Arrhenius temperature correction—the accepted method for translating a degradation rate measured at one temperature to the expected rate at another—and accounting for the cooler 95°F claim condition, the projected variation is only about 1.1%. A POSITA understood or found obvious to apply the Arrhenius correction here to quantify the degradation of Watanabe's test, which ran at the hotter 122°F (50°C), to the milder 95°F temperature recited in the claim. EX-1003, ¶¶178, 182-185.

To run the Arrhenius evaluation, a POSITA would have sought the activation energy of the principle molecular type (capsanthin), which was reported in Palma—

a study of the same molecule in spray-dried, dark-stored, microencapsulated paprika oleoresin, tested across 104–158°F (40–70°C, bracketing Watanabe’s 122°F) under confirmed first-order kinetics. EX-1014. Applying that value, the cooler 95°F claim temperature slows capsanthin’s degradation enough that its projected variation over the full 1,000-hour period is only about 1.1%, comfortably within the ***about 5%*** claimed loss threshold. EX-1003, ¶¶179, 182-185.

Although Palma postdates the priority date, its reported activation energy is a fixed physicochemical property (activation energy) of the capsanthin molecule that was present in Example 5 powder when it was made. EX-1003, ¶180. Palma is therefore evidence of an inherent property of that powder. *Id.* Nevertheless, this Arrhenius result is independently corroborated by pre-priority-date literature, including Bechoff. EX-1012 (Bechoff), 354. Applying Bechoff’s activation energy to capsanthin by structural analogy yields essentially the same projected variation—about 1.1%—confirming that the conclusion holds even applying Bechoff’s pre-priority date activation energy. EX-1003, ¶¶180, 182-185.

Third, even using the lowest temperature dependence in the food-stability literature, capsanthin’s projected variation stays within the ceiling. Q_{10} — the factor by which a degradation rate changes per 18°F (10°C) of temperature—provides a simplified temperature correction; a larger Q_{10} gives more credit for the lower claim

temperature. Applying a conservative Q_{10} of about 1.5, capsanthin's projected variation at 95°F over 1,000 hours is no more than about 2.0%. EX-1013 (Labuza), Ch. 16 & Tbl. 16.1; EX-1003, ¶¶181-185.

In summary, all three analyses are within the claimed 5% range—about 3.7% on the most conservative assumptions, about 2.0% at a conservative Q_{10} floor, and about 1.1% at capsanthin's measured activation energy. And, all three analyses leverage well known and conventional techniques (e.g., first-order kinetics, the Arrhenius relationship, and Q_{10} scaling) applied to Watanabe's own measured data together with parameters drawn from the literature. EX-1007; EX-1027; EX-1012; EX-1013; EX-1003, ¶186.

Alternatively, if “*during any period ... up to* about 1000 hours” sets the *maximum* length of the aging period rather than a minimum, Watanabe discloses the limitation. Reference Example 3 aged the Example 5 powder for 672 hours—a period “up to about 1000 hours” (i.e., about 1,000 hours or shorter)—with capsanthin varying by no more than about 2.5% across that entire period. That is well within the claimed ***about 5%*** ceiling. EX-1006, [0055]–[0058], Table 3. Watanabe thus discloses a ***principle molecular type*** whose weight percentage stays within about 5% of its starting value across an aging period of about 1,000 hours or less at the requisite elevated temperature. EX-1003, ¶187.

5. Claim 11

Watanabe-JECFA References render obvious that *the one or more principle molecular types are molecular types of a food composition* (capsanthin, the principle molecular type identified above for [10b] from which the paprika red color is obtained). EX-1003, ¶¶189-190.

Watanabe teaches that its powder compositions—including the Example 5 paprika-containing powder—are designed for incorporation into “various drinks, foods, cosmetics and the like.” EX-1006, [0001], [0006], [0053]. Capsanthin, the principle molecular type identified at [10b], is so incorporated into food and is itself a permitted food colorant in commercial paprika oleoresin used as a food coloring. Capsanthin is therefore a molecular type of *a food composition*. EX-1003, ¶190.

6. Claim 15

Watanabe-JECFA References render obvious that *the one or more principle molecular types are molecular types of a pigment composition* (capsanthin, the principle molecular type identified above for [10b]). EX-1003, ¶¶191-192.

Watanabe identifies “coloring agents” as a category of active component its powder compositions are designed to carry. EX-1006, [0001], [0006], [0008]–[0009]. Capsanthin is itself the principal red pigment of paprika oil — the molecule from which paprika’s characteristic red color is obtained—and is the principal constituent measured by Watanabe’s spectrophotometric “paprika pigment” assay

in Reference Example 3, used by Watanabe as the indicator of the powder's coloring performance. EX-1006, [0055]–[0056], Table 3. EX-1003, ¶192.

7. Claim 16

Watanabe-JECFA References render obvious claim 16 for the reasons in §IV.B.4 ([10c]). EX-1003, ¶193.

8. Claim 17

Watanabe-JECFA References render obvious claim 17 for the reasons in §IV.B.4 ([10c]). EX-1003, ¶¶194-195.

9. Claim 19

Watanabe-JECFA References render obvious that *the carrier comprises starch* (the trehalose/water-soluble hemicellulose carrier of Watanabe's powder composition may further contain starch, starch syrup, and starch hydrolyzates and starch derivatives such as dextrin, as taught at EX-1006, [0018]). EX-1003, ¶¶196-197.

As shown for [10a], the *carrier* of Watanabe's spray-dried powder is the carbohydrate matrix that surrounds the paprika oil active ingredient. §IV.B.4 ([10a]); EX-1006, [0008]–[0009], [0044], [0053]. Watanabe teaches that the carrier composition “may further contain sugars such as sucrose, lactose, glucose, starch syrup and reduced starch syrup; sugar alcohols; various starch hydrolyzates and starch derivatives (e.g., dextrin); starch; gelatin; natural gums such as gum arabic;

and the like.” EX-1006, [0018]. Watanabe further teaches that “[t]he contents of these additives may be suitably chosen according to the properties desired for the powder composition.” EX-1006, [0018]. EX-1003, ¶197.

C. GROUND 3: Subramaniam

1. Subramaniam

Subramaniam is directed to spray dried powders created using well-known techniques (EX-1004, [0004]-[0005]) using agar agar as an encapsulating carrier (EX-1004, [0009]-[0013], [0020]-[0022]) to create spray-dried compositions with “particularly advantageous stability in a medium of high relative humidity.” EX-1004, [0022]. EX-1003, ¶¶78-80.

Subramaniam’s preparation method (set in Example 1, used in Examples 2-7), prepares an aqueous emulsion of the carrier and active ingredient and spray-dries the homogenized feed. EX-1004, [0050]-[0051], [0064]. EX-1003, ¶81.

Subramaniam reports seven examples, the first five of which measure active ingredients’ *retention* in spray-dried powders after spray-drying. Examples 1-4 prepare spray-dried compositions containing lemon oil in a carrier and report flavor retention of 85% to 87.8%. EX-1004, [0049]-[0059]. Example 5 prepares a spray-dried composition containing fragrance in the same carriers, and reports 100% retention. EX-1004, [0060]-[0061]. EX-1003, ¶82.

Unlike Examples 1-5, Example 7 measures active ingredients’ *stability* in the

spray-dried powder after it is exposed to an aging/storage test. Example 7 presents a comparative stability example: two spray-dried compositions—Formulation A (with 3% agar agar) and Formulation B (without agar agar)—are prepared, each containing a five-component perfume composition encapsulated in a carrier; the powders are admixed with a detergent containing a percarbonate-TAED bleaching system, stored at 37°C/70% relative humidity for four weeks, and the residual perfuming ingredients are quantified by GC-MS. EX-1004, [0064]-[0067], Table 1. EX-1003, ¶83.

2. Claim 1

(a) [1pre]

If the preamble is limiting, Subramaniam discloses *a spray-dried powder*. EX-1003, ¶¶198-201, EX-1004, [0001], [0004]-[0005], [0014], [0016], [0020], [0048].

Subramaniam's stated invention is "a composition in the form of a spray-dried powder, comprising one or more active ingredients dispersed in a carrier." EX-1004, [0020]; *see also id.*, [0014]. *See also* EX-1004, [0050]-[0051], [0060]-[0061] (describing preparation method of spray-dried powders). EX-1003, ¶¶200-201.

(b) [1a]

Subramaniam discloses *a plurality of dried particles* (Example 5's spray-dried particulates) *which individually contain an amount of final active ingredient* (an amount of fragrance recovered from Example 5's spray-dried powder)

encapsulated within a carrier (maltodextrin/sucrose/agar agar carrier) *resulting from drying a slurry* (drying of Example 5's aqueous emulsion) *containing an initial active ingredient* (fragrance as loaded into the aqueous emulsion), *a liquid solvent* (water) *and the carrier* (maltodextrin, sucrose and agar agar carrier materials dispersed in aqueous emulsion). EX-1003, ¶¶202-208.

Subramaniam's stated invention is "a composition in the form of a spray-dried powder, comprising one or more active ingredients dispersed in a carrier, wherein said carrier comprises at least one wall-forming carbohydrate material and from 1 to 30% of agar agar, relative to the dried weight of the composition." EX-1004, [0020]. Subramaniam further teaches that, "[i]n spray-dried powders, an active ingredient such as a flavor or a fragrance, usually hydrophobic, is entrapped as liquid droplets in a solidified matrix of a dehydrated carrier." EX-1004, [0005]. EX-1003, ¶203.

Subramaniam's composition is initially prepared as a *slurry*—specifically, an "aqueous emulsion." with water as the *liquid solvent*. EX-1004, [0030], [0050]. EX-1003, ¶205.

Example 5's aqueous emulsion is prepared from a composition consisting of maltodextrin 18DE, sucrose, agar agar, lecithin (together, the carrier materials), and fragrance and homogenized in water. EX-1004, [0060]-[0061]. The fragrance is the

initial active ingredient. EX-1004, [0060] EX-1003, ¶205.

The aqueous emulsion is then spray dried, producing dried particles in which ***an amount of the active ingredient is encapsulated within the carrier.*** EX-1004, [0005], [0051], [0053], [0060]. The resulting particulates' microscopy confirms that the active ingredient is encapsulated within the carrier on a particle-by-particle basis. EX-1004, [0053]. EX-1003, ¶206.

The below table shows Example 5's pre-spray-dried composition ("% dry" column) and when made into a slurry ("Grams" column, adding water). Ex-1004, [0059]-[0060]. The table lists relative weights of the active ingredient (fragrance) and the carrier materials. EX-1004, [0020], [0060]-[0061]. EX-1003, ¶207.

Subramaniam (EX-1004), Example 5 (annotated)		
Ingredients	Grams	% dry
Maltodextrin 18DE	2079	29.70
Sucrose	2079	29.70
Agar agar	700	10.00
Fragrance ¹⁾	2100	30.00
Lecithin	42	0.60
Water	13600	
Total		100.00
1) origin : Firmenich SA, Geneva, Switzerland		
□ = carrier (maltodextrin, sucrose, agar agar)		
■ = active ingredient (fragrance)		

(c) [1b]

Subramaniam discloses or renders obvious that *the initial active ingredient* (Example 5's fragrance loaded into the aqueous emulsion) *includes one or more constituent components* (the fragrance's constituent perfuming ingredients), *at least one of which is among one or more principle molecular types* (one or more principal, character-imparting constituent of the fragrance) *from which at least one of a desirable fragrance is obtained* (Example 5's fragrance). EX-1003, ¶¶209-216.

Subramaniam (EX-1004), Example 5 (annotated)		
Ingredients	Grams	% dry
Maltodextrin 18DE	2079	29.70
Sucrose	2079	29.70
Agar agar	700	10.00
Fragrance 1)	2100	30.00
Lecithin	42	0.60
Water	13600	
Total		100.00
1) origin : Firmenich SA, Geneva, Switzerland		
= carrier (maltodextrin, sucrose, agar agar) = active ingredient (fragrance)		

Example 5's fragrance (initial active ingredient) *includes one or more constituent components*. A POSITA understood a commercial “fragrance” of the

type used in Example 5 is a multi-component mixture of perfuming ingredients (e.g., aroma chemicals, essential oils, and aromatic isolates) formulated together to produce a characteristic odor profile. EX-1003, ¶¶210-211. Each such perfuming ingredient is a constituent component of the fragrance. *Id.*

That understanding is consistent with and corroborated by Subramaniam's Example 7, showing that a "perfume composition" has multiple perfuming ingredients (there, five), EX-1004, [0065], n.1. Subramaniam also refers to such perfuming ingredients as active-ingredient constituents quantified in its retention analyses. EX-1004, [0067] (describing GC-MS analysis "to determine the residual quantity of the various perfuming ingredients"); EX-1004, [0068] (referring to "the highest proportion of perfuming ingredients which have not been decomposed"); EX-1004, Table 1 (title). This understanding is consistent with the '776Patent, which discloses that "[t]he active ingredient may be any desirable single constituent component or a combination of constituent components," "desirable" meaning at least one component provides the desired "flavor," etc. EX-1001, 6:10–15. For example, the '776Patent identifies D-limonene as a "particular constituent component" of orange oil. EX-1001, 12:18–21, Fig. 11; *see also id.* 12:9–34 (Fig. 11 accelerated-aging discussion). EX-1003, ¶212.

At least one of the constituent components of Example 5's initial active

ingredient (the fragrance) *is among one or more principle molecular types from which the desirable fragrance is obtained*. While Subramaniam does not expressly list each constituent component of Example 5's active ingredient (fragrance from "Firmenich SA"), a POSITA understood or found obvious the fragrance includes *one or more constituent components* from which the desired fragrance (i.e., scent) is obtained. For example, Subramaniam confirms that the fragrance, i.e., its active ingredient, "may include volatile perfuming or flavoring ingredients, as well as other kinds of ingredients such as pharmaceutical actives." EX-1004, [0015]. Subramaniam further discloses that "the active ingredient" of its "spray-dried composition" is "preferably ... [a] fragrance ingredient or composition" (EX-1004, [0031]), which includes a "variety" of "fragrance materials," both natural and synthetic origin, that may be "single compounds and mixtures" and that are "well known" in the art for "imparting an odor and/or a flavor or taste to a consumer product traditionally perfumed or flavored" or for "modifying the odor and/or taste." EX-1004, [0032] Consistently, Subramaniam identifies Fenaroli's Handbook of Flavor Ingredients as a source of "specific examples of such components," (EX-1004, [0032]), which catalogs individual flavor and aroma compounds and confirms the materials Subramaniam encapsulates are molecular types from which a composition's flavor or fragrance is obtained. EX-1036, 99; EX-1004, [0032]. EX-

1003, ¶¶213-214.

Further, [1b] does not require the specific identification of each constituent component of Example 5's active ingredient (fragrance from "Firmenich SA"); it requires only that the active ingredient include at least one constituent that is a principle molecular type—a principal, character-imparting constituent from which the desired fragrance is obtained (*see* §§IV.A, IV.B). A POSITA understood or found obvious a commercial fragrance of the type used in Example 5 contains at least one such principal character-imparting constituent, because a fragrance is formulated around the constituents that produce its characteristic odor. EX-1003, ¶215. Even if specific identification were required, it would have been obvious to a POSITA that the fragrance includes at least one principal, character-imparting constituent from which its fragrance is obtained. *Id.* A POSITA formulating or evaluating an encapsulated fragrance would have been motivated to characterize its principal odor-bearing constituents, because those constituents define the product's commercial value and are the constituents a formulator monitors. *Id.* And a POSITA would have had a reasonable expectation of success in doing so: identifying the principal character-imparting constituents of a fragrance was routine analytical work (e.g., GC-MS of the type Subramaniam performs to quantify "the various perfuming ingredients," EX-1004, [0067]), and fragrances would typically have such

constituents. EX-1003, ¶¶215-216.

(d) [1c]

Subramaniam discloses that *the final active ingredient* (fragrance recovered from Example 5's spray-dried powder) *includes one or more of the constituent components* (fragrance's constituent perfuming ingredients) *corresponding with those of the initial active ingredient* (same perfuming ingredients as loaded into Example 5's aqueous emulsion) *as modified by the drying of the slurry*. EX-1003, ¶¶217-221.

In Example 5, the spray-dried product has a fragrance retention of 100% after spray-drying. EX-1004, [0061] ("The final product (after the process) had a flavor retention of 100% (solid content of the emulsion introduced into the spray-drier of 33.98%)."); §IV.C.2 ([1d]) (explaining that the [0061] reference to "flavor retention" should be understood to include "fragrance retention", consistent with Subramaniam's express reference to "flavor or fragrance retention" (EX-1004, [0047])); EX-1003, ¶218.

Subramaniam (EX-1004), Example 5 (annotated)

Example 5

Preparation of a spray-dried composition according to the invention

Formulation :

[0060]

Ingredients	Grams	% dry
Maltodextrin 18DE	2079	29.70
Sucrose	2079	29.70
Agar agar	700	10.00
Fragrance 1)	2100	30.00
Lecithin	42	0.60
Water	13600	
Total		100.00

1) origin : Firmenich SA, Geneva, Switzerland

Preparation : The spray-dried powder was prepared as described in Example 1.

[0061] The final product (after the process) had a flavor retention of 100% (solid content of the emulsion introduced into the spray-drier of 33.98%).

= carrier (maltodextrin, sucrose, agar agar)

= active ingredient (fragrance)

= 100% flavor retention of the final product

The reported 100% recovery is a *bulk* fragrance retention: a single percentage for the “Fragrance” as loaded, with no per-constituent breakdown, and a value at the level of the whole fragrance can be 100% only if each constituent perfuming ingredient was likewise recovered. EX-1003, ¶219. A POSITA understood the fragrance’s 100% bulk retention through the spray-drying process indicates that the fragrance recovered from the spray-dried Example 5 powder—the final active ingredient—contains the same constituent perfuming ingredients that were loaded into the aqueous emulsion as the initial active ingredient. *Id.* Indeed, a constituent perfuming ingredient of the fragrance cannot be selectively lost during spray drying

while retaining the bulk fragrance at 100%. *Id.*

Additionally, the ***final active ingredient*** (fragrance recovered in the spray-dried powder) includes ***one or more of the constituent components*** corresponding with those of the initial active ingredient ***as modified by the drying of the slurry***. Per the '776 Patent, the “as modified by the drying of the slurry” limitation encompasses scenarios where the final active ingredient reflects modifications occurring from spray drying (e.g., encapsulation). For example, the specification describes that modifications from drying the slurry include heat-induced modifications occurring during the spray-drying process itself. EX-1001, 2:31–37. Here, the specification contemplates modification due to the spray-drying process, and not being limited to a modification in a separate, quantified value in the final active ingredient. *See id.* Congruently, Subramaniam discloses that the final active ingredient in Example 5’s spray-dried powder is the fragrance as modified by the heat induced modification stemming from the spray-drying step that resulted in fragrance encapsulation. EX-1004, [0050]-[0051], [0060]-[0061]. EX-1003, ¶220.

(e) [1d]

Subramaniam discloses or renders obvious ***a weight percentage of at least one of the one or more principle molecular types*** (at least one principle molecular type of Example 5’s fragrance) ***in the final active ingredient*** (the fragrance as

recovered from Example 5's spray-dried powder) *is within about 5%* (0% variation—100% retention) *of a weight percentage of the corresponding principle molecular types in the initial active ingredient* (the fragrance as loaded into Example 5's spray-drier emulsion at 30% dry weight). EX-1003, ¶¶222-227.

Subramaniam reports Example 5's "[t]he final product (after the [spray drying] process) had a flavor retention of 100% (solid content of the emulsion introduced into the spray-drier of 33.98%)." EX-1004, [0061]. As a starting point, a POSITA understood the phrasing "flavor retention" is intended to include "fragrance retention." EX-1003, ¶¶224-225. That is because Example 5's active ingredient is identified as "Fragrance" (not "flavor") in the composition table at paragraph [0060], and Subramaniam separately uses "flavor retention" for its lemon-oil examples (Examples 1-4), where the active ingredient is a flavorant. EX-1004, [0052] (Example 1), [0055] (Example 2), [0057] (Example 3), [0059] (Example 4). EX-1003, ¶¶223-224.

Example 4⁷

⁷ EX-1004, [0058]-[0059]. Examples 1-3 likewise describe "flavor retention" of "lemon oil". EX-1004, [0049], [0052], [0054]-[0057].

Example 4

Preparation of a spray-dried composition according to the invention

Formulation :

[0058]

Ingredients	Grams	%dry
Maltodextrin 18DE	798	11.40
Sucrose	1554	22.20
Agar agar	1050	15.00
Gum Arabic	798	11.40
Lemon oil ¹⁾	2800	40.00
Water	18200	
Total		100.00

1) see Example 1

Preparation : The spray-dried powder was prepared as described in Example 1.

[0059] The final product (after the process) had a flavor retention of 87.00% (solid content of the emulsion introduced into the spray-drier of 27.78%).

Example 5

Example 5

Preparation of a spray-dried composition according to the invention

Formulation :

[0060]

Ingredients	Grams	% dry
Maltodextrin 18DE	2079	29.70
Sucrose	2079	29.70
Agar agar	700	10.00
Fragrance ¹⁾	2100	30.00
Lecithin	42	0.60
Water	13600	
Total		100.00

1) origin : Firmenich SA, Geneva, Switzerland

Preparation : The spray-dried powder was prepared as described in Example 1.

[0061] The final product (after the process) had a flavor retention of 100% (solid content of the emulsion introduced into the spray-drier of 33.98%).

Moreover, Subramaniam's general teaching at paragraph [0047] confirms the dual usage of "flavor or fragrance retention" and the meaning of "retention" as the proportion of active ingredient recovered in the spray-dried product: "the

compositions of the invention provide a flavor or fragrance retention of up to 85% after the encapsulation process.” EX-1004, [0047]; EX-1003, ¶225.

Because Example 5 discloses that its spray-drying process results in the fragrance’s 100% bulk retention (i.e., the final active ingredient) (EX-1004, [0061]), a recovery at or near 100% means little or no constituent was lost (as spray drying does not create constituent components not already present). EX-1003, ¶¶226-227. Additionally, since the principal, character-imparting constituents make up most of the fragrance, a POSITA understood or found obvious those constituents were recovered substantially intact. *Id.* A reported recovery at/near 100% leaves the “share” of the principal constituents substantially unchanged. *Id.* It follows that the weight percentage of at least one component within Subramaniam’s recovered fragrance is within about 5% of its weight percentage in the initial fragrance. *Id.*

3. Claim 2

Subramaniam discloses or renders obvious that *the one or more principle molecular types* (at least one perfuming ingredient of Example 5’s fragrance from which the fragrance is obtained) are molecular types *of a food composition* (used for the perfuming or flavoring food compositions). EX-1003, ¶¶228-229.

Subramaniam teaches that its spray-dried “active ingredient” (here, fragrance) “may include volatile perfuming or flavoring ingredients, as well as other kinds of

ingredients such as pharmaceutical actives” (EX-1004, [0015]) and that the spray-dried products “can be advantageously used ... for the perfuming or flavoring of food compositions” including “chewing gums or chewing sweets, savoury food or baking in the field of flavours” (EX-1004, [0044]-[0045]). §IV.C.2 ([1b]); EX-1003, ¶229; *see also* EX-1001, 12:19–30, 15:18–24, 18:13–18, FIG. 11 (similarly explaining that the same molecular type can be of a food or flavor composition, depending on end use; treating orange oil’s D-limonene as simultaneously a flavor and a food principle molecular type). EX-1003, ¶229.

4. Claim 3

Subramaniam discloses or renders obvious that *the one or more principle molecular types* (at least one perfuming ingredient of Example 5’s fragrance from which the fragrance is obtained) are molecular types *of a flavor composition* (used for the flavoring food compositions). EX-1003, ¶¶230-231.

Subramaniam’s spray-dried “active ingredient” (here, fragrance) “may include volatile perfuming or flavoring ingredients” (EX-1004, [0015]), and the invention’s second object is “the flavoring or perfuming of consumer products such as foods, beverages, or perfuming compositions” (EX-1004, [0017]). Subramaniam’s Examples 1-4 prepare spray-dried compositions where lemon oil—a flavor—is the active ingredient. EX-1004, [0049]-[0059]; §IV.C.2 ([1b]); EX-

1003, ¶231. *See also* EX-1001, 12:19–30, 15:18–24, 18:13–18, FIG. 11 (similarly explaining that the same molecular type can be of a food or flavor composition, depending on end use; treating orange oil’s D-limonene as simultaneously a flavor and a food principle molecular type). EX-1003, ¶231.

5. Claim 4

Subramaniam discloses or renders obvious that *the one or more principle molecular types* (at least one perfuming ingredient of Example 5’s fragrance from which the fragrance is obtained)) are molecular types *of a fragrance composition* (used for perfuming a product). EX-1003, ¶¶232-233.

Example 5’s active ingredient is “Fragrance” (origin: Firmenich SA, Geneva, Switzerland), present at 30% dry weight. EX-1004, [0060]; §IV.C.2 ([1b]); EX-1003, ¶233. A fragrance is, by definition, a perfuming composition the constituent perfuming ingredients of which are principle molecular types of the fragrance. §IV.C.2 ([1b]); EX-1003, ¶233 (citing EX-1032 (Pybus)). Accordingly, a POSITA understood at least one perfuming ingredient of Example 5’s fragrance (i.e., *one or more principle molecular types*) itself is a *molecular type of a fragrance composition*. EX-1003, ¶233.

6. Claim 5

Subramaniam discloses or renders obvious that *the one or more principle molecular types* (at least one pharmaceutical active Subramaniam teaches its spray-

dried active ingredient may include, from which the medical/therapeutic effect is obtained) is a molecular type *of a medicament composition* (used as/in a medicament). EX-1003, ¶¶234-235.

Subramaniam teaches that its spray-dried composition's active ingredient "may include ... pharmaceutical actives." EX-1004, [0015]; §IV.C.2 ([1b]); EX-1003, ¶¶234-235. Accordingly, a POSITA understood at least one such pharmaceutical active included in Subramaniam's spray-dried active ingredient (i.e., *one or more principle molecular types*) is itself a *molecular type of a medicament composition*. EX-1003, ¶235.

7. Claim 7

Subramaniam discloses claim 7 for the reasons in §IV.C.2 ([1d]). EX-1003, ¶236.

8. Claim 8

Subramaniam discloses claim 8 for the reasons in §IV.C.2 ([1d]). EX-1003, ¶237.

9. Claim 9

Subramaniam discloses claim 9 for the reasons in §IV.C.2 ([1d]). EX-1003, ¶238.

10. Claim 10

(a) [10pre]

If the preamble is limiting, Subramaniam discloses *a spray-dried powder* (spray-dried powder described in the context of Example 7's Formulation A). Example 7's Formulation A is similar to that of Example 5 because both were "prepared according to the method described in Example 1." EX-1004, [0001], [0004]-[0005], [0014], [0016], [0020], [0048], [0050]-[0051], [0064]-[0065]; §IV.C.2 ([1pre]); EX-1003, ¶239.

(b) [10a]

Subramaniam discloses *a plurality of dried particles* (Formulation A's spray-dried particulates) *which individually contain an amount of active ingredient* (Formulation A's perfume composition) *encapsulated within a carrier* (a maltodextrin/sucrose/agar agar carrier). EX-1003, ¶¶240-245.

Subramaniam discloses *a plurality of dried particles* (Formulation A's spray-dried particulates). In Example 7, Formulation A's aqueous emulsion is prepared by combining maltodextrin 18DE, sucrose, perfume composition, agar agar, lecithin, and water. EX-1004, [0065]. EX-1003, ¶241.

Subramaniam (EX-1004), Example 7 (annotated)

Example 7

Comparative example on the stability of a spray-dried composition according to the invention, in a detergent base

[0064] Two spray-dried compositions having the following formulations were prepared according to the method described in Example 1 :

Formulation A :

[0065]

Ingredients	Grams	% dry
Maltodextrin 18DE	2865	38.20
Sucrose	2865	38.20
Perfume composition ¹⁾	1500	20.00
Agar agar	225	3.00
Lecithin	45	0.60
Water Total	9600	100.00

1) composition consisting of a mixture of benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial® (origin: Givaudan-Roure, Vernier, Switzerland) and hexyl salicylate ; each ingredient being present in the same weight percentage in the composition

 = carrier (maltodextrin, sucrose, agar agar)

 = active ingredient (perfume composition)

Example 1's method is used to prepare Example 7's Formulation A, which involves preparing the emulsion with the ingredients and spray drying it, producing dried particles. EX-1004, [0005], [0051], [0064]; EX-1003, ¶242.

Formulation A's spray-dried particulates *individually contain an amount of active ingredient* (Formulation A's perfume composition). The perfume composition is the active ingredient. EX-1004, [0031] ("The active ingredient dispersed in the carrier of the spray-dried composition of the invention will

preferably be a hydrophobic flavor or fragrance ingredient or composition of current use.”), [0015] (“What is meant here by ‘active ingredient’, is a liquid ingredient, preferably hydrophobic, which is sought to be protected by way of solid encapsulation. It may include volatile perfuming or flavoring ingredients, as well as other kinds of ingredients such as pharmaceutical actives for instance.”), [0065], [0067]; EX-1003, ¶243.

Formulation A’s spray-dried particulates (EX-1004, [0051], [0064]) that individually contain perfume composition (***active ingredient***) are ***encapsulated within a carrier*** (a maltodextrin/sucrose/agar agar carrier). EX-1004, [0005], [0053], [0065]. Subramaniam teaches that, “[i]n spray-dried powders, an active ingredient such as a flavor or a fragrance, usually hydrophobic, is entrapped as liquid droplets in a solidified matrix of a dehydrated carrier, generally consisting of carbohydrates, such as starches, hydrolyzed starches (maltodextrin), chemically modified starches, emulsifying polymers (gum arabic) and in certain instances monomers and dimers of simple aldohexoses, or any combination thereof.” EX-1004, [0005]. The carrier of the resulting dried particles comprises wall-forming carbohydrate materials (maltodextrin, sucrose) together with agar agar. EX-1004, [0020], [0065]; EX-1003, ¶244.

Subramaniam (EX-1004), Example 7 (annotated)

Example 7

Comparative example on the stability of a spray-dried composition according to the invention, in a detergent base

[0064] Two spray-dried compositions having the following formulations were prepared according to the method described in Example 1 :

Formulation A :

[0065]

Ingredients	Grams	% dry
Maltodextrin 18DE	2865	38.20
Sucrose	2865	38.20
Perfume composition 1)	1500	20.00
Agar agar	225	3.00
Lecithin	45	0.60
Water Total	9600	100.00

1) composition consisting of a mixture of benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial® (origin: Givaudan-Roure, Vernier, Switzerland) and hexyl salicylate ; each ingredient being present in the same weight percentage in the composition

 = carrier (maltodextrin, sucrose, agar agar)

 = active ingredient (perfume composition)

(c) [10b]

Subramaniam discloses that *the active ingredient* (Formulation A's perfume composition) *includes one or more constituent components* (benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial®, and hexyl salicylate), *at least one of which is among one or more principle molecular types from which at least one of a desirable fragrance is obtained* (the desirable scent of Formulation A's perfume composition). EX-1003, ¶¶246-250.

Formulation A's **active ingredient**—the perfume composition—includes the following **constituent components**: “benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial® (origin: Givaudan-Roure, Vernier, Switzerland) and hexyl salicylate; each ingredient being present in the same weight percentage in the composition.” EX-1004, [0065], n.1. EX-1003, ¶247.

Subramaniam (EX-1004), Example 7 (annotated)

Example 7

Comparative example on the stability of a spray-dried composition according to the invention, in a detergent base

[0064] Two spray-dried compositions having the following formulations were prepared according to the method described in Example 1 :

Formulation A :

[0065]

Ingredients	Grams	% dry
Maltodextrin 18DE	2865	38.20
Sucrose	2865	38.20
Perfume composition ¹⁾	1500	20.00
Agar agar	225	3.00
Lecithin	45	0.60
Water Total	9600	100.00

1) composition consisting of a mixture of benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial® (origin: Givaudan-Roure, Vernier, Switzerland) and hexyl salicylate ; each ingredient being present in the same weight percentage in the composition

- = carrier (maltodextrin, sucrose, agar agar)
 = active ingredient (perfume composition)
 = constituent components (footnote 1)

Each constituent component is a perfuming ingredient from which the fragrance of Formulation A's perfume composition is obtained. EX-1004, [0065], n.1, [0067]-

[0068], Table 1. Subramaniam refers to Example 7's constituent components as "perfuming ingredients." EX-1004, [0067] (describing GC-MS analysis "to determine the residual quantity of the various perfuming ingredients"); EX-1004, [0068] (referring to "the highest proportion of perfuming ingredients which have not been decomposed"); EX-1004, Table 1 (titled "Percentage of perfume ingredients recovered"); EX-1003, ¶248.

Each of the five constituent components is a *principle molecular type from which the desirable fragrance* (fragrance of Formulation A's perfume composition) *is obtained*. EX-1003, ¶249. Per the '776 Patent, a "principle molecular type" (from which a characteristic (here, fragrance/scent) of the active ingredient is obtained) encompasses a constituent component that ultimately contributes to the desired characteristic (here, scent), such that when the component's concentration decreases in the overall spray-dried composition, the desired characteristic (here, scent) is likewise undesirably altered. EX-1001, 2:31–37. Here, each of the five perfume composition's constituent components are "perfuming ingredients"—each present in the "same weight percentages"—contributing to the desirable fragrance. EX-1004, [0065], n.1, [0067]–[0068], Table 1. The claim's "one or more"/"at least one of" language confirms that the perfume composition may have multiple principle molecular types simultaneously. EX-1001, 17:66–18:1, 18:8 (claim 1); *see also id.*

at 18:47–52 (claim 10), 20:7–21 (claim 20), 18:19–21 (claim 4), 18:63–65 (claim 13); EX-1003, ¶249.

(d) [10c]

Subramaniam discloses or renders obvious *a weight percentage of at least one of the one or more principle molecular types* (hexyl salicylate) *in the active ingredient* (Formulation A’s perfume composition) *does not vary by more than about 5% during aging of the dried powder* (0% variation—100% recovery) *during any period of elevated temperature of about 95°F up to about 1000 hours*. EX-1003, ¶¶251-276.

As a threshold, this limitation encompasses stability over time—whether a principle molecular type’s weight percentage in the active ingredient changes during aging relative to its weight before aging. EX-1001, claim 10. The ’776Patent confirms this, describing the allegedly inventive property as a principle molecular type that “does not vary significantly ... during aging of the dried powder” at the recited temperature and duration. EX-1001, 2:31–40. This is a before-and-after comparison of the same molecule across aging, not a comparison of one constituent’s “share” against the others. Congruently, Subramaniam discloses or renders obvious the instant limitation. EX-1003, ¶252.

Example 7 prepares a spray-dried powder (“Formulation A”) containing a

perfume composition of benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial®, and hexyl salicylate, encapsulated in a maltodextrin/sucrose/agar agar carrier. EX-1004, [0064]-[0065]. Each perfuming ingredient is a *principle molecular type from which a desirable fragrance is obtained*. EX-1004, [0064]; §IV.C.10 ([10b]); EX-1003, ¶253.

The weight percentage of hexyl salicylate, one of *the principle molecular types in the active ingredient* (Formulation A's perfume composition) *did not vary by more than about 5% during aging of the dried powder*. EX-1003, ¶254. In Example 7, Subramaniam admixed the Formulation A powder with a standard European detergent containing a percarbonate-TAED bleaching system, stored the mixture for four weeks at 37°C and 70% relative humidity, then separated the powder from the detergent and quantified the residual perfuming ingredients by GC-MS. EX-1004, [0067]. Table 1 reports the percentage of each perfuming ingredient recovered after this storage period relative to the amount of each perfuming ingredient incorporated into the composition. EX-1004, [0067], Table 1. Subramaniam separately reports, as a comparative control, a reference detergent onto which the pure perfume composition was sprayed directly and stored at 3°C. EX-1004, [0067], Table 1; EX-1003, ¶254.

Subramaniam (EX-1004), Example 7 — Table 1 (annotated)			
Table 1 : Percentage of perfume ingredients recovered after storage for four weeks at 37° and 70% RH			
Perfuming ingredient	Sprayed-on detergent powder	Formulation B (no agar agar)	Formulation A (3% of agar agar)
Benzyl acetate	3	6	6
Phenyl ethyl acetate	10	18	23
Cyclamen aldehyde	31	45	67
Lilial®	0.0	2	10
Hexyl salicylate	25	48	100

= perfuming ingredient (active ingredient)
 = hexyl salicylate (constituent component) / 100% recovery
 = Formulation A (3% agar agar)

For hexyl salicylate in Formulation A, Table 1 lists a 100% recovery. EX-1004, Table 1. A POSITA understood 100% recovery indicates a 0% weight variation of hexyl salicylate before and after aging, within the claimed variance of *no more than about 5% during aging*. EX-1003, ¶255.

Hexyl salicylate's weight percentage did not vary by more than 5% *during aging of the dried powder* during any period of *elevated temperature of about 95°F* up to about 1000 hours. Formulation A was stored at an elevated temperature of 37°C/98.6°F, which is about (and in excess of) 95°F as claimed. EX-1004, [0067], Table 1. Moreover, because degradation slows as temperature falls, an ingredient retained at 37°C would be retained at least as well at 95°F. EX-1007 at 14–15, 19, Fig. 1.9. EX-1003, ¶256.

Further, Subramaniam discloses or renders obvious that the weight percentage

of hexyl salicylate did not vary by more than 5% *during any period* of the claimed elevated temperature and duration, for the reasons noted below. EX-1003, ¶257.

If the claim limitation “*during any period* ... up to about 1000 hours” is interpreted as setting a minimum duration of about 1000 hours for aging, [10c] would have been obvious to a POSITA in view of Subramaniam and the well-known principles of accelerated stability testing. A POSITA, reading Subramaniam’s Example 7 and the reported 100% recovery of hexyl salicylate after four weeks at 37°C/70% relative humidity in a percarbonate-TAED detergent, would have found it obvious that hexyl salicylate would satisfy the milder *elevated temperature of about 95°F* test of [10c] over a period of about 1000 hours with a reasonable expectation of success. Subramaniam (EX-1004), [0067], Table 1; EX-1003, ¶258.

Subramaniam subjected hexyl salicylate to materially more stressful storage conditions than [10c] specifies, on three independent axes: higher temperature, added humidity, and a harsher bleach-testing environment. (1) Subramaniam’s storage temperature (37°C (98.6°F)) is higher than [10c] requires. EX-1003, ¶¶259-260. A POSITA understood chemical degradation rates increase with temperature, so an ingredient retained at 37°C would be retained at least as well at about 35°C (95°F) over an equal or longer period. *Id.* (2) Subramaniam adds elevated humidity (70% relative humidity) that [10c] aging does not require, and the ’776Patent’s only

disclosed embodiment is a dry hot box (would not be subject to any humidity). EX-1001, 14:21-40; EX-1003, ¶261. A POSITA understood 70% relative humidity accelerates a dried powder's degradation relative to that dry baseline, making the moisture in Subramaniam an additional stressor absent from [10c]. EX-1003, ¶261 (citing EX-1008; EX-1009). (3) Subramaniam further adds an active chemical oxidant, admixing the encapsulated powder with a detergent containing a percarbonate-TAED bleaching system, understood to generate peracetic acid (a strong oxidant capable of degrading fragrance materials, and one the industry used as a standardized accelerated bleach-stability screen because of its severity). EX-1003, ¶262 (citing EX-1010).

Each of these conditions is a significant stressor and resulted in degradation of certain components, e.g., benzyl acetate fell to 6%, phenyl ethyl acetate to 23%, cyclamen aldehyde to 67%, and Lilial® to 10%. EX-1004, Table 1. But hexyl salicylate was recovered at 100% (0% variation). *See id.* That hexyl salicylate showed no degradation despite these stressors demonstrates that it would also show no degradation in a milder environment that is broadly encompassed by the claim—i.e., one without humidity, *lower temperature*, and without a chemical oxidant. EX-1003, ¶263.

Subramaniam aged Formulation A for 672 hours; [10c] recites a period up to

about 1,000 hours. EX-1004, [0067]. If “*during any period*” requires aging for the about 1000 hours, that limitation would have been obvious to a POSITA, applying the routine predictive tools of the formulation art to Subramaniam’s teaching, the claimed variation would remain within about 5% out to about 1,000 hours (for reasons similar to those noted above in the Watanabe ground; *see* Section IV.B.3, IV.B.4.[10c]). Additionally, a POSITA evaluating an encapsulated fragrance would have wanted to confirm stability across a commercial shelf life on the order of 1,000 hours, because retention over that period determines whether the product remains usable. EX-1003, ¶264. Projecting a shorter stability (the 672 hours of testing in Subramaniam) result to a longer period (1000 hours as claimed) is the conventional and ordinary purpose of accelerated testing. *Id.*; EX-1007 at 14–19. Having survived the harsher, shorter test intact, a POSITA would reasonably expect hexyl salicylate to remain within [10c]’s about-5% threshold across the milder conditions the claim encompasses and for the longer ~1,000-hour duration. EX-1003, ¶264, ¶274.

The following comparison summarizes the relationship between the two tests.

Subramaniam Test vs. '776 Patent Test

Degradation Mechanism	Subramaniam Test	'776 Patent Test [10c]
Active peroxygen oxidant (PAA generated by percarbonate-TAED)	Present	Not specified in claim (absent from dry-oven test in the specification)
Elevated humidity (70% RH)	Present	Not specified in claim (absent from dry-oven test described in specification)
Elevated temperature	37°C (98.6°F)	~35°C (95°F)
Duration	672 hours (4 weeks)	Under one interpretation, up to about 1000 hours
Result for hexyl salicylate after aging	100% retained (0% variation)	Required: $\leq$ about 5% variation
Stress on conditions the claim specifies	Greater (heat, humidity, and oxidant)	Milder/fewer specified stressors; longer maximum duration

Note that Arrhenius or Q10 modeling is neither necessary nor available to establish the hexyl salicylate stability under claim 10's conditions. First, Arrhenius and Q10 modeling do not alter this result just explained in detail (that a POSITA would have expected hexyl salicylate to stay within about 5% at 1,000 hours given its 100% retention under harsher conditions). EX-1003, ¶¶265-273. Second, such modeling cannot be performed here because those tools scale a measured, nonzero degradation rate to a different temperature or time, and Subramaniam's single endpoint reports a degradation rate of zero, leaving nothing to scale. Scaling zero by any temperature factor over any duration yields zero. *Id.* Third, even an illustrative bounding exercise using a deliberately conservative linear extrapolation treatment, confirms that hexyl salicylate would remain within the threshold of [10c] at about 1000 hours. *Id.*

In the alternative, if “*during any period* ... up to about 1000 hours” sets the maximum length of the aging period rather than a minimum, Subramaniam discloses [10c] outright. Subramaniam's hexyl salicylate's weight percentage did not vary by more than about 5% across the 672 hours of testing imposed by Subramaniam. EX-1003, ¶275.

11. Claim 11

Subramaniam discloses or renders obvious that *the one or more principle*

molecular types (benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial®, and hexyl salicylate of Formulation A's perfume composition from which a fragrance is obtained) are molecular types **of a food composition** (used for perfuming or flavoring food compositions). EX-1003, ¶¶277-278.

Subramaniam's spray-dried "active ingredient" (here, fragrance) "may include volatile perfuming or flavoring ingredients, as well as other kinds of ingredients such as pharmaceutical actives" (EX-1004, [0015]) and the spray-dried products "can be advantageously used ... for the perfuming or flavoring of food compositions" including "chewing gums or chewing sweets, savoury food or baking in the field of flavours" (EX-1004, [0044]-[0045]). §IV.C.2 ([1b]); EX-1003, ¶278. This '776 Patent likewise contemplates that the same molecular type can be that of a food or flavor composition, depending on end use. EX-1001, 12:19–30, 12:43–45, 15:18–24, 18:13–18, FIG. 11 (treating orange oil's D-limonene as simultaneously a flavor and a food principle molecular type). EX-1003, ¶278.

12. Claim 12

Subramaniam discloses or renders obvious that **the one or more principle molecular types** (benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial®, and hexyl salicylate of Formulation A's perfume composition from which the fragrance is obtained) are molecular types **of a flavor composition** (used for

flavoring food compositions). EX-1003, ¶¶279-280.

Subramaniam's spray-dried "active ingredient" (here, fragrance) "may include volatile perfuming or flavoring ingredients" (EX-1004, [0015]), and the invention's second object is "the flavoring or perfuming of consumer products such as foods, beverages, or perfuming compositions" EX-1004, [0017]; EX-1003, ¶280. *See also* EX-1001, 12:19–30, 15:18–24, 18:13–18, FIG. 11 (similarly explaining that the same molecular type can be of a food or flavor composition, depending on end use; treating orange oil's D-limonene as simultaneously a flavor and a food principle molecular type). EX-1003, ¶280.

13. Claim 13

Subramaniam discloses or renders obvious that *the one or more principle molecular types* (benzyl acetate, phenylethyl acetate, cyclamen aldehyde, Lilial®, and hexyl salicylate of Formulation A's perfume composition from which the fragrance is obtained) are molecular types *of a fragrance composition* (used for perfuming a product). EX-1003, ¶¶281-282.

The active ingredient of Example 7's Formulation A is a "[p]erfume composition" consisting of five perfuming ingredients. EX-1004, [0065], n.1, [0067]-[0068], Table 1; §IV.C.10 ([10b]); EX-1003, ¶282. Per §IV.C.10 ([10b]), each of those five perfuming ingredients is a principle molecular type of the perfume

composition—a fragrance composition. EX-1003, ¶282.

14. Claim 14

Subramaniam discloses or renders obvious that *the one or more principle molecular types* (at least one of the pharmaceutical actives Subramaniam teaches its spray-dried active ingredient may include, from which the medical/therapeutic effect is obtained) are molecular types *of a medicament composition* (used as/in a medicament). EX-1003, ¶¶283-284.

Subramaniam teaches that its spray-dried composition’s active ingredient “may include ... pharmaceutical actives.” EX-1004, [0015]; §IV.C.2 ([1b]); EX-1003, ¶¶283-284. Accordingly, a POSITA understood that at least one such pharmaceutical active included in Subramaniam’s spray-dried active ingredient is itself a *molecular type of a medicament composition*. EX-1003, ¶284.

15. Claim 16

Subramaniam discloses or renders obvious claim 16 for the reasons in §IV.C.10 ([10c]). EX-1003, ¶285.

16. Claim 17

Subramaniam discloses or renders obvious claim 17 for the reasons in §IV.C.10 ([10c]). EX-1003, ¶286.

17. Claim 18

Subramaniam discloses or renders obvious claim 18 for the reasons in

§IV.C.10 ([10c]). EX-1003, ¶287.

18. Claim 19

Subramaniam discloses claim 19. EX-1003, ¶¶288-289. Subramaniam teaches that its *carrier* includes “[s]uitable wall-forming carbohydrate materials,” which includes “natural or modified starch” and that in a particular embodiment, the carbohydrate material is selected from the group consisting of a maltodextrin ... a chemically modified starch, a hydrogenated starch hydrolysate and a succinylated or hydrolyzed starch.” EX-1004, [0024]; *also* EX-1004, [0005]. The *carrier* of Formulation A in Example 7 includes maltodextrin 18DE, a hydrolyzed *starch*. EX-1004, [0005], [0065]; §IV.C.10 ([10a]). EX-1003, ¶¶288-289.

V. CONCLUSION, FEES

IPR of the Challenged Claims is requested. EX-1003, ¶¶290-291. Please charge fees to Deposit Account 06-1050.

VI. MANDATORY NOTICES UNDER 37 C.F.R § 42.8(a)(1)

A. Real Party-In-Interest Under 37 C.F.R. § 42.8(b)(1)

McCormick & Company, Inc. is the petitioner. McCormick & Company, Inc., FONA Technologies, LLC, and Fona International, LLC are the real parties-in-interest.

B. Related Matters Under 37 C.F.R. § 42.8(b)(2)

Petitioner is not aware of any disclaimers, reexamination certificates, or petitions for *inter partes* review for the '776Patent.

However, U.S. Patent No. 8,939,388, the grandparent of the '776Patent, was the subject of an *inter partes* review filed June 6, 2015, challenging claims 1, 16, 19, and 28–30. On December 14, 2016, the Board issued a final written decision holding each challenged claim unpatentable over the prior art. The Federal Circuit affirmed that decision on January 16, 2018.

The '776Patent is the subject of the following civil action: *ZoomEssence, Inc. v. McCormick & Company, Inc. et al*, 2-25-cv-01240 (EDTX), filed December 19, 2025.

C. Lead And Back-Up Counsel Under 37 C.F.R. § 42.8(b)(3)

Petitioner provides the following designation of counsel.

Lead Counsel	Backup counsel
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D. Service Information

Please address all correspondence and service to the address listed above.

Petitioner consents to electronic service by email at IPR61529-0003IP1@fr.com.

Respectfully submitted,

Dated 7/9/2026

/Karan Jhurani/

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(Control No. IPR2026-00416)

Counsel for Petitioner

CERTIFICATION UNDER 37 CFR § 42.24

Under the provisions of 37 CFR § 42.24(d), the undersigned hereby certifies that the word count for the foregoing Petition for *Inter Partes* Review totals 13,936 words, which is less than the 14,000 allowed under 37 CFR § 42.24.

Dated 7/9/2026

/Karan Jhurani/

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CERTIFICATE OF SERVICE

Pursuant to 37 CFR §§ 42.6(e)(4)(i) *et seq.* and 42.105(b), the undersigned certifies that on July 9, 2026, a complete and entire copy of this Petition for *Inter partes* Review, Power of Attorney, and all supporting exhibits were provided via USPS Priority Express Mail, to the Patent Owner by serving the correspondence address of record as follows:

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