Commentary

***Food Chemicals Codex* (*FCC*)**
Eighth Edition, Third Supplement

August 30, 2013

In accordance with USP's Rules and Procedures of the 2010-2015 Council of Experts, and except as provided in Section 8.01(e) Immediate Standards, USP publishes proposed revisions to the *Food Chemicals Codex (FCC)* for public review and comment in the *FCC Forum* (*FCCF)*, USP's journal for public notice and comment for *FCC*. After comments are considered and incorporated as the Food Ingredients Expert Committee (FIEC) deems appropriate, the proposal may advance to official status or be republished in *FCCF* for further notice and comment, in accordance with the Rules and Procedures. In cases when proposals advance to official status without republication in *FCCF*, a summary of comments received and the FIEC's responses are published on the *Commentary* section of the USP Website at the time the revision is published.

The *Commentary* is not part of the text of the monograph or general test or assay. Rather, it explains the basis of the FIEC's response to public comments. If there is a difference between the contents of the *Commentary* section and the monograph or general test or assay, the text of the monograph prevails. In case of a dispute or question of interpretation, the language of the monograph text, alone and independent of the *Commentary* prevails.

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Monographs:

No comments were received for the following proposals:

Monographs:

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Butyl Cinnamate
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Caffeine
Chromium Picolinate
L-Histidine Monohydrochloride
Isobutyraldehyde
Olestra
Phytic Acid Solution
Sucrose
Xylitol

Monograph/Section: Benzyl Mercaptan/Description
Expert Committee: Monographs—Food Ingredients
Number of Comments: 1
Comment summary # 1: The commenter suggested replacing the text "Repulsive garlic-like" in the *Description* section with "Powerful, roasted, burnt beef."
Response: Comment incorporated.

Monograph/Section: Benzyl Methyl Sulfide/Description
Expert Committee: Monographs—Food Ingredients
Number of comments: 2
Comment summary # 1: The commenter suggested correcting the spelling of "2-propen-1-yl 3-phenyl 2-propenoate."
Response: Comment not incorporated. The synonym 2-Propen-1-yl 3-phenyl 2-propenoate was deleted from the synonym list.
Comment summary # 2: The commenter suggested replacing the word "stench" in the *Description* section with "Powerful, roasted, burnt beef."
Response: Comment incorporated.

Monograph/Section(s): Brilliant Blue/*Chromium*
Expert Committee(s): Monographs—Food Ingredients
No. of Commenters: 1
Comment Summary #1: The commenter requested changing the chromium limit of NMT 0.0005% (5 mg/kg) in the current *FCC* monograph to NMT 0.005% (50 mg/kg) to be consistent with limit for the equivalent batch certified color in the USA, FD&C Blue No. 1, as published in 21 *CFR* 74.101.
Response: Comment incorporated.

Monograph/Sections: Butyl Heptanoate/Specific tests, Specific gravity
Expert Committee: Monographs—Food Ingredients
Expert Committee-initiated Change #1: The Food Ingredients Expert Committee revised the *Specific gravity Acceptance criterion* in the *Specific tests* section from 0.8656 to 0.866 (+ 0.002) to more accurately reflect commercial practices.

Monograph/Section(s): Calcium L-5-Methyltetrahydrofolate/Multiple Sections
Expert Committee(s): Monographs—Food Ingredients
Expert Committee Initiated Change #1: The FIEC standardized the names of the USP reference standards in the monograph.

Monograph/Section(s): Chitosan
Expert Committee(s): Monographs—Food Ingredients
Expert Committee Initiated Change #1: The FIEC moved the test for *Degree of Deacetylation by 1H NMR Spectroscopy* from the *Assay* section of the monograph to the *Specific Tests* section, because this test does not truly give a measurement of the purity of the sample being tested.

Expert Committee Initiated Change #2: The FIEC deleted the test for *Protein Content*, because this determination is not necessary for chitosan used in foods.

Monograph/Section(s): Alpha-Damascone/Chemical Information
Expert Committee(s): Monographs—Food Ingredients
Expert Committee Initiated Change #1: The FIEC removed the synonym "Dihydro Floriffone A" from the monograph, because it is a trade name associated with a single manufacturer.

Monograph/Section(s): Beta-Damascone/Chemical Information
Expert Committee(s): Monographs—Food Ingredients
Expert Committee Initiated Change #1: The FIEC removed the synonym "Dihydro Floriffone B" from the monograph, because it is a trade name associated with a single manufacturer.

Monograph/Section(s): Delta-Damascone/Chemical Information
Expert Committee(s): Monographs—Food Ingredients
Expert Committee Initiated Change #1: The FIEC removed the synonym "Dihydro Floriffone TD" from the monograph, because it is a trade name associated with a single manufacturer.

Monograph/Section: Gold/Description
Expert Committee(s): Monographs—Food Ingredients
Number of commenters: 1
Comment summary # 1: The commenter suggested indicating that the gold sheets should be free from creases and folds.
Response: Comment Incorporated.
Comment summary # 2: The commenter suggested including a specific range of thickness for the gold foils or sheets.
Response: Comment partially incorporated. In absence of reliable information on a specific thickness range for gold foils or sheets, the words "a few micrometers thick" were deleted from the sentence: "It is typically made by hammering gold into foil or sheets that are ~~a few micrometers thick and~~ backed with paper support; this paper is peeled away before use."

Monograph/Section(s): Krill Oil/Multiple Sections
Expert Committee(s): Monographs—Food Ingredients
No. of Commenters: 10

Description
Comment Summary #1: The commenter requested that "Antarctic krill" and the Latin binomial "*Euphasia superba*" be removed from the *Description*, because krill oil may be obtained by extraction from different forms of krill meal, phospholipid-peptide complexes, or other processed forms of krill.

Response: Comment not incorporated. The proposed monograph reflects products approved for use in foods, which are sourced from the organism stated.
Comment Summary #2: The commenter requested that the phrase "…including numerous constituent fatty acids…" be removed from the *Description*, because fatty acids are a known natural part of phospholipids and triglycerides.
Response: Comment not incorporated. The FIEC intends for this statement to reflect that the fatty acids contained in the phospholipid and triglyceride fractions of Krill Oil are specific constituents characteristic to Krill Oil.
Comment Summary #3: The commenter requested replacing the phrase "using acetone as the extraction solvent" with "either using a solvent-free process (or physical extraction process) or using a suitable extraction solvent" in the *Description,* because this statement is more accurate.
Response: Comment not incorporated. Krill oil products that currently are approved for use in foods are extracted using food-grade solvents.
Comment Summary #4: The commenters requested that the sentence "It is obtained through cold extraction solely from crushed Antarctic krill (*Euphasia* superba) using acetone as the extraction solvent" be replaced with "It is obtained through extraction of Antarctic krill (*Euphasia superba*) biomass using a suitable extraction solvent," because the proposed change provides a better description of the range of approved processes for manufacturing krill oil.
Response: Comment incorporated.
Comment Summary #5: The commenter requested that the sentence "The whole lipid portion of the extract is then separated and concentrated through evaporation of the acetone, followed by filtration" be removed from the *Description*, because it is too specific to describe all approved processes for manufacturing krill oil.
Response: Comment incorporated.
Expert Committee Initiated Change #1: The FIEC added the phrase "using a suitable extraction solvent" to the *Description,* because while acetone may not be the only approved extraction solvent for manufacturing krill oil, all products are currently manufactured using some type of extraction solvent.

Identification
Comment Summary #6: The commenters requested numerous changes in the *Fatty Acid Composition* table, including the addition of many fatty acids that may be "none detected" in the ingredient and changes to the ranges of the existing constituent fatty acids. The commenters indicated that the original ranges are too narrow and requested the specification for eicosapentaenoic acid (EPA) be changed from "12.0 – 16.0%" to "> 10%" and that the specification for docosahexaenoic acid (DHA) be changed from "7.0-11.0%" to "> 5%".
Response: Comment incorporated. The FIEC made numerous edits to the specifications in the *Fatty Acid Composition* table based on comments and data received.
Comment Summary #7: The commenter requested the removal of the *UV-Visible Absorption Spectrum* test and requirement from the *Identification* section of the monograph, because this requirement is not typically part of a krill oil

specification and many types of coloring matters would potentially give rise to the results described.
Response: Comment incorporated.
Comment Summary #8: The commenter requested that "Moroctic Acid" be replaced with "Stearidonic Acid" in the *Fatty Acid Composition* table, because stearidonic acid is a more widely-used term.
Response: Comment incorporated.
Comment Summary #9: The commenter requested that the phrase "a single maximum at about 468 nm" be replaced with "a broad maximum in the range of 460 to 490 nm" in the *Acceptance criteria* for the *Identification* test for *UV-Visible Absorption Spectrum*, because this would more accurately reflect the UV-visible absorption spectrum of krill oil and of astaxanthin itself.
Response: Comment not incorporated. The FIEC removed this test and requirement entirely, as requested in a separate comment received.
Expert Committee Initiated Change #2: The FIEC added text to the *Acceptance criteria* and the table in the *Fatty Acid Composition* test to clarify that all of the specifications within that test are in units of g/100 g of oil.
Expert Committee Initiated Change #3: The FIEC combined the requirements for all 18:1 monounsaturated fatty acids (formerly proposed as "Vaccenic acid" and "Oleic acid") and re-labeled them "Octadecenoic Acid, 18:1 (all isomers)" in the table under *Fatty Acid Composition*. This change was made because data used to develop the proposed specifications for these monounsaturated fatty acids did not always include individual analyses for the isomers.

Assay
Comment Summary #10: The commenters requested changes to the minimum amounts of EPA and DHA required in the *Assay* on the basis that the original specifications do not accurately reflect the EPA and DHA content of all approved products.
Response: Comment incorporated. The FIEC changed the minimum requirement for EPA from "NLT 12.0% (w/w)" to "NLT 10.0%". The DHA requirement is changed from "NLT 7.0% (w/w)" to "NLT 5.0%". The FIEC also added wording to clarify that the percentage of EPA and DHA should be calculated in the units of g/100 g of oil.

Impurities
Comment Summary #11: The commenters requested the removal of the *Residual Solvent* test and requirement from the *Organic Impurities* section of the monograph, because there are a range of approved solvents that may be used in processing krill oil and no one specific limit (for one solvent) should be required.
Response: Comment incorporated.

Specific Tests
Comment Summary #12: The commenter requested that a method to separate and quantify mono- and diesters of astaxanthin be added to the monograph.

Response: Comment not incorporated. The Expert Committee will consider further revisions to the monograph upon the receipt of the necessary supporting data.

Comment Summary #13: The commenters suggested removing the *Anisidine Value* test and requirement from the *Specific Tests* section of the monograph, because krill oil cannot be tested using the method cited, due to the presence of interferences.

Response: Comment incorporated.

Comment Summary #14: The commenters requested the removal of the *Total Oxidation* test and requirement, because the *Anisidine Value* test (upon which the *Total Oxidation* calculation is dependent) cannot reliably be determined on krill oil.

Response: Comment incorporated.

Comment Summary #15: The commenter requested the removal of the thin-layer chromatographic procedure from the *Astaxanthin Esterification* test, because there are HPLC methods that are easier to use and give equivalent results.

Response: Comment not incorporated. The Expert Committee will consider further revisions to the monograph upon the receipt of the necessary supporting data.

Comment Summary #16: The commenters requested that the proposed *Content of Astaxanthin* test, which is spectrophotometric, be replaced with a HPLC method, because spectrophotometric methods may be difficult on krill oil and there are existing chromatographic techniques that are commonly used for quantitation of astaxanthin.

Response: Comment not incorporated. The Expert Committee will consider further revisions to the monograph upon the receipt of the necessary supporting data.

Comment Summary #17: The commenters requested lowering the *Acceptance criteria* in the test for *Content of Astaxanthin*, because the original proposed limit was not based on the true content of astaxanthin in krill oil (calculated as free astaxanthin and not esterified astaxanthin). The commenters also indicated that the importance of astaxanthin in krill oil lies in its natural antioxidant properties, which help stabilize the extracted oil and that the levels of astanxanthin required to prevent oxidation of the extracted oil is much lower than required by the original proposal. Finally, the commenters requested that the upper limit for the *Content of Astaxanthin* be deleted entirely, because seasonal variation and variations arising from different extraction procedures do give rise to a wide range of astaxanthin concentrations and there is no reason to limit higher amounts of naturally-occurring astaxanthin in krill oil.

Response: Comment incorporated. The FIEC changed the specification from “0.10% - 0.15%” to “NLT 100 µg/g”. Further changes were made to clarify that the limit is no longer proposed as a percentage. The FIEC also changed the format of the absorptivity of astaxanthin described in the equation to support the units of the new limit.

Comment Summary #18: The commenters requested the removal of the *Iodine Value* test and requirement from the *Specific Tests* section of the monograph, because the degree of unsaturation in the oil can already be determined from the fatty acid profile that is already required in the *Identification* section. The commenters also indicated that the red color of the oil may cause difficulties in performing the analysis.
Response: Comment incorporated.
Comment Summary #19: The commenter requested that the *Acceptance criteria* in the test for *Iodine Value* be changed, because the proposed limit is too restrictive and the tests lacks precision as there may be difficulties in performing this test due to the color of krill oil.
Response: Comment not incorporated. The FIEC removed this test and requirement entirely based on other comments received.
Comment Summary #20: The commenters requested that the *Acceptance criteria* for the *Peroxide Value* test be changed from "less than 0.2 mEq/kg" to "max 5" because the proposed requirement would be difficult to determine in a reproducible manner on a colored oil and is inconsistent with the *FCC* monographs for *Menhaden Oil* which is also a marine sourced oil.
Response: Comment incorporated.
Comment Summary #21: The commenter suggested removing the *Peroxide Value* test under *Specific Tests,* because this test includes at least one color change step that cannot be seen in krill oil samples due to the dark color of the oil.
Response: Comment not incorporated. Data received from multiple sources indicated that the test could be reproducibly performed on krill products.
Comment Summary #22: The commenters requested the removal of the *Saponification Value* test and requirement from the *Specific Tests* section of the monograph, because the test is never used in commercial sales of krill oil and does not indicate the quality of the ingredient. The commenter also indicated that the requirement was unnecessary, because a fatty acid profile is already required in the *Identification* section. Commenters also stated that the red color of the oil may cause difficulties in performing the analysis.
Response: Comment incorporated.
Comment Summary #23: The commenter requested that the *Acceptance criteria* in the test for *Saponification Value* be changed, because the proposed limit is too restrictive and the test lacks precision as there may be difficulties in performing this test due to the color of the krill oil.
Response: Comment not incorporated. The FIEC removed this test and requirement entirely based on other comments received.
Comment Summary #24: The commenter requested that a specification be added to the monograph indicating the percentage of omega-3 fatty acids bound to the phospholipids in krill oil, because this parameter is necessary to prevent adulteration of krill oil with fish oil in order to standardize fatty acid content.
Response: Comment not incorporated. The Expert Committee will consider further revisions to the monograph upon the receipt of the necessary supporting data.

Comment Summary #25: The commenter requested that the limit of *Total phospholipids* under *Phospholipids* be changed from "28%-52% (w/w)" to ">28% w/w" to accommodate products with a phospholipids content that exceeds the proposed upper limit.
Response: Comment not incorporated. The Expert Committee will consider further revisions to the monograph upon the receipt of the necessary supporting data.
Expert Committee Initiated Change #4: The FIEC changed the concentration of the *Sample solution* in the test for *Astaxanthin Esterification* from "10 mg/mL in acetone" to "250 mg/mL in acetone" to correct an erratum in the original proposal.

Other Requirements
Expert Committee Initiated Change #5: The FIEC added a sentence: "Indicate the type and level of solvents present from extraction" to the existing *Labeling* section in *Other Requirements*. This change was made in order to inform users of the potential presence of specific extraction solvents, because individual solvent limits could not be determined based on data and information received.

Monograph/Section: Silver/Description
Expert Committee(s): Monographs—Food Ingredients
Number of commenters: 1
Comment summary # 1: The commenter suggested indicating that the silver sheets should be free from creases and folds.
Response: Comment incorporated.
Comment summary # 2: The commenter suggested including a specific range of thickness for the silver foils or sheets.
Response: Comment partially incorporated. In absence of reliable information on a specific thickness range for silver foils or sheets, the phrase "a few micrometers thick" was deleted from the sentence: "It is typically made by hammering silver into foil or sheets that are ~~a few micrometers thick and~~ backed with paper support; this paper is peeled away before use."

Monograph/Section(s): Steviol Glycosides/Multiple Sections
Expert Committee(s): Monographs—Food Ingredients
No. of Commenters: 1
Comment Summary #1: The commenter requested removing the *Identification* test requirement that the principal peak in the chromatogram of the Sample solution correspond to either the stevioside or rebaudioside A peak, because this requirement will limit introduction into the marketplace of next generation stevia sweeteners that have one of the other nine named steviol glycosides as the principal component.
Response: Comment not incorporated. The analytical methods and specifications proposed in the FCC monograph were demonstrated to be suitable for use with ingredient compositions composed primarily of stevioside or

rebaudioside A, consistent with the monograph *Description*, but may not be suitable for ingredients with different compositions. Stakeholders producing steviol glycoside ingredients with different compositions are encouraged to submit to FCC a request for revision to add their ingredient's composition to the scope of the monograph, or to create a new monograph.

Comment Summary #2: The commenter indicated that the proposed HPLC method does not sufficiently separate stevioside from a known impurity "Iso Reb A," which is present in some commercial products. The commenter provided batch data and supporting chromatograms on twenty-two commercial samples found to contain "Iso Reb A" with estimate concentrations ranging from 0.04 to 2.8% and a mean of 0.37%.

Response: Comment not incorporated. A validated alternative method was not provided by the commenter that was capable of a baseline resolution of "Iso Reb A," stevioside, and rebaudioside A. Stakeholders are encouraged to submit such methods and supporting validation packages to USP for consideration as future revisions.

Comment Summary #3: The commenter suggested changing the *Standard solution* concentration used in the *Assay* procedure from 0.5 mg/mL to 1.2 mg/mL, so that the analyte concentrations in sample solutions prepared from ingredients with a high level of rebaudioside A or stevioside will match the *Standard solution*.

Response: Comment not incorporated. The concern raised by the commenter is already addressed in the proposed procedure, which uses a single concentration calibration and was validated to be accurate when testing ingredients containing high levels of rebaudioside A and stevioside.