



# Concentrating Omega 3's

(Summary)

This paper presents and **OVERVIEW** of the concentration of Omega 3 Fatty Acids in ester forms = EPA & DHA.

The source is strictly fish oil(s) and not marine organisms (algae), nor will it cover indirect source of Omega 3 via ALA (alpha-linolenic acid) conversion through body metabolism sourcing ALA from plants.

The concentration, again, will detail use of Esterification/Molecular Distillation only whereas various other techniques and approaches are used or exist.



## Concentrating Omega 3's

### Chemistry

Most classes of lipids contain one or more fatty acid molecules. All fatty acids are comprised of a carbon chain varying between 2 to 20 or more carbons with an acid or carboxyl group ( $\text{HO} - \text{C} \equiv \text{O}$ ) at one end and a methyl group ( $\text{CH}_3$ ) at the other end.

Fatty acids are classified as

Monounsaturated (only one double bond)

example: oleic acid 18 : 1n – 9

Polyunsaturated (two or more double bonds)

Omega – 3 and Omega – 6 PUFA

example: linoleic acid 18, 2n – 6 (Omega 6)

alpha-linolenic 18 : 3n – 3 (Omega 3)

Saturated (no double bonds)

example: Lauric 12 : 0, etc.

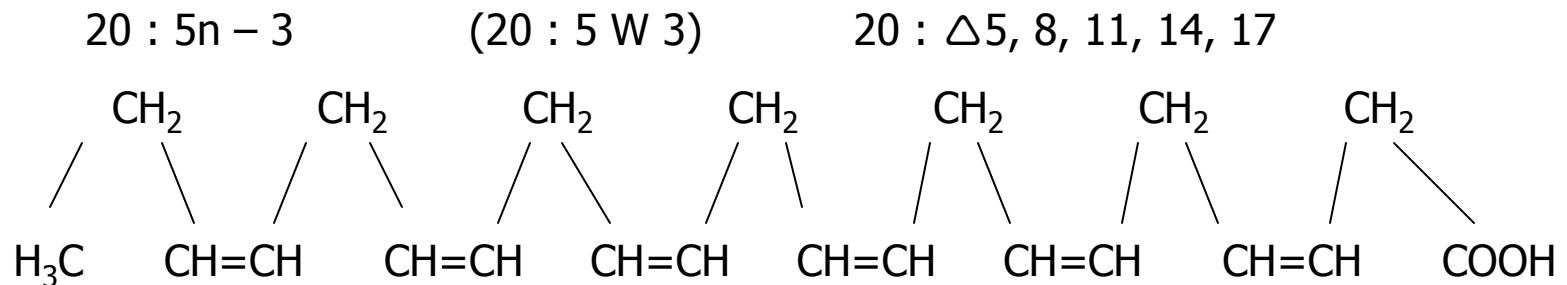


## Concentrating Omega 3's

### Nomenclature

The names of fatty acids are abbreviated according to their chemical structures

Example: EPA is eicosapentaenoic acid



First number 20 refers to number of Carbon atoms in the chain.

Number after " (5) refers to number of double bonds.

N-3 or Omega-3 refers to location of first double bond along the Carbon chain starting with methyl end.

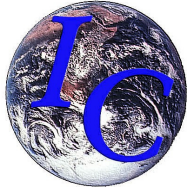
DHA is docosahexaenoic acid  $22 : 6n - 3$



## Concentrating Omega 3's

### Principal Polyunsaturated Fatty Acids

Trivial Name	Shorthand Designation	Abbreviation
<b>Omega-6 PUFA</b>		
Linoleic acid	18:2n6	LA
Gamma-linolenic acid	18:3n6	GLA
Dihomo-gamma linolenic acid	20:3n6	DGLA
Arachidonic acid	20:4n6	AA
Docosatetraenoic acid	22:4n6	DTA
Docosapentaenoic acid	22:5n6	DPA(n-6)
<b>Omega-3 PUFA</b>		
Alpha-linolenic acid	18:3n3	LNA
Eicosapentaenoic acid	20:5n3	EPA
Docosapentaenoic acid	22:5n3	DPA(n-3)
Docosahexaenoic acid	22:6n3	DHA



## Concentrating Omega 3's

### **Why the Interest in Omega 3 PUFA Medical Data on**

- Normal development of the placenta and fetus throughout pregnancy requires adequate supply of PUFAs
- Reduction on ventricular arrhythmia (irregular heartbeat).
- n3/PUFA affect in channels in excitable tissue – brain neuron phenomenon.
- n3/PUFA reduce myocardial oxygen requirement (increase the energy efficiency of the heart) so heart withstands periods of ischemia (high workload).
- Reduction in platelet activity and fibrinogen concentration (anti-thrombotic).  
Reduction of cell growth factors responsible for proliferation in artery walls.
- Benefits in ADHD (Attention Deficit / Hyperactivity Disorder).
- n3/PUFA reduces thromboxane production.
- n3/PUFA reduces level of plasma triglycerol and VLDL.
- n3/PUFA will reduce blood pressure in mildly hypertensive subjects.
- n3/PUFA will reduce total cholesterol.
- Postpartum Depression treatment.



## Concentrating Omega 3's

### Average Daily Dose (USA)

1.1 grams as Pure n-3, PUFA

(2 oz of salmon or mackerel, 3 oz of herring, trout, tuna, sturgeon and anchovy will give 1,100 milligrams per day)



## Concentrating Omega 3's

### Sources

<b>Fish Oils</b>	<b>EPA</b>	<b>DHA</b>
	g/100 gm	
Cod	12	37
Herring	9	11
Haddock	14	24
Mackerel	8	8
Sardine	10	9
Anchovy	18	11
Scallop	21	26
Sole	12	7
Tuna	7	18
Menhaden	12-13	8
Salmon	9	11



# Process of Concentration

## **Step 1: Production of Fish Oil**

Raw fish is cleaned and cooked in pressure cookers, and then passed through screw presses to remove the oil. The fish meal recovered is used for other applications via further processing.

The crude oil is centrifuged to remove solid matter removed as sludge. The oil is further “desludged” to remove smaller particulate to obtain clean, clear crude oils.

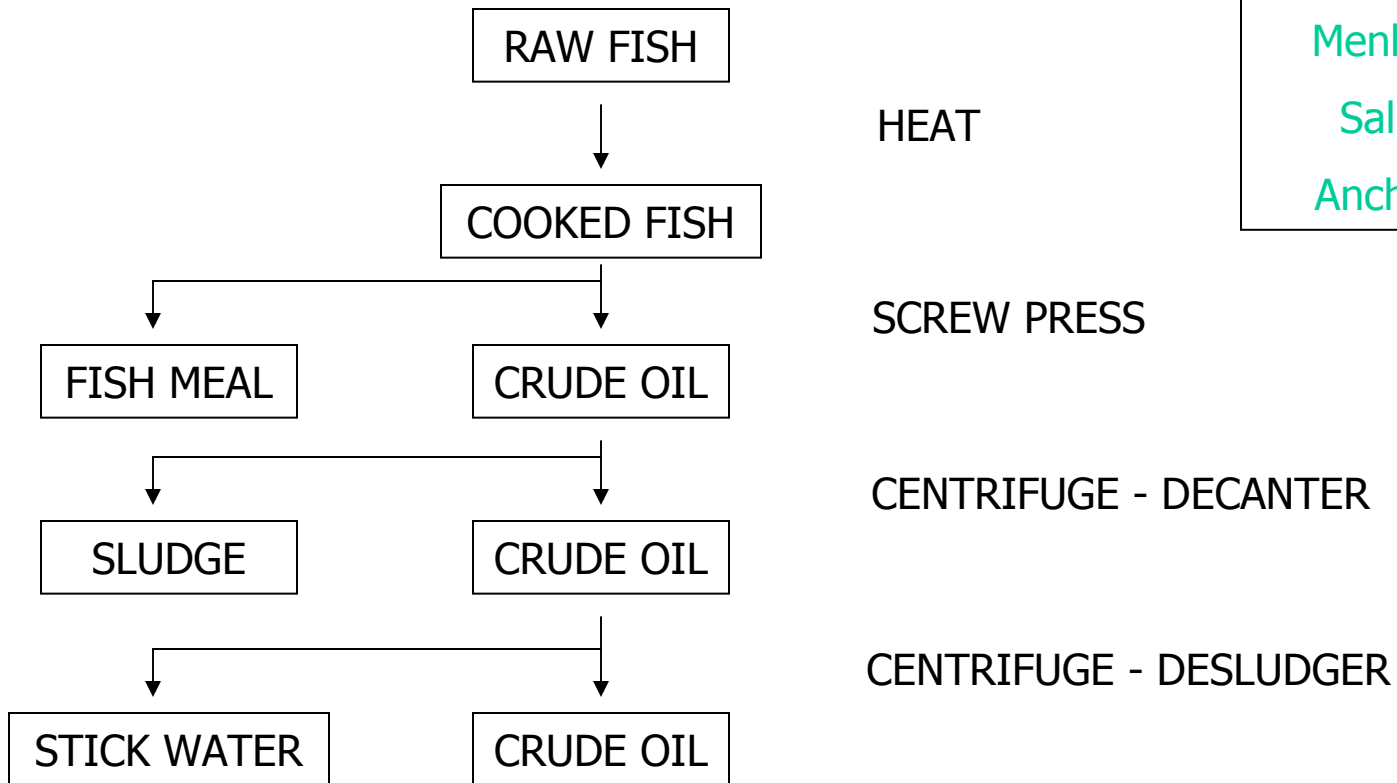




## Process of Concentration

**The schematic for each category is shown:**

### A. Production of Fish Oil



Cod liver  
Herring  
Menhaden  
Salmon  
Anchovies



## Process of Concentration

### **Refining of Crude Fish Oil**

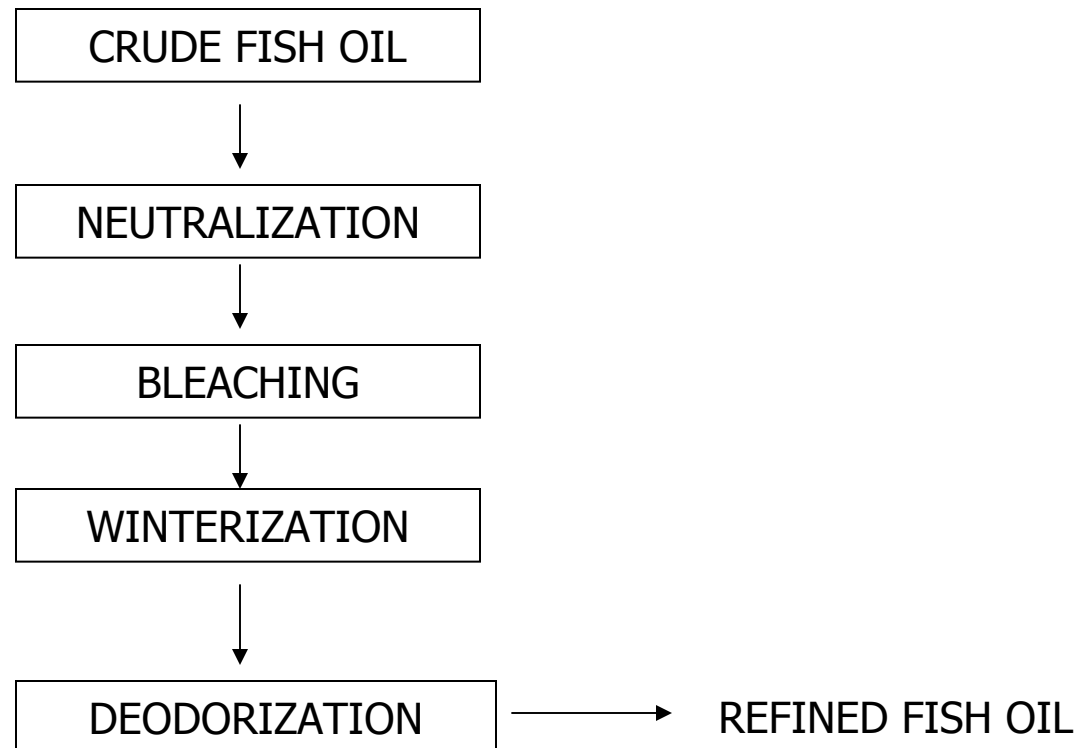
Crude fish oil is refined very much like edible plant oils. The crude fish oil is neutralized (addition of inorganic acids), bleached, winterized and steam deodorized just as soya or corn or other oils. The objective is to remove any metallic and other impurities and phospholipids and odor.

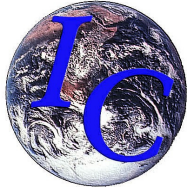
The resulting oil specifications are shown in the next chart.



## Process of Concentration

### B. Refining of Crude Fish Oil





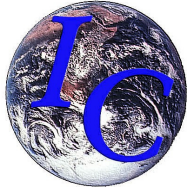
## Process of Concentration

### Product Qualify (Specifications)

#### Refined Fish Oil

(Obtained as feed, but can also be marketed)

Total EPA/DHA (EPA : DHA 1 : 1)	≥	25% wt
Moisture	<	1.0% wt
FFA (as oleic)	<	3.0% wt
Color		12 max (Gardner scale)
Iodine Value		165 - 185



## Process of Concentration

### **Concentration / Purification of Refined Fish Oil into High Concentration**

(EPA / DHA > 55% wt) Omega-3 Fish Oil Esters

#### Urea / Ethanol Crystallization

Most widely used way to separate multi-double bond materials from unsaturated and single or double-olefins. Properly done, it is highly selective and, again, a first-pass crystallization and separation takes the PUFA concentration to 60+% range. In this technique, the esters are mixed with hot solution of urea dissolved in ethanol (solvent). The mixture is then cooled to a final temperature to obtain the desired product composition. Lower unsaturated FA stay as crystals while poly unsaturated remain dissolved.

Disadvantage is it requires large volumes of flammable liquids, requiring storage, heating, chilling, recirculation and filtration.



## Process of Concentration

### **Supercritical Fluid Extraction and Fractionation**

Fish oil esters are subjected to CO<sub>2</sub> extraction. This process exploits the solubility differences between various components of the mixture (reflected in values of partition coefficients). The process can yield 90% EPA / DHA with EPA / DHA of 95% purity.

Disadvantage is the high cost and complex operation of system.



## Process of Concentration

### **CCSE (Counter Current Solvent Extraction)**

In this tried-and-true technique is the workhorse of the entire industry for organic liquid separation. However, the attendant problems of large solvent recovery plants, vapor content, etc., are its disadvantages.

In CCSE, the entire of fish oil is fed continuously through vertical columns while  $\text{CO}_2$  moves in the opposite direction. Esters flow downward while  $\text{CO}_2$  flows in an upward direction.



## Process of Concentration

### **Esterification / Molecular Distillation**

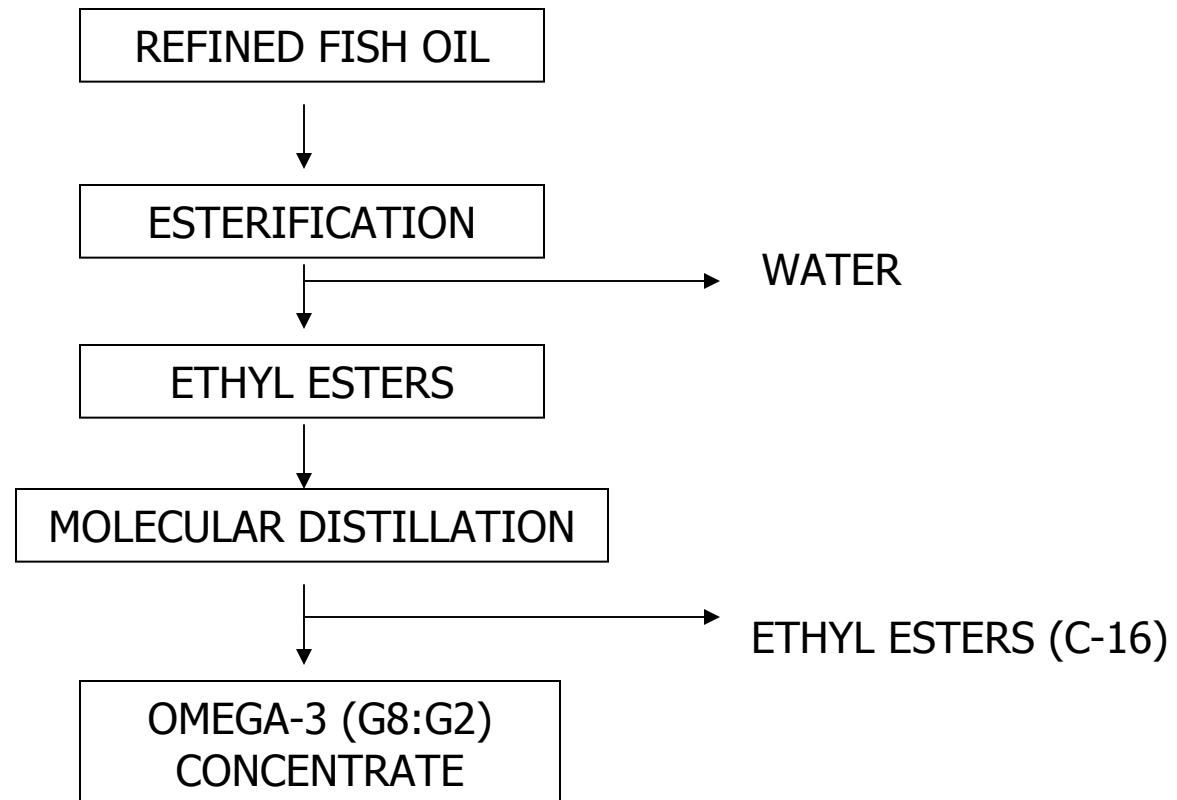
The refined bleached deodorized fish oil containing 18 / 12 EPA / DHA is first subjected to esterification. The procedure involves charging the oil into a reactor with 1:0.5 volume ethanol (anhydrous) and 100 ppm antioxidant (BHT), sodium ethoxide (or KOH), agitate and "cook" for 4-5 hours with a N<sub>2</sub> purge. Sulfuric acid (20%) is added. After ½ hour or so, the reactants cooled, water washed and aqueous phase separated from oil. The oil is vacuum-dried.





## Process of Concentration

### C. Concentration / Purification of Refined Fish Oil into High Concentration (EPA/DHA >55%) Omega-3 Fish Oil Esters





## Process of Concentration

### Molecular Distillation

The basic concept is simple. Distill the  $C_{18}$  fraction. Then, distill the  $C_{20} - C_{22}$  fraction (EPA/DHA concentrate) leaving  $C_{24}^{++}$  as residue. The equipment utilizes 2-stage molecular distillation (2 stills) with a degasser. The degasser removes the moisture left after esterification. The first still removes the 20-50%  $C_{10} - C_{18}$ . The second still removes 40-80%  $C_{20} - C_{22}$ . 5-10% wt constitutes the second still residue. Operating pressures required are 0.005 to 0.01 torr. Temperatures needed are  $170^{\circ}\text{C} - 190^{\circ}\text{C}$ . The yield is  $\sim 70\%$  with EPA/DHA concentration of 55-65% wt.



## Product Data Sheet

### **PRODUCT**

Omega-3 Fish Oil Ester Containing 55% DHA/EPA

### **DESCRIPTION**

A cold-water concentrated fish oil containing 55% DHA/EPA in low-odor easy-to-digest ester form. Selection of fish and proprietary processing techniques produce a pesticide and heavy metal free product. Product is 100% natural and is stabilized with natural mixed tocopherols (Vitamin E).

### **SPECIFICATIONS**

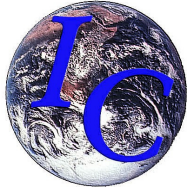
Docosahexaenoic Acid (DHA)	%	min	25
Eicosapentaenoic Acid (EPA)	%	max	30
Total $\omega$ -3 Fatty Acid Content	%	min	55
Acid Value	mg KOH/g	max	2
Peroxide Value	meq O/kg	max	10
Anisidine Value		max	30



## Product Data Sheet

### **SPECIFICATIONS (continued)**

Unsaponifiable Matter	%	typ	2	
Colour (Lovibond)	1"	typ	3	R30Y
Cholesterol	mg/ml	max	5	
Vitamin A	I.U./ml	max	100	
Vitamin D	I.U./ml	max	50	
Vitamin E	PPM	max	2500	
Total Heavy Metals	PPM	max	10	



## Double Blind Smell Test of Omega-3 Fish Oils

Results of smell test from anchovy and salmon oil. The low and high odor comparison between Chile and InCon deodorization of anchovy and salmon oils. Chile deodorization is standard industry deodorization whereas InCon deodorization is molecular still deodorization.

Anchovy Oil Deodorized at Chile	High Odor	(9 High out of 10)
Anchovy Oil Deodorized at InCon	Low Odor	(9 Low out of 10)
Salmon Oil Deodorized at Chile	High Odor	(10 High out of 10)
Salmon Oil Deodorized at InCon	Low Odor	(10 Low out of 10)



# Molecular Still

