GOED - SUPPORTING EPA/DHA PRODUCERS AND FINISHED PRODUCT MANUFACTURERS ACHIEVE PROFICIENCY IN CONTROLLING OXIDATION AND PROVIDING QUALITY PRODUCTS TO CONSUMERS

GERARD BANNENBERG

GLOBAL ORGANIZATION FOR EPA AND DHA OMEGA-3S (GOED), USA
TOPICS THAT WILL BE ADDRESSED IN THIS PRESENTATION

GOED & THE GOED VOLUNTARY MONOGRAPH

ACHIEVING COMPLIANCE:

- ANALYTICAL METHODS WE SUGGEST TO OUR MEMBERS
- RANDOMIZED PRODUCT TESTING
- LABORATORY PROFICIENCY TESTING

INDUSTRY ASSOCIATION WORK - THE NEW ZEALAND FISH OIL REPLICATION STUDY
GLOBAL ORGANIZATION FOR EPA AND DHA OMEGA-3S

ASSOCIATION OF 200+ COMPANIES IN THE OMEGA-3 SECTOR AIMING FOR QUALITY EPA/DHA-CONTAINING OILS AND FINISHED PRODUCTS
Product quality:

- Fatty acid content
- Oxidation state
- Environmental contaminants

Maximum oxidation levels for products that fall within the scope of GOED’s monograph:

- Peroxide Value (PV): max 5 meq O₂/kg
- Para-Anisidine Value (p-AV): max 20
- TOTOX: max 26 - “Total oxidation value” calculated: (2 x PV) + p-AV
SCOPE

Applicable to omega-3 EPA and DHA fatty acids obtained from marine, plant, or microbial sources esterified as triglycerides, re-esterified as triglycerides, or esterified as ethyl esters. Applicable to bulk refined oil products sold as ingredients, refined finished liquid oil products, and finished encapsulated oils intended for use as dietary supplements or foods. Not applicable to crude unrefined oils, formulations, special delivery systems (including solid forms such as microencapsulation), and oils with total omega-3 content below 10% wt/wt. The specifications described herein apply throughout the stated lifetime (shelf-life) of the product.

http://www.goedomega3.com/
METHODS

RECOMMENDED ANALYTICAL METHODS

Oxidation:

- Peroxide Value (PV)
  AOCS Official Method Cd 8b-90
  Ph.Eur. 2.5.5 “Peroxide Value”

- Para-Anisidine Value (p-AV)
  AOCS Official Method Cd 18-90
  Ph.Eur. 2.5.36 “Anisidine Value”

- TOTOX Calculated: (2 x PV) + p-AV

EPA/DHA content:

- The GOED Analytical Method “Assay for EPA and DHA”
- Ph.Eur. 2.4.29 “Composition of Fatty Acids in Oils rich in Omega-3 Acids”
- AOCS Official Method Ce 1i-07 “Determination of Saturated, cis-
  Monounsaturated, and cis-Polyunsaturated Fatty Acids in Marine and Other Oils
  Containing Long-Chain Polyunsaturated Fatty Acids (PUFAs) by Capillary GC”
- USP 401 “Fats and Fixed Oils”
RANDOMIZED TESTING OF EPA/DHA FINISHED PRODUCTS

Products selected randomly from GOED members listing, by an external consultant from stores and internet, and by selection under California Prop 65 regulations. Analyzed by major laboratory (GOED member).

2012 (12 prop 65 / RTP 8)
2013 (24 prop 65 / 23 RTP)
2014 (24 Prop 65 / 9 RTP)

Total 100 finished products, of which 34 from GOED members.

2012: Oxidation, EPA/DHA content not tested.
2013: Oxidation, EPA/DHA content not tested.

2014: EPA/DHA: 9 GOED member products (26 non-member)

Oxidation:

<table>
<thead>
<tr>
<th>Product origin</th>
<th>Tested</th>
<th>PV &gt; 5</th>
<th>P-AV &gt; 20</th>
<th>TOTOX &gt; 26</th>
</tr>
</thead>
<tbody>
<tr>
<td>GOED member</td>
<td>10</td>
<td>6</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>Non-member</td>
<td>26</td>
<td>13</td>
<td>8</td>
<td>9</td>
</tr>
</tbody>
</table>

2016: Focus on NZ fish oil samples / 2017: Focus on US products.
LABORATORY PROFICIENCY TESTING

AOCS-GOED Nutraceutical Oil LPP – run since 2006. Six blinded oil samples/year. Participants measure omega-3 LCPUFA (EPA, DHA, total) and oxidation (PV, p-AV)

Evaluates deviation from the mean measured value. The lab will get an approval when reported results are within specific ranges. Useful feedback for labs to see how they compare to other labs.
BACKGROUND

IN 2015 A STUDY WAS PUBLISHED THAT REPORTED A MARKED NON-COMPLIANCE OF FISH OIL PRODUCTS IN NEW ZEALAND

Fish oil supplements in New Zealand are highly oxidised and do not meet label content of n-3 PUFA


Reported:

- Only 3 of 32 tested fish oils contained EPA plus DHA ≥ label claim
- 69% of products contained less than 67% of the EPA+DHA label claim
- 83% exceeded the recommended PV levels (5 meq O₂/kg)
- 25% exceeded the recommended p-AV levels (20)
- 50% exceeded the recommended TOTOX levels (26)
- Only 8% met all criteria for oxidative quality
BACKGROUND

CONSEQUENCES OF THE 2015 NEW ZEALAND FISH OIL STUDY

Observations surprising and at odds with GOED’s internal randomized testing of member products

- Marked media attention:
  - and little critical assessment.
  - Stuff.co.nz (Jan. 22, 2015): “Fish oil sold in NZ is actually more like snake oil”
  - NZ Herald (Jan. 22, 2015): “Consumers sold short on omega-3 oil”
  - The Guardian (Jan. 21, 2015): “Fish oil rules reviewed as study reveals consumers are being hoodwinked”

- NZ fish oil sales fell 12% following the publication

- Citations: 12 publications by 10 different research groups

Need for a reassessment to confirm results
OBJECTIVE:

A NEW ASSESSMENT OF THE EPA/DHA CONTENT AND OXIDATION STATE OF FISH OIL SUPPLEMENTS IN NEW ZEALAND

Replication study

*Scientific Reports* May 3, 7(1), 1488, 2017

**OMEGA-3 LONG-CHAIN POLYUNSATURATED FATTY ACID CONTENT AND OXIDATION STATE OF FISH OIL SUPPLEMENTS IN NEW ZEALAND.**

METHODS

STUDY SETUP

• Five participating laboratories:

  All participate in the AOCS Laboratory Proficiency Program “GOED Omega-3 Nutraceutical Oils”

  - DSM Nutritional Products, Columbia, MD, U.S.A.
  - Omega Protein, Houston, TX, U.S.A.
  - Eurofins Central Analytical Laboratories, New Orleans, LA, U.S.A.
  - Nutrasource Diagnostics Inc, Guelph, ON, Canada
  - Alkemist Labs, Costa Mesa, CA, U.S.A.

• Tested products: 47 fish oil supplements from the New Zealand market

• Each fish oil was blinded and tested by at least two participating laboratories

• Analysis was asked to be carried out using GOED’s recommended methods
### Summary

Percentage of tested products that complied with the GOED quality criterion for oxidative status (PV max. 5 meq O$_2$/kg; p-AV max. 20; TOTOX max. 26), and that adhered to labeled content of EPA plus DHA. (* : only for unflavored fish oils)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Current study (n=47)</th>
<th>Albert et al 2015 (n=36)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PV</td>
<td>72%</td>
<td>17%</td>
</tr>
<tr>
<td>p-AV</td>
<td>86% *</td>
<td>75%</td>
</tr>
<tr>
<td>TOTOX</td>
<td>77% *</td>
<td>50%</td>
</tr>
<tr>
<td>EPA+DHA content compliance to label claim</td>
<td>91%</td>
<td>9%</td>
</tr>
</tbody>
</table>
# RESULTS

## EPA/DHA CONTENT

*Note, only a subset of the 47 samples is shown here*

<table>
<thead>
<tr>
<th>Company</th>
<th>Brand</th>
<th>Product Name</th>
<th>Alkemist</th>
<th>DSM</th>
<th>Eurofins</th>
<th>Nutra source</th>
<th>Omega Protein</th>
<th>Average</th>
<th>Label Claim</th>
<th>% of Label Claim</th>
</tr>
</thead>
<tbody>
<tr>
<td>BioBalance New Zealand</td>
<td>BioBalance</td>
<td>Omega 3 Fish Oil</td>
<td></td>
<td>285</td>
<td>260</td>
<td>273</td>
<td>300</td>
<td>91%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Fish Oil 1000*</td>
<td></td>
<td>289</td>
<td>296</td>
<td>292</td>
<td>300</td>
<td>97%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Odourless Fish Oil 1000</td>
<td>290</td>
<td>288</td>
<td>264</td>
<td>281</td>
<td>300</td>
<td>94%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Omega Brain</td>
<td>636</td>
<td>585</td>
<td>610</td>
<td>600</td>
<td>102%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Omega Daily</td>
<td>611</td>
<td>559</td>
<td>528</td>
<td>566</td>
<td>600</td>
<td>94%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Omega Joint</td>
<td>747</td>
<td>698</td>
<td>722</td>
<td>670</td>
<td>108%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Omega Triple</td>
<td>851</td>
<td>844</td>
<td>784</td>
<td>826</td>
<td>900</td>
<td>92%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comvita New Zealand</td>
<td>Comvita</td>
<td>Omega 3 Fish Oil</td>
<td></td>
<td>286</td>
<td>269</td>
<td>278</td>
<td>300</td>
<td>93%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>GO Healthy New Zealand</td>
<td>GO Healthy</td>
<td>GO Fish Oil 1,000mg Odourless</td>
<td></td>
<td>280</td>
<td>257</td>
<td>269</td>
<td>300</td>
<td>90%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**EPA/DHA content: 91% of samples compliant with label claim**
## RESULTS

### Peroxide Value

*subset of the 47 samples shown here*

<table>
<thead>
<tr>
<th>Company</th>
<th>Brand</th>
<th>Product Name</th>
<th>Eurofins</th>
<th>Nutrasource</th>
<th>Omega Protein</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sanofi Consumer Healthcare</strong></td>
<td><strong>Nature's Own</strong></td>
<td>Fish Oil 1000mg</td>
<td>8.34</td>
<td>9.81</td>
<td>9.08*</td>
<td></td>
</tr>
<tr>
<td><strong>Sanofi Consumer Healthcare</strong></td>
<td><strong>Nature's Own</strong></td>
<td>Fish Oil 2000mg</td>
<td>7.98</td>
<td>9.02</td>
<td>8.50*</td>
<td></td>
</tr>
<tr>
<td><strong>Sanofi Consumer Healthcare</strong></td>
<td><strong>Nature's Own</strong></td>
<td>Odourless Fish Oil 1500mg</td>
<td>4.86</td>
<td>5.83</td>
<td>5.35*</td>
<td></td>
</tr>
<tr>
<td><strong>Sanofi Consumer Healthcare</strong></td>
<td><strong>Nature's Own</strong></td>
<td>Odourless Fish Oil 2000mg</td>
<td>3.40</td>
<td>4.06</td>
<td>3.73</td>
<td></td>
</tr>
<tr>
<td><strong>Sanofi Consumer Healthcare</strong></td>
<td><strong>Nature's Own</strong></td>
<td>Odourless Fish Oil 1000mg</td>
<td>5.57</td>
<td>6.59</td>
<td>6.08*</td>
<td></td>
</tr>
<tr>
<td><strong>Sanofi Consumer Healthcare</strong></td>
<td><strong>Nature's Own</strong></td>
<td>Concentrated Fish Oil 1-A-Day Odourless</td>
<td>0.99</td>
<td></td>
<td>0.99</td>
<td></td>
</tr>
<tr>
<td><strong>Swisse Wellness</strong></td>
<td><strong>Swisse</strong></td>
<td>Ultiboost Odourless Wild Fish Oil</td>
<td>6.37</td>
<td>7.39</td>
<td>6.88*</td>
<td></td>
</tr>
<tr>
<td><strong>Good Health Products</strong></td>
<td><strong>Good Health</strong></td>
<td>Omega 3 Fish Oil 1500mg</td>
<td>2.47</td>
<td>3.92</td>
<td>3.20</td>
<td></td>
</tr>
<tr>
<td><strong>Good Health Products</strong></td>
<td><strong>Good Health</strong></td>
<td>Omega 3 Fish Oil Health Guard 1000mg</td>
<td>3.30</td>
<td>4.13</td>
<td>3.72</td>
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</tr>
<tr>
<td><strong>Good Health Products</strong></td>
<td><strong>Good Health</strong></td>
<td>Super Omega 3 Health Guard Fish Oil</td>
<td>1.05</td>
<td>1.96</td>
<td>1.51</td>
<td></td>
</tr>
<tr>
<td><strong>Good Health Products</strong></td>
<td><strong>Good Health</strong></td>
<td>O-Mega 3 Bursts</td>
<td>4.16</td>
<td>3.29</td>
<td>3.73</td>
<td></td>
</tr>
<tr>
<td><strong>Integria Healthcare</strong></td>
<td><strong>Thompson's</strong></td>
<td>Fish Oil 1500</td>
<td>3.21</td>
<td>3.22</td>
<td>3.22</td>
<td></td>
</tr>
</tbody>
</table>

**PV: 72% of samples compliant with PV < 5 meq/kg**
## RESULTS

### PARA-ANISIDINE VALUE

*subset of the 47 samples shown here*

<table>
<thead>
<tr>
<th>Company</th>
<th>Brand</th>
<th>Product Name</th>
<th>Eurofins</th>
<th>Nutrasource</th>
<th>Omega Protein</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Unflavored Oils</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sanofi Consumer Healthcare</td>
<td>Nature's Own</td>
<td>Fish Oil 1000mg</td>
<td>12.40</td>
<td>15.96</td>
<td>14.18</td>
<td></td>
</tr>
<tr>
<td>Sanofi Consumer Healthcare</td>
<td>Nature's Own</td>
<td>Fish Oil 2000mg</td>
<td>13.40</td>
<td>16.89</td>
<td>15.15</td>
<td></td>
</tr>
<tr>
<td>Good Health Products</td>
<td>Good Health</td>
<td>Omega 3 Fish Oil 1500mg</td>
<td>10.80</td>
<td>12.61</td>
<td>11.71</td>
<td></td>
</tr>
<tr>
<td>Good Health Products</td>
<td>Good Health</td>
<td>Omega 3 Fish Oil Health Guard 1000mg</td>
<td>9.90</td>
<td>12.04</td>
<td>10.97</td>
<td></td>
</tr>
<tr>
<td>Good Health Products</td>
<td>Good Health</td>
<td>Super Omega 3 Health Guard Fish Oil</td>
<td>3.60</td>
<td>5.74</td>
<td>4.67</td>
<td></td>
</tr>
<tr>
<td>Integria Healthcare</td>
<td>Thompson's</td>
<td>Salmon Oil Plus 1000mg</td>
<td>24.10</td>
<td>24.27</td>
<td>24.19*</td>
<td></td>
</tr>
<tr>
<td>Vitaco Health</td>
<td>NutraLife</td>
<td>Fish Oil 1000mg</td>
<td>11.30</td>
<td>12.31</td>
<td>11.81</td>
<td></td>
</tr>
<tr>
<td>Vitaco Health</td>
<td>NutraLife</td>
<td>Fish Oil 1500mg</td>
<td>11.60</td>
<td>12.28</td>
<td>11.94</td>
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</tr>
<tr>
<td>Vitaco Health</td>
<td>NutraLife</td>
<td>Fish Oil 1500mg Plus Vitamin D</td>
<td>11.10</td>
<td>12.23</td>
<td>11.67</td>
<td></td>
</tr>
<tr>
<td>Comvita New Zealand</td>
<td>Comvita</td>
<td>Omega 3 Fish Oil</td>
<td>13.10</td>
<td>13.16</td>
<td>13.13</td>
<td></td>
</tr>
</tbody>
</table>

**p-AV:** Unflavored fish oils (n=22): 86% compliant with p-AV < 20

(flavored fish oils (n=25): 40% compliance‡)

‡ for comparison only
## RESULTS

### TOTOX

**subset of the 47 samples shown here**

<table>
<thead>
<tr>
<th>Company</th>
<th>Brand</th>
<th>Product Name</th>
<th>Eurofins</th>
<th>Nutra source</th>
<th>Omega Protein</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Unflavored Oils</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BioBalance New Zealand</td>
<td>BioBalance</td>
<td>Omega 3 Fish Oil</td>
<td>19.16</td>
<td>16.19</td>
<td>17.68</td>
<td></td>
</tr>
<tr>
<td>Blackmores</td>
<td>Blackmores</td>
<td>Fish Oil 1000*</td>
<td>18.36</td>
<td>22.73</td>
<td>20.55</td>
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</tr>
<tr>
<td>Comvita New Zealand</td>
<td>Comvita</td>
<td>Omega 3 Fish Oil</td>
<td>17.86</td>
<td>19.16</td>
<td>18.51</td>
<td></td>
</tr>
<tr>
<td>GO Healthy New Zealand</td>
<td>GO Healthy</td>
<td>GO Fish Oil 1,000mg Odourless</td>
<td>7.96</td>
<td>15.63</td>
<td>11.80</td>
<td></td>
</tr>
<tr>
<td>GO Healthy New Zealand</td>
<td>GO Healthy</td>
<td>GO Fish Oil 1,500mg Odourless</td>
<td>10.88</td>
<td>21.38</td>
<td>16.13</td>
<td></td>
</tr>
<tr>
<td>GO Healthy New Zealand</td>
<td>GO Healthy</td>
<td>GO Fish Oil 1-A-Day + Vitamin D3 1,000IU</td>
<td>12.82</td>
<td>20.64</td>
<td>16.73</td>
<td></td>
</tr>
<tr>
<td>Good Health Products</td>
<td>Good Health</td>
<td>Omega 3 Fish Oil 1500mg</td>
<td>15.74</td>
<td>20.45</td>
<td>18.10</td>
<td></td>
</tr>
<tr>
<td>Good Health Products</td>
<td>Good Health</td>
<td>Omega 3 Fish Oil Health Guard 1000mg</td>
<td>16.5</td>
<td>20.3</td>
<td>18.40</td>
<td></td>
</tr>
<tr>
<td>Good Health Products</td>
<td>Good Health</td>
<td>Super Omega 3 Health Guard Fish Oil</td>
<td>5.7</td>
<td>9.66</td>
<td>7.68</td>
<td></td>
</tr>
<tr>
<td>Health &amp; Herbs International</td>
<td>Radiance</td>
<td>Omega 3 EPA &amp; DHA</td>
<td>16.82</td>
<td>20.12</td>
<td>18.47</td>
<td></td>
</tr>
<tr>
<td>Integria Healthcare</td>
<td>Thompson's</td>
<td>Salmon Oil Plus 1000mg</td>
<td>25.52</td>
<td>26.79</td>
<td>26.16*</td>
<td></td>
</tr>
<tr>
<td>Lighthouse Supplements</td>
<td>Lighthouse</td>
<td>Salmon Oil 1000</td>
<td>23.58</td>
<td>27.55</td>
<td>25.57</td>
<td></td>
</tr>
<tr>
<td>PharmaCare Laboratories</td>
<td>Bioglan</td>
<td>Super Fish Oil</td>
<td>64.2</td>
<td>45.15</td>
<td>54.68*</td>
<td></td>
</tr>
</tbody>
</table>

**TOTOX:** Unflavored fish oils (n=22): 77% compliant with TOTOX < 26
(flavored fish oils (n=25): 32% compliance ‡)

‡ for comparison only
EXPLORING THE RELATIONSHIP BETWEEN PV AND P-AV

RESULTS

THIS STUDY

UNFLAVORED FISH OILS

FLAVORED FISH OILS
RESULTS

THIRD-PARTY PRODUCT QUALITY MONITORS / CONSUMER PRODUCT WATCHDOGS

LabDoor.com

IFOS (International Fish Oil Standards program)
A comparatively high and unusual level of primary oxidation in Albert et al 2015 samples
RESULTS

DOES OXIDATION AT THE LIMIT OF NON-COMPLIANCE CONTRIBUTE TO LOSS OF EPA/DHA?
RESULTS

DOES OXIDATION AT THE LIMIT OF NON-COMPLIANCE CONTRIBUTE TO LOSS OF EPA/DHA?
A NOTE ON THE EXPRESSION OF EPA/DHA LEVELS AND OXIDATION

To avoid different rates of compliance in reports:

✓ Express EPA/DHA content in absolute weights (not area % obtained from chromatograms), and in the same chemical form if content is to be compared across products. Ideally, content should be calculated as free fatty acid equivalents on a w/w basis (e.g. mg EPA/g oil product), and then expressed on the label according to applicable regulation (e.g. as triglycerides)

✓ The assay for p-AV measurement is susceptible to interference by flavors, leading to false positive overestimation of p-AV values, and incorrect attribution of non-compliance due to oxidation of PUFAs

✓ PV is expressed by weight (not by volume)

✓ Compliance with EPA and DHA contents claimed on product labels should be assessed against a relevant regulatory threshold. In Australia and New Zealand the regulations indicate that a product should contain at least 90% of claimed content (not 100%)
SUMMARY

The analysis of all available fish oil supplements on the NZ market by at least two independent laboratories, has shown that:

1. 91% are within label claims for EPA and DHA
2. 72% comply with PV, 86% with p-AV, 77% with TOTOX (GOED limits)
3. If Australian regulatory limits are followed, 98% comply with PV (max 10 meq/kg), 100% with p-AV (max 30), 96% with TOTOX (max 50)
4. An inspection of the PV to p-AV relationship suggests that fish oil samples analyzed by Albert et al 2015 had experienced recent exposure to air
5. There is no evidence that measurable oxidative deterioration at the strictest acceptable limits of fish oil supplement quality affects EPA/DHA content
CONCLUSIONS

- Standardization of methods and working towards compliance is being undertaken.
- Improved and new methods are needed that can be used and implemented across the industry.
- The results of the 2015 study by Albert and colleagues could not be confirmed. The majority of fish oil products on the New Zealand market comply with reported EPA/DHA content and strictest industry standards for oxidation. Nearly 100% of products comply with quality standards set by regional regulations.

- The GOED replication study highlights potential issues in the execution of the methods (and deficiencies in reporting) that may have contributed to reporting of high rates of fish oil product non-compliance.

- It is very important that producers of fish oils and finished product manufacturers set product specifications and claims with a reliable test method in mind, and use or select a laboratory with accreditation or demonstrated proficiency for analysis.
THANK YOU

PLEASE CONTACT GOED FOR MORE INFORMATION

GERARD@GOEDOMEGA3.COM