

# Impact of new diet formulations on food safety

J. Nácher-Mestre

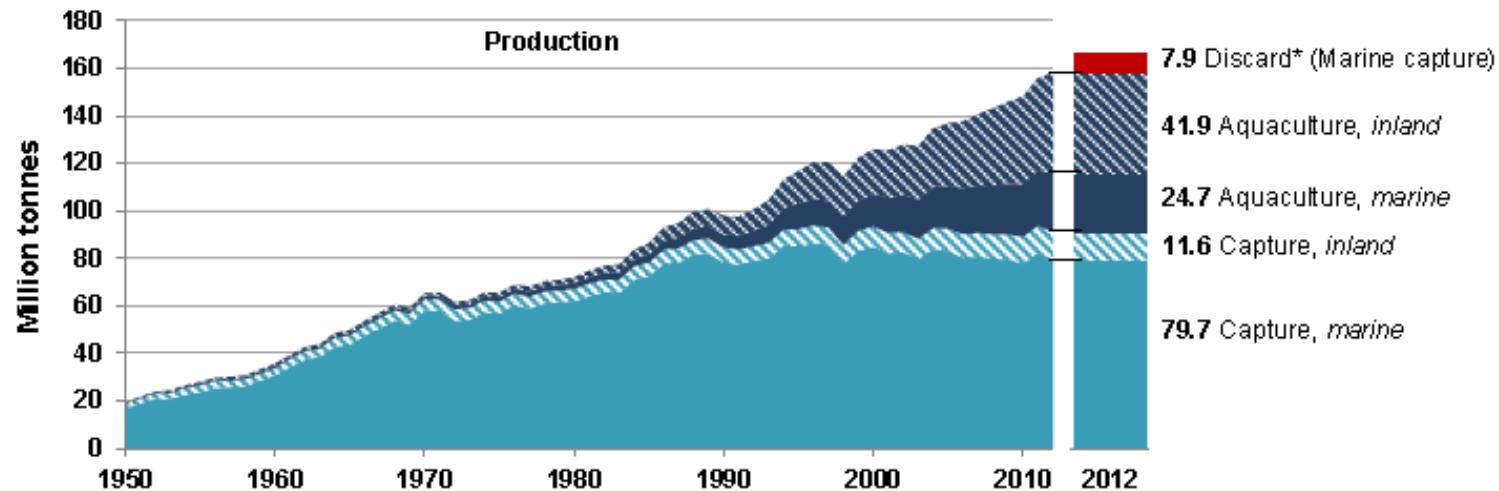
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# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Aquaculture

At a global level, aquaculture is increasing while fisheries remains stable



Sustainable fisheries and aquaculture for food security and nutrition. A report by the High Level Panel of Experts on Food Security and Nutrition of the Committee on World Food Security, Rome 2014.

Marine origin sources for aquaculture are limited  
The validation of new alternatives are advisable

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

Low fish meal & low fish oil availability

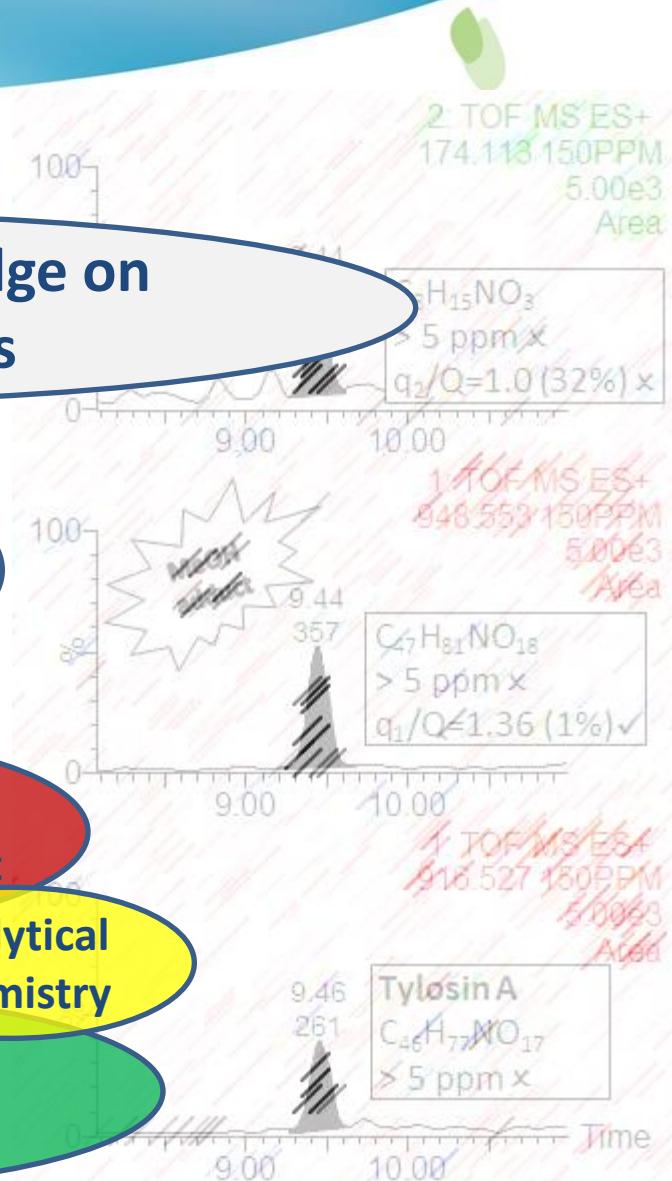
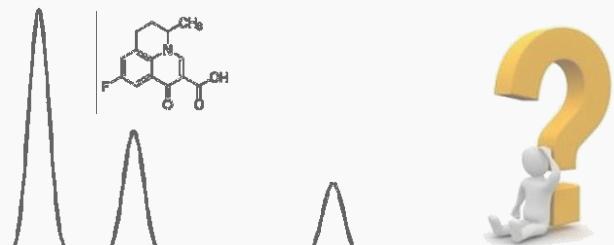
Necessity of knowledge on  
new alternatives

New feed  
formulations

Contaminants  
Ingredient-feed-fillet

Analytical  
Chemistry

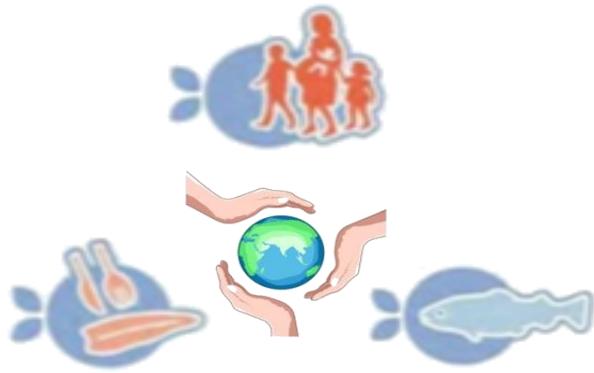
Feed safety & Public  
health Implications



## Why this research is essential?

**EU-legislation on maximum residue levels (MRL) throughout food production chain**

**Legislation goals on feed ingredients and feed are directed towards:**



**Protect consumer safety**

**Protect animal welfare**

**Protect the environment**

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

Why the need of feed-to-fillet transfer assessment?

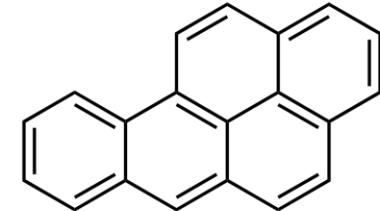
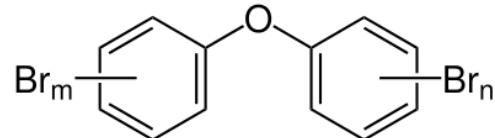
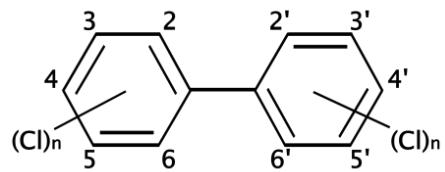
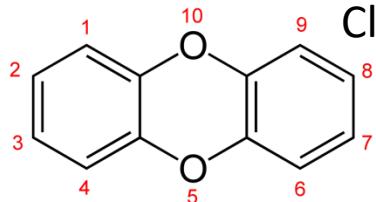
**There are some differences between contaminant groups and chemicals in the group**

**Contaminant groups:**

- Dioxins
- Polychlorinated biphenyls - PCBs
- Brominated flame retardants - PBDEs
- Polyaromatic hydrocarbons - PAHs
- Organophosphate pesticides
- Polar pesticides

**Differences inside of a group:**

- Dioxins: 4 Cl vs 5 Cl vs 6 Cl vs 7 Cl
- PCBs: mono-ortho, non-ortho
- PBDEs: 4 Br vs 5 Br vs 6 Br
- PAHs: light vs heavy PAHs, nº rings



**There are still some unknown TF differences between fish species**

## CONTAMINANTS CLASSIFICATION (aquaculture perspective)

### Classical contaminants

(from classical marine ingredients)

- POPs (Stockholm Convention)  
*([www.pops.int](http://www.pops.int))*
- ✓ OCPs (aldrin, chlordane, heptachlor, HCB,...)
- ✓ PCBs
- ✓ Dioxins, furans
- ✓ PBDEs
- ✓ Non-polar pesticides
- New POPs (HCH, HBCD,...)
- PAHs

### “New” contaminants

(from novel ingredients)

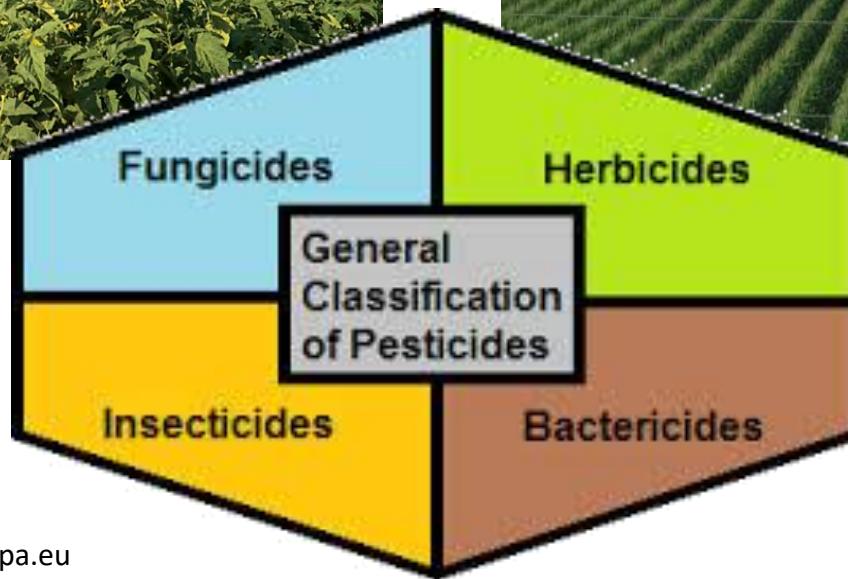
- Polar pesticides (pirimiphos methyl, chlorpyrifos, boscalid, azoxystrobin,...)
- Mycotoxins (AF, FB, DON,...)
- Therapeutic agents
- Dyes

Real advantage of marine substitution by plants --> POPs reduction

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## RELEVANT CONTAMINANTS FROM PLANTS

### Pesticides



## RELEVANT CONTAMINANTS FROM PLANTS

### Mycotoxins



Fusarium fungi species



Aspergillus niger species

13 EU regulated mycotoxins in food (AFB1,  $\Sigma$ AFB1-AFB2-AFG1-AFG2, AFM1, DON, ZEN, OTA, PAT, FB1 + FB2, T-2 and HT-2)

8 EU regulated mycotoxins in feed and feed ingredients (AFB1, DON, ZEN, OTA, FB1 + FB2, T-2 and HT-2)

Commission Recom. Nº 2006/576. Off. J. Eur. Union L229, 7-9

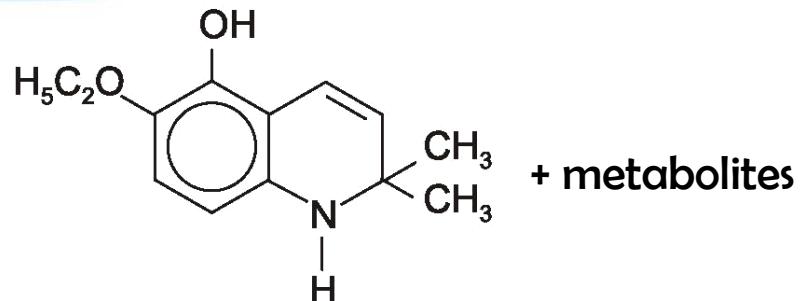
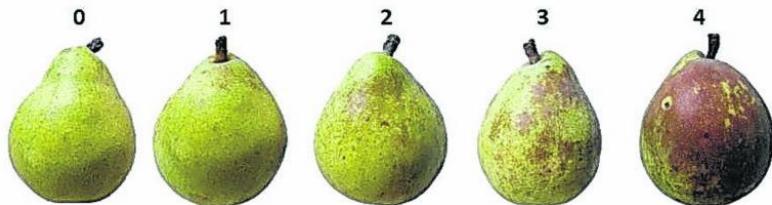
Commission Reg. Nº 1881/2006. Off. J. Eur. Union L364, 5-24

Nácher-Mestre, J. et al., Chemosphere 2015, 128, 314-320

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## OTHER UNDESIRABLES IN AQUACULTURE

### Ethoxyquin (ETQ)



Therapeutic agents – salicylic acid, enrofloxacin, trimethoprim,...



*“Extraordinary disease - veterinary treatment”*



PAP residues - antibiotics, hormones, stimulants, dyes,...



*Pork meal*

*Poultry blood*

*Feather meal*

*Acetaminophen, crystal violet, norgestimate, fluoxetine,...*

Nácher-Mestre, J. et al., JAFC 2013, 2014

Nácher-Mestre, J. et al., Chemosphere 2016, 154, 231-239

Commission regulation (EU) N° 56/2013

Love, D et al., Environ. Sci. Technol. 2012, 46, 3795-3802

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

What contaminants are important for...

...feed-to-fillet transfer and food safety?

depends on  
where it  
comes from

## Marine Ingredients

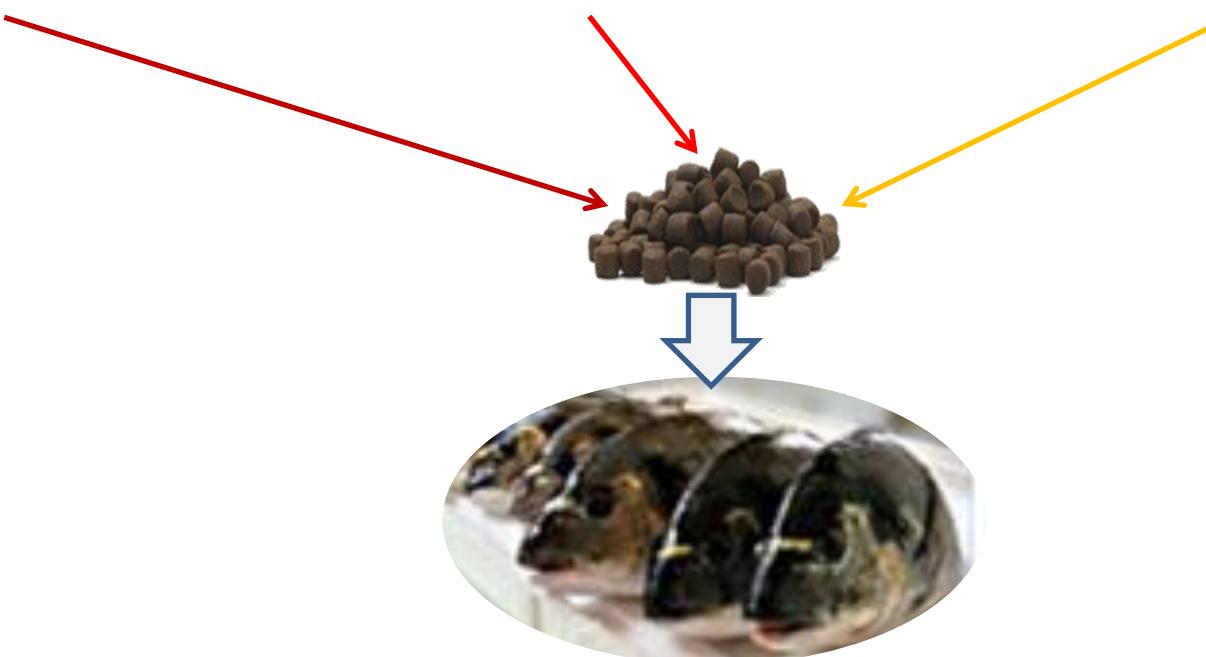
POPs  
Chlorinated pesticides  
PAHs

## Plant Ingredients

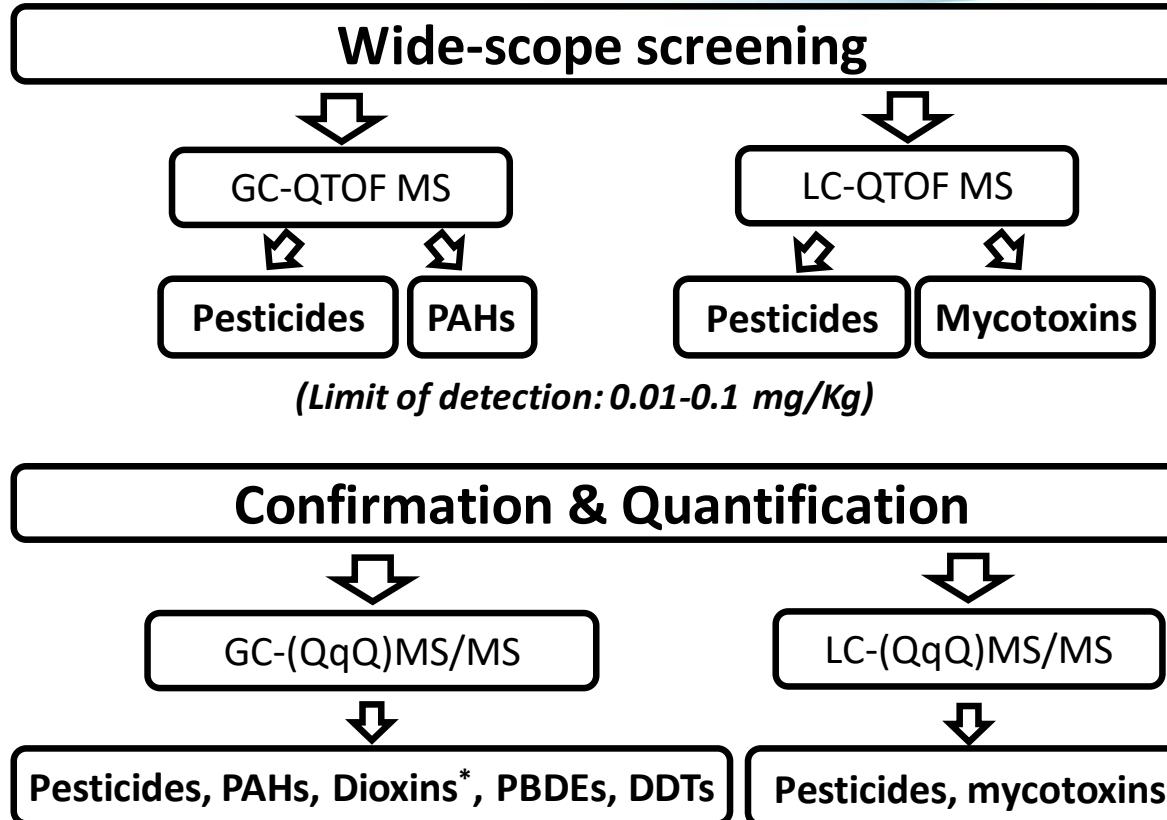
Polar pesticides  
Mycotoxins  
PAHs

## Processed Animal Products (PAP)

Therapeutical agents      PAHs  
Pharmaceuticals  
Hormones  
Dyes



# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY



# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY



AQUACULTURE  
AWARENESS

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## EU INTEREST/PROPOSALS FOR FISH SAFETY:



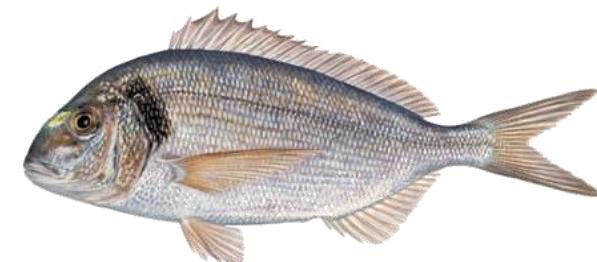
**Development of adequate analytical methods to detect chemical residues and contaminants for fish safety assessment**

## IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## CONTAMINANTS IN AQUACULTURE-data generated

- Knowledge and key information of contaminants on:
    - Marine ingredients
    - Plant meals and plant oils
    - PAPs
  - Established MRLs >> [ ] findings (after plant-based feed)
  - Feed-to-fillet Transfer Factor
  - Fish safety conclusions – future trends

**COMPREHENSIVE DATA FROM  
ACTIVE FEEDING TRIALS ON  
ATLANTIC SALMON AND GILTHEAD SEA BREAM**

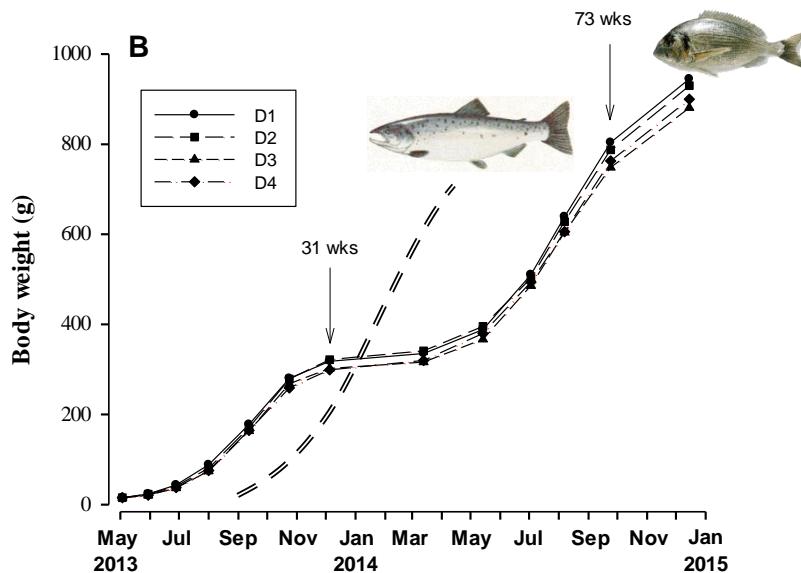
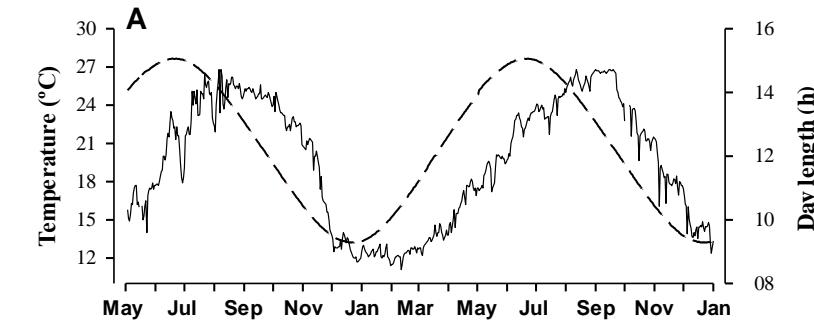


# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Alternative feed reared on plant sources

Ingredients (% of total)	Atlantic salmon	Sea bream
<b>Vegetable meal mixture</b>	65	73.84
<i>Wheat</i>	6	6.64
<i>Wheat gluten</i>	15	7.3
<i>Corn gluten</i>	4	25
<b>Soybean protein</b>	<b>18</b>	<b>25</b>
<i>Pea protein</i>	13	
<i>Rapeseed meal</i>		9.9
<i>Field peas</i>	9	
<b>Vegetable oil mixture</b>	15.8	13.0
<i>Linseed oil</i>	2.2	
<i>Palm oil</i>	4.8	6.5
<i>Rapeseed oil</i>	8.8	6.5
<b>Marine sources</b>	14.4	7.5
<i>Fish meal</i>	8	3
<i>Fish oil</i>	4.4	2.5
<i>Krill meal</i>	2	
<i>Fish protein</i>		2
<b>Vitamin and mineral supplementation</b>	4.8	5.66

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY



# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Undesirables overview throughout salmon and sea bream feeding

Sample \ Compound	PESTICIDES		PAHs	MYCOTOXINS	POPs	ETQ* / ETQ-D
	POLAR	NON-POLAR				
Plant meal	✓		✓	✓		✓
Plant oil	✓		✓	✓		✓
Marine ingredients	✓	✓	✓		✓	✓

✓ = identified

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Plant-based Feed	✓		✓	✓		✓

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Fish fillet			✓			✓

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# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

Undesirables overview throughout salmon and sea bream feeding

ARRAINA 2012-2016



## Pesticides

Concentrations  
(ng/g, ww)

Chlorpiriphos-methyl

Pirimiphos-methyl

Boscalid

Azoxystrobin

### Sea bream FEED

D1      D2      D3      D4

<5      5,1      7,0      <5

### Salmon FEED

A      B      D      G

8,8      49,0      5,8      6,6

8,6      <5      <5      <5

9,0      <5      <5      <5

800 pesticides were studied by means of GC & LC coupled to HRMS

No feed-to-fillet transfer of pesticides

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Undesirables overview throughout salmon and sea bream feeding

**PAHs**

AQUAMAX 2006-2010



ARRAINA 2012-2016



Average  
Concentrations  
(ng/g, ww)

$\Sigma$ PAH

	FEED		FILLET		FEED			FILLET		
	Sea bream	Salmon	Sea bream	Salmon	Sea bream -1	Sea bream -2	Salmon	Sea bream (300g)	Sea bream (700g)	Salmon (800g)
$\Sigma$ PAH	234	59	6,3	6,5	156	163	198	164	161	168

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Undesirables overview throughout salmon and sea bream feeding

**PAHs**

AQUAMAX 2006-2010



ARRAINA 2012-2016



Average Concentrations (ng/g, ww)

$\Sigma$ PAH

BaPEs

	FEED		FILLET		FEED			FILLET		
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$\Sigma$ PAH	234	59	6,3	6,5	156	163	198	164	161	168
BaPEs	1,01	9,45	0,01	0,57	0,15	0,16	0,22	0,16	0,16	0,17

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Undesirables overview throughout salmon and sea bream feeding

### PAHs

AQUAMAX 2006-2010



ARRAINA 2012-2016



Average Concentrations (ng/g, ww)

$\Sigma$ PAH

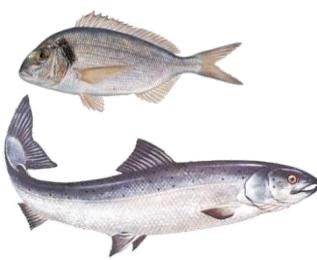
BaPEs

EU-BaPE limits

	FEED		FILLET		FEED			FILLET		
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$\Sigma$ PAH	234	59	6,3	6,5	156	163	198	164	161	168
BaPEs	1,01	9,45	0,01	0,57	0,15	0,16	0,22	0,16	0,16	0,17
EU-BaPE limits			2,0					2,0		

PAHs transfer has increased when using plant-based diets at higher %

Despite of these conditions, fillet concentrations remain below EU limits



# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

## Undesirables overview throughout salmon and sea bream feeding

Mycotoxins (ng/g, ww)	Sunflower meal	Rapeseed meal	wheat	wheat gluten	corn gluten	Pea protein	SOY PROTEIN CONCENTRATE
	(n=1)	(n=1)	(n=3)	(n=4)	(n=3)	(n=3)	(n=4)
AFG2	-	-	-	-	-	-	-
AFG1	-	-	-	-	-	-	-
AFB2	-	-	-	-	-	-	-
<b>AFB1</b>	-	-	-	-	-	-	-
NIV	-	-	-	-	-	-	-
Fus X	-	-	-	-	-	-	-
<b>DON</b>	-	-	53-371 (3)	17-504 (4)	139-814 (3)	-	-
3-AcDON	-	-	-	-	-	-	-
15-AcDON	-	-	-	-	53-452	-	-
NEO	<b>No feed-to-fillet transfer of mycotoxins</b>						
DIA	-	-	-	-	-	-	-
<b>HT-2</b>		-	4-8.1 (2)	4 (2)	67 (1)	-	-
T-2	-	-	4 (1)	4 (2)	2.8 (1)	-	-
ZEN	-	-	-	14-17 (2)	8-13 (3)	-	-
<b>OTA</b>	0.4	0.4	0.4 (1)	2.0-5.2 (4)	0.4 (3)	1.8 (1)	-
<b>FB1</b>	-	-	-	0.4-8.2 (2)	0.4-2319 (3)	-	0.4 (2)
<b>FB2</b>	-	-	-	2.9 (1)	2.9-1943 (3)	-	0.5 (1)
FB3	-	-	-	2.1 (1)	7.8-638 (3)	-	-
<b>Sum FB1+FB2+FB3</b>	-	-	-	13.2	11.1-4901	-	-

# IMPACT OF NEW DIET FORMULATIONS ON FOOD SAFETY

Undesirables overview throughout salmon and sea bream feeding

ARRAINA 2012-2016

## Ethoxyquin and its dimer



Average  
Concentrations  
(ng/g, ww)

ETQ

ETQ dimer

### FEED

	Sea bream -1	Sea bream -2	Salmon	Sea bream (300g)	Sea bream (700g)	Salmon (800g)
ETQ	7934	7334	5801	440	418	312
ETQ dimer	10228	13718	1391	383	703	94

All ingredients, feeds and fish were positive for ETQ and ETQ dimer

ETQ/ETQ dimer are transferred from ingredients to edible fillets

TF between 0.02-0.11



## Undesirables OVERVIEW throughout salmon and sea bream feeding

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Fish fillet			✓			✓
Transfer factor = [fish] / [feed]						
Sea bream	-	-	0.5-2.0	-	-	0.05-0.07/0.03-0.07
Atlantic salmon	-	-	0.4-1.3	-	-	0.02-0.07/0.02-0.11

\*preservative

 No feed-to-fillet transfer of pesticides and mycotoxins (after plant feeding)

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Ingredient-to-feed dilution factor contributes to the absence of POPs and non-polar pesticides in fillet

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ETQ and its dimer were found at concentrations around mg/kg in all samples

# Teşekkür ederim!



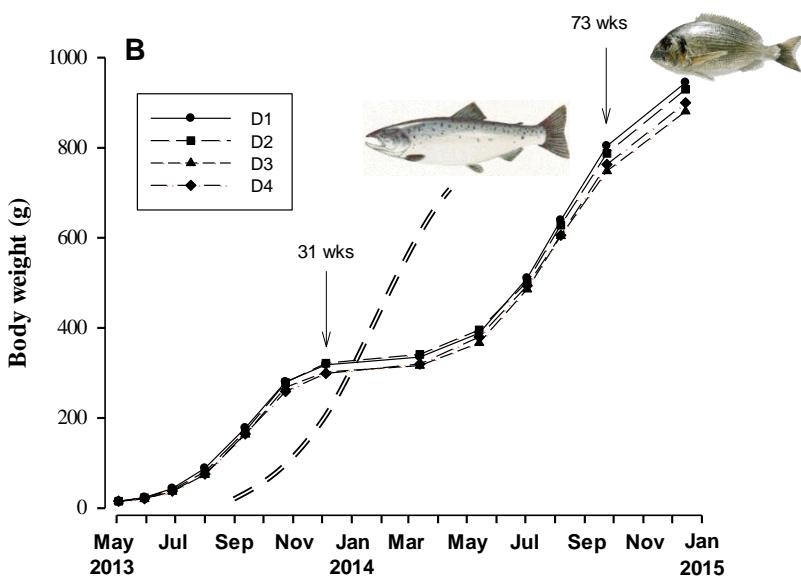
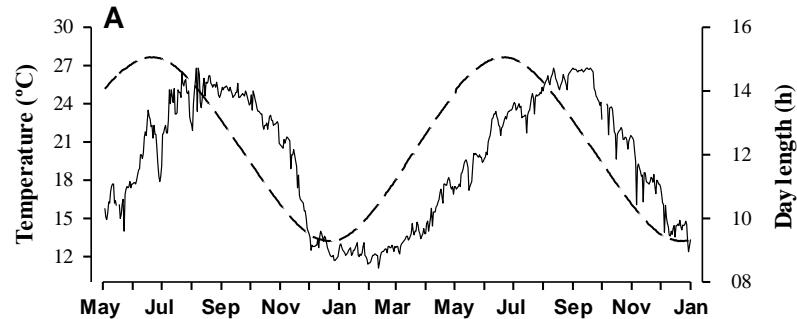
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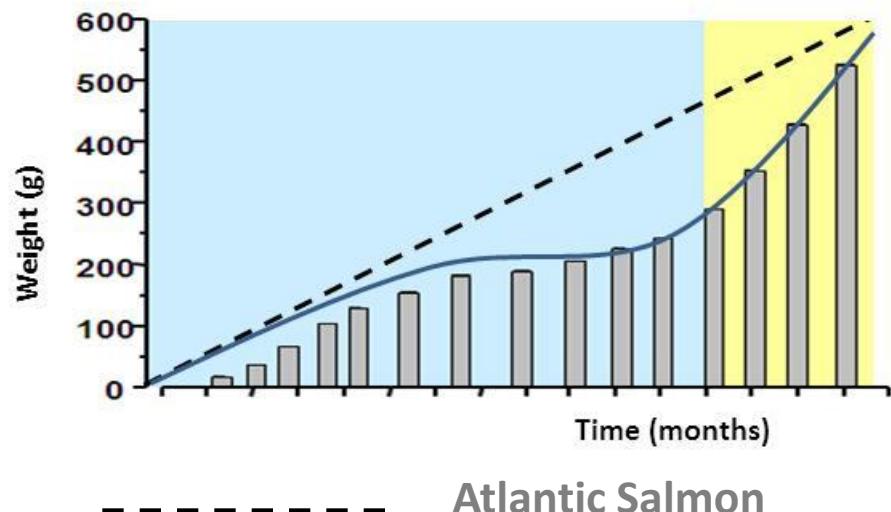
# GROWTH IMPLICATIONS ON FISH SAFETY



Gilthead sea bream growth \_Nutrigroup IATS

**Stationary growing**  
 vs  
**non-stationary growing stages**

Risk dependent on fish species



Atlantic Salmon

Gilthead sea bream

Recibidos (1) - nacher@u... X EU Pesticides database - X Jaime

ec.europa.eu/food/plant/pesticides/eu-pesticides-database/public/?event=pesticide.residue.selection&language=EN

Aplicaciones Google Universitat Jaume I JAIME WordReference.com Traductor de Google Alfresco Canciones infantiles Estación Meteorológica Otros marcadores

European Commission > Food Safety > Plants > Pesticides > Pesticides Database

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## PESTICIDES

### EU Pesticides database

Search active substances  
Search products  
**Search pesticide residues**  
Download MRLs data

Sustainable use of pesticides  
Approval of active substances  
Authorisation of Plant Protection Products  
Maximum Residue levels

« ALL TOPICS

## Search pesticide residues

**1 Select pesticide residues (5 max)**

Search: gly

Pesticide residues
F
<input type="checkbox"/> Fluoroglycofene
G
<input type="checkbox"/> Glyphosate
T
<input type="checkbox"/> Trimethyl-sulfonium cation, resulting from the use of glyphosate (F)

**2 Select products**

Search: soya

Code	Groups and examples of individual products to which the MRLs apply (a)
0251040	Cresses and other sprouts and shoots
0260010	Beans (with pods)
0260020	Beans (without pods)
0300010	Beans
0401070	Soyabeans

**3 Select**

Current MRLs  
 MRLs evolution (max 1 pesticide)

**4 Display +**

Pesticides selected

Products selected

All X

M Recibidos (1) - nacher@... X EU Pesticides database - X Jaime

ec.europa.eu/food/plant/pesticides/eu-pesticides-database/public/?event=pesticide.residue.displayMRL&language=EN

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## EU Pesticides database

European Commission > Food Safety > Plants > Pesticides > Pesticides Database

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### Glyphosate

Newer Historic of publications Older

Search:

Export to Excel

Showing 1 to 1 of 1 entries 50 records per page

Pesticide residues and maximum residue levels (mg/kg)

Code number	Groups and examples of individual products to which the MRLs apply (a)	Reg. (EU) No 293/2013	Reg. (EU) No 441/2012	Reg. (EC) No 839/2008	Reg. (EC) No 149/2008
0401070	Soyabean	20.0	20.0	20.0	20.0

Showing 1 to 1 of 1 entries

Table legend: Category Group Subgroup Main product Others Clickable Footnotes N/A Not published yet \* Indicates lower limit of analytical determination

« ALL TOPICS

## Current MRL values

to Excel

5 of 5 entries

50 records per page

Search:

« < 1 > »

Pesticide residues and maximum residue levels (mg/kg)

Products to which MRLs apply (Part A of Annex I to Reg. 396/2005)	Azoxystrobin i	Chlorpyrifos (F) i	Chlorpyrifos-methyl (F) i	Pirimiphos-methyl (F) i	Boscalid (F) (R) (A) i
<input type="checkbox"/> Sunflower seeds	0.5	0.05*	0.05*	0.5	1.0 
<input type="checkbox"/> Rapeseeds/canola seeds	0.5	0.05*	0.05*	0.5	1.0 
<input type="checkbox"/> Soyabbeans	0.5	0.05*	0.05*	0.5	3.0 
<input type="checkbox"/> Maize/corn	0.02	0.05	3.0	0.5	0.15 
<input type="checkbox"/> Wheat	0.5	0.05*	3.0	5.0 	0.8 