Received: 4 April 2014

Revised: 16 May 2014

(wileyonlinelibrary.com) DOI 10.1002/jsfa.6816

A comparison of actual versus stated label amounts of EPA and DHA in commercial omega-3 dietary supplements in the United States

Alison C Kleiner, Dennis P Cladis and Charles R Santerre^{*}

Abstract

BACKGROUND: Eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) are associated with health benefits throughout life and are obtained primarily through fish and fish oil supplements. Due to the growing popularity of dietary supplements, 47 commercial fish, krill, and algal oil supplements were analyzed for EPA, DHA, and other fatty acids.

RESULTS: For fish- and krill-based supplements, the range of EPA was 81.8 to 454.6 mg g⁻¹ oil and DHA was 51.6 to 220.4 mg g⁻¹ oil. For algal oil supplements, EPA ranged from 7.7 to 151.1 mg g⁻¹ oil and DHA ranged from 237.8 to 423.5 mg g⁻¹ oil. The percentage of the stated label amount for EPA and DHA ranged from 66 to 184% and 62 to 184%, respectively. Only 10 supplements (21% of those tested) had at least 100% of the stated label amount of EPA, while 12 supplements (25% of those tested) had at least 100% of the stated label amount of EPA, while 12 supplements (25% of those tested) had at least 100% of the stated label amount of EPA, or DHA.

CONCLUSIONS: These results indicate that the quality of fish oil supplements is not being adequately monitored by manufacturers or government agencies and increased testing is needed to ensure regulatory compliance. © 2014 Society of Chemical Industry

Keywords: fish oil; dietary supplement; fatty acids; EPA; DHA

INTRODUCTION

Fatty fish and fish oil supplements are the primary dietary sources of long-chain omega-3 polyunsaturated fatty acids (LC-PUFAs),¹⁻³ including eicosapentaenoic acid (C20:5n-3, EPA) and docosahexaenoic acid, (C22:6n-3, DHA). EPA and DHA have been associated with many health benefits throughout life.⁴ During fetal and infant development, EPA and DHA supplementation has been associated with the development of cognition and photoreceptors.⁵⁻¹³ EPA and DHA supplementation during pregnancy not only benefits the fetus, but also leads to healthier pregnancies by increasing gestation duration and infant birth weights.^{14,15} The nutritional status of the mother is not only important during pregnancy, but also after birth, as the composition of the mother's breast milk influences the infant's development.¹⁶ In aging populations, EPA and DHA supplementation has been associated with improved cardiovascular health,^{17–19} maintenance of cognitive function,^{20,21} retention of visual abilities,^{22,23} and a decreased inflammatory response to injury.24

Over the past 40 years, dietary supplement intake has increased and become an increasingly important component of human diets.^{2,3} There are many factors influencing this increase, but the most important reasons behind the rise in fish oil intake are increased consumer awareness of the health benefits associated with EPA and DHA²⁵ and the fact that dietary supplements are the most cost effective way to increase EPA and DHA intakes.¹ Given the plethora of fish oils available to consumers, the objective of this study was to assess EPA and DHA label declarations of fish, krill and algal oil dietary supplements in the USA.

EXPERIMENTAL

Chemicals

Methanol (ChromAR grade) was purchased from Macron Fine Chemicals (Center Valley, PA, USA). Sodium chloride (ACS grade), sodium hydroxide (ACS grade), and isooctane (pesticide grade) were purchased from Fisher Scientific (Pittsburgh, PA, USA). Butylated hydroxytoluene (BHT) was purchased from United States Biochemical Corp. (Cleveland, OH, USA). BF₃-methanol (10% w/w) was purchased from Sigma-Aldrich (St Louis, MO, USA). Methyl tricosanoate (>99% pure) was purchased from Nu-Chek Prep, Inc. (Elysian, MN, USA).

* Correspondence to: Charles R Santerre, Department of Nutrition Sciences, Purdue University, Stone Hall, 700 W. State St, West Lafayette, IN 47907-2059, USA. E-mail: santerre@purdue.edu

Department of Nutrition Sciences, Purdue University, Stone Hall, West Lafayette, IN, USA

Table 1. Conc	Concentrations of major fatty acids (mg g^{-1} oil)												
Vendor	Product name	Fish ^a	14:0	16:0	18:0	18:1 <i>n</i> 9	18:2 <i>n</i> 6	18:3 <i>n</i> 6	20:4 <i>n</i> 6	18:3 <i>n</i> 3	20:5 <i>n</i> 3	22:5n3	22:6n3
Arctic Naturals	Daily Omega-3	Anchovy, sardine	64.4	145.2	28.2	82.0	12.0	2.3	9.1	6.8	156.3		95.8
			± 3.2	± 6.9	± 1.5	± 4.4	± 2.6	± 13.3	± 0.8	± 3.3	± 9.2		± 5.8
	Krill Omega-3	Krill	66.0	139.5	18.2 2.	76.0 2.2	12.9	1.6	6.5 2.2	7.3	148.6		83.1
	Total Omega	Anchovy, sardine	+ 4./	± 5.0 136.7	± 0.4 31.6	± 2.2	± 3./ 65.4	± 0.1	± 0.2	± 0.4	± 1.2		± 1.0
			± 4.1	+ 9.1	± 2.0	± 5.8	+ 4.0	+ 1.9	± 0.5	+ 0.8	± 7.4		± 4.2
	Ultra Omega-3	Anchovy, sardine	2.2	9.5	23.1	48.9	9.4	0.9	16.5	4.1	285.0		189.1
		nombra nedaelA [AliM	± 1.5	± 6.3	± 3.9	± 6.9	± 2.6	± 4.5	± 1.9	± 2.2	± 30.5		± 21.7
			+0.2 ± 1.4	± 8.5	∠4.0 ± 3.0	± 10.4	+.c1 ± 1.6	+ 	4.0 ± 0.2	9.2 I + 0.6	c.00 ± 4.8	22.0 ± 1.4	± 2.9
	100% Pure 300 mg, Omega-3 Krill Oil	Krill	70.5	137.5	7.6	67.6	10.4	1.0	1.5	11.6	112.7		52.0
		- /	± 6.9	± 3.7	± 0.3	± 4.5	± 0.6	± 0.1	± 0.2	± 4.9	± 2.1		+ 1.3
	Natural Officiaa-3 Fishly Frax and Bolage Off	11/9	+ 0.1	+ 3.1	+ 1.3	+ 6.4	+ 5.2	00.2 + 2.3		4 7.1 +	+ 4.1		+ 5.8
	Odorless Fish Oil 1200 mg, Heart, Joint and Skin Health	n/a	57.2	130.2	26.7	82.2	10.1	2.5	10.3	5.7	152.1		100.0
			± 4.1	± 8.5	± 3.5	± 4.0	± 0.7	± 0.1	± 1.4	± 0.8	± 5.8		± 5.2
Equaline	Omega-3 Fish Oil, 1200 mg	Sardine, anchovy, mackerel	43.1	145.3	32.2	91.6	11.1	3.1	10.2	9.9	170.1		85.9
:			± 35.2	± 4.5	± 8.6	± 15.9	± 2.8	± 0.6	± 1.9	± 1.2	± 11.5		± 24.0
Finest Nutrition	Cod Liver Oil	Cod	50.1	128.8	25.2	137.8	18.5	1.4	5.4	8.3	97.7		100.0
			± 3.2	± 2.0	± 1.6	± 11.8	± 4.3	± 0.1	± 0.2	± 0.1	± 1.3		± 9.0
	DHA Algal-900, Triple Strength	Algae (Schizochytrium spp.)	51.5	144.4	8.5	128.6	12.8	2.2	9.6	0.9	15.4		316.3
			± 2.3	+ 6.3	+ 1.2	± 5.4	+ 1.3	± 0.1	+ 0.6	± 0.4	± 6.0		± 7.6
	Double Strength 1200 mg, Fish Oil	Mackerel, sardine, anchovy	2.9	33.8	33.9	/.5/	8.4	2.0	1.61	τ.τ 	261.4		180.8
	Regular Strength 600 mg. Fish Oil	Mackerel. sardine. anchovv	± 0.8	± 12.3	± 0.0	±.cl ±	± 2.0	± 0.5	± 2.0	± 0.9	± 16.01 2.57.9		± 0.9 172.5
			+ 1.0	+ 6.9	+ 1.3	± 13.1	+ 1.8	± 0.6	± 2.1	+ 1.2	± 2.4		± 5.1
	Triple Strength, 1400 mg, Fish Oil	Mackerel, sardine, anchovy	0.8	3.1	16.3	26.7	2.9	0.6	23.2	2.0	410.7		158.5
			± 0.2	± 0.9	± 0.6	± 1.6	± 0.1	± 0.1	± 2.5	± 0.2	± 9.6		± 4.2
	Wild Alaskan Salmon Oil	Salmon	45.4	142.7	21.6	150.3	17.3	0.4	4.1	11.9	84.6		84.8
N	Fish Oil FEA was Handle Handleb Omeans 2	Andrew and and and and	± 2.1	± 7.1	+ 0.8	+ 10.1	± 0.8	± 0.5	± 0.2	± 0.4	± 4.0		± 2.9
N OGEI		אוונווטעץ, אמומוויב, ווומראבובו	+ 0.2	4.0 +	+ 9.2	± 12.8	+ 0.5	+ 2.5	+ 2.4	+ 0.2	+ 30.4		+ 3.0
Meijer Naturals	Algal-900 DHA	Algae (Schizochytrium spp.)	62.5	147.6	8.0	130.0	12.5	2.1	7.6	0.8	7.7		328.7
			± 5.5	± 3.3	± 0.7	± 12.4	± 1.2	± 0.2	± 2.4	± 0.1	± 2.3		± 11.7
	Cod Liver Oil	Cod	45.6	112.6	21.5	140.3	20.3	1.4	4.4	7.5	90.9		99.1
			± 1.4	± 2.0	± 0.2	± 1.5	± 0.2	± 0.0	± 0.0	± 0.1	± 0.9		± 0.9
	Omega 3 High EPA Fish Oil	Salmon, mackerel, sardine	5.6	12.4	8.5	43.4	5.4	0.8	21.9	2.8	421.4		167.0
			± 0.4	± 0.5	± 0.3	± 3.2	± 0.3	± 0.1	± 0.8	± 0.4	± 13.3		± 6.3
Natrol	Omega-3 Krill Oil	Krill	71.1	146.2	7.0	67.2	10.2	1.3	2.5	8.9	125.3		73.3
			± 1.9	± 3.5	± 0.2	± 1.4	± 0.4	± 0.2	± 0.2	+ 	± 3.2		± 1.0
]

www.soci.org

Table 1. Continued	Ū												
Vendor	Product name	Fish ^a	14:0	16:0	18:0	18:1 <i>n</i> 9	18:2 <i>n</i> 6	18:3 <i>n</i> 6	20:4 <i>n</i> 6	18:3 <i>n</i> 3	20:5 <i>n</i> 3	22:5n3	22:6n3
Nature's Bounty	Dual Spectrum Omega-3	Krill, anchovy, sardine	20.2	39.8	7.8	45.2	7.1	1.1	16.7	4.1	349.4	25.9	141.2
			± 0.5	+ 1.1	± 0.6	± 1.2	± 0.2	± 0.6	± 0.4	± 0.1	± 4.6	± 1.0	± 1.8
	Odor-Less Double Strength Fish Oil, 2400 mg	Anchovy, sardine, mackerel	3.8	28.2	30.1	89.3	10.3	2.5	16.5	6.6	252.9	33.4	164.4
	Odor-1 acc Trinla Stranoth Eich Oil 1400 ma	Anchora cardina macharal	+ 0.	± 0.4	± 0.4	+	+ 0.+	0.0 1	10.7 76.4	+ 0. 0.	± 3.8	± 0.5 53 5	± 2.7 158 3
			+ 0.2	+ 0.3	+ 5.6	+ 9.6	; 	+ 0.3	+ 7.0-4 1.4	± 0.5	+ 9.0 + +	± 5.0	± 3.2
	Red Krill Oil	Krill	76.2	140.9	7.5	70.4	15.5	1.0	2.2	7.0	104.3	2.4	52.4
			± 3.0	± 6.9	± 0.3	± 2.6	± 0.8	± 0.0	± 0.2	± 0.7	± 7.3	± 0.1	± 2.4
	Triple Strength Red Krill Oil	Krill	82.3	128.7	18.4	82.9	11.2	1.5	5.1	7.2	91.9 . 6 E	9.9 - 1 E	55.2
Nature Made	Burp-Less Fish Oil, 1200 mg	Anchovy, sardine	± 29.0 66.2	± 42.2	± 27.0	± 21.2 73.9	± 1.9	± 0.1	H 6.	± 1.3	± 0.5 147.8	± 1.5	± 3.1 92.5
			± 4.3	± 7.8	± 1.3	± 3.9	± 0.7	± 0.2	± 0.5	± 0.5	± 6.8	± 0.8	± 5.7
	Fish Oil, 1200 mg	Anchovy, sardine, mackerel	72.9	153.6	28.8	80.1	9.8	2.9	9.8	5.4	156.6	17.6	99.1
		<u>-</u>	+ 0.9	+ 1.0	± 0.3	± 0.4	± 0.1	+ 0.1	+ 0.1	± 0.1	± 4.9	± 0.5	± 2.9
	Fish Oil Pearls	Anchovy, sardine	5.1 '	14.0	22.8	45.1	5.0	ر آ.	17.4	3.0	2/1.9	49.5	220.4
	Eull Strandth Minis Sunar Omada-3	Anchow sarding mackerel	+ 	± 3.7	± 1.5	+ 1.8 8.1 +	+ 0.6	+ 0.3 1 6	± 4.0	± 0.5	± 9.8 431.0	+ 1.4 306	土 /.6 155.8
			+ 0.0	+ 2.7	6.0 +	+ 7.2	; ; +	+ 1.0	+ 1.8	+ 0.9	+ 15.9	+ 14.3	+ 9.0
	Krill Oil	Krill	71.1	138.7	6.9	68.1	11.1	1.2	2.2	10.7	115.2	2.6	64.1
			± 3.0	± 4.5	± 0.2	± 2.0	± 0.3	± 0.0	± 0.0	± 1.8	± 2.0	± 0.1	± 1.6
	Ultra Omega-3 Fish Oil, 1400 mg	Anchovy, sardine	0.6	4.9	16.4	35.2	4.2	2.3	24.7	3.3	454.6	21.0	168.8
:		-	± 0.2	± 0.3	± 0.9	± 2.4	± 0.2	± 0.0	± 0.7	± 0.3	± 8.4	± 0.5	± 2.7
Nordic Naturals	Daily DHA	Anchovy, sardine	1.9	13.9	35.1	61.7	9.2	2.9	23.5	2.5	101.3	21.7	423.5
			± 0.2	+ 0.6	± 5.5	± 4.0	+ 0.0	± 1.6	± 0.2	± 0.2	+ 8.8	± 2.8	± 11.6
		Alleliovy, salulle	+ 0.8	+ 2.8	0.4c	+ 1.9	+ 0.4	+ 0.7	+ 1.1	orc +	292.2 + 10.5	+ 1.9	+ 1-25-
Pure Alaska Omega	Pure Alaska Omega 100% Natural Omega-3 Wild Alaskan Salmon Oil	Wild alaskan salmon	52.0	129.8	24.7	_ 133.6	_ 15.2	- - -:-	4.5	13.3	84.1	22.3	102.3
			± 6.9	± 3.6	+ 1:1	± 13.4	± 0.6	± 0.0	± 0.1	± 0.8	± 6.2	± 1.5	± 3.1
Purelife Naturals	Fresh Alaskan Extra Strength Omega-3 Fish Oil	n/a	0.1	1.0	0.2	35.2	13.9	3.5	6.9	7.7	353.6	44.4	183.4
ReNew I ife	Omega Smart Super Krill	Krill and fish oils	± 0.0 45.4	± 0.1 87.6	± 0.0 7 4	± 0.6	± 0.2 10.8	± 0.1	± 0.1	± 0.2	± 7.0	± 0.9	± 3.2 117.9
			+ 4.	± 2.9	± 1.3	± 2.5	± 1.0	± 0.2	± 1.0	± 0.5	+ 3.8	± 1.5	± 2.3
	Omega Smart Ultimate Fish Oils, Super Critical	n/a	0.4	1.4	3.7	10.8	2.2	2.2	31.2	1.9	581.4	12.7	88.7
			± 0.0	± 0.0	± 0.1	± 0.2	± 0.0	± 0.0	± 0.7	± 0.0	± 12.2	± 0.3	± 2.0
Schiff	MegaRed Omega-3 Krill Oil	Krill	68.1	137.9	7.4	64.0	10.2	1.1	1.4	13.0	120.8	2.4	53.5
		•	± 3.4	± 4.3	± 0.3	± 2.9	± 0.5	± 0.0	± 0.0	± 0.5	± 2.0	± 0.0	± 0.7
Simply Right	Fish Oil 1200 mg, Vitamin D-3	Mackerel, sardine, anchovy	2.8	23.6	33.0	60.9	7.6	1.5	13.0	4.6	266.3	42.8	186.9
		-	+ 1:0	± 2.1	± 0.7	+ 0.8	+ 0.1	+ 0.0	± 0.2	± 0.1	± 4.7	± 0.7	± 3.7
	Fish Oil 600 mg, with 300 mg Omega-3	Mackerel, sardine, anchovy	4.9	31.7	32.1	58.1	6.2 2.2	1.6	13.2	3.9	278.1	46.0 2 2	168.1
			± 1.7	± 4.0	± 1.0	± 5.3	± 0.3	± 0.1	± 0.2	± 0.3	± 11.0	± 0.4	土 5.4

Sample Preparation

Two cod liver oil dietary supplements, four algal oil dietary supplements, 10 krill oil dietary supplements, and 31 fish oil dietary supplements were purchased from 15 drug stores and grocery stores around the Lafayette, IN, and Chesterfield, MO, areas. Two lots of each dietary supplement were purchased. From each bottle, the oil contained in half of the capsules was removed and combined in a 50 mL plastic centrifuge tube (Fisher Scientific) to create a composite sample. The oil was removed from the capsule by puncturing the capsule and using pressure to eject the oil out of the capsule into the tube. The samples were blanketed with nitrogen (99.995% pure; Indiana Oxygen Co., Indianapolis, IN, USA), sealed, and refrigerated at 4 °C until analysis. All analyses were performed within 1 week of opening the capsules, and each sample was analyzed in duplicate.

Derivatization

Fatty acids were measured using a variation of the AOAC Official Method, 991.39, for 'Fatty Acids in Encapsulated Fish Oils'.²⁶ Methyl tricosanoate (10 mg) as an internal standard, supplement oil (200 mg), and methanolic sodium hydroxide (2 mL of a 0.5 N solution) were combined in a Kimax screw-cap glass culture tube (Fisher Scientific). The tube was agitated (Fisher Vortex Genie 2; Fisher Scientific) and heated for 10 min at 105 °C in a heating block (VWR International, Radnor, PA, USA). During heating, the tube was agitated once, and then cooled to room temperature. Boron trifluoride (3 mL) was added, the tube was agitated, and heated again at 105 °C for 30 min. The tube was agitated once during heating, allowed to cool to 30-40 °C, and then isooctane (1 mL) was added and the tube was agitated for 30 s. Saturated sodium chloride (4 mL) was added, the tube was agitated for 30 s, and centrifuged for 5 min at $300 \times q$ (International Clinical Centrifuge Model CL; International Equipment Co., Needham Heights, MA, USA). The organic isooctane layer was transferred to a clean glass culture tube (VWR International). Butylated hydroxytoluene-methanol $(50 \,\mu\text{L}\text{ of a } 10 \,\text{mg}\,\text{mL}^{-1}\text{ solution})$ and isooctane $(1 \,\text{mL})$ were added to the original glass tube and agitated for 30 s. The tube was then centrifuged for 5 min before transferring the organic isooctane layer to the test tube containing the previously removed isooctane layer. Approximately 1 mL of the combined isooctane layers was transferred to a GC vial, (VWR International) flushed with nitrogen, and capped.

GC-FID determination of fatty acids

The derivatized fatty acid methyl esters were quantified using gas chromatography with split/splitless injector and a flame ionization detector (GC/FID, Varian 3900 GC, CP-8400 auto sampler, CP-8410 auto injector; Varian Analytical Instruments, Walnut Creek, CA, USA). A CP-52CB wax capillary column was used for analysis (CP 8843, 30 m × 0.32 mm I.D., DF-25 coating thickness 0.25 μ m; Agilent Technologies, Inc., Santa Clara, CA, USA). Operating conditions were: injection port temperature, 250 °C; detector temperature, 300 °C; oven programmed from 170 °C for 4 min to final hold temperature of 240 °C for 4 min, with an increase of 3 °C min⁻¹; helium carrier gas, 2.5 mL min⁻¹ (99.995% pure, Indiana Oxygen Co., Indianapolis, IN, USA). The FID operated with the following flow rates: helium, 25 mL min⁻¹; hydrogen, 30 mL min⁻¹ (99.8% pure; Inweld Corp., Indianapolis, IN, USA); compressed air, 300 mL min⁻¹ (commercial grade; Specialty Gases of America, Toledo, OH, USA).

Menhaden oil (PUFA No. 3; Supelco, Bellefonte, PA, USA) and GLC 462 (Nu-Chek Prep, Inc. Elysian, MN, USA) were used for peak

Table 1. Continued	per												
Vendor	Product name	Fish ^a	14:0	16:0	18:0	18:1 <i>n</i> 9	18:2 <i>n</i> 6	18:3 <i>n</i> 6	20:4 <i>n</i> 6	18:3 <i>n</i> 3	20:5 <i>n</i> 3	22:5n3	22:6n3
	Triple Strength Fish Oil	Mackerel, sardine, anchovy	1.0	2.3	15.8	24.8	2.9	1.2	20.5	1.8	404.1	45.9	152.7
			± 0.1	± 0.2	± 1.7	± 1.4	± 0.2	± 0.1	± 0.7	± 0.1	± 10.5	± 1.3	± 5.3
	Wild Alaskan Salmon Oil, 1200 mg	Salmon	46.0	141.6	22.0	154.9	16.8	1.0	4.0	11.6	81.8	18.2	79.8
			± 1.0	± 2.6	± 0.5	± 3.5	± 0.3	± 0.4	± 0.0	± 0.3	± 0.9	± 0.3	± 1.5
Spring Valley	Fish Oil with D ₃ , Double Strength, 1200 mg Mack	Mackerel, sardine, anchovy	2.2	27.5	33.4	64.1	7.9	1.7	13.2	4.8	267.5	41.8	176.4
			± 0.3	± 7.2	± 1.8	± 5.2	± 0.5	± 0.2	± 0.3	± 0.1	± 3.6	± 1.3	± 14.3
	Pure Krill Oil	Krill	76.2	139.4	8.1	72.6	11.2	1.1	1.6	9.6	111.0	2.2	51.6
			± 15.3	± 22.7	+ 1.1	± 10.4	± 1.6	± 0.2	± 0.4	± 2.7	± 24.1	± 0.4	± 11.5
	Triple Strength Fish Oil, 1400 mg	Mackerel, sardine, anchovy	1.4	3.8	17.9	26.3	3.0	0.7	20.4	2.0	406.6	44.6	151.5
			± 0.2	± 0.4	± 2.2	± 1.2	± 0.2	± 0.1	± 1.8	± 0.4	± 6.8	± 0.7	± 2.6
Sundown Naturals	s Plant Based, Omega-3	Algae (Schizochytrium spp.)	11.8	154.7	17.3	246.7	19.5	0.2	12.0	0.6	151.1	29.1	237.8
			± 0.5	± 5.2	± 0.5	± 7.2	± 0.6	± 0.0	± 0.3	± 0.0	± 4.3	± 0.8	± 6.3
Walgreens	100% Pure Omega-3 Krill Oil	Krill	74.7	134.8	6.4	63.3	12.1	1.1	2.2	7.6	109.7	2.8	58.1
			± 3.1	± 5.5	± 0.3	± 4.9	± 0.6	± 0.1	± 0.1	± 0.4	± 3.2	± 0.3	± 5.0
^a Indicates the fish	^a Indicates the fish or algae species used to make each product.												

Vendor	Product Name	SFA ^a	MUFA ^b	<i>n</i> -6 ^c	<i>n</i> -3 ^d
Arctic Naturals	Daily Omega-3	241.2	197.5	25.3	307.1
	Krill Omega-3	227.1	184.7	22.8	277.9
	Total Omega	228.6	204.2	106.4	253.5
	Ultra Omega-3	41.9	101.1	35.0	543.8
CVS Pharmacy	100% Natural Wild Alaskan Salmon Oil	201.7	315.3	26.9	261.0
	100% Pure 300 mg, Omega-3 Krill Oil	217.3	146.4	13.5	219.8
	Natural Omega-3 Fish, Flax and Borage Oil	92.3	173.5	245.7	399.4
	Odorless Fish Oil 1200 mg, Heart, Joint & Skin Health	217.7	207.2	26.1	303.1
Equaline	Omega-3 Fish Oil, 1200 mg	223.7	208.7	27.0	314.1
Finest Nutrition	Cod Liver Oil	206.0	349.9	28.1	249.3
	DHA Algal-900, Triple Strength	208.5	138.9	28.0	349.0
	Double Strength 1200 mg, Fish Oil	76.2	139.9	32.9	515.3
	Regular Strength 600 mg, Fish Oil	79.9	143.0	32.7	503.2
	Triple Strength, 1400 mg, Fish Oil	27.6	64.2	36.8	640.8
	Wild Alaskan Salmon Oil	211.0	336.6	26.1	241.7
Kroger	Fish Oil 554 mg, Hearth Health Omega-3	19.3	34.8	38.0	669.7
Meijer Naturals	Algal-900 DHA	222.9	135.9	25.8	351.1
	Cod Liver Oil	181.6	330.8	30.7	240.0
	Omega 3 High EPA Fish Oil	29.0	81.0	37.7	657.8
Natrol	Omega-3 Krill Oil	226.2	160.6	14.7	244.6
Nature's Bounty	Dual Spectrum Omega-3	69.9	93.6	31.2	549.8
,	Odor-Less Double Strength Fish Oil, 2400 mg	66.6	160.6	36.2	491.2
	Odor-Less Triple Strength Fish Oil, 1400 mg	28.9	62.3	38.8	632.8
	Red Krill Oil	226.5	158.8	19.6	188.9
	Triple Strength Red Krill Oil	234.0	193.9	19.6	194.8
Nature Made	Burp-Less Fish Oil, 1200 mg	239.0	189.7	23.7	290.4
	Fish Oil, 1200 mg	259.3	209.0	25.5	310.2
	Fish Oil Pearls	48.4	94.4	32.1	567.9
	Full Strength Minis, Super Omega-3	23.5	52.0	33.9	654.9
	Krill Oil	218.7	161.4	15.2	229.6
	Ultra Omega-3 Fish Oil, 1400 mg	26.6	62.0	37.2	690.0
Nordic Naturals	Daily DHA	61.0	96.6	45.0	562.3
	Daily Omega with Vitamin D ₃	69.4	122.8	36.2	567.9
Pure Alaska Omega	100% Natural Omega-3 Wild Alaskan Salmon Oil	208.4	303.0	26.2	267.2
Purelife Naturals	Fresh Alaskan Extra Strength Omega-3 Fish Oil	3.4	57.2	37.3	637.6
ReNew Life	Omega Smart Super Krill	142.2	137.0	26.2	401.4
	Omega Smart Ultimate Fish Oils, Super Critical	7.7	22.1	28.9	731.5
Schiff	MegaRed Omega-3 Krill Oil	214.9	140.0	13.2	233.1
Simply Right	Fish Oil 1200 mg, Vitamin D-3	65.6	118.1	28.8	529.7
	Fish Oil 600 mg, with 300 mg Omega-3	75.1	120.4	27.8	524.3
	Triple Strength Fish Oil	25.4	58.2	32.9	628.0
	Wild Alaskan Salmon Oil, 1200 mg	211.3	337.6	26.2	229.8
Spring Valley	Fish Oil with D_3 , Double Strength, 1200 mg	68.7	122.2	30.0	520.2
,	Pure Krill Oil	225.6	157.2	14.5	214.0
	Triple Strength Fish Oil, 1400 mg	29.7	61.7	32.6	630.2
Sundown Naturals	Plant Based, Omega-3	191.1	251.4	33.9	425.5
Walgreens	100% Pure Omega-3 Krill Oil	217.9	151.2	16.2	208.4

^a Total of all SFA, including 12:0, 14:0, 16:0, 18:0, 20:0, 22:0, and 24:0.

^b Total of all MUFA, including 14:1*n*5, 16:1*n*7, 18:1*n*7, 18:1*n*9, 20:1*n*9, 22:1*n*9, and 24:1*n*9.

^c Total of all omega-6 fatty acids, including 18:2*n*6, 18:3*n*6, 20:2*n*6, 20:3*n*6, 20:4*n*6, 22:2*n*6, and 22:4*n*6.

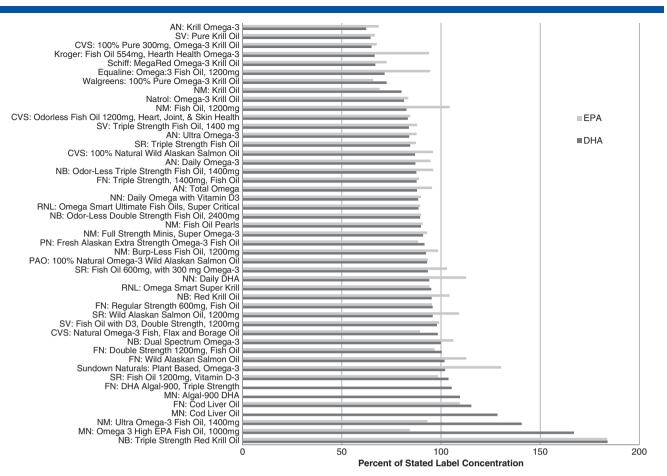
^d Total of all omega-3 fatty acids, including 18:3n3, 18:4n3, 20:3n3, 20:4n3, 20:5n3, 22:5n3, and 22:6n3.

identification. These standards were run every 30 samples during testing.

Calculations/quantitative analysis

Quantitative analysis was performed by comparing the area of each peak in the GC spectrum to the area of the internal standard peak (C23:0), according to AOAC method 991.39²⁶ and Tvrzická

*et al.*²⁷ A total of 28 fatty acids were measured, including saturated fatty acids (C12:0, C14:0, C16:0, C18:0, C20:0, C22:0, and C24:0), mono-unsaturated fatty acids (C14:1*n*5, C16:1*n*7, C18:1*n*7, C18:1*n*9, C20:1*n*9, C22:1*n*9, and C24:1*n*9), omega-3 fatty acids (C18:3*n*3, C18:4*n*3, C20:3*n*3, C20:4*n*3, C20:5*n*3, C22:5*n*3, and C22:6*n*3), and omega-6 fatty acids (C18:2*n*6, C18:3*n*6, C20:2*n*6, C20:4*n*6, C22:2*n*6, and C22:4*n*6).



www.soci.org

Figure 1. Stated label amounts (in %) of EPA and DHA in 47 dietary supplements. Abbreviations indicate vendors: AN, Arctic Naturals; CVS, CVS Pharmacy; FN, Finest Nutrition; MN, Meijer Naturals; NB, Nature's Bounty; NM, Nature Made; NN, Nordic Naturals; PAO, Pure Alaska Omega; PN, Purelife Naturals; RNL, ReNew Life; SR, Simply Right; and SV, Spring Valley.

RESULTS AND DISCUSSION

Fatty acids concentrations for two cod liver oil dietary supplements, four algal oil dietary supplements, 10 krill oil dietary supplements, and 31 fish oil dietary supplements are shown in Table 1 and Table 2. For the saturated fatty acids (SFAs), mysteric acid (C14:0), palmitic acid (C16:0), and stearic acid (C18:0) were present in the highest concentrations. Total SFA concentration in the supplements ranged from 3.4 to 259.3 mg g⁻¹ oil, with the average concentration being 134.7 mg g⁻¹ oil. Oleic acid (C18:1*n*9), the predominant monounsaturated fatty acid present, ranged in concentration from 10.8 to 246.7 mg g⁻¹ oil.

Of the long-chain omega-3 fatty acids, EPA was present in the highest concentrations in all of the supplements tested, except for the algal oil supplements which had higher levels of DHA. This is due to the fact that the fish used in the supplements tested (anchovies, cod liver, krill, mackerel, salmon, and sardines) have slightly higher concentrations of EPA than DHA,^{28,29} while algae (*Schizochytrium* spp.) has more DHA than EPA.³⁰ The concentration of EPA present in fish- and krill-based supplements ranged from 81.8 to 454.6 mg g⁻¹ oil, while the range for algal oil supplements was 7.7 to 151.1 mg g⁻¹ oil. DHA concentration in the algal oil dietary supplements ranged from 237.8 to 423.5 mg g⁻¹ oil, while the range for all other supplements was 51.6 to 220.4 mg g⁻¹ oil. Docosapentaenoic acid (DPA, C22:5*n*3) and α -linolenic acid (ALA, C18:3*n*3) were also present in all samples tested. DPA concentration ranged from 2.2 to 49.5 mg g⁻¹ oil and ALA ranged from 0.6

to 13.3 mg g⁻¹ oil, excluding the sample (CVS Pharmacy, Natural Omega-3 Fish, Flax and Borage Oil) containing flax and borage oil which had an average ALA concentration of 207.6 mg g⁻¹ oil. The total average concentration of omega-3 fatty acids was 418.2 mg g⁻¹ oil.

Omega-6 fatty acids were present in lower amounts than omega-3 fatty acids. The total concentration of omega-6 fatty acids ranged from 13.2 to 106.4 mg g⁻¹ oil, excluding the sample that contained flax and borage oil in addition to fish oil. The total average concentration of omega-6 fatty acids was 34.6 mg g⁻¹ oil. The most abundant omega-6 LC-PUFAs in the samples were linoleic acid (LA), γ -linolenic acid (GLA), and arachidonic acid (ARA). Excluding the sample with flax and borage oil, the concentration of LA did not exceed 10.3 mg g⁻¹ oil, and the concentration of ARA was below 31.2 mg g⁻¹ oil. Two supplements had high concentrations of GLA (30.6 and 66.2 mg g⁻¹ oil), while all others were in the range of 0.2 to 3.5 mg g⁻¹ oil.

All products included in this study had the total amount of EPA, DHA, or both listed on the bottle. Figure 1 illustrates the % of the stated label amount for EPA and DHA. EPA ranged from 66% to 184% of the stated label amount, while the range for DHA was 62% to 184%. Only 10 supplements contained at least 100% of the stated label claim for EPA, while 12 had at least 100% of the stated label claim for DHA. Thus, 74% of the supplements tested contained less than the stated label amount of EPA and DHA. To be compliant with U.S. Food and Drug Administration (FDA)

Table 3. Comparison of fish oil composition to the amounts stated on the labels

Study	<100% stated label amount ^a	<80% stated label amount ^b
Current work	74%	16%
Shim <i>et al.</i> ³²	91%	73%
Ackman <i>et al.</i> ³³	75%	16%
Sullivan Ritter <i>et al.</i> ³⁴	59%	13%

 $^{\rm a}\,\%$ of supplements with less than the stated label amount of EPA or DHA.

 $^{\rm b}$ % of supplements that would be considered non-compliant with labeling regulations enforced by the U.S. Food and Drug Administration.

labeling requirements,³¹ a supplement must contain at least 80% of the stated label amount of each component listed. Using this criterion, six supplements were non-compliant for EPA and eight were non-compliant for DHA.

Overall, these results are similar to previously published studies.³²⁻³⁴ A summary and comparison of the results in these studies is shown in Table 3. All studies found that a majority of omega-3 dietary supplements sold contain less than the stated label amount of EPA or DHA. However, two of the studies, along with this study, found that only a small percentage of the supplements tested were not compliant with current labeling regulations in the USA. The fourth study found that 73% of the supplements tested were non-compliant, though it is not clear why this was the case.³² This work was completed by previous members of our lab and supplements in both studies were purchased from some of the same vendors. Thus, it appears that while little improvement has been made in producing supplements containing as much EPA and DHA as the label states, the compliance of supplements with FDA regulations has increased dramatically over the past decade.

There are many possible reasons for the supplements containing less than the stated label amount of EPA and DHA. One reason may be the fluctuations in the fatty acid concentrations of fish during different times of the year.³⁵ Additionally, fish from different sources (i.e. farmed or wild) may have different dietary levels of fatty acids, based on feeding regimens.^{35,36} Finally, it is possible that manufacturers are aiming to meet the regulatory compliance threshold of 80% rather than 100% of the stated label amounts as a way to limit production costs associated with fish oils.

Overall, this study demonstrates the wide variability between the stated label amounts of EPA and DHA and those determined analytically. These results indicate that there is still room for improvement in the regulation of dietary supplements, as all of the non-compliant supplements were commercially available. In addition to government regulation, manufacturers must improve quality assurance processes of dietary omega-3 supplements to ensure their supplements contain at least the stated label amount of EPA and DHA. This study updates the current status of omega-3 supplements in the USA, and the results should inform consumers about the potential deficiencies in the regulation of labeling practices of commercial dietary omega-3 supplements.

ACKNOWLEDGEMENTS

This research was funded by a USDA grant, National Institute of Food and Agriculture, IND 0-2010-01295.

REFERENCES

- 1 Watters C, Edmonds C, Rosner L, Sloss K and Leung P, A cost analysis of EPA and DHA in fish, supplements and foods. *J Nutr Food Sci* **2**: doi:10.4172/2155-9600.1000159 (2012).
- 2 Radimer K, Bindewald B, Hughes J, Ervin B, Swanson C and Picciano M, Dietary supplement use by US adults: Data from the National Health and Nutrition Examination Survey, 1999–2000. *Am J Epidemiol* **160**:339–349 (2004).
- 3 Bailey R, Gahche J, Lentino C, Dwyer J, Engel J, Thomas P, et al., Dietary supplement use in the United States, 2003–2006. J Nutr 141:261–266 (2011).
- 4 Ackman R, Fatty Acids in Food and Their Health Implications. CRC Press, Boca Raton, FL (2007).
- 5 Swanson D, Block R and Mousa S, Omega-3 fatty acids EPA and DHA: health benefits throughout life. *Adv Nutr* **3**:1–7 (2012).
- 6 Dunstan J, Barden A, Beilin L, Holt P, Calder P, Taylor A, et al., Effects of n-3 polyunsaturated fatty acids supplementation in pregnancy on maternal and fetal erythrocyte fatty acid composition. Eur J Clin Nutr 58:429–437 (2004).
- 7 Palmer D, Sullivan T, Gold M, Prescott S, Heddle R, Gibson R, *et al.*, Effect of n-3 long chain polyunsaturated fatty acid supplementation in pregnancy on infants' allergies in first year of life: randomised controlled trial. *BMJ* **344**:e184 (2012).
- 8 Luchtman D and Song C, Cognitive enhancement by omega-3 fatty acids from child-hood to old age: Findings from animal and clinical studies. *Neuropharmacology* 64:550–565 (2013).
- 9 Milte C, Parletta N, Buckley J, Coates A, Young R and Howe P, Eicosapentaenoic and docosahexaenoic acids, cognition, and behavior in children with attention-deficit/hyperactivity disorder: a randomized controlled trial. *Nutrition* 28:670–677 (2012).
- 10 Colombo J, Carlson S, Cheatham C, Shaddy D, Kerling E, Thodosoff J, et al., Long-term effects of LCPUFA supplementation on childhood cognitive outcomes. Am J Clin Nutr 98:403–412 (2013).
- 11 Innis S, Essential fatty acid metabolism during early development, in *Biology of Metabolism in Growing Animals*, ed. by Burrin D and Mersmann H. Elsevier, Philadelphia, PA, USA, pp. 235–274 (2005).
- 12 Jensen C, Voigt R, Llorente A, Peters S, Prager T, Zou Y, et al., Effects of early maternal docosahexaenoic acid intake on neuropsychologial status and visual acuity at five years of age of breast-fed term infants. J Pediatr 157:900–905 (2010).
- 13 Jacques C, Levy E, Muckle G, Jacobson S, Bastien C, Dewailly E, *et al.*, Long-term effects of prenatal omega-3 fatty acid intake on visual function in school-age children. *J Pediatr* **158**:83–90 (2011).
- 14 Salvig J and Lamont R, Evidence regarding an effect of marine n-3 fatty acids on preterm birth: A systematic review and meta analysis. Acta Obstet Gynecol **90**:825–838 (2011).
- 15 Lucas M, Dewailly E, Muckle G, Ayotte P, Bruneau S, Gingras S, et al., Gestational age and birth weight in relation to n-3 fatty acids among Inuit (Canada). *Lipids* **39**:617–626 (2004).
- 16 Rogers L, Valentine C and Keim S, DHA supplementation: current implications in pregnancy and childhood. *Pharmacol Res* 70:13–19 (2013).
- 17 Musa-Veloso K, Binns M, Kocenas A, Chung C, Rice H, Oppedal-Olsen H, et al., Impact of low v. moderate intakes of long chain n-3 fatty acids on risk of coronary heart disease. Br J Nutr **106**:1129–1141 (2011).
- 18 Leaf A, Albert C, Josephson M, Steinhaus D, Kluger J, Kang J, et al., Prevention of fatal arrhythmias in high-risk subjects by fish oil n-3 fatty acid intake. *Circulation* **112**:2762–2768 (2005).
- 19 Albert C, Campos H, Stampfer M, Ridker P, Manson J, Willett, et al., Blood levels of long chain n-3 fatty acids and the risk of sudden death. N Engl J Med **346**:1113–1118 (2002).
- 20 Denis I, Potier B, Vancassel S, Heberden C and Lavialle M, Omega-3 fatty acids and brain resistance to ageing and stress: Body of evidence and possible mechanisms. *Aging Res Rev* 12:579–594 (2013).
- 21 Tan Z, Harris W, Beiser A, Au R, Himali J, Debette S, *et al.*, Red blood cell omega-3 fatty acid levels and markers of accelerated brain aging. *Neurology* 78:658–664 (2012).
- 22 Stough C, Downey L, Silber B, Lloyd J, Kure C, Wesnes K, *et al.*, The effects of 90-day supplementation with the omega-3 essential fatty acid docosahexaenoic acid (DHA) on cognitive function and visual acuity in a healthy aging population. *Neurobiol Aging* **33**:824.e1–824.e3 (2012).
- 23 Christen W, Schaumberg D, Glynn R and Buring J, Dietary ω-3 fatty acid and fish intake and incident age-related macular degeneration in women. Arch Ophthalmol **129**:921–929 (2011).

- 24 Kiecolt-Glaser J, Belury M, Andridge R, Malarkey W and Glaser R, Omega-3 supplementation lowers inflammation and anxiety in medical students: A randomized controlled trail. *Brain Behav Immun* 25:1725–1734 (2011).
- 25 Bailey R, Gahche J, Miller P, Thomas P and Dwyer J, Why US adults use dietary supplements. *JAMA Int Med* **173**:355–361 (2013).
- 26 Association of Official Analytical Chemists, Fatty acids in encapsulated fish oils, Method 991.39, in *Official Methods of Analysis*, 18th edition. AOAC International, Rockville, MD, USA (2007).
- 27 Tvrzická E, Vecka M, Staňková B and Žák A, Analysis of fatty acids in plasma lipoproteins by gas chromatography–flame ionization detection quantitative aspects. *Anal Chim Acta* **465**:337–350 (2002).
- 28 U.S. Department of Agriculture and U.S. Department of Health and Human Services. *Dietary Guidelines for Americans 2005, Appendix G-2.* Available: http://www.health.gov/dietaryguidelines/ dga2005/report/html/G2_Analyses.htm#tableg2-adda2 [15 May 2014].
- 29 Gigliotti J, Davenport M, Beamer S, Tou J and Janczynski J, Extraction and characterisation of lipids from Antarctic krill (*Euphausia superba*). Food Chem **125**:1028–1035 (2011).

- 30 Fedorova-Dahms I, Marone P, Bauter M and Ryan A, Safety evaluation of DHA-rich algal oil from *Schizochytrium* sp. *Food Chem Tox* 49:3310–3318 (2011).
- 31 U.S. Food and Drug Administration, *Code of Federal Regulations: Nutrition Labeling of Foods*. U.S. Government Printing Office, Washington, DC (2009).
- 32 Shim S, Santerre C, Burgess J and Deardorff D, Omega-3 fatty acids and total polychlorinated biphenyls in 26 dietary supplements. *J Food Sci* **68**:2436–2440 (2003).
- 33 Ackman R, Ratnayake W and Macpherson E, EPA and DHA contents of encapsulated fish oil products. J Am Oil Chem Soc 66:1162–1164 (1989).
- 34 Sullivan Ritter J, Budge S and Jovica F, Quality analysis of commercial fish oil preparations. *J Sci Food Agric* **93**:1935–1939 (2013).
- 35 Kaya Y and Erdem M, Seasonal comparison of wild and farmed brown trout (*Salmo trutta forma fario* L., 1758). *Int J Food Sci* **60**:412–423 (2009).
- 36 Hossain M, Fish as source of n-3 polyunsaturated fatty acids (PUFAs), which one is better – farmed or wild? *Adv J Food Sci Technol* **3**:455–466 (2011).