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GRN 000554

November 7, 2014

Via FedEx (Tracking No. 771777675441)

Antonia Mattia, Ph.D.
Director, Division of Biotechnology and
GRAS Notice Review (HFS-225)
Office of Food Additive Safety
Center for Food Safety and Applied Nutrition
Food and Drug Administration
4300 River Road
College Park, Maryland 20740

Re: GRAS Notification for Synthetic Amorphous Silica

Dear Dr. Mattia:

We respectfully submit the attached GRAS Notification on behalf of our client, Evonik Corporation (Evonik), for synthetic amorphous silica for use as a multipurpose direct additive in a variety of processed foods and beverages up to a maximum level of 2% in the finished product.[1] The GRAS Notification also covers the intended use of synthetic amorphous silica as an indirect additive. The attached GRAS Notification provides a review of the information related to the intended uses and manufacturing and safety of the ingredient. We have included three (3) hard copies of the GRAS Notification for your review.

Evonik has determined that synthetic amorphous silica is generally recognized as safe (GRAS) based on scientific procedures in accordance with 21 C.F.R. § 170.30(b) and in conformance with the guidance issued by the Food and Drug Administration (FDA) under proposed 21 C.F.R. § 170.36, 62 *Fed. Reg.* 18938 (Apr. 17, 1997). Therefore, the use of

[1] This GRAS Notification proposes the use of synthetic amorphous silica in a broader range of food categories than those included in the previously-filed GRAS Notification for synthetic amorphous silica. *See* Cabot Corporation, GRN No.000321 (filed Feb. 18, 2010), available at: http://www.accessdata.fda.gov/scripts/fcn/fcnDetailNavigation.cfm?rpt=grasListing&id=321.

synthetic amorphous silica in food as described in this GRAS Notification is exempt from the requirement of premarket approval as set forth in the Federal Food, Drug, and Cosmetic Act.

The analytical data, published studies, and information that are the basis for this GRAS determination are available for FDA review and copying at reasonable times at Keller and Heckman LLP, 1001 G Street, NW, Suite 500W, Washington, DC 20001, or will be sent to FDA upon request.

We look forward to FDA's review of this submission and would be happy to provide Agency officials with any information they may need to complete their assessment. Thank you for your attention to this matter.

Sincerely,

(b) (6)

Frederick A. Stearns

Enclosure

DEPARTMENT OF HEALTH AND HUMAN SERVICES
Food and Drug Administration

GENERALLY RECOGNIZED AS SAFE (GRAS) NOTICE

Form Approved: OMB No. 0910-0342; Expiration Date: 02/29/2016
(See last page for OMB Statement)

FDA USE ONLY

GRN NUMBER	DATE OF RECEIPT
000554	
ESTIMATED DAILY INTAKE	INTENDED USE FOR INTERNET
NAME FOR INTERNET	
KEYWORDS	

Transmit completed form and attachments electronically via the Electronic Submission Gateway *(see Instructions)*; OR Transmit completed form and attachments in paper format or on physical media to: Office of Food Additive Safety *(HFS-200)*, Center for Food Safety and Applied Nutrition, Food and Drug Administration, 5100 Paint Branch Pkwy., College Park, MD 20740-3835.

PART I – INTRODUCTORY INFORMATION ABOUT THE SUBMISSION

1. Type of Submission *(Check one)*

☒ New ☐ Amendment to GRN No. _____ ☐ Supplement to GRN No. _____

2. ☐ All electronic files included in this submission have been checked and found to be virus free. *(Check box to verify)*

3a. For New Submissions Only: Most recent presubmission meeting *(if any)* with FDA on the subject substance *(yyyy/mm/dd)*: _____

3b. For Amendments or Supplements: Is your amendment or supplement submitted in response to a communication from FDA? *(Check one)* ☐ Yes ☐ No If yes, enter the date of communication *(yyyy/mm/dd)*: _____

PART II – INFORMATION ABOUT THE NOTIFIER

1a. Notifier

Name of Contact Person	Position
Cynthia Llames	Regulatory Manager

Company *(if applicable)*: Evonik Corporation

Mailing Address *(number and street)*: 299 Jefferson Road

City	State or Province	Zip Code/Postal Code	Country
Parsippany	New Jersey	07054	United States of America

Telephone Number	Fax Number	E-Mail Address
973-929-8569	973-929-8502	cynthia.llames@evonik.com

1b. Agent or Attorney *(if applicable)*

Name of Contact Person	Position
Frederick A. Stearns	Partner

Company *(if applicable)*: Keller and Heckman LLP

Mailing Address *(number and street)*: 1001 G Street NW

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City	State or Province	Zip Code/Postal Code	Country
Washington	District of Columbia	20001	United States of America

Telephone Number	Fax Number	E-Mail Address
202-434-4288	202-434-4646	stearns@khlaw.com

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PART III – GENERAL ADMINISTRATIVE INFORMATION

1. Name of Substance

Synthetic amorphous silica

2. Submission Format: *(Check appropriate box(es))*

☐ Electronic Submission Gateway

☒ Paper

☐ Electronic files on physical media with paper signature page

If applicable give number and type of physical media

3. For paper submissions only:

Number of volumes 1

Total number of pages 19

4. Does this submission incorporate any information in FDA's files by reference? *(Check one)*

☐ Yes *(Proceed to Item 5)* ☒ No *(Proceed to Item 6)*

5. The submission incorporates by reference information from a previous submission to FDA as indicated below *(Check all that apply)*

☐ a) GRAS Notice No. GRN _____

☐ b) GRAS Affirmation Petition No. GRP _____

☐ c) Food Additive Petition No. FAP _____

☐ d) Food Master File No. FMF _____

☐ e) Other or Additional *(describe or enter information as above)* _____

6. Statutory basis for determination of GRAS status *(Check one)*

☒ Scientific Procedures *(21 CFR 170.30(b))* ☐ Experience based on common use in food *(21 CFR 170.30(c))*

7. Does the submission (including information that you are incorporating by reference) contain information that you view as trade secret or as confidential commercial or financial information?

☐ Yes *(Proceed to Item 8)*

☒ No *(Proceed to Part IV)*

8. Have you designated information in your submission that you view as trade secret or as confidential commercial or financial information *(Check all that apply)*

☐ Yes, see attached Designation of Confidential Information

☐ Yes, information is designated at the place where it occurs in the submission

☐ No

9. Have you attached a redacted copy of some or all of the submission? *(Check one)*

☐ Yes, a redacted copy of the complete submission

☐ Yes, a redacted copy of part(s) of the submission

☐ No

PART IV – INTENDED USE

1. Describe the intended use of the notified substance including the foods in which the substance will be used, the levels of use in such foods, the purpose for which the substance will be used, and any special population that will consume the substance *(e.g., when a substance would be an ingredient in infant formula, identify infants as a special population).*

Synthetic amorphous silica is intended to be used as a direct additive in a variety of processed foods and beverages (specified in the Notification with reference to Table 3 in the Codex Alimentarius General Standard for Food Additives) at a maximum level of 2% in the finished food. The substance will function as an anticaking agent; defoaming agent; stabilizer; adsorbent; carrier; conditioning agent; chillproofing agent; filter aid; emulsifying agent; viscosity control agent; and anti-settling agent.

Synthetic amorphous silica also is intended to be used as an indirect additive in the manufacture of adhesives; coatings; defoaming agents; greases and lubricants; paper and paperboard; and polymers that are then used as components of food-packaging materials.

2. Does the intended use of the notified substance include any use in meat, meat food product, poultry product, or egg product? *(Check one)*

☐ Yes ☒ No

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PART V – IDENTITY

1. Information about the Identity of the Substance

	Name of Substance[1]	Registry Used *(CAS, EC)*	Registry No.[2]	Biological Source *(if applicable)*	Substance Category *(FOR FDA USE ONLY)*
1	Synthetic amorphous silica	CAS No. 7631-86-9			
2					
3					

[1] Include chemical name or common name. Put synonyms *(whether chemical name, other scientific name, or common name)* for each respective item (1- 3) in Item 3 of Part V *(synonyms)*

[2] Registry used e.g., CAS *(Chemical Abstracts Service)* and EC *(Refers to Enzyme Commission of the International Union of Biochemistry (IUB), now carried out by the Nomenclature Committee of the International Union of Biochemistry and Molecular Biology (IUBMB))*

2. Description

Provide additional information to identify the notified substance*(s)*, which may include chemical formula*(s)*, empirical formula*(s)*, structural formula*(s)*, quantitative composition, characteristic properties *(such as molecular weight(s))*, and general composition of the substance. For substances from biological sources, you should include scientific information sufficient to identify the source *(e.g., genus, species, variety, strain, part of a plant source (such as roots or leaves), and organ or tissue of an animal source), and include any known toxicants that* could be in the source.

Synthetic amorphous silica (CAS No. 7631-86-9) can be divided into two categories depending on whether the manufacturing process involves a wet route (precipitated silica, silica gel) or a thermal route (pyrogenic silica). Colloidal silicas (silica sols) are stable dispersions of synthetic amorphous silicas in a liquid, usually water. Furthermore, synthetic amorphous silicas, which are generally hydrophilic, may be rendered hydrophobic by surface treatment. Synthetic amorphous silicas exist as highly pure, white, fluffy powders or milky-white dispersions of these powders in fluids (usually water).

All synthetic amorphous silica products covered by this GRAS Notification meet the Food Chemicals Codex (FCC) 9th edition monograph for silicon dioxide (INS 551).

3. Synonyms

Provide as available or relevant:

1	silica
2	silicon dioxide
3	

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PART VI – OTHER ELEMENTS IN YOUR GRAS NOTICE

(check list to help ensure your submission is complete – check all that apply)

- [x] Any additional information about identity not covered in Part V of this form
- [x] Method of Manufacture
- [x] Specifications for food-grade material
- [x] Information about dietary exposure
- [x] Information about any self-limiting levels of use *(which may include a statement that the intended use of the notified substance is not-self-limiting)*
- [ ] Use in food before 1958 *(which may include a statement that there is no information about use of the notified substance in food prior to 1958)*
- [x] Comprehensive discussion of the basis for the determination of GRAS status
- [x] Bibliography

Other Information

Did you include any other information that you want FDA to consider in evaluating your GRAS notice?

- [ ] Yes
- [x] No

[illegible]

- [ ] [illegible]
- [ ] [illegible]

PART VII – SIGNATURE

1. The undersigned is informing FDA that Evonik Corporation *(name of notifier)*

has concluded that the intended use(s)of Synthetic amorphous silica *(name of notified substance)*

described on this form, as discussed in the attached notice, is (are) exempt from the premarket approval requirements of section 409 of the Federal Food, Drug, and Cosmetic Act because the intended use(s) is (are) generally recognized as safe.

2. [x] Evonik Corporation *(name of notifier)* agrees to make the data and information that are the basis for the determination of GRAS status available to FDA if FDA asks to see them.

Evonik Corporation *(name of notifier)* agrees to allow FDA to review and copy these data and information during customary business hours at the following location if FDA asks to do so.

Keller and Heckman LLP, 1001 G Street NW, Suite 500W, Washington, DC 20001 *(address of notifier or other location)*

Evonik Corporation *(name of notifier)* agrees to send these data and information to FDA if FDA asks to do so.

OR

- [ ] The complete record that supports the determination of GRAS status is available to FDA in the submitted notice and in GRP No. _____ *(GRAS Affirmation Petition No.)*

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3. Signature of Responsible Official, Agent, or Attorney	Printed Name and Title	Date (mm/dd/yyyy)
(b) (6)	Frederick A. Stearns (Attorney)	11/07/2014

PART VIII – LIST OF ATTACHMENTS

List your attached files or documents containing your submission, forms, amendments or supplements, and other pertinent information. Clearly identify the attachment with appropriate descriptive file names (or titles for paper documents), preferably as suggested in the guidance associated with this form. Number your attachments consecutively. When submitting paper documents, enter the inclusive page numbers of each portion of the document below.

Attachment Number	Attachment Name	Folder Location (select from menu) (Page Number(s) for paper Copy Only)
	Cover letter to Antonia Mattia	N/A
	GRAS Notification for Synthetic Amorphous Silica	Pages 1-16
	Appendix (FCC monograph for silicon dioxide)	Pages 17-18
	References	Page 19

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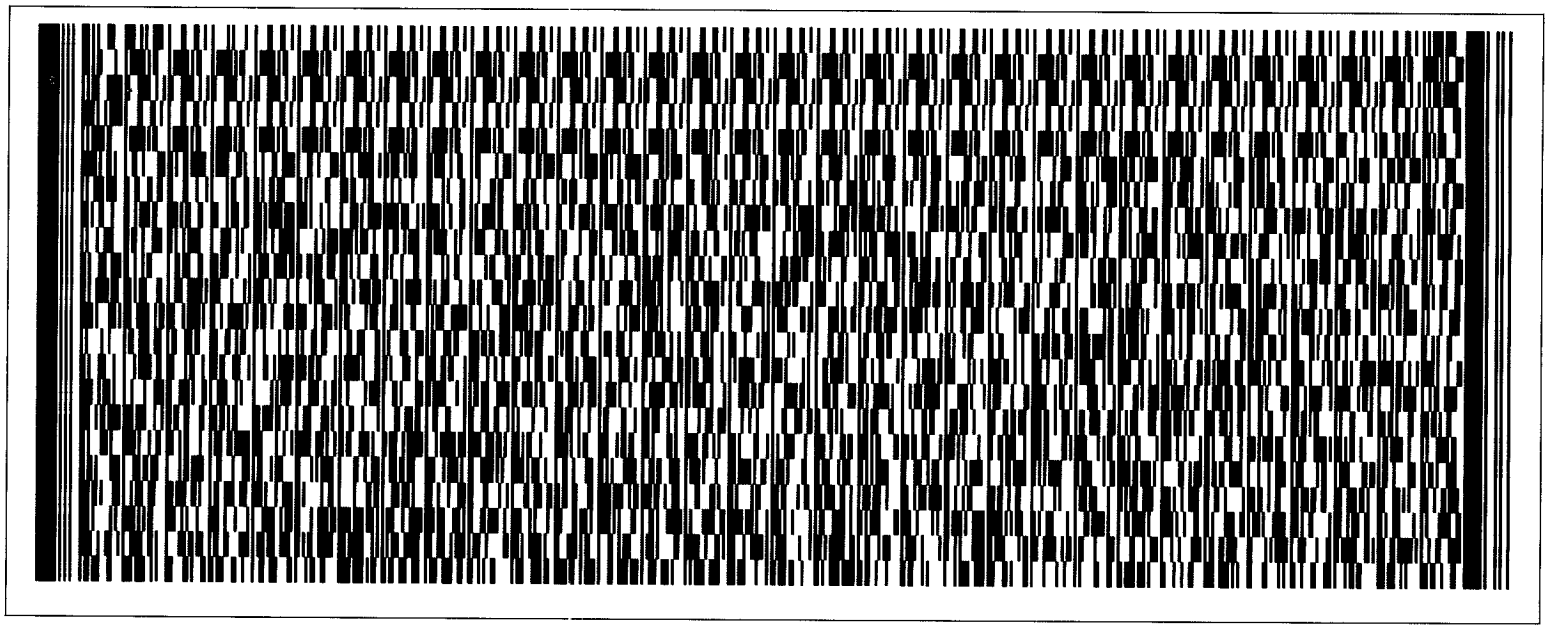
OMB Statement: Public reporting burden for this collection of information is estimated to average 150 hours per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to: Department of Health and Human Services,Food and Drug Administration, Office of Chief Information Officer, 1350 Piccard Drive, Room 400, Rockville, MD 20850. (Please do NOT return the form to this address.). An agency may not conduct or sponsor, and a person is not required to respond to, a collection of information unless it displays a currently valid OMB control number.



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GRAS Notification for Synthetic Amorphous Silica

Prepared for:	U.S. Food and Drug Administration Office of Food Additive Safety (HFS-200) Center for Food Safety and Applied Nutrition 5100 Paint Branch Parkway College Park, MD 20740-3835
Name of Notifier:	Evonik Corporation
Mailing Address:	All communications on this matter are to be sent to Counsel for the Notifier: Frederick A. Stearns Keller and Heckman LLP 1001 G Street, NW Suite 500W Washington, DC 20001 (202) 434-4288
Name of Substance and Intended Use:	Synthetic amorphous silica as a multipurpose direct and indirect additive up to a maximum level of 2% in finished products
Dated:	November 7, 2014

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Figure 1 Overview of the Various Forms of Silica

I. Introduction

Keller and Heckman LLP submits the enclosed information on behalf of our client, Evonik Corporation (Evonik), in support of this notification that synthetic amorphous silica is generally recognized as safe (GRAS) for use in multiple food applications. The products subject to this notification are SIPERNAT® and hydrophilic AEROSIL® products. We refer to these products collectively as "synthetic amorphous silica" throughout the document.

Synthetic amorphous silica is intended for use both in direct and indirect food additive applications.

Evonik has determined that synthetic amorphous silica is GRAS based on scientific procedures in accordance with 21 C.F.R. § 170.30(b) and in conformance with the guidance issued by the Food and Drug Administration (FDA) under proposed 21 C.F.R. § 170.36, 62 Fed. Reg. 18938 (Apr. 17, 1997).

In support of our GRAS notification, we submit information in the following areas:

- Identity and specifications for synthetic amorphous silica;
- Manufacturing process for synthetic amorphous silica;
- Comparability of Evonik's synthetic amorphous silica to the subject of GRN000321, for which FDA had no questions regarding GRAS status;
- Existing U.S. regulatory clearances for silica substances (both by FDA and the Environmental Protection Agency (EPA));
- Scientific safety evaluations on silica substances;
- Safety studies conducted on synthetic amorphous silica; and
- The intended uses and dietary exposure assessment for synthetic amorphous silica.

II. Administrative Information

A. Claim Regarding GRAS Status

Evonik hereby notifies the agency of its determination that synthetic amorphous silica is GRAS based on scientific procedures for use in direct and indirect food additive applications in certain specific categories of food.

B. Name and Address of the Notifier

Evonik Corporation
299 Jefferson Road
Parsippany, NJ 07054-0677

All communications on this matter are to be sent to Counsel for the Notifier:

Frederick A. Stearns
Keller and Heckman LLP
1001 G Street, NW Suite 500W
Washington, DC 20001
Telephone: (202) 434-4288
Facsimile: (202) 434-4646
Email: Stearns@khlaw.com

C. Common or Usual Name of GRAS Substance

The common or usual name for the GRAS substance is "synthetic amorphous silica" or "silicon dioxide, chemically prepared." All substances subject to this notification are chemically identical forms of silicon dioxide.

D. Intended Use of GRAS Substance

When directly added to food, synthetic amorphous silica is used at a maximum level of 2% in finished food products as an anticaking agent; defoaming agent; stabilizer; adsorbent; carrier; conditioning agent; chillproofing agent; filter aid; emulsifying agent; viscosity control agent; and anti-settling agent.

When used as an indirect additive, synthetic amorphous silica may be used in the manufacture of adhesives; coatings; defoaming agents; greases and lubricants; paper and paperboard; and polymers that are then used as components of food-packaging materials.

All segments of the population may have dietary exposure to synthetic amorphous silica from its direct use in food and/or by migration from impregnated food-packaging materials into food.

E. Self-Limiting Levels of Use

The use of synthetic amorphous silica as a direct food additive is limited to a maximum level of 2% in finished food products. As an indirect additive, it will be used only in amounts reasonably necessary to produce the intended effect in the manufacture of adhesives; coatings; defoaming agents; greases and lubricants; paper and paperboard; and polymers that are then used as components of food-packaging materials.

F. Statement of Availability of Data and Information

The analytical data, published articles, and information that are the basis for this GRAS determination are available for review and copying by FDA at reasonable times at Keller and Heckman LLP, 1001 G Street, NW, Suite 500W, Washington, DC 20001. These documents will be sent to FDA upon request.

III. Product Identity and Specifications

A. Product Identification

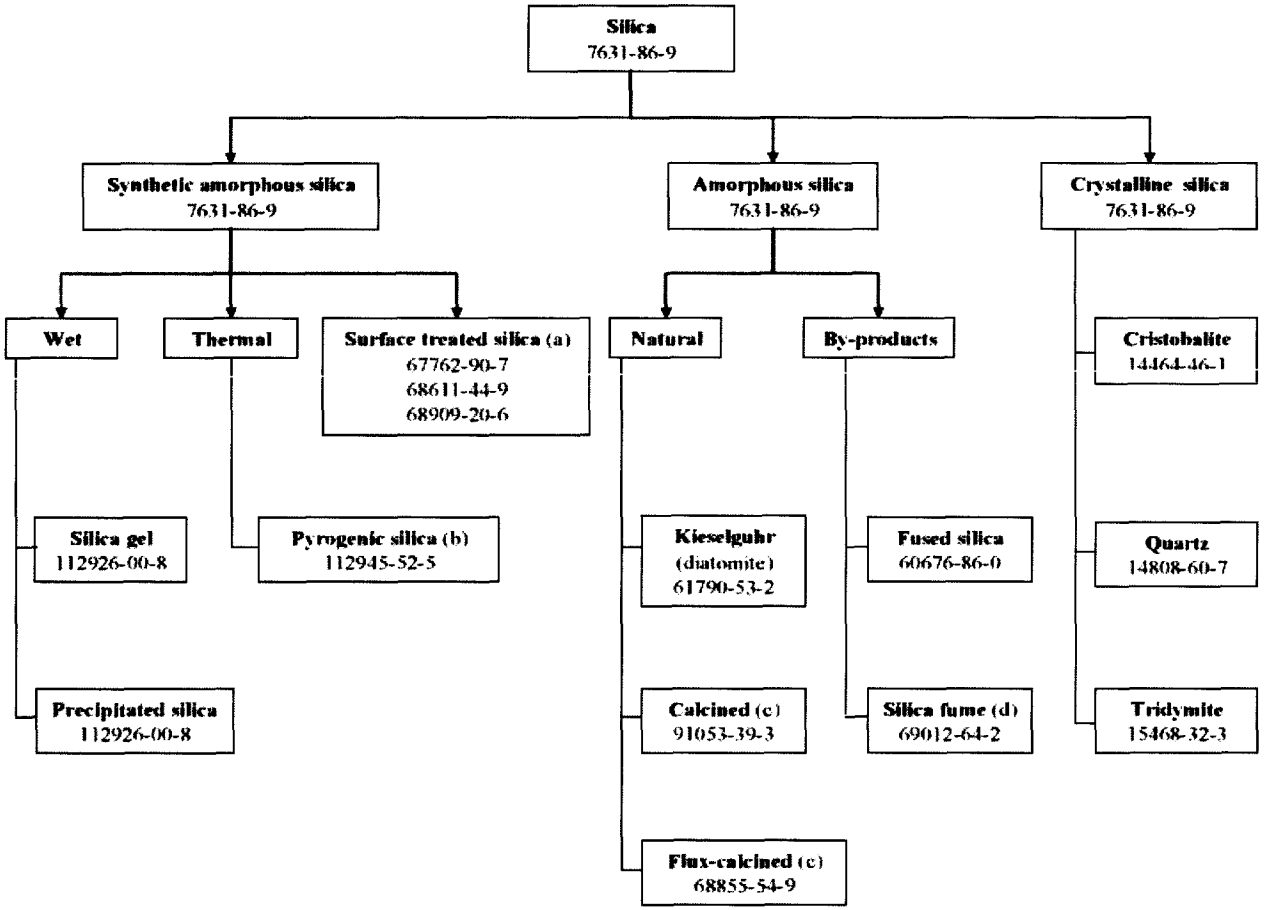
Synthetic amorphous silica is chemically identical to silicon dioxide (SiO_2) (which is also called "silica"), a term which includes both naturally occurring and synthetic silicas. Naturally occurring silicon dioxide has a crystalline structure whereas synthetic amorphous silica is non-crystalline.

Synthetic amorphous silica (CAS No. 7631-86-9) can be divided into two categories depending on whether the manufacturing process involves a wet route (precipitated silica, silica gel) or a thermal route (pyrogenic silica). Colloidal silicas (silica sols) are stable dispersions of synthetic amorphous silicas in a liquid, usually water. Furthermore, synthetic amorphous silicas, which are generally hydrophilic, may be rendered hydrophobic by surface treatment. Synthetic amorphous silicas exist as highly pure, white, fluffy powders or milky-white dispersions of these powders in fluids (usually water).

Figure 1 below provides an overview of the many forms of silica:[1]

[1] *Synthetic Amorphous Silica* (CAS No. 7631-86-9). European Centre for Ecotoxicology and Toxicology of Chemicals (ECETOC), JACC No. 51. ISSN-0773-6339-51. Brussels, Sept. 2006, available at: http://members.ecetoc.org/Documents/Document/JACC%20051.pdf.

Figure 1: Overview of the Various Forms of Silica



(a) All forms of SAS can be surface-treated either physically or chemically; most common treating agents are organosilicon compounds (Appendix B: Table B.2)

(b) Pyrogenic silica is also known as fumed silica in the English speaking countries

(c) Partial transformation into cristobalite

(d) By-product from electrical furnace

B. Product Specifications

Synthetic amorphous silica products are highly pure substances that meet the Food Chemicals Codex (FCC) (9th edition) monograph for Silicon Dioxide (INS 551) (attached to this notification as an Appendix).[2]

The solid powder forms of synthetic amorphous silica do not exist as nanoparticles (nanoparticles are particles with a diameter of < 100 nm). Synthetic amorphous silica products exist as aggregates or grow further to form agglomerates. The mean diameter is typically in the micro (μm) and millimeter (mm) scale range and far above 100 nm.

IV. Manufacturing Process

A. Manufacturing Process for SIPERNAT® Products

The production process for SIPERNAT® precipitated synthetic amorphous silica can be divided into the following general unit operations: raw material storage, synthesis, washing/solid-liquid filtration, drying, packaging, and storage. Optionally after the drying step, the product can be milled or granulated.

The primary raw material is water glass (CAS# 1344-09-8; $Na_2O_nSiO_2$, n=2 to 4), which is an aqueous sodium silicate solution. Industrial production occurs in two stages. In the first stage, water glass cullets are produced from sand and soda ash by melting both components at approximately 1300°C in a glass furnace. In the second stage, these cullets are then dissolved in an autoclave with water, producing water glass. Water glass is a colorless, odorless, non-flammable, strongly alkaline liquid (approximate pH 11). It is characterized by its molar ratio of SiO_2 and Na_2O, as well as by the concentration of these components.

During the precipitation process, water glass is reacted with sulfuric acid to form precipitated silica according to the equation below.

$$(nNa_2O.xSiO_2) + nH_2SO_4 \rightarrow nNa_2SO_4 + xSiO_2$$

Reaction conditions such as temperature (40 – 95°C), pH (4.5 – 12.5), flows, residence time, mixing energy, and reactor geometry are selected based on the intended application of the precipitated silica. The solid content of the precipitation step is typically between 50 and 200 g/L.

Next, the suspension produced in the precipitation step is filtered using a filter press, membrane filter press, or belt/drum filter depending on the properties of silica produced. The solid content of the filter cake is typically 15 to 35% by weight. After the initial filtration, the product is washed to remove salts. Washing is normally performed in the filtration equipment.

---

[2] Evonik has performed analytical testing on product batches to demonstrate conformity to the FCC monograph. Such data are available to FDA upon request.

Following filtration and washing, the silica is dried in a process dependent on the desired characteristics of the final product. Belt dryers, turbine dryers, recycle dryers, rotary drum dryers, and spray dryers may be used depending on the final characteristics. This step produces a finely divided powder with excellent absorption and free flow properties.

After drying, the product may be milled; the type of milling performed depends on the properties required for a particular product application. During this process, the particle size distribution and sieve residue characteristics of the product are modified.

For certain applications, a dust free product is required. To fulfill this requirement, the spray dried or milled product is granulated.

B. Manufacturing Process for AEROSIL® Products

The manufacture of AEROSIL® pyrogenic synthetic amorphous silicon dioxide and/or AEROSIL® colloidal silicon dioxide (fumed silica) occurs by hydrolysis of a volatile silane compound in an oxygen-hydrogen gas flame.

The raw material $SiCl_4$ (or as a mixture with other chlorosilanes of alkylchlorosilane) is mixed in gaseous form with air and hydrogen and is burned in a flame. The following chemical reaction takes place:

$$SiCl_4 + 2\ H_2O + O_2 \rightarrow SiO_2 + 4\ HCl$$

The result is silicon dioxide with an extremely fine particle size, which is characteristic of this process. The reaction parameters must be kept under strict control (air rate, temperature, $SiCl_4$ rate, hydrogen rate, etc.) in order to achieve uniform quality. It is possible to obtain variations of the product properties by appropriately controlled modifications to the reaction conditions.

All raw materials used are of high purity and synthetic origin. No raw materials of animal or plant origin and no organic solvents are used in the manufacture of these products.

The AEROSIL® agglomerates inside the cooling coil and the gas/solid mixture are separated at the cyclone system. Hydrophilic AEROSIL® pyrogenic synthetic amorphous silicon dioxide types and hydrophilic AEROSIL® colloidal silicon dioxide types are transferred from the cyclones to the deacidifier to remove HCl from the powder. Hydrophobic AEROSIL® pyrogenic synthetic amorphous silicon dioxide types are additionally treated with alkylchlorosilane at the hydrophobation unit. The powder is pneumatically conveyed into the final product silo. Undensified or densified AEROSIL® pyrogenic synthetic amorphous silicon dioxide and AEROSIL® colloidal silicon dioxide is then bagged, filled into containers, or loaded into silo cars.

HCl is removed from the offgas stream in the absorption unit and re-used in the production of more silicon dioxide.

V. Basis for GRAS Determination

As discussed in greater detail below, abundant support exists for the safety and GRAS status of synthetic amorphous silica for use in direct and indirect food additive applications.

A. Regulatory Status of Synthetic Amorphous Silica

1. Existing GRAS Notice for Synthetic Amorphous Silica

On August 18, 2010, FDA issued a letter stating that it had no questions regarding the GRAS status of synthetic amorphous silica for use in various food categories at levels up to 2% and as an indirect additive in food-packaging materials.[3] FDA issued this letter in response to GRAS Notice No. GRN000321 filed by Cabot Corporation for its synthetic amorphous silica.[4] Evonik's synthetic amorphous silica is chemically identical to the substance that was the subject of GRN000321, and we anticipate that FDA will similarly have no questions regarding the GRAS determination discussed herein.

2. FDA Clearances for Silica in Food

As described above, silicon dioxide (or silica) takes many forms, including that of synthetic amorphous silica. The safety of various forms of silica is already well-established, as shown by the fact that FDA has cleared silicon dioxide and various silicates for a wide range of direct and indirect food additive uses. For example, silicon dioxide may be used as an anti-caking agent in accordance with 21 C.F.R. § 172.480, and as a defoaming agent in accordance with 21 C.F.R. § 173.340. Silica gels with a minimum silica content of 89.5% are considered GRAS when used as anti-foaming agents in accordance with good manufacturing practice (21 C.F.R. § 182.1711). Additionally, silicon dioxides are considered GRAS as substances migrating from paper and paperboard products used in food packaging (21 C.F.R. § 182.90).

Table 1 below lists current FDA clearances for silica substances as direct and indirect food additives, all of which lend support to the current determination that silicon dioxide in the form of synthetic amorphous silica is GRAS.

Table 1: Current FDA Clearances for Silica Substances as Direct and Indirect Food Additives

21 CFR Reference	Direct Food Additive Clearances	Silica Form
160.105	Anti-caking in dried eggs	Silicon dioxide
160.185	Anti-caking in dried egg yolks	Silicon dioxide
172.230(a)	Microencapsulation of flavoring oils	Silicon dioxide
172.480	Principal section, free-flow and anti-caking in	Silicon dioxide

[3] Agency Response Letter, GRAS Notice No. GRN 000321 (Aug. 18, 2010), available at: http://www.fda.gov/Food/IngredientsPackagingLabeling/GRAS/NoticeInventory/ucm225016.htm.

[4] Cabot Corporation, GRN No.000321 (filed Feb. 18, 2010), available at: http://www.accessdata.fda.gov/scripts/fcn/fcnDetailNavigation.cfm?rpt=grasListing&id=321.

	foods	
173.340(a)	Defoamer agents	Silicon dioxide
182.1711	Dietary supplements	Silica aerogel
73.1	Diluents in color additive mixtures for food use exempt from certification (not more than 2% of the ink solids)	Silicon dioxide
73.575(a)(2)	Titanium dioxide color additive mixtures for food use exempt from certification (not more than 2% total)	Silicon dioxide
	Indirect Food Additive Clearances	
175.105	Adhesives	Silicon dioxide
175.300	Resinous and polymeric coatings	Silicones and silane coupled silica
175.320	Resinous and polymeric coatings for polyolefin films	Silioxanes and silicones
175.350	Vinyl acetate/crotonic acid copolymer	Silica
175.380	Xylem-formaldehyde resins	Silane coupled silica and silicones as adjuvants
175.390	Zinc-silicon dioxide matrix coatings	Zinc-silicon dioxide; silica gel; and sodium silicate
176.170	Components of paper and paperboard in contact with aqueous and fatty foods	Silica; silicate and other silica compounds; siloxanes and silicones
176.180	Components of paper and paperboard on contact with dry food	Sodium methyl siliconate
176.200	Defoaming agents used in coatings	Silica
176.210	Defoaming agents used in the manufacture of paper and paperboard	Silica; siloxanes and silicones
177.1200	Cellophane	Silica; silicic acid; sodium silicate; and aluminum silicate
177.1210	Closure with sealing gaskets for food containers	Substances that are subject to regulations in part 174, 175, 176, 177, 178, and 179.45
177.1350	Ethylene-vinyl acetate copolymers	Substances the use of which is permitted under applicable regulations in parts 170 through 189
177.1400	Hydroxyethyl cellulose film, water-insoluble	Silica; silicic acid; sodium silicate; and aluminum silicate (by reference to § 177.1200(c))

177.2250	Filters, microporous polymeric	Silicon dioxide
177.2420	Polyester resins, cross-linked	Silicon dioxide
177.2600	Rubber articles intended for repeated use	Silica; silicone; silicone elastomers; and aluminum silicate
178.3297	Colorants for polymers	Aluminum and potassium silicate; calcium silicate; magnesium silicate; silica; and titanium dioxide-magnesium silicate
178.3570	Lubricants with incidental food contact	Dialkyldimethylammonium aluminum silicate
182.90	Substances migrating to food from paper and paperboard products	Silicon dioxides and sodium silicate

Additionally, the Select Committee on GRAS Substances (SCOGS) evaluated the safety of dietary exposure to silicates (including silicon dioxide) and published a report in 1979 entitled "Evaluation of the Health Aspects of Certain Silicates as Food Ingredients."[5] The SCOGS report concluded that there was no evidence in the available literature that demonstrated or suggested reasonable grounds to suspect a hazard to the public when various silicates (including silicon dioxide) were used at levels that were then-current or that might reasonably be expected in the future.

3. EPA Clearances for Silica in Food

EPA has established exemptions from tolerance for various silicas in 40 C.F.R. § 180.950, which lists tolerance exemptions for minimal risk active and inert ingredients. Unless specifically excluded, residues resulting from the use of the listed substances as either inert or active ingredients in a pesticide chemical formulation, including antimicrobial pesticide chemicals, are exempt from the requirement of a tolerance under section 408 of the Federal Food, Drug, and Cosmetic Act if such use is in accordance with good agricultural or manufacturing practices. Accordingly, the list in 40 C.F.R. § 180.950 may be considered equivalent to a GRAS list. Table 2 below describes the list of silica substances included in 40 C.F.R. § 180.950.

Table 2: Tolerance Exemptions for Silica Substances in 40 C.F.R. § 180.950

Silica, amorphous, fumed (crystalline free)	112945-52-5
Silica, amorphous, precipitated and gel	7699-41-4
Silica gel	63231-67-4
Silica gel, precipitated, crystalline-free	112926-00-8

[5] Select Committee on GRAS Substances (SCOGS). Evaluation of the Health Aspects of Certain Silicates as Food Ingredients (1979).

Silica, hydrate	10279-57-9
Silica, vitreous	60676-86-0

Due to their negligible toxicity, various silica compounds are exempt from the requirement for tolerance when applied to growing crops and raw agricultural commodities after harvest[6] and when applied to animals,[7] provided that they are used in accordance with good agricultural practice.

The clearances described above, all of which contribute to human dietary exposure to silica, support the view that synthetic amorphous silica, when used as intended, is GRAS.

4. Additional Evaluations of Silica

A number of agencies have reviewed the human health effects of silica, including the carcinogenicity of synthetic amorphous silica. Silica is not listed as carcinogenic or hazardous by the International Agency for Research on Cancer (IARC) or by the American Conference for Governmental Industrial Hygienists (ACGIH).[8]

In the European Union, silicon dioxide is permitted as a food additive in a wide range of foods.[9] In 1990, the Scientific Committee on Food (SCF) established a group Acceptable Daily Intake (ADI) "not specified" for silicon dioxide. The Joint FAO/WHO Expert Committee on Food Additives (JECFA) evaluated the use of amorphous silica, calcium silicate, and sodium alumino-silicate in food and concluded that their acceptable use is "not limited except for good manufacturing practice."[10] Five years later, JECFA re-evaluated silicon dioxide and a number of

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6 40 C.F.R. §§ 180.910, 180.1017.

7 40 C.F.R. §§ 180.930, 180.1017.

8 *See Synthetic Amorphous Silica* (CAS No. 7631-86-9). European Centre for Ecotoxicology and Toxicology of Chemicals (ECETOC), JACC No. 51. ISSN-0773-6339-51. Brussels, Sept. 2006, available at: http://members.ecetoc.org/Documents/Document/JACC%20051.pdf.

9 Scientific Opinion of the Panel on Food Additives and Nutrient Sources added to Food on calcium silicate, silicon dioxide and silicic acid gel added for nutritional purposes to food supplements following a request from the European Commission. *The EFSA Journal*. (2009): 1132, 1-24, available at: http://www.efsa.europa.eu/en/scdocs/doc/ans_ej1132_Inorganicsiliconsources_op_en.pdf. *See also* Regulation (EU) No. 1129/2011 of 11 November 2011, which took effect June 1, 2013 and established a Union list of food additives. OJ EC No. L 295/1 (12/11/2011), available at: http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2011:295:0001:0177:EN:PDF.

10 WHO, 1969. Silicon Dioxide Amorphous, Calcium, Magnesium, and Sodium Alumino Silicates. Thirteenth report of the Joint FAO/WHO Expert Committee on Food Additives, FAO Nutrition Meetings Report Series; Wld. Hlth. Org. techn. Rep. Ser., available at: http://www.inchem.org/documents/jecfa/jecmono/v46aje60.htm. JECFA develops an ADI "not specified" or "not limited" for food substances of very low toxicity for which the available data (chemical, biological, toxicological, and other) indicate that the total dietary intake of the substance based on its use at levels necessary to achieve the desired effects and from its background levels in food do not represent a hazard to health. For this reason, and for reasons specified in individual evaluations, the establishment of a numeric ADI is not deemed necessary.

silicates, and determined that a human ADI was not limited, except for magnesium silicate and talc.[11]

The European Centre for Ecotoxicology and Toxicology of Chemicals (ECETOC) Joint Assessment of Commodity Chemicals (JACC) programme evaluated the physico-chemical properties, toxicology, ecotoxicology and environmental fate and impact of (non-crystalline) synthetic amorphous silica.[12] The Organization for Economic Co-operation and Development (OECD) Screening Information Data Set (SIDS) also performed a comprehensive toxicological evaluation of multiple forms of silica.[13] Both reports confirm the safety of silicates and provide further support that silica is GRAS when added either directly or indirectly to food.

B. Safety Overview and Common Use of Silica in Food

1. Silica in Food

Silicon ranks next to oxygen in abundance in the Earth's crust, where it takes the form of silicate minerals. It is also present as the oxide (silica) in soils/sediments, in some organisms such as diatoms, and in some plants. Silica is also a natural element found in food. It occurs as a trace element in the human biological system, both in soft tissues and in bones.

Silicon dioxide and silicates have a long history of use in food without detrimental effects. Silicon is used in consumer products, including cosmetics, pharmaceuticals and foods. Average daily intakes of silica range from 43 to 107 mg SiO_2/d, with the lower values for animal based diets and the highest values for plant based diets. Foods with high silica content include grains, especially oats, barley, and some rice fractions.[14] As shown by the FDA clearances above, silica is incorporated into a variety of food products (such as beverage mixes, salad dressing, sauces, gravy mixes, seasoning mixes, soups, spices, snack food, sugar substitutes and desserts) as an anti-caking agent, thickener, and absorber, at levels typically up to 2% by weight. Silica is also used as an anti-caking agent and as an excipient in pharmaceuticals for various drug and vitamin preparations.[15]

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[11] WHO, 1974. Silicon Dioxide and Certain Silicates. Seventeenth Report of the Joint FAO/WHO Expert Committee on Food Additives, Wld. Hlth. Org. techn. Rep. Ser. No. 539, available at: http://www.inchem.org/documents/jecfa/jecmono/v05je04.htm.

[12] *See Synthetic Amorphous Silica* (CAS No. 7631-86-9). European Centre for Ecotoxicology and Toxicology of Chemicals (ECETOC), JACC No. 51. ISSN-0773-6339-51. Brussels, Sept. 2006, available at: http://members.ecetoc.org/Documents/Document/JACC%20051.pdf.

[13] OECD High Production Volume Chemicals Programme, SIDS Dossier, synthetic amorphous silica and silicates. Berlin, Germany, 19-22 Oct. 2004, available at: http://www.chem.unep.ch/irptc/sids/oecdsids/Silicates.pdf.

[14] Pennington, JA. "Silicon in foods and diets." *Food Additives and Contaminants*. 8.1 (1991): 97-118.

[15] Silica, some silicates, coal dust and para-aramid fibrils. *IARC monographs on the evaluation of carcinogenic risks to humans* 68 (1997): 41-242, available at: http://monographs.iarc.fr/ENG/Monographs/vol68/mono68.pdf.

2. Safety Studies on Synthetic Amorphous Silica

As stated above, in 2006, ECETOC issued a comprehensive review of the safety of synthetic amorphous silica, including discussions of significant sources of peer-reviewed, published data.[16] The results of acute oral toxicity studies indicate a very low order of toxicity for synthetic amorphous silica; no deaths and no signs of toxicity were observed at doses of up to 5,000 mg SiO_2/kg bw. No difference was found between LD_{50} values for the various types of synthetic amorphous silica studied.

The data available regarding repeat dose toxicity confirm the absence of significant toxicity for synthetic amorphous silica by oral routes of exposure. A number of repeat dose oral toxicity studies were conducted in rats and mice. The toxicity of a precipitated synthetic amorphous silica product (trade name, SIPERNAT® 22) was evaluated in a 13-week feeding study in Wistar rats, which received synthetic amorphous silica in the daily diet at 0 (control), 0.5, 2, and 8% (approx. 0, 250, 1,000, and 4,000 mg/kg bw/d). General condition, behavior, survival, body weights, water intake and hematological, and urinary parameters were not adversely affected at any dose. Increased food intake associated with decreased food efficiency occurred in the highest-dose group. These changes were attributed to the high amount of (inert) synthetic amorphous silica in the diet. Other slight changes were not of toxicological significance. Gross and microscopic pathological examination did not reveal any abnormalities that could be ascribed to the ingestion of for synthetic amorphous silica. The highest dose (8% in the diet or 4,000 mg/kg bw/d) was considered to be the no-observed effect level (NOEL).

Chronic administration of synthetic amorphous silica for up to 24 months at a concentration of up to 5.0% in the diet to mice and rats caused no alteration in survival rate, body weight, food consumption, behavior, organ weights, or blood chemistry. Similar administration did not cause gross or microscopic changes or neoplasms in any examined tissues. The authors of these studies concluded that there were no signs of carcinogenicity and no other significant treatment-related adverse effects. Furthermore, synthetic amorphous silica is non-genotoxic in a variety of *in vitro* and *in vivo* tests. Finally, there were no signs of reproductive or developmental toxicity in studies in rats, mice, hamsters, and rabbits administered silica orally during gestation.

C. Dietary Intake of Silica

As stated above, synthetic amorphous silica is used at a maximum level of 2% in finished food products as an anticaking agent; defoaming agent; stabilizer; adsorbent; carrier; conditioning agent; chillproofing agent; filter aid; emulsifying agent; viscosity control agent; and anti-settling agent. With respect to direct additive uses, Table 3 below identifies various food categories from the current Codex Alimentarius General Standard for Food Additives in which

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[16] *Synthetic Amorphous Silica* (CAS No. 7631-86-9). European Centre for Ecotoxicology and Toxicology of Chemicals (ECETOC), JACC No. 51. ISSN-0773-6339-51. Brussels, Sept. 2006, available at: http://members.ecetoc.org/Documents/Document/JACC%20051.pdf. Approximately one-third of the references cited in this comprehensive review consist of peer-reviewed, published studies. The remaining references include agency and association meta-reviews and industry-generated data and reports.

synthetic amorphous silica may be used.[17] Table 3 below encompasses a broader range of food categories than those included in the previously-filed GRAS Notice for synthetic amorphous silica (GRN000321).[18] When used as an indirect additive, synthetic amorphous silica may be used in the manufacture of adhesives; coatings; defoaming agents; greases and lubricants; paper and paperboard; and polymers that are then used as components of food-packaging materials.

All segments of the population may have dietary exposure to synthetic amorphous silica from its direct use in food and/or by migration from impregnated food- packaging materials into food.

Table 3: Subset of Codex Alimentarius Food Categories Included in the Intended Conditions of Use

Codex Alimentarius Food Category or Subcategory	
Number	Description
01.1.2	Dairy-based drinks, flavoured and/or fermented (e.g., chocolate milk, cocoa, eggnog, drinking yoghurt, whey-based drinks)
01.3	Condensed milk and analogues (plain)
01.4.3	Clotted cream (plain)
01.4.4	Cream analogues
01.5	Milk powder and cream powder and powder analogues (plain)
01.6.5	Cheese analogues
01.7	Dairy-based desserts (e.g., pudding, fruit or flavoured yoghurt)
01.8.1	Liquid whey and whey products, excluding whey cheeses
02.2.2	Fat spreads, dairy fat spreads and blended spreads
02.3	Fat emulsions mainly of type oil-in-water, including mixed and/or flavoured products based on fat emulsions
02.4	Fat-based desserts excluding dairy-based dessert products of food category 01.7
03.0	Edible ices, including sherbet and sorbet
04.1.2	Processed fruits
04.2.2.2	Dried vegetables (including mushrooms and fungi, roots and tubers, pulses and legumes, and aloe vera), seaweeds, and nuts and seeds
04.2.2.3	Vegetables (including mushrooms and fungi, roots and tubers, pulses and legumes, and aloe vera), and seaweeds in vinegar, oil, brine, or soybean sauce
04.2.2.4	Canned or bottled (pasteurized) or retort pouch vegetables (including mushrooms and

[17] Codex Alimentarius Commission, General Standard for Food Additives (GSFA) Online Database: Silicon dioxide, amorphous (551), available at: http://www.codexalimentarius.net/gsfaonline/additives/details.html?id=284.

[18] The additional categories are: (1) semi-preserved fish and fish products (including mollusks, crustaceans, and echinoderms); (2) fully preserved, including canned or fermented fish and fish products (including mollusks, crustaceans, and echinoderms); and (3) yeast and like products.

Table 3: Subset of Codex Alimentarius Food Categories Included in the Intended Conditions of Use

Codex Alimentarius Food Category or Subcategory	
Number	Description
	fungi, roots and tubers, pulses and legumes, and aloe vera), and seaweeds
04.2.2.5	Vegetable (including mushrooms and fungi, roots and tubers, pulses and legumes, and aloe vera), seaweed, and nut and seed purees and spreads (e.g., peanut butter)
04.2.2.6	Vegetable (including mushrooms and fungi, roots and tubers, pulses and legumes, and aloe vera), seaweed, and nut and seed pulps and preparations (e.g., vegetable desserts and sauces, candied vegetables) other than food category 04.2.2.5
04.2.2.8	Cooked or fried vegetables (including mushrooms and fungi, roots and tubers, pulses and legumes, and aloe vera), and seaweeds
05.0	Confectionery
06.3	Breakfast cereals, including rolled oats
06.4.3	Pre-cooked pastas and noodles and like products
06.5	Cereal and starch based desserts (e.g., rice pudding, tapioca pudding)
06.6	Batters (e.g., for breading or batters for fish or poultry)
06.7	Pre-cooked or processed rice products, including rice cakes (Oriental type only)
06.8	Soybean products, excluding soybean-based seasonings and condiments of food category 12.9
07.0	Bakery wares
09.3	Semi-preserved fish and fish products, including mollusks, crustaceans, and echinoderms
09.4	Fully preserved, including canned or fermented fish and fish products, including mollusks, crustaceans, and echinoderms
10.4	Egg-based desserts (e.g., custard)
11.6	Table-top sweeteners, including those containing high-intensity sweeteners
12.2.2	Seasonings and condiments
12.3	Vinegars
12.4	Mustards
12.5	Soups and broths
12.6	Sauces and like products
12.7	Salads (e.g., macaroni salad, potato salad) and sandwich spreads excluding cocoa- and nut-based spreads of food categories 04.2.2.5 and 05.1.3
12.8	Yeast and like products
12.10	Protein products other than from soybeans
13.3	Dietetic foods intended for special medical purposes, excluding products of food

Table 3: Subset of Codex Alimentarius Food Categories Included in the Intended Conditions of Use

Codex Alimentarius Food Category or Subcategory	
Number	Description
	category 13.1
13.4	Dietetic formulae for slimming purposes and weight reduction
13.5	Dietetic foods (e.g., supplementary foods for dietary use) excluding products of food categories 13.1 - 13.4 and 13.6
13.6	Food supplements
14.1.4	Water-based flavoured drinks, including "sport," "energy," or "electrolyte" drinks and particulated drinks
14.2.1	Beer and malt beverages
14.2.2	Cider and perry
14.2.4	Wines (other than grape)
14.2.5	Mead
14.2.6	Distilled spirituous beverages containing more than 15% alcohol
14.2.7	Aromatized alcoholic beverages (e.g., beer, wine and spirituous cooler-type beverages, low alcoholic refreshers)
15.0	Ready-to-eat savouries
16.0	Prepared foods

Due to the ubiquitous occurrence of silicas in the environment and in food, the proposed uses of synthetic amorphous silica in this GRAS notification will not result in an increase in daily human dietary intake of silica. The direct use of synthetic amorphous silica in food will not increase dietary intake over the present 2% threshold permitted under current FDA clearances for silica substances.

VI. Summary of Basis for GRAS Determination

Evonik has determined that synthetic amorphous silica is Generally Recognized as Safe (GRAS) based on the following:

- The fact that Evonik's synthetic amorphous silica is chemically identical to the subject of GRN000321, for which FDA had no questions regarding GRAS status;
- The fact that numerous FDA and EPA clearances currently exist for silica, demonstrating the safety and wide range of acceptable uses for this compound;
- Published scientific data establishing the safety of synthetic amorphous silica and other silica substances;
- The ubiquitous natural occurrence of silica and its wide-ranging use in food;

- The intended uses and an estimate of silica consumption; and
- The fact that specifications for synthetic amorphous silica meet appropriate food grade specifications.

VII. Conclusion

Based on the documentation provided in this GRAS notification, and as discussed above, Evonik has concluded that synthetic amorphous silica is GRAS via scientific procedures for use in a variety of foods up to 2% and in food packaging materials.

Appendix

Food Chemicals Codex (9th edition) monograph for Silicon Dioxide (INS 551)

chloride, and cover both cavities with an inverted watch glass.

Acceptance criteria: No purple or deep indigo blue color appears in or above the liquid containing the sample residue.

- **WAX**

Sample: 10 g, finely ground

Analysis: Transfer the *Sample* and 2.5 g of sodium carbonate into a 200-mL tall-form beaker. Add 150 mL of hot water, immerse the beaker in a boiling water bath, and stir until the *Sample* is dissolved. Cover the beaker with a watch glass, heat for 3 h without agitation, and cool in a cold water bath. When the wax has floated to the surface, filter the mixture through medium-speed, quantitative, ashless filter paper, thus transferring the wax to the paper. Wash the filter with water. Pour 5 to 10 mL of alcohol onto the filter to accelerate drying. Wrap the paper loosely in a larger piece of filter paper, bind with a piece of fine wire, and dry with the aid of gentle heat. Extract with chloroform in a suitable continuous extraction apparatus for 2 h, using a previously dried and accurately weighed flask to receive the extracted wax and solvent. Evaporate the solvent, dry the wax at 105° to constant weight, and calculate the percentage of wax in the sample taken.

Acceptance criteria: NMT 0.2%

Silicon Dioxide

First Published: Prior to FCC 6

Synthetic Amorphous Silica

SiO_2 Formula wt 60.08
INS: 551 CAS: [7631-86-9]
UNII: ETJ7Z6XBU4 [silicon dioxide]

DESCRIPTION

Silicon Dioxide occurs as an amorphous substance that shows a noncrystalline pattern when examined by X-ray diffraction. It is produced synthetically, either by a vapor-phase hydrolysis process, yielding *fumed silica*, or by a wet process, yielding *precipitated silica, silica gel, colloidal silica,* or *hydrous silica. Fumed silica* is produced in an essentially anhydrous state, whereas the wet-process products are obtained as hydrates or contain surface-adsorbed water. *Fumed silica* occurs as a white, fluffy, non-gritty powder of extremely fine particle size and is hygroscopic. The wet-process silicas occur as white, fluffy powders or as white, microcellular beads or granules and are hygroscopic or absorb moisture from the air in varying amounts. All of these forms of Silicon Dioxide are insoluble in water and in organic solvents, but they are soluble in hydrofluoric acid and in hot, concentrated solutions of alkalies.

Function: Anticaking agent; defoaming agent; carrier; conditioning agent; chillproofing agent in malt beverages; filter aid

Packaging and Storage: Store in well-closed containers.

IDENTIFICATION

- **A. PROCEDURE**

Sample: 5 mg

Analysis: Place *Sample* into a platinum crucible, mix with 200 mg of anhydrous potassium carbonate, and ignite over a burner at a red heat for about 10 min. Cool, dissolve the melt in 2 mL of freshly distilled water, warming if necessary, and slowly add 2 mL of ammonium molybdate TS.

Acceptance criteria: A deep yellow color appears.

- **B. PROCEDURE**

Sample: Solution remaining from *Identification* test *A*

Analysis: Place 1 drop of *Sample* from *Identification* test *A* on a filter paper, and evaporate the solvent. Add 1 drop of a saturated solution of *o*-tolidine in glacial acetic acid, and place the paper over ammonium hydroxide.

Acceptance criteria: A green-blue spot develops.

ASSAY

- **PROCEDURE**

Sample: 1 g, previously dried

Analysis: Transfer *Sample* into a tared platinum crucible, ignite as directed in the test for *Loss on Ignition* (below), cool in a desiccator, and weigh to obtain the ignited sample weight (W). Moisten the residue with 3 or 4 drops of alcohol, add 2 drops of sulfuric acid, and then add enough hydrofluoric acid to cover the wetted sample. [**CAUTION**—Handle hydrofluoric acid in an appropriate fume hood.] Evaporate to dryness on a hot plate, using medium heat (95° to 105°), then add 5 mL of hydrofluoric acid, swirl the dish carefully to wash down the sides, and again evaporate to dryness. Ignite the dried residue over a Meker burner to a red heat, cool in a desiccator, and weigh to obtain the residual weight (w). The difference between the ignited sample weight and the residual weight (W – w) represents the weight, in g, of SiO_2 in the ignited sample.

Acceptance criteria

Fumed Silica: NLT 99.0% of SiO_2 on the ignited basis

Precipitated Silica, Silica Gel, and **Hydrous Silica:** NLT 94.0% of SiO_2 on the ignited basis

IMPURITIES

Inorganic Impurities

- **LEAD**

Standard stock solution: 100 µg/mL Pb prepared as follows: Dissolve 159.8 mg of lead nitrate, $Pb(NO_3)_2$, in 100 mL of water containing 1 mL of nitric acid, dilute to 1000.0 mL with water, and mix. [NOTE—Prepare and store this solution in glass containers that are free from lead salts.]

Standard solution: 0.25 µg/mL Pb: from *Standard stock solution.* [NOTE—Prepare on the day of use.]

Sample solution: Transfer 5.0 g of sample into a 250-mL beaker, add 50 mL of 0.5 N hydrochloric acid, cover with a watch glass, and slowly heat to boiling. Boil gently for 15 min, cool, and let the undissolved material settle. Decant the supernatant liquid through a Whatman No. 3, or equivalent, filter paper into a 100-mL volumetric flask, retaining as much as possible of the insoluble material in the beaker. Wash the slurry and beaker with three 10-mL portions of hot water, decanting each washing through the filter into the flask. Finally, wash the filter paper with 1.5 mL of hot water, cool the filtrate to room temperature, dilute with water to volume, and mix.

Analysis: Set a suitable atomic absorption spectrophotometer to a wavelength of 217 nm and adjust the instrument to zero absorbance against water. Measure the absorbance of the *Standard solution* and of the *Sample solution*.

Acceptance criteria: The absorbance obtained from the *Sample solution* is not greater than that obtained from the *Standard solution*. (NMT 5 mg/kg)

SPECIFIC TESTS

- **Loss on Drying,** Appendix IIC: 105° for 2 h
  Acceptance criteria
  Fumed Silica: NMT 2.5%
  Precipitated Silica and **Silica Gel:** NMT 7.0%
  Hydrous Silica: NMT 70.0%
  Colloidal Silica: NMT 85.0%
- **Loss on Ignition**
  Sample: 1 g, previously dried
  Analysis: Transfer the *Sample* into a suitable tared crucible, place in a cold muffle furnace and raise the temperature to 900° to 1000° during a 1-h period. Ignite at this temperature for 1 h, cool in a desiccator, and weigh.
  Acceptance criteria
  Fumed Silica: NMT 2.0% on the dried basis
  Precipitated Silica, Silica Gel, and **Hydrous Silica:** NMT 8.5% on the dried basis
- **Soluble Ionizable Salts (as Na_2SO_4)**
  Sample: 5 g, previously dried
  Control solution: 1 mg/mL anhydrous sodium sulfate, made to 250 mL
  Analysis: Stir *Sample* with 150 mL of water for at least 5 min in a high-speed mixer. Filter with the aid of suction, and wash the mixer and filter with 100 mL of water in divided portions, adding the washings to the filtrate. Dilute the filtrate to 250 mL with water. Determine the conductances of the diluted filtrate and of the *Control solution* with a suitable conductance bridge assembly.
  Acceptance criteria
  Precipitated Silica, Silica Gel, and **Hydrous Silica:** The conductance produced by the *Sample* is not greater than that produced by the *Control solution*. (equivalent to NMT 5.0%)

Silver

First Published: Third Supplement, FCC 8

Argentum
Silver
Chandi-ka-vark

Ag — Atomic weight 107.87
CAS: [7440-22-4]

UNII: 3M4G523W1G [silver]

DESCRIPTION

Silver occurs as silver-color paper thin foil or sheets. The foil or sheets are typically free from creases and folds. It is typically made by hammering silver into foil or sheets and backed with paper for support; this paper is peeled away before use. It is extremely fragile and breaks into smaller pieces if touched.

Function: Coloring agent; decorative ingredient
Packaging and Storage: Store in well-closed containers.

IDENTIFICATION

- **Solubility Test**
  [**Caution**—Wear proper safety gear and follow appropriate safety instructions while handling the chemicals.]
  Analysis: Place a small portion of silver in an appropriate container, and carefully add 3% nitric acid to dissolve completely. Add hydrochloric acid TS, diluted. Gently add 2 M ammonium hydroxide solution in the container to dissolve the precipitate, and promptly add 2 M nitric acid.
  [**Caution**—The solution obtained after dissolving the precipitate in 2 M ammonium hydroxide should be acidified quickly with 2 M nitric acid and discarded properly to avoid an explosion.]
  Acceptance criteria: Formation of white precipitate after adding hydrochloric acid TS that dissolves in 2 M ammonium hydroxide solution confirms presence of silver.

ASSAY

- **Procedure**
  Standard: NIST traceable silver standard solution, 1000 mg/L
  Standard solution: Dilute aliquots of the *Standard* with water to make at least four standard solutions with concentrations ranging from 2.5–100.0 µg/mL. [Note—Silver solution is sensitive to light. Store in an amber glass bottle. Prepare daily.]
  Sample: 1.0 g
  Sample solution
  [**Caution**—Carry out this preparation in a well ventilated fume hood, and adhere to proper safety procedure.]
  Transfer the *Sample* to a 250-mL beaker. Slowly add 10 mL of concentrated nitric acid into the beaker, and mix gently. Cover the beaker with a watch glass, heat to 95°, and let the solution reflux for 10–15 min. Make sure the *Sample* is completely dissolved. Allow the

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SUBMISSION END