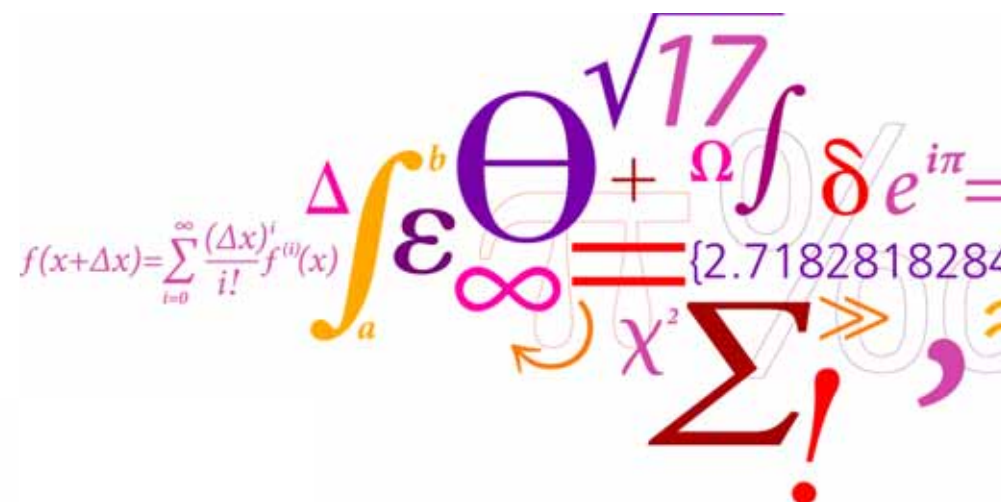


# Novel developments and future trends in methods of analysis for trace element species in food and feed control

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*Jens J. Sloth*

National Food Institute (DTU Food)  
 Technical University of Denmark

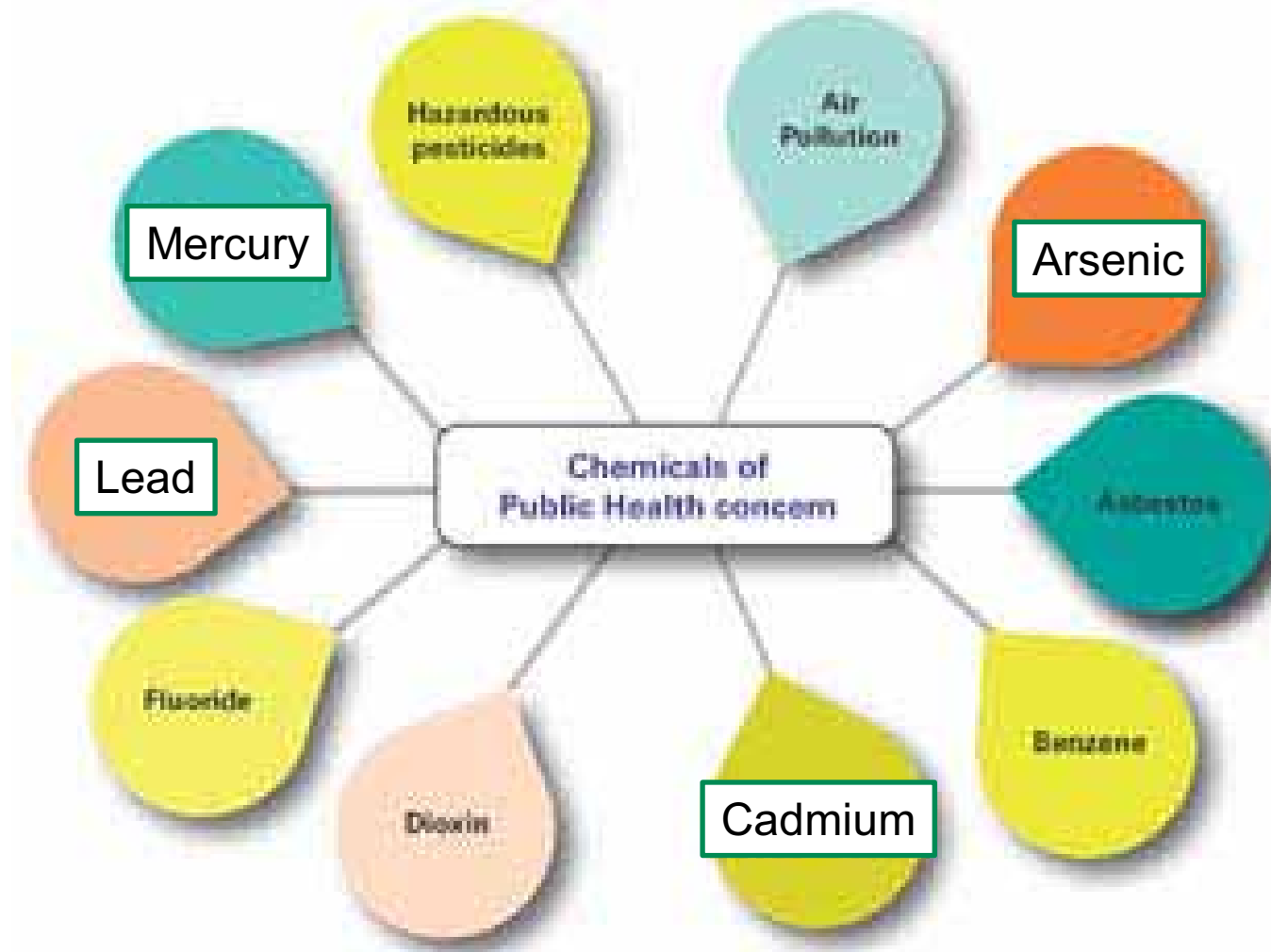


# Agenda

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- Speciation in relation to food and feed safety – where are we now??
- Current legislation and standardisation issues
- Selected examples
  - Arsenic speciation analysis (importance of inorganic arsenic)
  - Organotin speciation analysis (food contact materials)
  - Selenium speciation analysis (food fraud!)
- Future developments and needs



## Current situation in EU legislation:

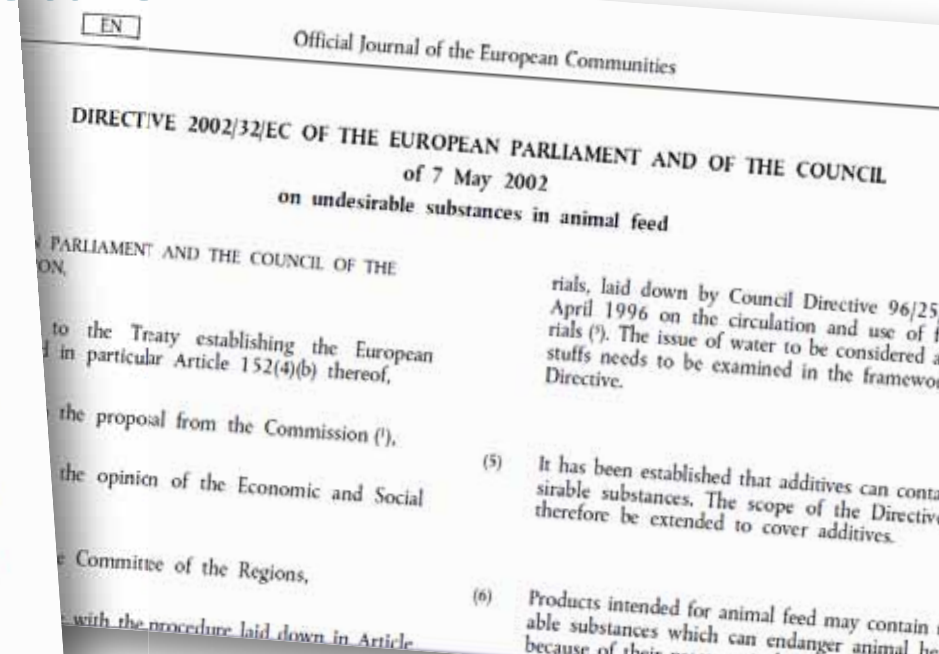
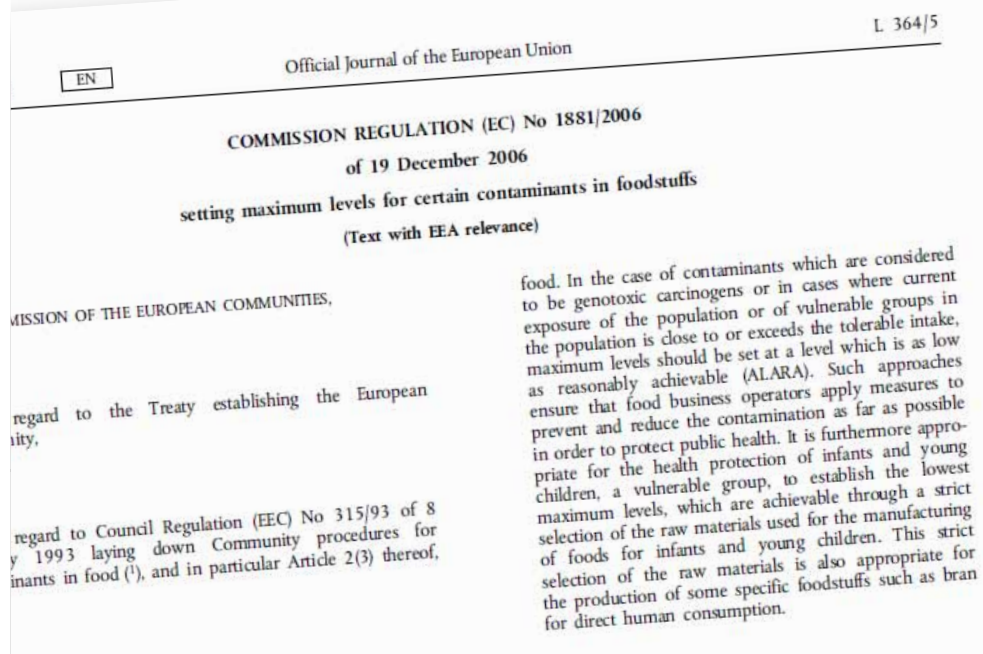
### Foodstuffs

**MLs for Pb, Cd, Hg and Sn**  
**EU directive 2006/1881/EC**

### Animal feedingstuffs


**MLs for As, Pb, Cd and Hg**  
**EU directive 2002/32/EC**

**Only maximum levels for  
total concentration of the  
metals**



# Speciation and regulation - some historical viewpoints

**1998**



ELSEVIER

Spectrochimica Acta Part B 53 (1998) 169–175

SPECTROCHIMICA  
ACTA  
PART B

Speciation analysis: where is it going? An attempt at a forecast<sup>1</sup>

Bernhard Welz

Department of Applied Research, Bodenseewerk Perkin-Elmer GmbH, D-87564

Fresenius J Anal Chem (1999) 363:431–434

© Springer-Verlag 1999

CONFERENCE CONTRIBUTION

**1999**

Torsten Berg · Erik H. Larsen

**Speciation and legislation –  
Where are we today and what do we need for tomorrow?**

**Handbook of  
Elemental  
Speciation II**

Species in the Environment,  
Food, Medicine and  
Occupational Health

**Speciation and the Emerging Legislation**

Nicole Proust  
Editor-in-Chief *THALES Research and Technology France, Orsay, France*

Rita Cornelis  
Associate Editors *University of Münster, Münster, Germany*

Wolfgang Buscher  
Associate Editors *University of Münster, Münster, Germany*

Joe Caruso  
Associate Editors *University of Münster, Münster, Germany*

Helen Crews  
Associate Editors *University of Münster, Münster, Germany*

Michael Sperling  
Associate Editors *University of Münster, Münster, Germany*

Klaus Heumann  
Associate Editors *University of Münster, Münster, Germany*

**Wiley 2005**

SECTION: FORUM

[www.rsc.org/analyst](http://www.rsc.org/analyst) | The Analyst

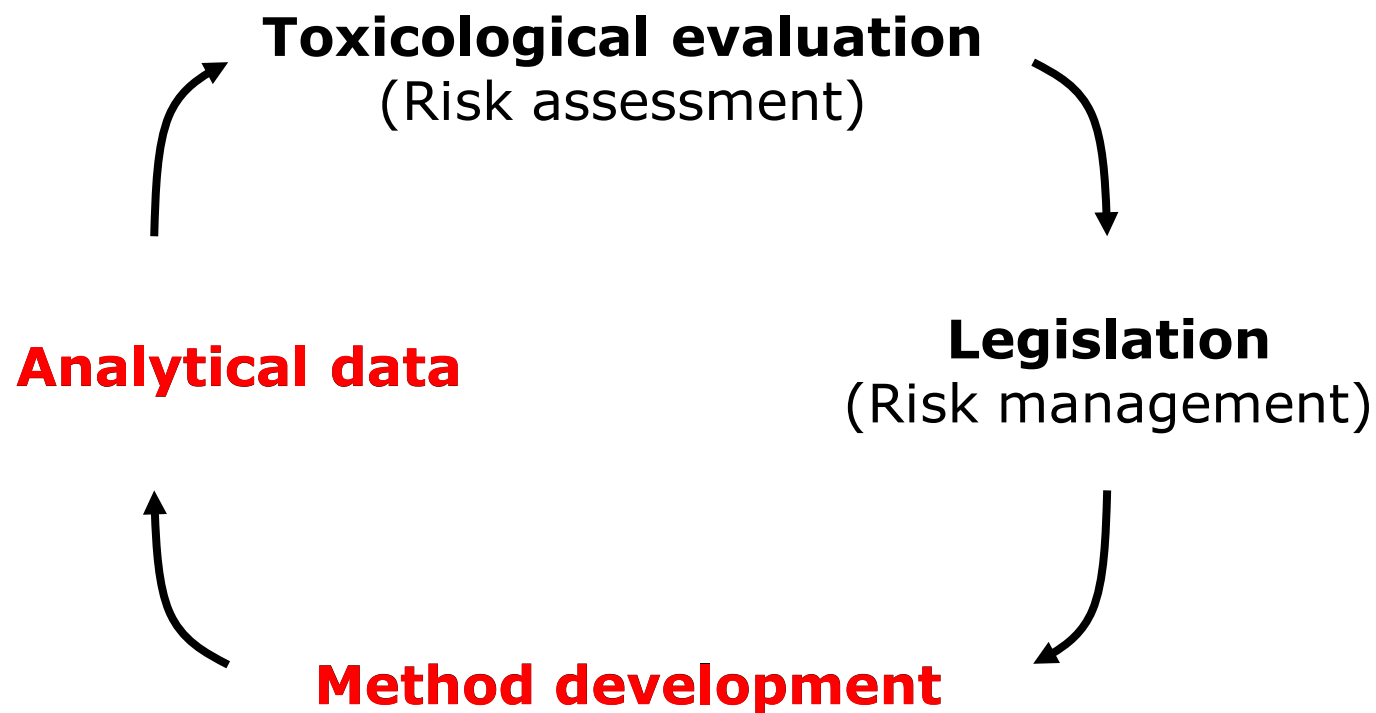
Toxic metal species and food regulations—  
making a healthy choice

**2007**

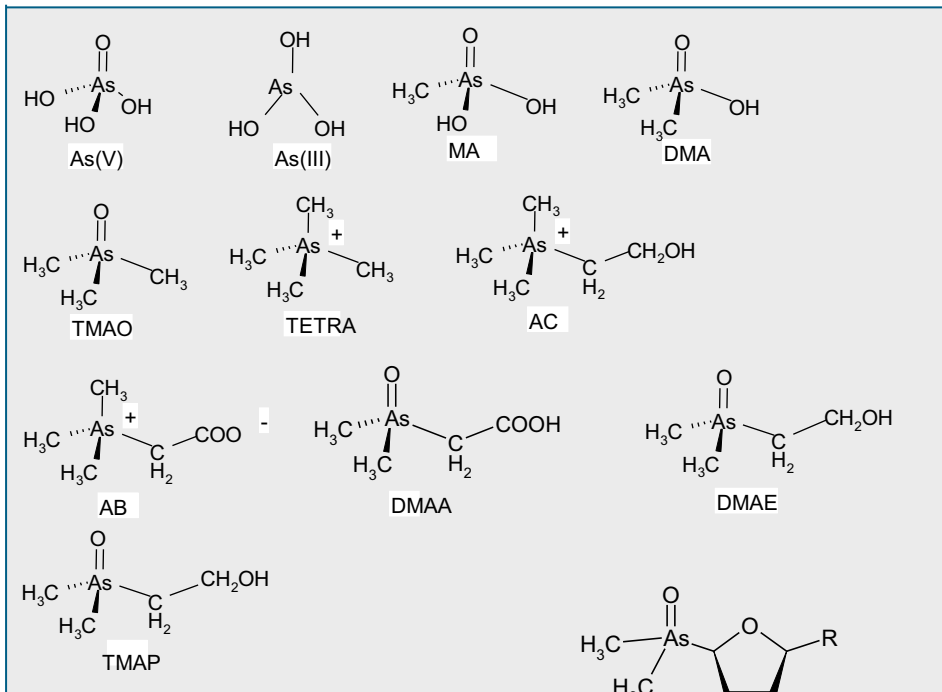
Kevin A. Francesconi

## Vicious circle of progress

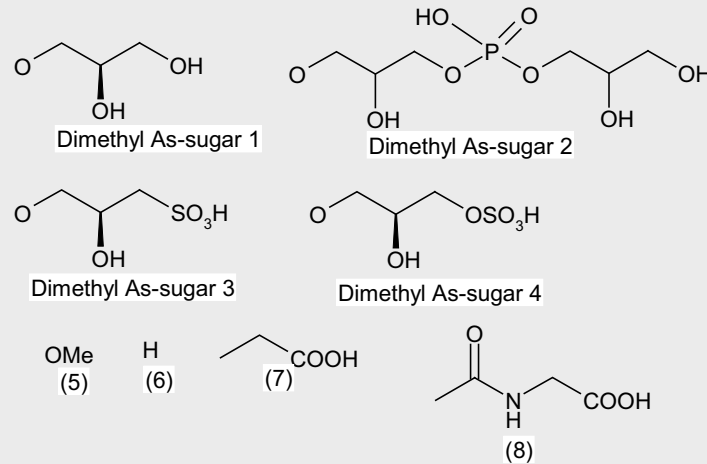
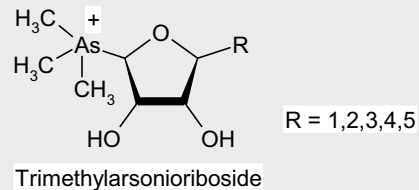
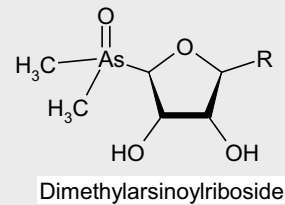
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# Arsenic compounds in the marine environment



More than **50** different arsenic species have been found in the marine environment – incl. lipid-soluble As compounds (*arsenolipids*).



## Example – arsenic speciation Important for correct risk assessment



**1 kg rice => 50-300 µg As**



**1 kg fish => 3000-10000 µg As**

**There is most focus on rice from a food safety point of view – why???**



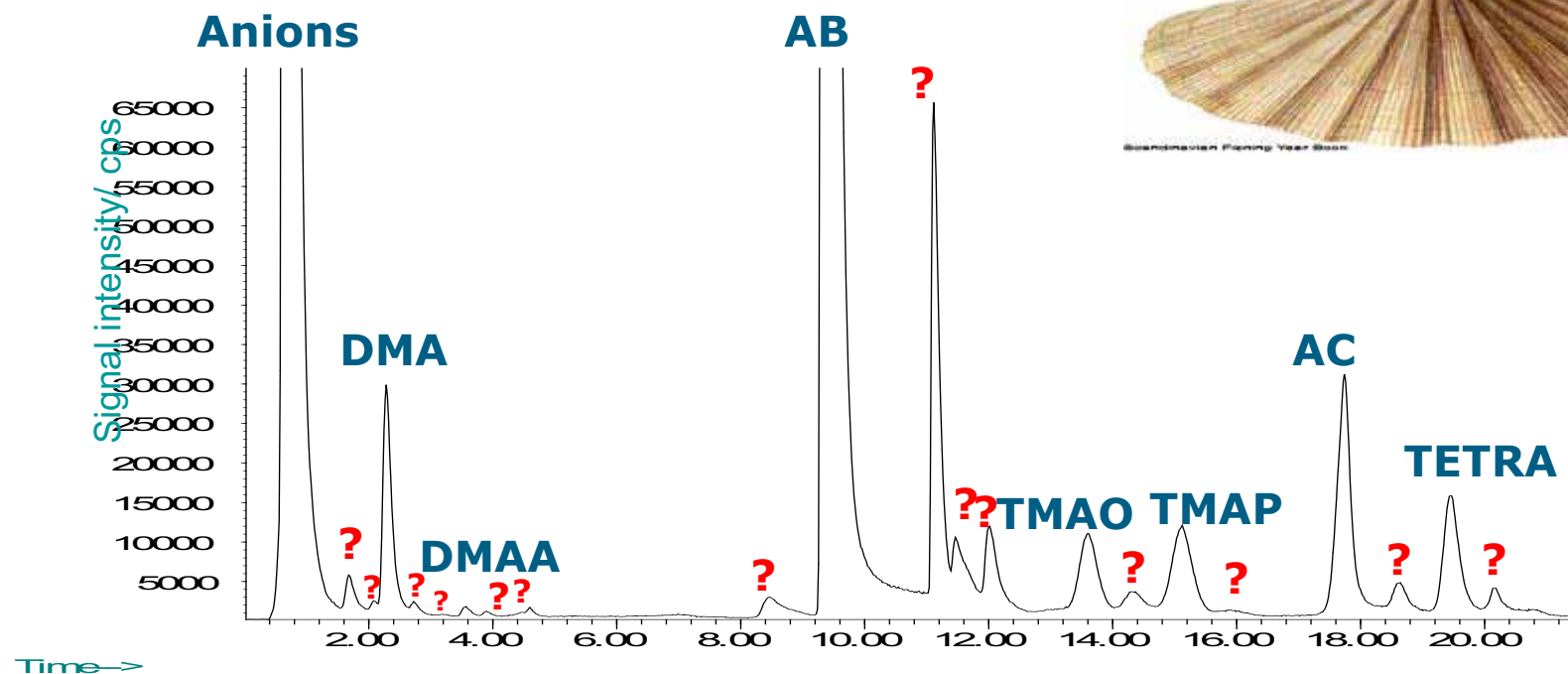
**The chemical form of arsenic is important  
=> Arsenic speciation**

# Speciation analysis of arsenic of scallop kidney

Cation-exchange with gradient elution – extraction with aqueous methanol

Column: Chrompack Ionospher 5C; Mobile phase: Pyridine; pH = 2.7

- seven compounds identified by coelution with available standards
- **16** non-identified peaks

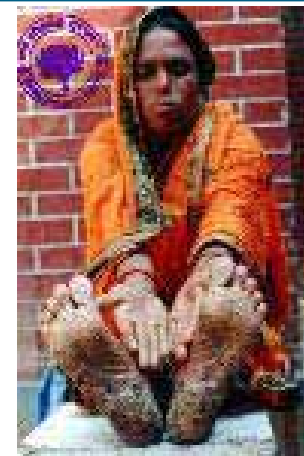


# Food safety and arsenic – toxicity

**Focus on inorganic arsenic; As(III) and As(V)**

**Long term exposure => skin diseases**

- Keratosis, gangrene, melatosis
- Skin cancer
- ... and also
- lung, kidney, liver, bladder cancers



**Cancer slope factor:  $1.5 \text{ (mg kg}^{-1} \text{ day}^{-1})^{-1}$  (for inorganic As)** (US EPA 2005)

~~WHO (1988) PTWI for inorganic arsenic:  $15 \text{ } \mu\text{g/kg bw/week}$   
(Provisional Tolerable Weekly Intake)~~

~~For a 70 kg person =>  $150 \text{ } \mu\text{g / day}$~~

**No longer appropriate**

## EFSA (2009) and JECFA (2010) opinions on arsenic in food

- Old PTWI value (WHO, 1988) was withdrawn
- **NEW!**  $BMDL_{1.0} = 0.3 - 8 \mu\text{g}/\text{kg bw per day}$  for inorganic arsenic
- => EU dietary exposures within this range
- => Risk to some consumers cannot be excluded



- **NEW!**  $BMDL_{0.5} = \underline{3 \mu\text{g}/\text{kg bw per day}}$  for inorganic arsenic
- => *0.5% increased incidence of lung cancer for 12 y exposure*



- “...there is a need to produce speciation data for different food commodities to support dietary exposure assessment...”
- “...more accurate information on the inorganic arsenic content of foods is needed to improve assessments of dietary exposures to inorganic arsenic”
- “...need for validated methods for selective determination of inorganic arsenic in food matrices”



Sample identification	Inorganic arsenic	Total arsenic
Salmon ( <i>Salmo salar</i> )	< 0.0006	1.9 0.2
Cod ( <i>Gadus morhua</i> )	< 0.0006	17 2
Cod ( <i>Gadus morhua</i> )	< 0.0006	15 2
Wolffish ( <i>Anarhichas lupus</i> )	< 0.0006	4.1 0.5
Wolffish ( <i>Anarhichas lupus</i> )	< 0.0006	31 4
Anglerfish ( <i>Lophius piscatorius</i> )	< 0.0006	15 2
Anglerfish ( <i>Lophius piscatorius</i> )	< 0.0006	44 6
Atlantic halibut ( <i>Hippoglossus hippoglossus</i> )	< 0.0006	12 1
Mackerel ( <i>Scomber scombrus</i> )	< 0.0006	1.7 0.2
Mackerel ( <i>Scomber scombrus</i> )	< 0.0006	2.8 0.4
Herring ( <i>Clupea harengus</i> )	< 0.0006	1.5 0.2
Herring ( <i>Clupea harengus</i> )	< 0.0006	1.7 0.2
Herring ( <i>Clupea harengus</i> )	< 0.0006	1.7 0.2
Tuna fish ( <i>Thunnus alalunga</i> )	0.008 0.001	0.9 0.1
Lobster tail meat ( <i>Homarus gammarus</i> )	< 0.0006	14 2
Loose, head and thorax meat ( <i>Homarus gammarus</i> )	0.017 0.005	22 3
Crab, white meat ( <i>Cancer pagurus</i> )	0.016 0.002	12 2
Crab, head and thorax meat ( <i>Cancer pagurus</i> )	0.010 0.009	26 3
King crab, white meat ( <i>Paralithodes camschaticus</i> )	0.005 0.001	26 3
Norway lobster ( <i>Nephrops norvegicus</i> )	0.020 0.003	21 3
Shrimp ( <i>Pandalus borealis</i> )	< 0.0006	3.8 0.5
Shrimp ( <i>Pandalus borealis</i> )	< 0.0006	60 8
Shrimp ( <i>Pandalus borealis</i> )	< 0.0006	67 8
Horse mussel ( <i>Modiolus modiolus</i> )	0.0012 0.002	3.4 0.4
Scallop muscle ( <i>Pecten maximus</i> )	0.008 0.001	3.1 0.3
Oyster ( <i>Ostrea edulis</i> )	0.014 0.002	1.8 0.2
Mink whale ( <i>Balaenoptera Acutorostrata</i> )	< 0.0006	0.61 0.08
Harp seal ( <i>Pagophilus groenlandicus</i> )		0.9 0.1
Hooded seal ( <i>Cystophora cristata</i> )		0.22 0.03

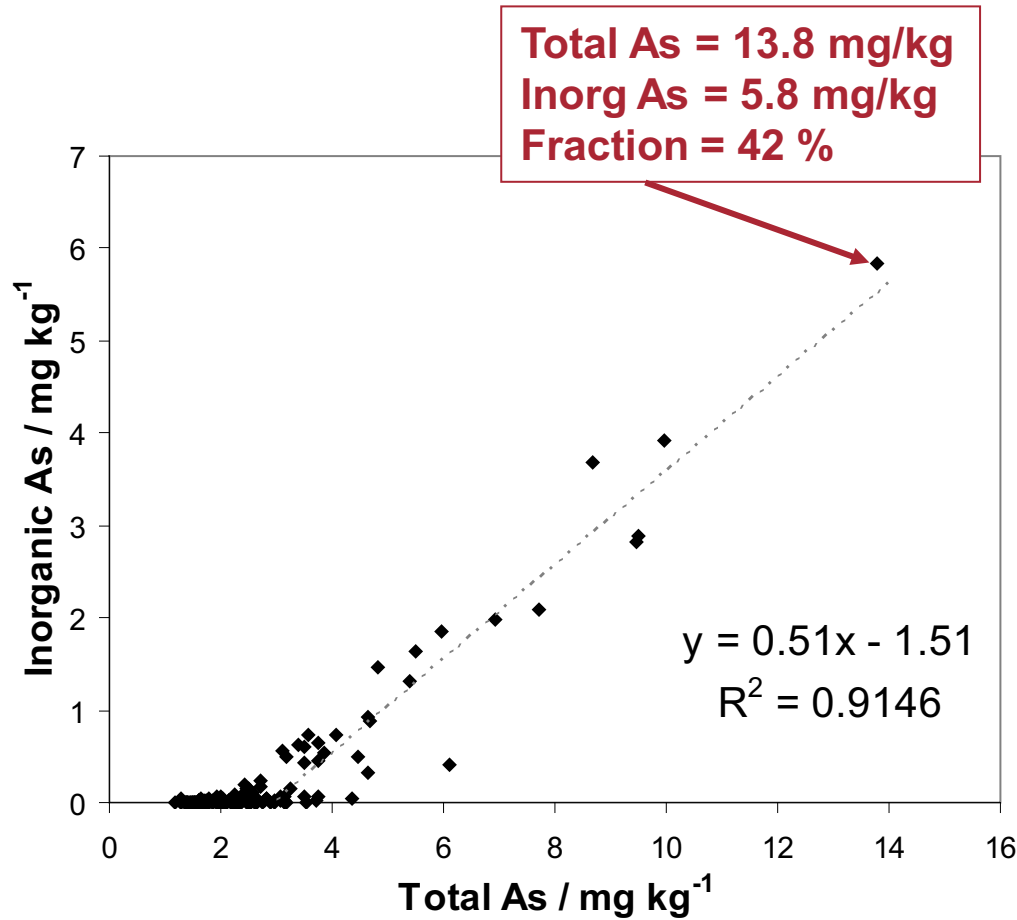
**Fish muscle**

**Crustaceans & bivalves**

**Marine mammals**

For all samples inorganic arsenic constitutes less than 1% of total arsenic

# ...but unusual high contents in some samples...



Data from 175 blue mussel (*Mytilus edulis*) samples collected along the Norwegian Coastline.



# Arsenic in rice – an emerging health issue?

Environmental Pollution 152 (2008) 746–749

Rapid communication

## Inorganic arsenic levels in baby rice are of concern

Andrew A. Meharg<sup>a,\*</sup>, Guoxin Sun<sup>b</sup>, Paul N. Williams<sup>a,b</sup>, Eureka Adomako<sup>a</sup>,  
Claire Deacon<sup>a</sup>, Yong-Guan Zhu<sup>b</sup>, Joerg Feldmann<sup>c</sup>, Andrea Raab<sup>c</sup>

<sup>a</sup> School of Biological Sciences, University of Aberdeen, Cruickshank Building, St. Machar Drive, Aberdeen AB24 3UU, UK

<sup>b</sup> Research Centre for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, China

*Median consumption of organic arsenic levels for UK babies from baby rice is above threshold considered safe.*

- **17 samples from supermarkets in Aberdeen**
- **Total arsenic levels: 0.12 – 0.47 mg/kg**
- **Inorganic arsenic: 0.06 – 0.16 mg/kg (33 – 69 % of tAs)**
- **35% above Chinese max level of 0.15 mg/kg iAs**
- **No regulation on As in food in EU (yet!)**

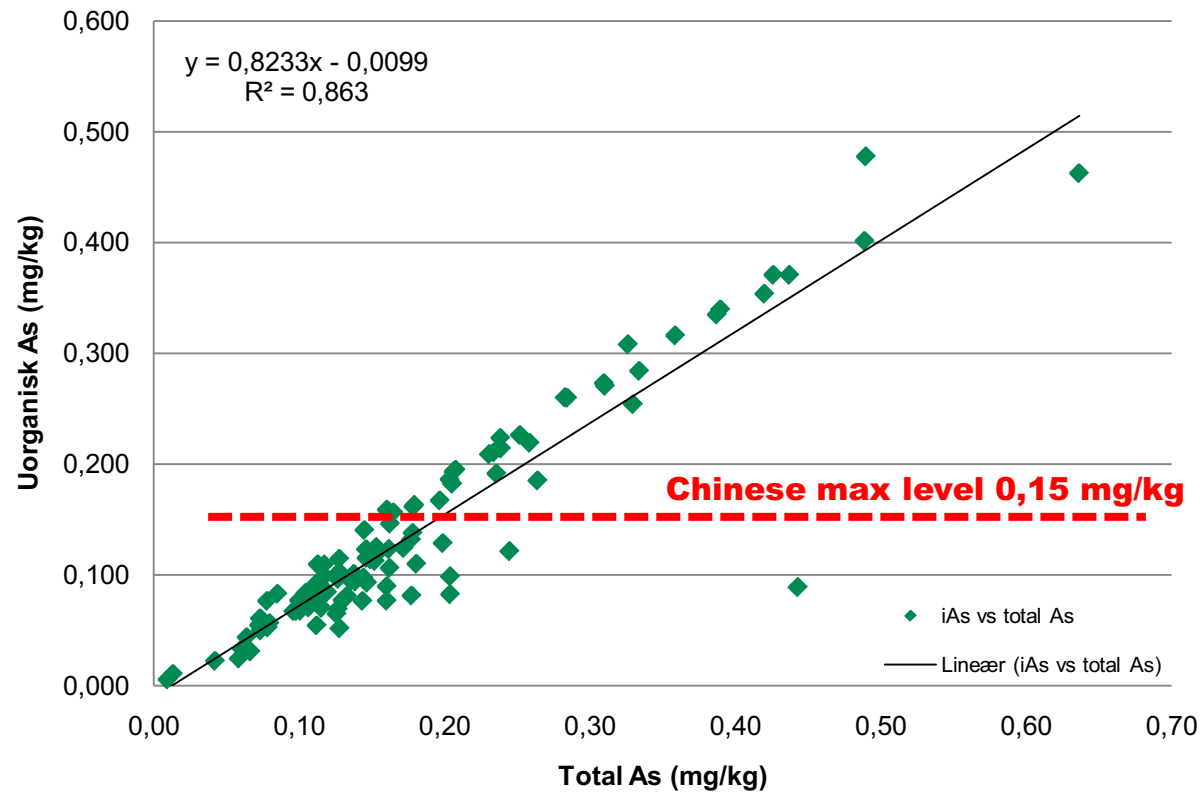
**Comment on cereals Cubadda**





# Arsenic in rice products

iAs vs total As



## 105 prøver i alt

- Hvide ris (white rice)
- Brune ris (brown rice)
- Røde ris (red rice)
- Sorte ris (black rice)
- Ris kiks (rice crackers)

## 33 prøver > 0,15 mg/kg

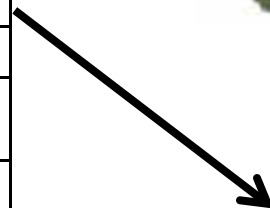
- 2 parboiled (20%)
- 4 brune (50%)
- 4 røde (50%)
- 5 sorte (71%)
- 1 Basmati (10%)
- 1 Grød (9%)
- 1 vilde (20%)
- 15 ris kiks (100%)

**Rice cracker mean: 0,31 mg/kg – intake 50 g/dag => 15 µg iAs (~1 µg/kg)  
> EFSA BMDL<sub>01</sub> på 0,3-8 µg/kg bw/dag**

# Inorganic arsenic in chinese food supplements



Name of Food supplement	Total Arsenic ( $\mu\text{g/g}$ )	Inorganic arsenic ( $\mu\text{g/g}$ )
Xiao Yao Wan	0.82	0.85
Bu Zhong Yi Qi Wan	0.62	0.50
Da Bu Yin Wan	0.59	0.55
Six Flavor teapills	0.72	N.D.
Golden Book Teapills	0.58	0.57
Xiang Sha Liu Jun Zi Wan	0.94	0.80
Gan Mao Ling	1.24	1.01
Chuan Xin Lian	5.00	3.17
Bi Yan Pian	0.70	0.58
Arouse power	1.12	1.02
Bio Chlorella	0.62	0.21
Unik Spirulina Kapsler	2.59	0.13
Chlorella	0.58	0.03
Ez-Biloba	0.63	0.67
Qvinde Dong Quai	0.68	0.48



**Gan Mao Ling**  
**Rec dose: 18 pills per day**  
 $\Rightarrow$  **iAs ~ 13  $\mu\text{g/day}$**   
 $\Rightarrow$  **0.22  $\mu\text{g/kg bw/day}$  (60 kg)**  
**Close to EFSA BMDL !!**



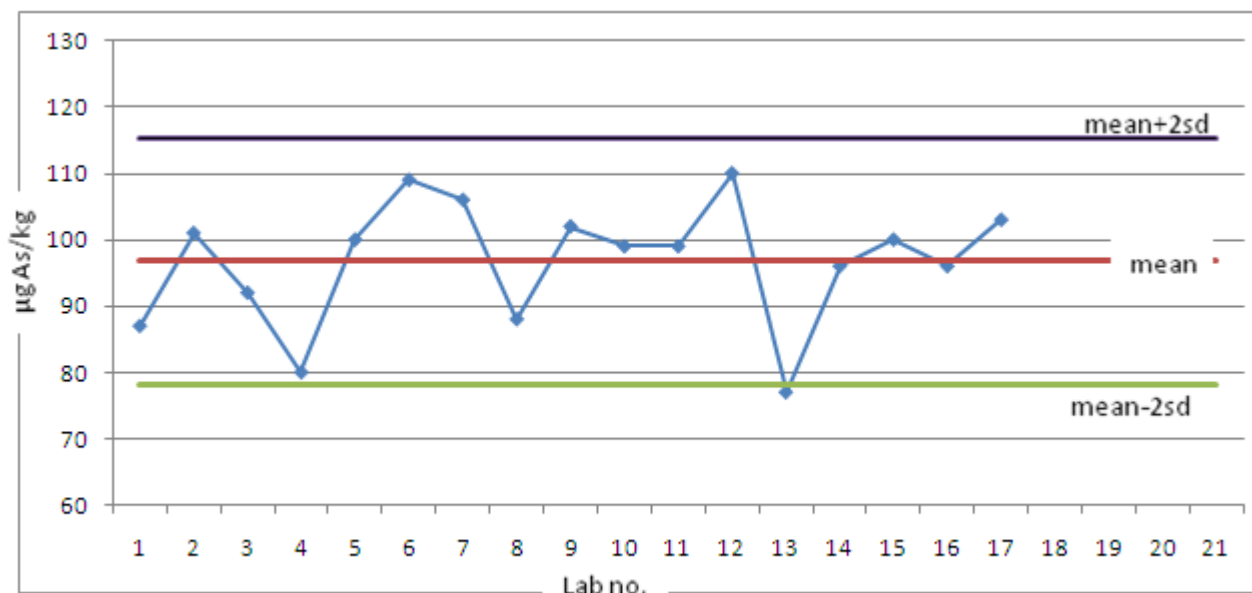
## Recent meeting: the Expert Committee “Industrial and Environmental Contaminants”

**“Based on the data processed so far, a maximum level of 0.2 mg/kg inorganic arsenic for rice as a category seems achievable”**

**“MSs are asked to reflect on the need for a maximum level for cereals other than rice”**

**“MSs are encouraged to concentrate on levels for inorganic arsenic in rice (collecting information on country of origin and rice variety), food supplements (algae) and infant food (rice based)”**

# Inorganic As in SRM NIST1586a

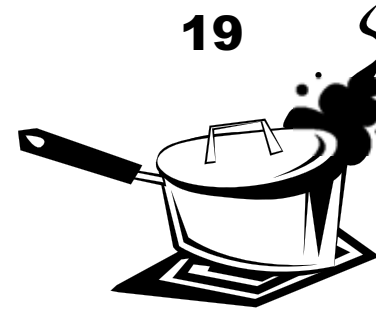


**0.097 mg/kg**

Extraction solution	Detection	iAs (µg/kg)	Reference
1 2M TFA	LC-ICPMS	87 +/- 9	Ackermann (2005)
2 Enzymatic, pepsin and pancreatin	LC-ICPMS	101 +/- 7	Ackermann (2005)
3 2M TFA	LC-ICPMS	92 +/- 2	Heitkemper (2001)
4 2M TFA	LC-ICPMS	80 +/- 16	Williams (2005)
5 2M TFA	LC-ICPMS	100 +/- 10	Williams (2006)
6 MeOH:H2O	LC-ICPMS	109 +/- 3	D'Amato (2004)
7 Enzymatic, alfa-amylase	LC-ICPMS	106 +/- 7	Kohlmeyer (2003)
8 Enzymatic, protease and alfa-amylase	LC-ICPMS	88 +/- 6	Sanz (2005)
9 1M H3PO4	HG-AFS	102 +/- 2	Matosreyes (2007)
10 1% HNO3	LC-ICPMS	99 +/- 4	Raab (2009)
11 1% HNO3	LC-ICPMS	99 +/- -	Sun (2008)
12 1% HNO3	LC-ICPMS	110 +/- 10	Sun (2009)
13 0,5 M TFA	LC-ICPMS	77 +/- -	Heitkemper (2009)
14 enzymatic, proteas and alfa-amylase	LC-ICPMS	96 +/- 9	Mar (2009)
15 2M TFA	LC-ICPMS	100 +/- 12	Meharg (2008)
16 water	LC-ICPMS	96 +/- 3	Narukawa (2008)
17 0,07M HCL and 10%H2O2	LC-ICPMS	103 +/- 15	DTU Food (2009)

Good agreement between labs and methods

+ lecture by M. de la Calle



## Processing

- Processing or storage may alter the arsenic species pattern
- No transformation of organoarsenic compounds to inorganic arsenic by normal cooking procedures (**IMPORTANT!**)
- Loss/uptake to/from boiling water possible
- Blue mussels => loss of AB after storage by freezing



# Commission directive 2009/114/EC (amendment)

Undesirable substances	Products intended for animal feed	Maximum content in mg/kg (ppm) relative to a feedingstuff with a moisture content of 12 %
(1)	(2)	(3)
1. Arsenic (*) (**)	Feed materials with the exception of:	2
	— meal made from grass, from dried lucerne and from dried clover, and dried sugar beet pulp and dried molasses sugar beet pulp,	4
	— palm kernel expeller,	4 (**)
	— phosphates and calcareous marine algae,	10
	— calcium carbonate,	15
	— magnesium oxide,	20
	— feedingstuffs obtained from the processing of fish or other marine animals, including fish,	25 (**)
	— seaweed meal and feed materials derived from seaweed,	40 (**)
	Iron particles used as tracer,	50
	Additives belonging to the functional group of compounds of trace elements except:	30
Complete feedingstuffs with the exception of:	Complete feedingstuffs with the exception of:	2
	— complete feedingstuffs for fish and complete feedingstuffs for fur animals,	10 (**)
	Complementary feedingstuffs with the exception of:	4
	— mineral feedingstuffs,	12



**Max levels for undesirable substances in animal feed**

**Only max levels for total arsenic**

## FOOTNOTE

(\*) Upon request of the competent authorities, the responsible operator must perform an analysis to demonstrate that the content of inorganic arsenic is lower than 2 ppm in products of particular interest, such as seaweed species *Hizikia fusiforme*.

**Inorganic arsenic lower than 2 ppm**

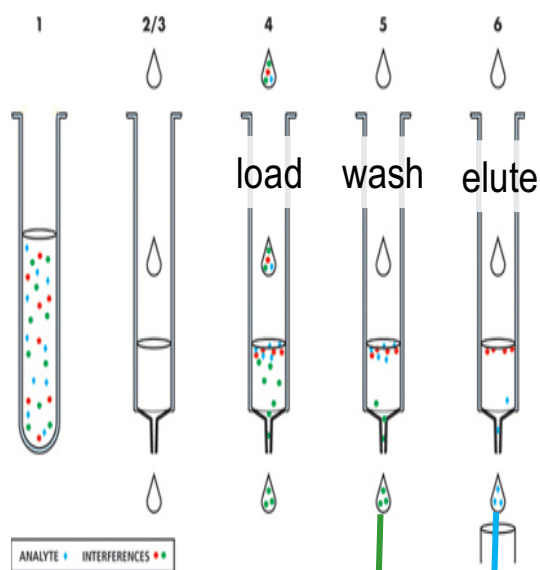
**Speciation analysis is required !!**

# SPE-HG-AAS – a speciation alternative...

μ-wave extraction

Separation by SPE

Detection by HG-AAS



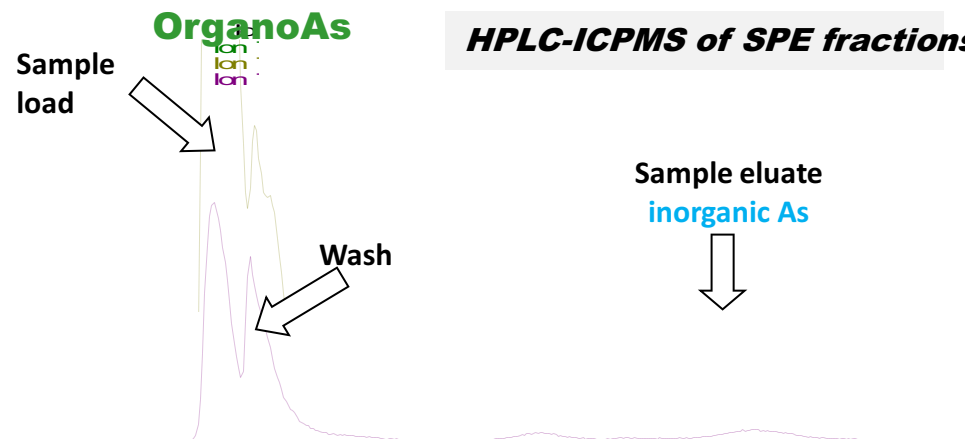
**Sequential elution for selective off-line separation of inorg As from organo As species by SPE**

**OrganoAs compounds**

**Inorganic As**

Abundance

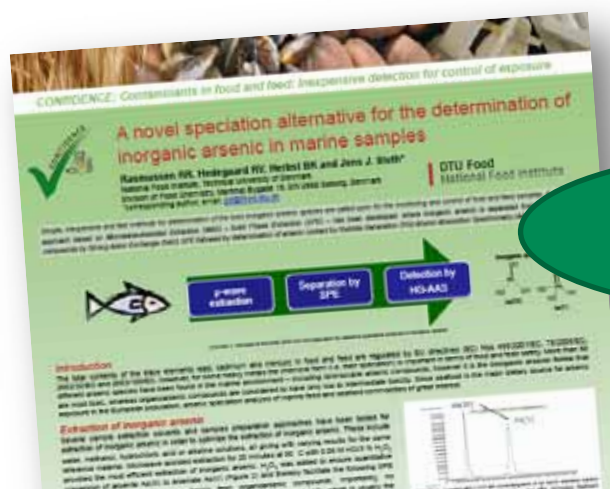
75000  
70000  
65000  
60000  
55000  
50000  
45000  
40000  
35000  
30000



# Performance characteristics from in-house validation



Parameter	Result
Analysis time	2 x 7 h for 24 samples
LoD (mg/kg)	0.08
LoQ (mg/kg)	0.16
Repeatability (%RSD)	3 - 7
Accuracy (%)	90 - 104



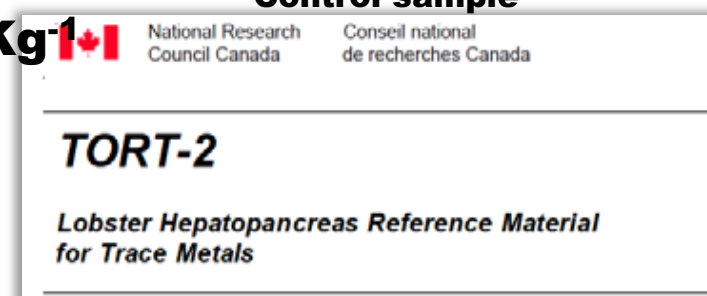
Further details on poster and handouts

# Samples for collaborative trial

Sample no	Sample type	Spiked / unspiked	Mean concentration (mg Kg <sup>-1</sup> )
IMEP32-1	Complete fish feedingstuff	Unspiked	0.07
IMEP32-2	Complete fish feedingstuff	Spiked	0.71
IMEP32-3	Fish meal	Unspiked	0.19
IMEP32-4	Fish meal	Spiked	1.06
IMEP32-5	Fish fillet	Spiked	2.64
IMEP32-7	Fish meal	Spiked	0.43
IMEP32 Control sample	CRM TORT-2 Lobster Hepatopancreas	Unspiked	0.54

**Concentration range: 0.07 – 2.64 mg Kg<sup>-1</sup>**

### Control sample



National Research Council Canada / Conseil national de recherches Canada

**TORT-2**

*Lobster Hepatopancreas Reference Material for Trace Metals*

Trace Metals (milligrams/kilogram)	
Arsenic (g,h,m)	21.6 ± 1.8

- **Total As**
- Not certified for iAs

# Results - overview

Matrix	Units	IMEP 32-1	IMEP 32-2	IMEP 32-3	IMEP 32-4	IMEP 32-5	IMEP 32-7	IMEP 32 Control Sample
N° of participating laboratories		10	10	10	10	10	10	10
Remaining data after outlier elimination		29	35	28	36	36	30	34
N° of remaining laboratories		9	10	9	10	10	9	10
Outliers	%	12.1	2.8	12.5	0.0	0.0	11.8	0.0
Overall mean $X_{obs} \pm u_{obs}$	mg Kg <sup>-1</sup>	0.071 ± 0.041	0.713 ± 0.117	0.189 ± 0.060	1.062 ± 0.140	2.643 ± 0.506	0.432 ± 0.066	0.544 ± 0.162
$S_r$	mg Kg <sup>-1</sup>	0.016	0.054	0.014	0.105	0.277	0.023	0.095
<b>RSD<sub>r</sub></b>	%	<b>22.8</b>	<b>7.6</b>	<b>7.5</b>	<b>9.9</b>	<b>10.8</b>	<b>5.4</b>	<b>17.5</b>
$r_L$	mg Kg <sup>-1</sup>	0.046	0.153	0.040	0.294	0.776	0.065	0.266
$S_R$	mg Kg <sup>-1</sup>	0.041	0.117	0.060	0.140	0.506	0.066	0.162
<b>RSD<sub>R</sub></b>	%	<b>57.6</b>	<b>16.4</b>	<b>31.9</b>	<b>13.2</b>	<b>19.1</b>	<b>15.3</b>	<b>29.7</b>
$R_L$	mg Kg <sup>-1</sup>	0.115	0.327	0.169	0.391	1.416	0.185	0.453
$\sigma_H$	mg Kg <sup>-1</sup>	0.017	0.120	0.039	0.168	0.365	0.078	0.095
<b>HorRat</b>		<b>2.4</b>	<b>1.0</b>	<b>1.6</b>	<b>0.8</b>	<b>1.4</b>	<b>0.8</b>	<b>1.7</b>



**HorRat >2**  
**Low concentration!!**

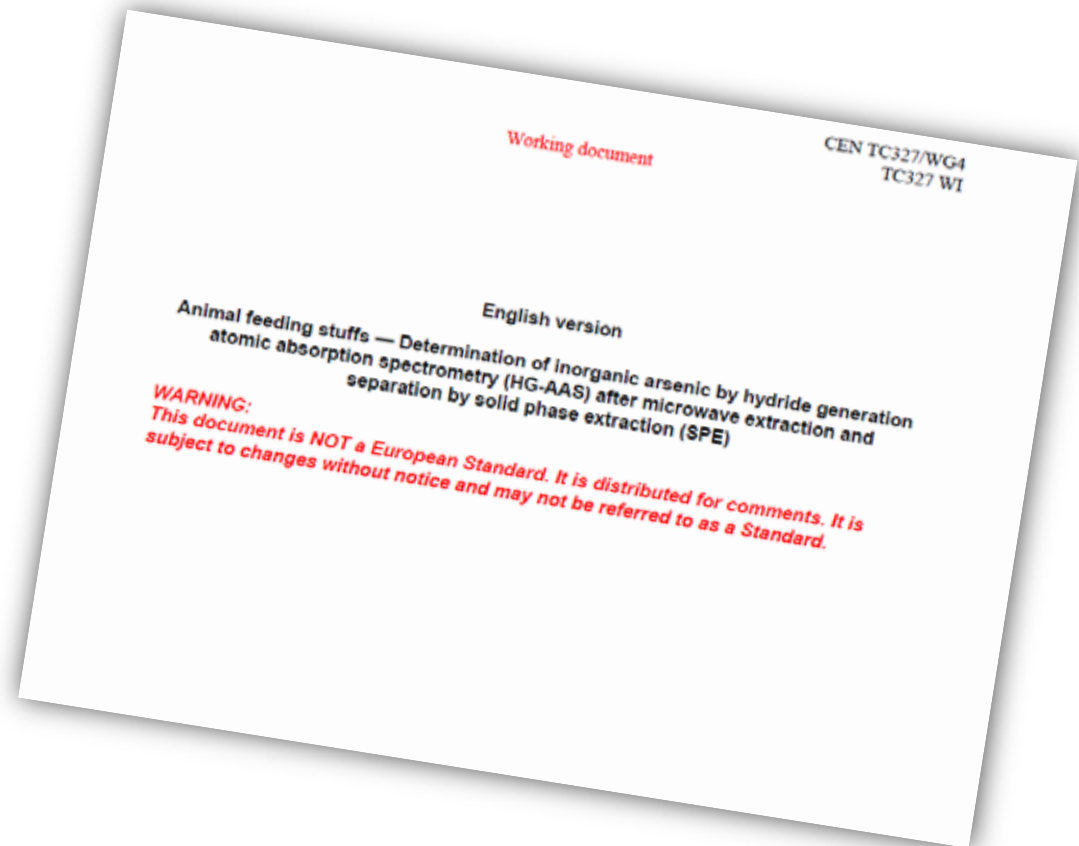
**All HorRat values < 2**

## Conclusions - I

- **A method for determination of iAs in feed of marine origin based on SPE-HG-AAS has been developed**
- **homogeneous and stable test samples were prepared**
- **a collaborative trial was conducted in Oct-Nov 2010**
- **Ten laboratories were evaluated as compliant**
- **Max 1 outlier lab was identified per sample**
- **HorRat values < 2 (0.8 – 1.7) for samples in the concentration range 0.19 – 2.6 mg Kg<sup>-1</sup>**
- **HorRat value > 2 (2.4) for S01 at concentration 0.07 mg Kg<sup>-1</sup>**
- **-Accuracy from control sample, mean = 0.544±0.162 mg Kg<sup>-1</sup>**  
**-Assigned value from HPLC-ICPMS determinations = 0.599±0.07 mg Kg<sup>-1</sup>**  
**-0.544/0.599 = 91 % (no significant difference)**

## Conclusions - II

- **EU directive "guideline" maximum level is 2 ppm (EU directive 2009/141/EC on animal feed)**
- **Method working range tested in ILC: 0.1 – 2.6 mg Kg<sup>-1</sup>**
- **HorRat values <2 in the working range tested**
- **The method is fit for purpose**



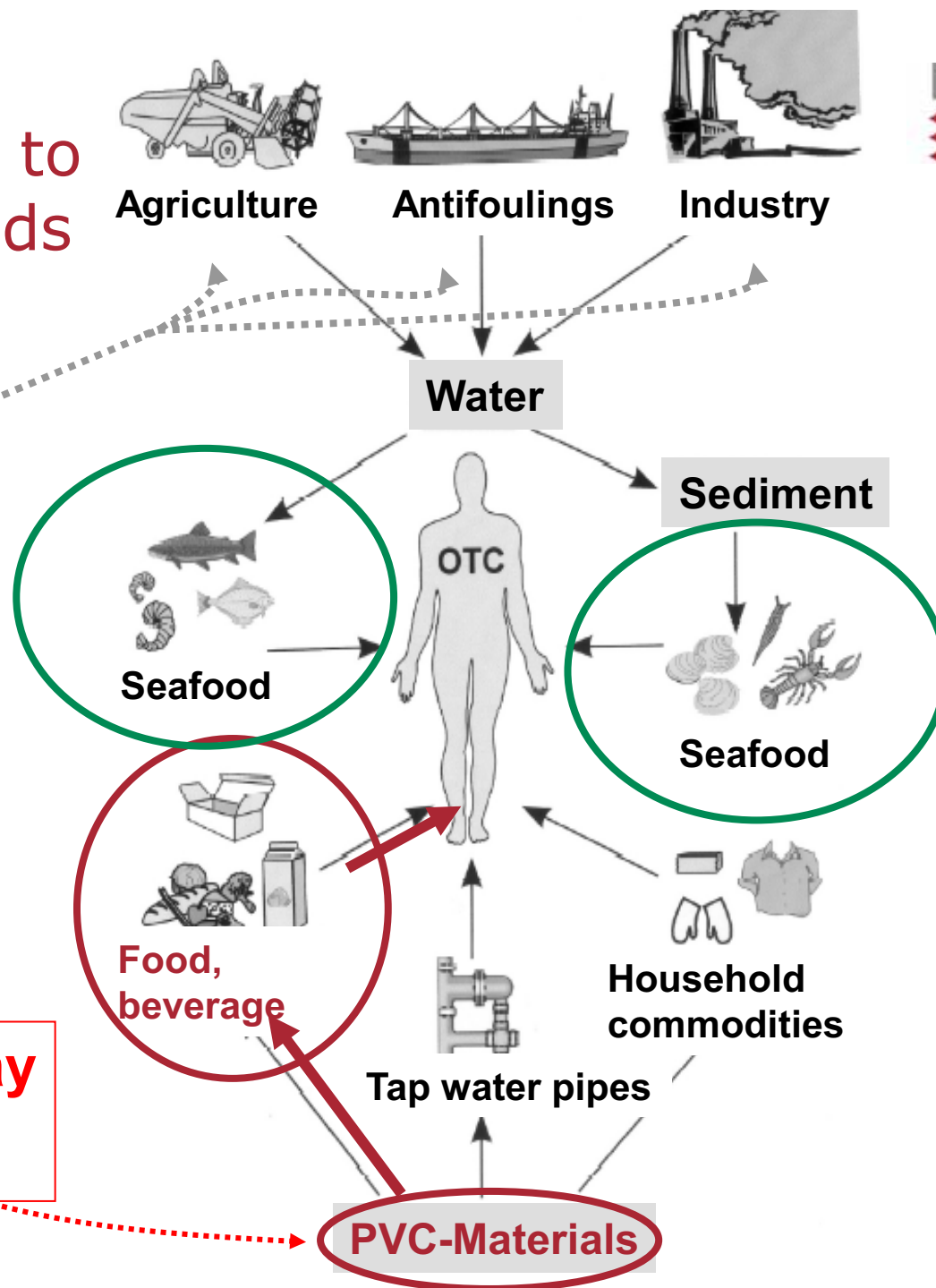
# Routes of exposure to organotin compounds

Used in

- Agriculture
- Antifoulings
- Industry

- PVC-Materials

**TDI: 0.25  $\mu\text{g}/\text{kg}$  bw/day**  
 $\sum$  TBT, DBT, TPhT and DOT



# Legislation on OTCs in Food Contact Materials



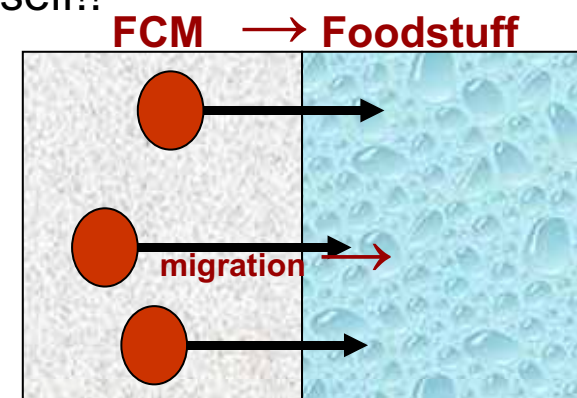
Compounds	Maximum level ( $\mu\text{g Sn/kg}$ foodstuff)
$\sum$ DBT, TBT, TPhT and DOT	40 (6)
$\sum$ MMT, DMT	180
MOT	1200
MDDT	12000 (50)
DDDT	24000 (50)

## Assumptions:

- 1 kg food per 6 dm<sup>2</sup>
- 100 mL in contact with 0.6 dm<sup>2</sup>

Ref: EFSA (2005); proposed EFSA values in parenthesis

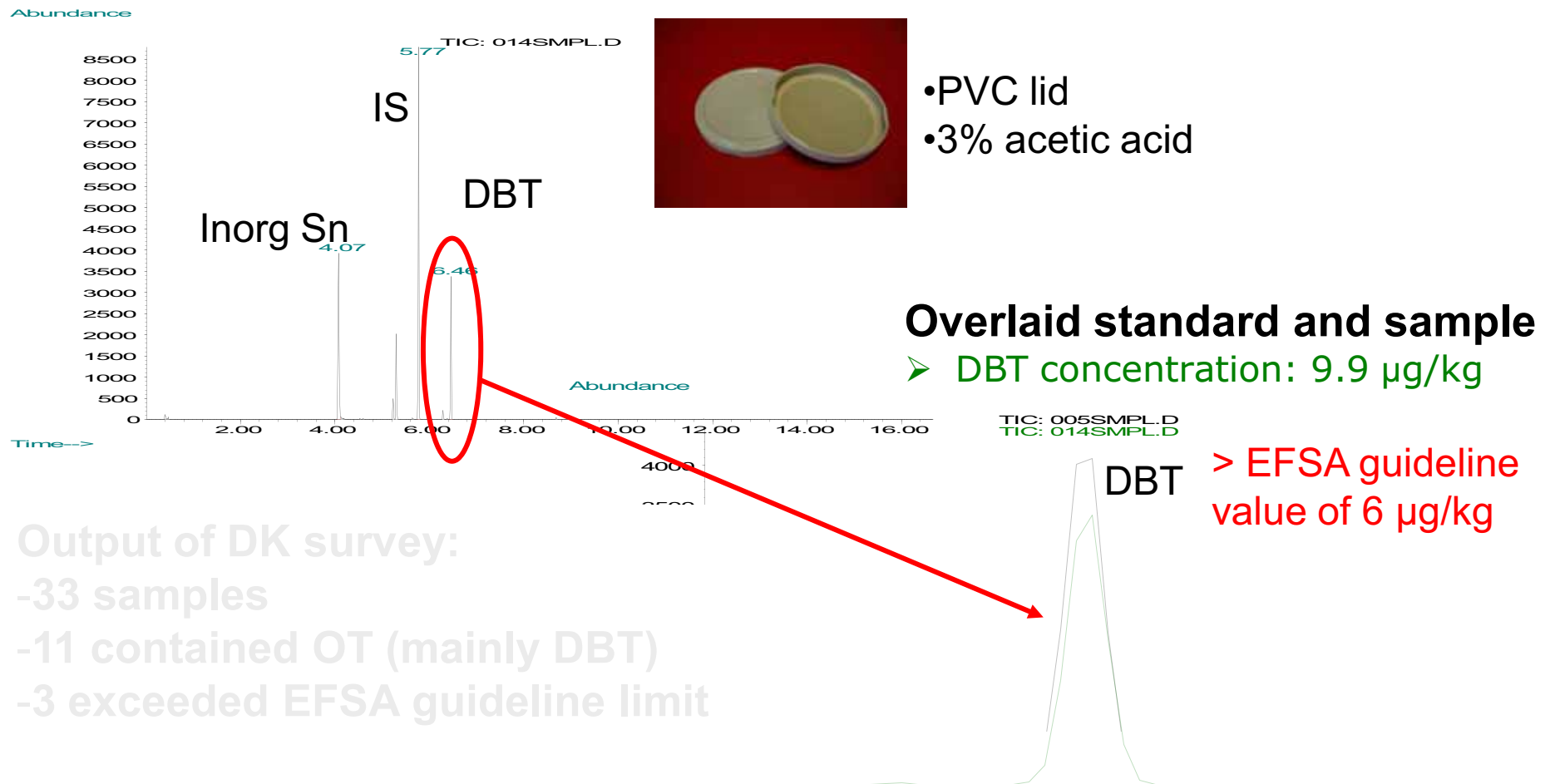
- Max levels on organotin migrating from the packaging material
- Testing by the use of food simulators (water, acid, oil, alcohol etc)
- BUT no maximum levels on organotins in the foodstuff itself!!



# Organotin migration from Food Contact Materials II

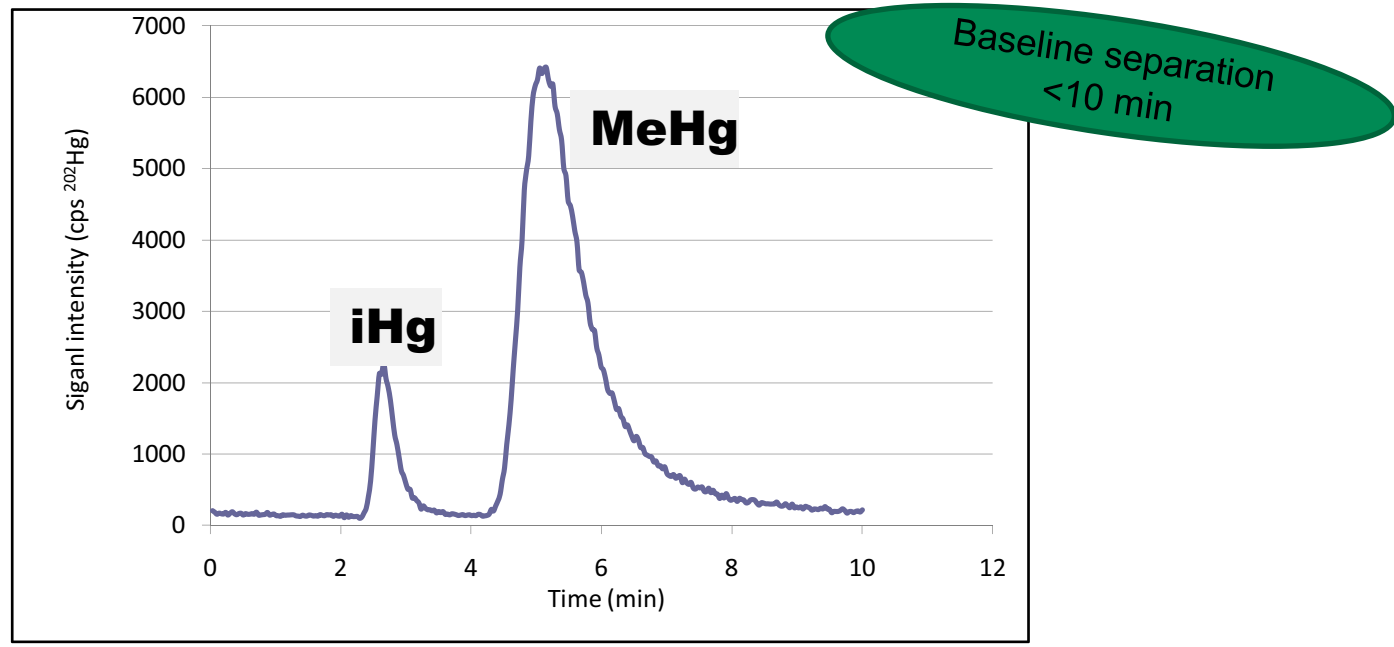
## Small scale survey on 33 FCMs

Baking paper, PVC cling films, silicone baking forms, lids with PVC gaskets  
PUR-agglomerated cork wine stoppers



Output of DK survey:  
-33 samples  
-11 contained OT (mainly DBT)  
-3 exceeded EFSA guideline limit

# Speciation analysis of Mercury by HPLC-ICPMS



***HPLC-ICPMS chromatogram of DORM-3 (Dogfish muscle)***

CRM	Certified (mg/kg)	Result (mg/kg)
DORM-2 (dogfish muscle)	4.47 +/- 0.32	4.21
DORM-3 (dogfish muscle)	0.355 +/- 0.056	0.35
TORT-2 (Lobster hepatopancreas)	0.152 +/- 0.013	0.16

**Relevance for regulation ?? But Codex opinion**

# Selenium in commercial food supplements

*Organic bound Se?*



*Selenite?*

*Selenate?*



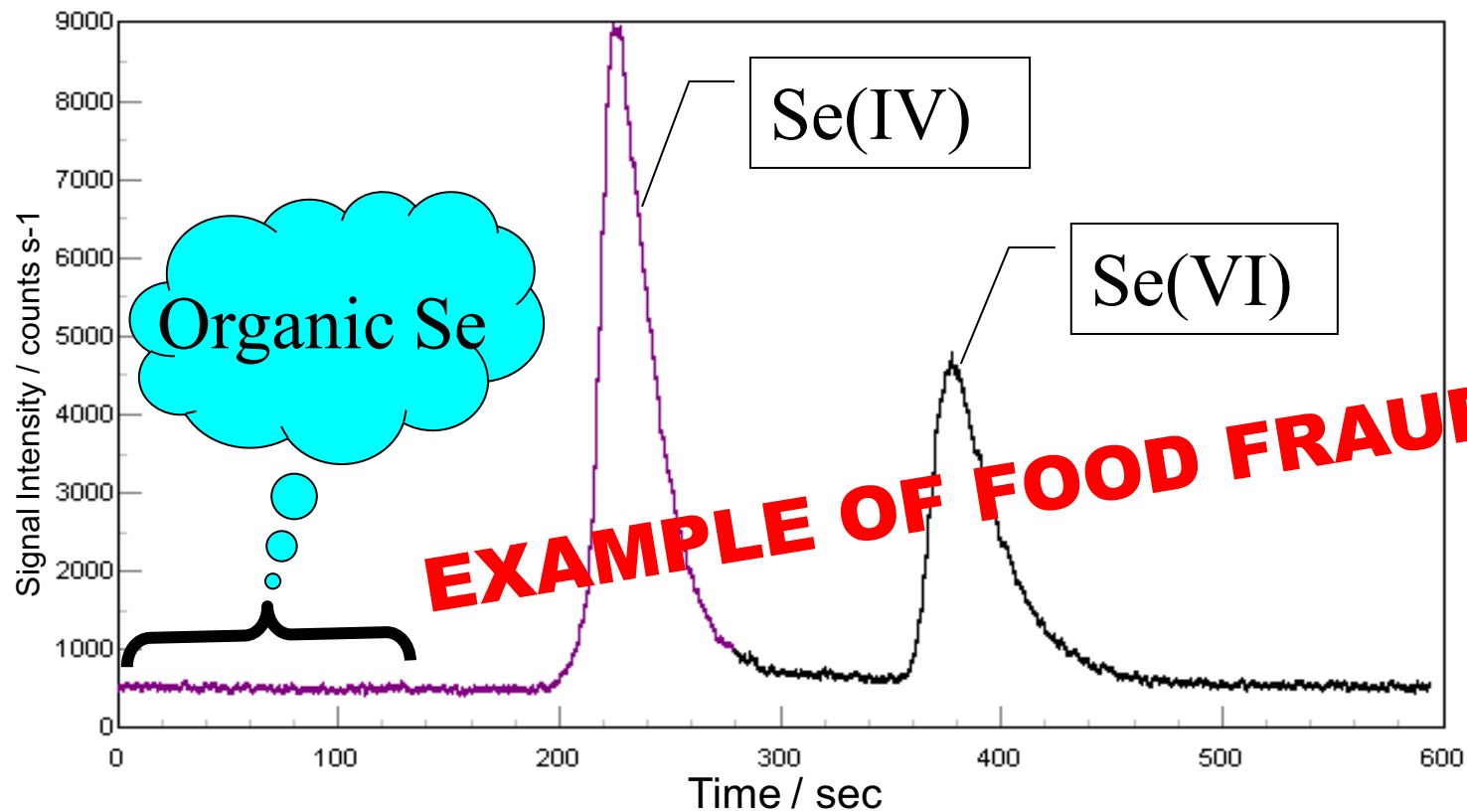
*Se yeast?*

*Selenomethionine?*

*Amino chelated Se?*

# Se speciation by HPLC-ICPMS

Supplement declared as organic bound Selenium (125  $\mu\text{g}$ /tablet)



Larsen, unpublished data.

Identification number of the additive	Name of the holder of authorisation	Additive	Composition, chemical formula, description, analytical method	Species or category of animal	Maximum age	Minimum content	Maximum content	Other provisions
						Maximum content of element (Se) in mg/kg of complete feedstuff with a moisture content of 12 %		
Category of nutritional additives. Functional group: compounds of trace elements								
358.12	—	Selenomethionine Selenomethionine produced by <i>Saccharomyces cerevisiae</i> CNCM I-3399 (Selenised yeast inactivated)	Characterization of the additive: Organic selenium mainly selenomethionine (63 %) content of 2 000-2 400 mg Se/kg (97-99 % of organic selenium) Characterization of the active substance: Selenomethionine produced by <i>Saccharomyces cerevisiae</i> CNCM I-3399 (Selenised yeast inactivated) Analytical method (1): Zeeman graphite furnace atomic absorption spectrometry (AAS) or hydride AAS	All species	—		0,50 (total)	1. The additive shall be incorporated in to feed in form of a premixture. 2. For user safety: breathing protection, safety glasses and gloves should be worn during handling.

(1) Details of the analytical methods are available at the following address of the Community Reference Laboratory: [www.irmm.jrc.be/crl-feed-additives](http://www.irmm.jrc.be/crl-feed-additives)

## Selenium in feed Incurred content vs added

**Organic bound minerals vs inorganic minerals**  
**Feed additives and food supplements**  
**EFSA opinions**

## Speciation summary

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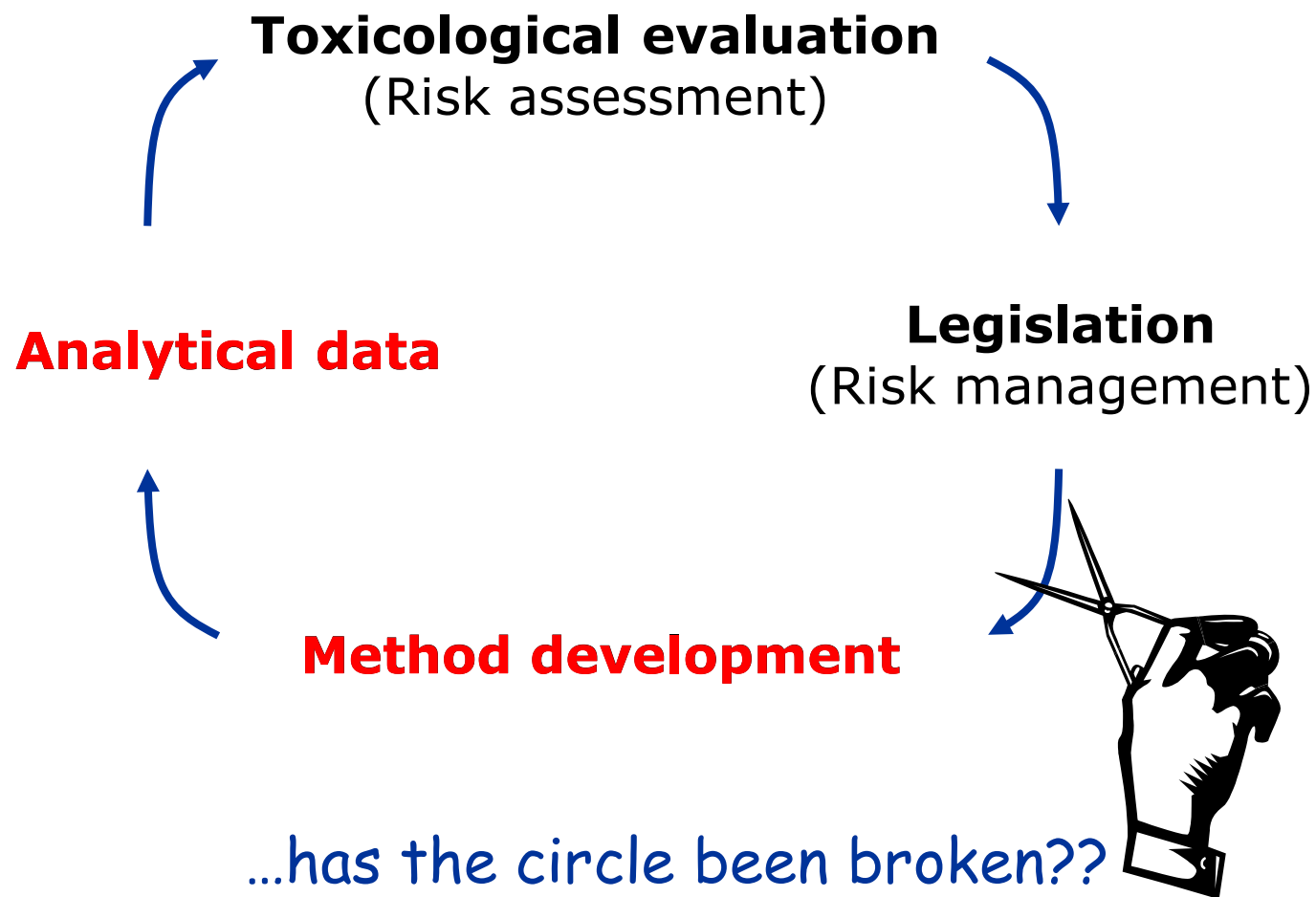
- ✓ speciation methods are more and more commonly used
- ✓ instrumentation is widely available
- ✓ legislation on species has started
- ✓ ...and more is expected in the future!



- ✓ standardised methods are not ready!
- ✓ ...but the need is known by authorities
- ✓ legislation is still behind!
- ✓ Lack of CRMs (e.g. for iAs)

## ...how to proceed?

---



## Perspectives I

- Maximum levels in the legislation are needed
- Standardised methods for official control is needed
- On-site screening methods for fast answers
  - biosensor based => answer: result>ML?
  - can a batch of food/feed be released for trade or is further analysis required?
- Fraud cases in food supplements / feed additives
  - do you get what you pay for?

## Perspectives II – trace elements in nanoform

**AEROSIL®** *Evonik*

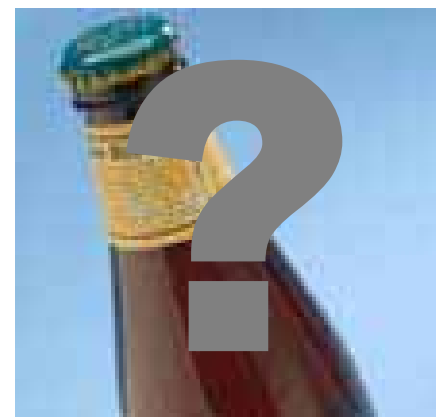


**silica nanoparticles as food additive for powdered food (e.g. soup, coffee creamer)**

<http://www.aerosil.com/>



**silver nanoparticles as food supplement**  
[www.fairvital.com](http://www.fairvital.com)



**nanoclay in PET beer bottles**

[www.honeywell.com](http://www.honeywell.com)

# NanoLyse Project

"Nanoparticles in Food: Analytical methods for detection and characterisation"

Validated methods for the determination of inorganic ENP in food extracts, based on size separation, size determination and specific detection

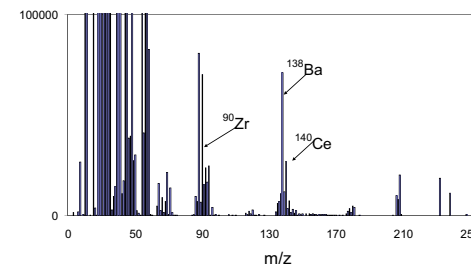
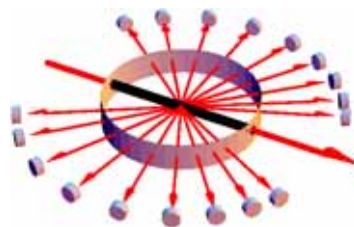
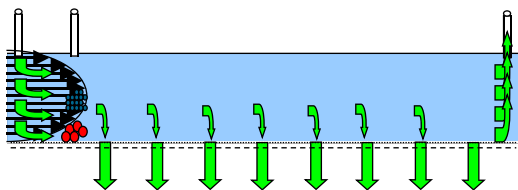
***silver nanoparticles in lean meat***



***silica nanoparticles in tomato soup***



# Our platform: AF4-MALS/DLS-ICP-MS



**asymmetric flow  
field flow  
fractionation  
(AF4)**

**particle separation  
according to their  
size (1nm – few  $\mu\text{m}$ )**

**optical detection  
multi angle (MALS)  
and dynamic light  
scattering (DLS), UV-  
vis absorption and  
fluorescence**

**particle detection  
size determination**

**inductively coupled  
plasma mass  
spectrometry  
(ICP-MS)**

**elemental detection  
chemical identity  
quantification**

**Schmidt, B.; Loeschner, K.; Hadrup, N.; Mortensen, A.; Sloth, J.J.; Koch, C.B. and Larsen, E.H.:  
“Quantitative Characterization of Gold Nanoparticles by Field-Flow Fractionation Coupled Online with  
Light Scattering Detection and Inductively Coupled Plasma Mass Spectrometry”, Analytical Chemistry,  
vol. 83 (7), 2461-2468 (2011).**

# Acknowledgements

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## **Coworkers:**

**Rie R. Rasmussen, Rikke V. Hedegaard,  
Xenia T. Trier, Katrin Löschner and Erik H. Larsen**



**Inge Rokkjær, Dorthe L. Cederberg**



**Veronika Sele, Heidi Amlund, Siri Bargaard, A.K. Lundebye  
Kåre Julshamn**



**Fernando Correiro-Raposo, Håkan Emteborg and Beatriz de la Calle**



## **Funding sources:**

**European Community's Seventh Framework Programme**

**European Committee for standardisation (CEN)**

**Danish Food Administration (DFVA)**





**Thanks for your attention**