Status: Currently Official on 14-Feb-2025
Official Date: Official as of 01-Dec-2022
Document Type: Dietary Supplement Monographs
DocId: GUID-B2C1B38E-606D-4103-857F-4DBF2B9F18EC_3_en-US
DOI: https://doi.org/10.31003/USPNF_M7099_03_01
DOI Ref: v0ib1

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Krill Oil Capsules

DEFINITION

Krill Oil Capsules contain NLT 95.0% of the labeled amount of Krill Oil calculated through the content of eicosapentaenoic acid (EPA), docosahexaenoic acid (DHA), and total phospholipids.

IDENTIFICATION

• **A. Fatty Acid Profile**

Antioxidant solution, System suitability solution 1, and **Chromatographic system:** Proceed as directed in *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile*.

Standard solution: Prepare as directed in *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile, Content of EPA and DHA, Test Solution 1*, except use 250 mg of USP Krill Oil RS.

Sample solution: Using the portion of oil from NLT 10 Capsules, prepare as directed in *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile, Content of EPA and DHA, Test Solution 1*.

System suitability

Samples: *System suitability solution 1* and *Standard solution*

Suitability requirements

Chromatogram similarity: The chromatogram from the *Standard solution* is similar to the reference chromatogram provided with the lot of USP Krill Oil RS being used.

Resolution: NLT 1.0 between the methyl oleate and methyl *cis*-vaccinate peaks, *Standard solution*

Theoretical area percentages: Meet the requirements for *System suitability solution 1*

Analysis

Sample: *Standard solution* and *Sample solution*

Identify the retention times of the peaks corresponding to the relevant fatty acid methyl esters by comparing the chromatogram of the *Standard solution* to the reference chromatogram provided with the lot of USP Krill Oil RS used.

Calculate the area percentage for each fatty acid as methyl esters in the portion of oil taken from the Capsules:

$$\text{Result} = (r_A/r_B) \times 100$$

r_A = peak area of each individual fatty acid from the *Sample solution*

r_B = total area of all peaks, except the solvent and butylated hydroxytoluene peaks, from the *Sample solution*

Acceptance criteria: See *Table 1*.

Table 1

Fatty Acid	Short-Hand Notation	Lower Limit (Area %)	Upper Limit (Area %)
Saturated fatty acids			
Myristic acid	14:0	5.0	13.0
Palmitic acid	16:0	17.0	24.6
Palmitic acid:myristic acid ratio	16:0/14:0	1.6	3.6
Monounsaturated fatty acids			
Palmitoleic acid	16:1 n-7	2.5	12.0
cis-Vaccenic acid	18:1 n-7	4.7	8.0
Oleic acid	18:1 n-9	6.0	14.5

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Fatty Acid	Short-Hand Notation	Lower Limit (Area %)	Upper Limit (Area %)
Eicosenic acid	20:1 n-9	0.0	2.0
Erucic acid	22:1 n-9	0.0	1.5
Polyunsaturated fatty acids			
Linoleic acid	18:2 n-6	0.0	3.0
Eicosapentaenoic acid	20:5 n-3	14.0	28.0
Docosapentaenoic acid	22:5 n-3	0.0	0.7
Docosahexaenoic acid	22:6 n-3	7.1	15.7

• **B. Phospholipid Profile**

Solution A, Internal standard, Sample stock solution, Sample solution, Standard solution, Instrumental conditions, System suitability, and **Analysis:** Proceed as directed in the test for *Content of Total Phospholipids* in *Strength*.

Acceptance criteria: The *Sample solution* shows ^{31}P nuclear magnetic resonance (NMR) spectra similar to those obtained with USP Krill Oil RS. The main signal in the ^{31}P NMR is due to phosphatidylcholine (PC); a signal due to phosphatidylcholine ether has an intensity of about 10% of that due to PC; and the second signal in intensity is due to 2-lysophosphatidylcholine (2-LPC). Minor signals due to phosphatidylethanolamine (PE), *N*-acylphosphatidylethanolamine (NAPE), lysophosphatidylethanolamine (LPE), and 1-lysophosphatidylcholine (1-LPC) among others are also observed.

STRENGTH

• **Content of EPA and DHA**

(See *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile*).

Standard solution 1a, Standard solution 1b, Standard solution 2a, Standard solution 2b, System suitability solution 1, and **Chromatographic system:** Proceed as directed in the chapter.

Test solution 1: Weigh NLT 10 Capsules in a tared weighing bottle, and carefully open the Capsules without loss of shell material. Transfer the combined contents to a 100-mL beaker. Remove any adhering substance from the emptied Capsules by washing with several small portions of acetone. Discard the washings, and allow the empty Capsules to dry in a current of air until the acetone is completely evaporated. Weigh the empty Capsules in the original tared weighing bottle, and calculate the average fill weight per Capsule. Take 250 mg of the combined Capsule contents and proceed as directed in *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile, Content of EPA and DHA, Test Solution 1*.

Test solution 2: Prepare as directed in *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile, Content of EPA and DHA, Test Solution 2*, using 250 mg of the combined Capsule contents.

Analysis: Proceed as directed in *Fats and Fixed Oils ⟨401⟩, Procedures, Omega-3 Fatty Acids Determination and Profile, Content of EPA and DHA, Analysis (for triglycerides)*.

Calculate the percentage of the labeled amounts of EPA and DHA in the portion of Capsules taken:

$$\text{Result} = A \times W_{Av} \times 100/L$$

A = content of EPA or DHA in the portion of Capsules taken (mg/mg)

W_{Av} = average fill weight (mg/Capsule)

L = label claim of EPA or DHA (mg/Capsule)

Acceptance criteria: NLT 95.0% of the labeled amounts of EPA and DHA

• **Content of Total Phospholipids**

(See *Nuclear Magnetic Resonance Spectroscopy ⟨761⟩, Qualitative and Quantitative NMR Analysis*.)

[Note—All deuterated solvents used in this method should be NLT 99.8 atom % D.]

Solution A: 0.2 M ethylenediaminetetraacetic acid (EDTA) adjusted with a 1 M cesium carbonate solution to a pH of 7.2–7.5. [Note—Use cesium carbonate of a sufficient grade for trace metals analysis.]

Internal standard: Use a triphenyl phosphate nuclear magnetic resonance (NMR) reference standard with NLT 99% purity.

Sample stock solution: Transfer a number of Capsules (NLT 5) to a conical flask with stopper. Add 2 mL/Capsule of water and melt the gelatin shells at 50°–60°. Add 10 mL/Capsule of chloroform and methanol (2:1), insert the stopper and shake intensively for 30 min. Transfer to a separation funnel fitted with glass wool. Collect the organic phase. Wash the conical flask with chloroform and methanol (2:1) and transfer to a separation funnel. Collect the organic phase and combine with previous extract in a suitable volumetric flask. Dilute with chloroform and methanol (2:1) to volume.

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